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# CHEMICAL ABSTRACTS

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No. 1

## 1—APPARATUS, PLANT EQUIPMENT AND UNIT OPERATIONS

W. L. RUDGER

Some early chemical balances. A. Rarich. *J. Soc. Chem. Ind.* 54, No. 42, 2-10S (1933). E. H.

Some modern chemical balances. M. Dunbar. *J. Soc. Chem. Ind.* 54, No. 42, 10-15S (1933). E. H.

A gas specific gravity balance. J. S. Hales and W. C. Moss. *J. Sci. Instrum.* 12, 201-13 (1935).—The app. is based on counterbalancing with riders the upthrust on a glass ball when successively surrounded by air and the gas. It is portable, accurate to 1 or 2 in the 3rd place, requires a small quantity of gas, and a detm. can be made in 3-4 min. J. H. Moore

New apparatus for determining molecular weight by the camphor method. Carl Tiedcke. *Mikrochemie* 15, 253-6 (1935).—In detg. mol. wts. by the method of Rast (C. A. 16, 2240), the substance is usually mixed with camphor in a melting tube, heated in a bath of  $H_2SO_4$  and the m. p. detd. with a thermometer graduated in fifths of a degree. The app. here described consists of a 250-cc flask with ground-glass connection in the neck with a thermometer, as is usual with pycnometers. The flask is also provided with a stirrer and a Pt hook for holding the tube con'g. the sample + camphor. W. T. H.

The modern vacuum filter. Hans Paul. *Eng. Progress* 16, 250-2 (1935).—A discussion of the working principle, basic and special types, and fields of use of vacuum filters. E. H.

Micro filtration apparatus. Herman Yacoda. *Mikrochemie* 15, 294-302 (1935).—The app. described works on the same principle as the Buchner funnel. W. T. H.

Recent accomplishments in the field of fractional ultrafiltration. Pierre Grabar. *Bull. soc. chim. bel.* 17, 1245-1307 (1935); cf. C. A. 29, 6481P.—A review, with over 200 references. L. E. Gilson

Technic of ultrafiltration through Cellophane membranes. Lucien Brull. *Compt. rend. soc. ch.* 120, 68-9 (1935).—In the app. described ultrafiltration through ordinary sheet Cellophane under pressures of 30-40 atm. is possible. L. E. Gilson

A method for withdrawing single portions from continuous filtration and vacuum distillation. Walter Schmidt-Lange. *Arch. Hyg. Exptl.* 114, 304-10 (1935). F. S.

A simple modification of the Cottrell apparatus for measuring elevations of boiling points. H. C. Palmer. *Soc. Sci. Res.* 17, 138 (1935). O. Reinmuth

A sound box for the electrically-driven laboratory centrifuges. N. Poillard. *Analyst* 60, 753-3 (1935).—To prevent the unpleasant noise of the centrifuge a box just large enough to hold the app. was made and lined with rubber sponge mats. The box is covered with wire. W. T. H.

Vacuum in laboratory and technology. H. Biermann. *Chem. Fabrik* 1935, 393-404.—The present status of vacuum technic is reviewed under the headings: vacuum production (types of pumps, production by absorption), vacuum-measuring instruments, sealing and connecting vacuum app., purifying and taking out vacuum app., some automatic devices to facilitate work with vacuum installations, application of vacuum in lab. and technology (distn., evapn., crystn., disinfection and sterilization, filtration). One hundred and forty-one references. J. H. Moore

A high-vacuum microdesiccator. Josef Unterwiesing. *Mikrochemie* 15, 315-18 (1935).—The app. shown is of stout construction and permits the use of a high vacuum

and an elevated temp. for the drying. It is of the revolving type and is provided with a receptacle in which the capsule con'tg. the substance is weighed before and after the drying. The app. is especially suited for work with substances which are hard to dry and are hygroscopic. W. T. H.

Modern drying machinery. T. J. Morgan. *Chemistry & Industry* 1935, 913-24. E. H.

How to use pilot plants in process development. F. C. Villard. *Chem. & Met. Eng.* 42, 534-5 (1935). E. H.

Fractional distillation of extremely small volumes of liquids. Anton A. Benedetti-Pichler and Julian R. Rachele. *Mikrochemie* 15, 1-5 (1935).—The distn. of a few cc. of liquid is best accomplished by heating below the b. p. in a closely covered space with an efficiently cooled condenser of small surface area in close proximity to the evap. liquid. The arm of the app. described, which is still in the exptl. stage, is to collect the distillate at a cold point in the space immediately above the evap. surface. A brass rack and pinion device is provided for raising and lowering the condensing system. By means of a vacuum, ice-cold water is made to circulate in an outer tube but since the water rises but little, only the lower end of the condenser tube is cooled. An Al heating block is provided + a thermometer. W. T. H.

Continuous-action water still. Roberto F. Bouch. *Indus. Farm. Espana* 6, 33-2 (1935).—A constant-level device, supplied with hot water from the condenser overflow, is attached to an inlet tube fused into the side of the glass distn. flask just below the middle. L. E. Gilson

Continuous production of distilled water free from carbon dioxide and ammonia. Frederick G. Straub. *Ind. Eng. Chem., Anal. Ed.* 7, 433-4 (1935).—Fifteen l. of condensed steam per hr. mixed with 30 cc. per min. of acid chromate soln. con'g. 9 cc. of concd.  $H_2SO_4$  and 10 g. of  $Na_2CrO_4$  to 5 l. of water, was fed to a gas-fired Barnstead still. Nine l. per hour of distd. water was generated which had a conductance of  $0.5 \times 10^{-4}$  and  $0.5 \times 10^{-4}$  mho with a  $p$ -value of 6.8. Sketch of equipment is shown. J. L. E.

A simple automatic cut-off for electric stills. M. R. Aravamudan Rao and Basant Sanjiva Rao. *Ind. Eng. Chem., Anal. Ed.* 7, 577 (1935).—An app. for preventing the burning out of elec. heating units in water stills is described and a drawing is given. J. L. E.

Simple microburet without stopcock. Karl Schwarz. *Mikrochemie* 15, 304-11 (1935); cf. C. A. 27, 2847.—A no. of improvements have been made, particularly with respect to providing several jets. The use of the pipet, obtainable from Haack of Vienna, is suggested. W. T. H.

Improved screw plungers for use with mercury piston microburets. S. J. Folley and E. A. Rowell. *Mikrochemie* 15, 335-7 (1935).—The advantage of the new design is that there is no opportunity for leakage of  $Hg$  along the thread of the screw. W. T. H.

A new absorption apparatus for the microchemical determination of carbon and hydrogen. A. Frodrich. *Mikrochemie* 15, 25-37 (1935).—The use of anhydrous and anoxic, which are more effective than  $CaCl_2$  and soda lime, permits smaller and higher absorption tubes. Each tube consists of 2 parts; the inside part contains the absorber and the outside mantle is provided with

and exit tubes for the gas. The 2 parts fit together and when properly placed the gas enters through an opening, passes down through the absorbent and back to a second opening in the stopper, where it passes out. W. T. H.  
A home made Kipp apparatus. J. H. H. MacRae and T. T. Richards. *School Sci. Rev.* 17, 137 (1935).

A self-starting flow regulating siphon. G. Mönch. *Z. tech. Physik* 16, 314 (1935).  
Centralized measuring system for liquids in tanks. W. S. Gilfoil. *Chem. & Met. Eng.* 42, 571 (1935).

An oil manometer. G. H. Van Hengel and J. D. Stark-weather. *Mech. Eng.* 57, 633 5 (1935).—A new instrument is described in detail, with 1 cut, for measuring the abs. pressure in steam turbine condensers. J. H. M.  
Calculations for hydrogen-liquefaction apparatus with the help of s-T (heat content temperature) diagrams. V. Fischer. *Z. ges. Kolloid Ind.* 42, 174-8 (1935).

Burlap bags for bulk chemicals. R. W. Lahey. *Chem. & Met. Eng.* 42, 544-8 (1935).  
Hydrogenation apparatus for small quantities of substance. J. Erdos. *Mikrochemie* 18, 305-8 (1935).—The app. described serves for the hydrogenation of a few cg. of substance and is designed so that it is easily possible to det. by measurement whether any H<sub>2</sub> has been absorbed by the sample and how much. The glass vessel in which the hydrogenation takes place is connected with a mech. shaker. W. T. H.

Water-cooled stone mill, its introduction and improvement. James W. Kent. *Paint, Oil & Chem. Rev.* 97, No. 21, 26-7 (1935).  
An all glass valve. John Willard. *J. Am. Chem. Soc.* 57, 2328-9 (1935).—An all-glass valve for use in systems where stopcocks are objectionable is described and illustrated. C. J. West.

Bin shapes and feeders. Harlow F. Harding. *Ind. Eng. Chem.* 27, 1338-41 (1935).—Bins of ordinary type, with truncated prism bottoms and single discharge openings, are subject to (a) segregation and (b) bridging of bin contents, causing variations of 20-30% in wt./cu. ft./hr. If 2 sides are vertical and others sloping, such troubles are lessened, if 4 sides are vertical and multiple discharge openings are used, difficulties are minimized. Bridging is prevented by striking the sides of the bin, using compressed air intermittently between double linings of the bin, employing oscillators, scrapers, or belt conveyors within the bin. Six types of volumetric feeders are described and discussed, but better results are to be had from feeders giving const. wt./unit time. A counter-weighted hopper may load and then be discharged at const. time intervals or a traveling belt may act in a similar way—both essentially batch methods. An improved type uses a balanced belt and counterweight operating continually, oscillating about a pivot support, and by a linkage partly opening or closing a gate as needed. The actual weight of material delivered from the end of the belt remains the same per unit of time regardless of its wt./cu. ft., or whether it is coarse or fine. Grinders fed thus show increased capacity of 10% as compared with const. vol. feed types, and reduced cost/ton and lessened wear on grinding parts. W. C. Fbaugh.

An improvement in the Sander conductivity apparatus. F. Pradal. *Lesty. Cukrovár.* 53, 490-2 (1935).—The S. cond. app. uses a high current intensity, heats the soln., demands very quick readings and necessitates a dark room with special lighting. P. used a c., increased the resistances so that only 0.02 of the previous current passed through the soln., took readings with a d.-c. milliammeter protected by a Se. rectifying tube, and with a separate made coarse adjustments quickly before taking the final readings leisurely under a small current intensity. With this arrangement P. detected the changes in cond. brought by a 0.5° change in the soln. temp. F. M.

A micro cell for the measurement of electrolytic conductivities. Alfred Fink and Philipp Gross. *Mikrochemie* 18, 109-74 (1935).—The cond. cell here described has a

capacity of 0.45-0.80 cc. and has been used for detg. the cond. of electrolytes dissolved in heavy water. The elec. part of the measurement has been described previously, C. A. 28, 2246\*. The cell is essentially composed of a cylinder of 4 mm diam and 1.5 cm length. At both ends there are attached, at right angles, capillary tubes of 0.8 mm which run upward parallel to the walls of the cell and, at the top, the tubes are bent into curves which are constricted at the open ends. W. T. H.

Auxiliary apparatus for luminescence analysis. W. Kern. *Appl. Phys.* 50, 1212-3 (1935).—An illustrated article. W. O. E.

A mass spectrometer. S. H. Bauer. *J. Phys. Chem.* 39, 659-65 (1935).—It is proposed that a simple mass spectrometer can be constructed, based on a principle first discussed by Smythe (C. A. 21, 1057), and later used by him and Mattauch (C. A. 26, 4256) in the development of an efficient velocity filter for ions. The expl. arrangement suggested differs from these in that the oscillating field is directly made to be the mass analyzer, it being assumed that a condenser can be treated as ideal. This assumption is discussed and justified. The characteristics of such a mass spectrometer are discussed. H. G.

A hydrogen discharge tube for absorption spectroscopy. W. H. Watson and D. G. Hurst. *Can. J. Research* 13, A, 19-21 (1935).—An inexpensive, easily assembled tube of rugged construction is described. All parts exposed to the discharge are water cooled. The capillary with its water-cooling sheath is the only glass part, is a self-contained unit and is easy to make. The tube has been operated without trouble at over 2 kw input at 1000 v.

Making light-pencils and furnaces with Nernst paste. C. Tingwaldt. *Physik. Z.* 36, 627-9 (1935).—Our methods of making pencils and tubes of various mixts. of ZrO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, CeO<sub>2</sub>, and ThO<sub>2</sub> are given. For electrodes, thin, interwoven wires of Pt or Pt-Rh were wound around the tube and cemented on with a paste of Zr nitrate. Bands were used for higher temps. The paste was ignited over a flame. The elec. resistances of tubes of the same size and shape, prep'd under the same conditions, but contg. resp. 2.7, 4.3 and 7.3% of Y<sub>2</sub>O<sub>3</sub>, were measured at 1600° and 1770° and a min. was found at about 4.3%. The forming of tubes to make small furnaces is described. A tube 70 mm long will heat up to 2000° on 220 v in free air. The ends, to which the electrodes are attached, must be kept below the m. p. of Pt, but the tube can be heated to 2370° without forming large crystals on the surface, as with com. Nernst pencils. The first lighting requires a higher temp. than afterward, since the elec. resistance is reduced by the 1st heating. E. R. Rushton.

High temperature conveyors—A novel type for heating furnaces. W. E. Rhyne. *Iron Steel Engr.* 12, 16-22 (Oct., 1935).—A high flexibility of design is described and illustrated. W. H. Boynton.

A home made portable assay furnace. James P. Sloss and Tillman M. Patten. *Mining and Met.* 16, 451 2 (1935).—By use of standard shapes of A. P. Green G-2 bricks, fire-clay splits and a 6 X 8 muffle, an entirely satisfactory assay furnace costing \$9.50 was constructed. I. or heating, a torch is employed similar to those used to preheat large castings. Gasoline, kerosene or distillate under 20-30 lb. pressure (using a hand pump) is supplied. Consumption is 1/4 gal. per hr. Fusing and cupelling can be done simultaneously. Alden H. Lmery.

Minimizing hazards in operation of refrigerating systems. L. S. Morse. *Ice and Refrigeration* 89, 187-9 (1935).—Temp.-pressure relationships and refrigerants are discussed in relation to hazards in refrigerating systems. A. H. Johnson.

New investigations in absorption refrigeration. B. H. Jennings. *Refrigerating Eng.* 30, 87-93 (1935).

Acid-stable lining instead of lead for receptacles. Grigor'ev. *Zhurnal So. No. 26094, Ser. 62, Referatbary Silikhatil'ner No. 1338 (1935)*.—Products coated with water glass and a siliceous material are resistant to acids in the presence of Na fluosilicate. They are attacked only

by HF and some higher fatty acids. Finely pulverized siliceous material is mixed with 4% Na silicate and liquid water glass (36° to 38°Bé.) and applied on metallic or wooden receptacles; 3 or 4 days are necessary for the coat to dry and harden. The coated ware may be treated with  $H_2SO_4$  to improve their resistance to fatty acids.

M. V. Kondoidy

Apparatus for distilling liquids. Edwin M. F. Guignard. Swiss 171,029, May 1, 1935 (Cl. 36a).

Apparatus for continuous distillation of liquids. Edwin M. F. Guignard. Fr. 784,520, July 22, 1935.

Continuous still, with preheating device. Barrag-Meguin A-G. Ger. 618,100, Sept. 2, 1935 (Cl. 12a. 5).

Crystallization. The Dorr Co., Inc. Brit. 431,812, July 16, 1935. Minor crystallizable constituents or impurities that show decreasing soly. with increasing concn. of the main soln are removed by continuously concg the soln within the metastable range of said minor constituents so as not to permit spontaneous crystal. thereof and then mixing with fresh feed in such proportions that the mixt. is supersatd. to such a degree that the minor constituents are pptd. The process is applicable to the removal of fluosilicates from a soln of  $H_3PO_4$ . App. is described.

Crystallizing manganese nitrate hydrate. Howard S. McQuand (to Grasselli Chemical Co.) U. S. 2,017,960, Oct. 22. Crystals resembling light, dry snow are obtained by stirring a melt of Mn nitrate hydrate with solid  $CO_2$  in direct intimate contact.

Gas or liquid meters. Arne Zachariassen and Marius E. Petersen. Brit. 431,667, July 12, 1935. App. for measuring liquids or gases comprises 2 or more chambers for the fluid to be measured and a bellows or like extensible member in each chamber, the bellows being connected in pairs directly through conduits and filled with working liquid.

Thermohydrometer suitable for testing liquids such as "anti-freeze" solutions. Leo Edelmann (to E. Edelmann & Co.). U. S. 2,018,441, Oct. 22. Structural details.

Electronic temperature-indicating and -control system suitable for use with laboratory ovens. Wm. S. Halstead. U. S. 2,017,839, Oct. 22. Various elec. and operative details.

Thermometer suitable for floating in water. Herbert Hastings. U. S. 2,019,221, Oct. 29. Structural details.

Rotary-drum filter. Harry W. Denhard (to Oliver United Filters Inc.). U. S. 2,018,398, Oct. 22. Structural details.

Filter for liquids. E. Häny & Cie. Swiss 173,030, June 1, 1935 (Cl. 36d).

Air filter. Richard R. Halstead (to Halstead Corp.). U. S. reissue 19,732, Oct. 22. A reissue of original pat. No. 1,948,791 (C. A. 28, 2576).

Air filter. Harry S. Kaiser (to H. S. Kaiser Co.). U. S. 2,019,186, Oct. 29.

Air filter. Verner Dahlman (to Independent Air Filter Co.). U. S. 2,019,213, Oct. 29.

Multi-stage air filter. Herbert G. Kamrath (to General Motors Corp.). U. S. 2,018,131, Oct. 22. Structural details.

Filter for gases. Wilhelm Rehfus. Fr. 784,533, July 22, 1935. The filter is formed of bands twisted to spiral form and placed side by side to form a layer which is superimposed by another layer or layers.

Centrifugal separators. Gutehoffnungshütte Oberhausen A.-G. Brit. 431,257, July 3, 1935. A centrifuge with nozzles or hollow outlet bodies leading to outlets in the wall of the drum has a distributing plate with guiding or distributing passages.

Magnetic separator. Karl Sittig. Ger. 618,156, Sept. 3, 1935 (Cl. 15. 4 01).

Pneumatic separators with spinner disks for spreading the material in a casing where it is subjected to a rising air-current. British "Rema" Manufacturing Co. Ltd. and Peter Howden. Brit. 431,147-S, July 2, 1935.

Baffle apparatus for separating dust from gases. Mas-

chinen- und Filterbau G. m. b. H. Ger. 618,493, Sept. 9, 1935 (Cl. 12a. 2 01).

Separating solid materials. Khas Prins. Brit. 431,801, July 16, 1935. In the septn of refuse from comparatively large-sized coal, a flowing stream of small coal is formed in a trough which has a series of baffles by which upward surges in the stream are produced, and the material to be septd is fed onto the top of the stream, the coal floating on the upper surface and being skimmed off at intervals while the refuse sinks and is carried away by the stream, from which it is ultimately screened. App. is described.

Apparatus for separating coarse and fine solids by the action of air currents. Charles E. Hermann. U. S. 2,018,669, Oct. 29. Structural details.

Apparatus (employing air currents) for separating heavy granular particles from comminuted diatomaceous earth. McKinley Stockton (to Dicalco Co.). U. S. 2,018,039, Oct. 22. Various structural and operative details.

Apparatus for separating materials of different specific gravities such as coal and refuse by agitation and use of a blast of air. Henry M. Chance. U. S. 2,018,010, Oct. 22. Structural, mech and operative details.

Apparatus for separating liquids of different densities. Buderus'sche Eisenwerke. Ger. 618,020, Aug. 31, 1935 (Cl. 85a. 9 03).

Sifting-apparatus. Thomas Robinson and Son Ltd. Ida Robinson and Eric Nascau Molesworth (legal representatives of Wilfrid N. Robinson (deceased)). Brit. 432,060, July 19, 1935.

Sieves for grading, classifying, etc. Louis Herrmann. Brit. 431,841, July 16, 1935.

Devices for dispersing gases in liquids. The Distillers Co. Ltd. and James Lockey. Brit. 431,674, July 12, 1935. Addn. to 387,486 (C. A. 27, 4446) and 393,531 (C. A. 27, 5386).

Treating gases with liquids. Chemische Fabrik Carlthius A.-G. Brit. 431,788, July 15, 1935. In a reaction tower, the filling consisting of elongated plate-shaped or hollow bodies is poured in from the walls so that the bodies arrange themselves, with respect to the incoming liquid, at an acute angle toward the axis of the tower, whereby the downflowing liquid is directed toward the center.

Gas washers. Wilhelm Francke. Brit. 431,309, July 4, 1935. Addn. to 393,117 (C. A. 27, 5383). Wet filters used for purifying gases as described in 393,117 are composed of layers of small hollow bodies located between sieves or netting, the thickness of a layer being, e. g., 20 mm.

Means for detecting impurities in gases. Bernhard Dräger. Brit. 431,804, July 16, 1935. An adsorbent, e. g., silica or alumina gel, impregnated with an indicator soln. is used.

Device for centrifuging liquids or suspensions in swinging holders. Ewald Collatz. U. S. 2,018,837, Oct. 29. Mech. details.

Apparatus for transferring liquefied gases from transporting containers. Wm. F. Mesinger (to Linde Air Products Co.). U. S. 2,018,144, Oct. 22. Various structural, mech and operative details.

Dispensing measured quantities of liquefied gases such as liquefied ammonia from tank cars. Walter H. Kuehn (to Atmospheric Nitrogen Corp.). U. S. 2,018,857, Oct. 29. App. and various operative details are described.

Dispensing measured quantities of liquefied ammonia or the like from tank cars, etc. Frank C. Clark (to Atmospheric Nitrogen Corp.). U. S. 2,018,833, Oct. 29. Various features of app. and operation are described.

Siphon for conducting liquids. Jose P. Anter. U. S. 2,018,110, Oct. 22. Various structural and operative details.

Sheet-metal containers for ether and other volatile substances. John Feaver Ltd., Howards & Sons Ltd., Arthur Swann and Frederick W. Williams. Brit. 430,776, June 25, 1935.

Tanks for storing volatile liquids in the open air. Askan Wilf Svendgaard. Brit. 431,001, June 28, 1935. Sech

tanks comprise a vessel having metal walls and having the top and sides covered with a layer of concrete or other material in direct contact therewith, said layer serving as a heat-insulator and as a reinforcement for the tank.

Apparatus for the recovery of solvent vapor from solutions employed in an installation for the making of films, foils, coated papers, etc. Gennady Frenkel Brit. 432,176, July 22, 1935.

Apparatus for carbonating liquids in bottles. Geo. F. Sargent (to Sodax (1923) Ltd.) U. S. 2,018,424, Oct. 22. Various structural and operative details.

Emulsifying devices. Wm. M. Melmore and Alice L. Melmore Brit. 431,978, July 18, 1935.

Recovering solids from solutions. Firma Rud. O. Meyer Ger. 618,107, Sept. 2, 1935 (Cl. 12b). In processes in which solids are sprayed into a tower against a stream of hot gas, the dust carried away by the gas is sepd. and returned to the tower at a point where the temp. of the gas stream is sufficiently high to dry the dust. The process is particularly useful in recovering anhyd.  $MgSO_4$  or  $MgSO_4 \cdot H_2O$  from  $MgSO_4$  solns. App. is described.

Means for feeding air to drying towers. Willy Kuhles. Ger. 618,426, Sept. 13, 1935 (Cl. 12a 3).

Rotary-drum drying apparatus. Jørgen Brabæk. Ger. 618,160, Sept. 3, 1935 (Cl. 12a 2). See Brit. 326,829 (C. A. 24, 4876).

Conveyor-band apparatus for drying ammonium sulfate. Elektra A.-G. für Elektrotechnik. Ger. 618,363, Sept. 6, 1935 (Cl. 12a 2).

Apparatus for concentrating a solution or suspension by spraying. Johan M. Visser. Brit. 431,756, July 15, 1935. The soln. is sprayed onto the bottom of an evacuated container, the bottom being already covered with a dry substance, scrapers being provided to agitate the dry material and a roller being provided to smooth down the dry substance after agitation.

Bulk evaporators. James Müller. Brit. 431,301, July 4, 1935.

Evaporating solutions or emulsions, particularly of materials sensitive to heat or oxygen. Johan Marie Visser. Ger. 618,105, Sept. 2, 1935 (Cl. 12a 2). See Fr. 746,203 (C. A. 27, 4321).

Apparatus for evaporating salt solutions. Escher Wyss Maschinenfabriken A.-G. Swiss 175,336, May 1, 1935 (Cl. 36f).

Evaporating device for humidifying air. Robert C. Yost and Cyril G. Schelly (to Yost-Schelly Patent Estate, Inc.) U. S. 2,018,990, Oct. 29. Structural and operative details.

Air-dehumidifying and -conditioning apparatus. Robert H. Polson (to Young Radiator Co.). U. S. 2,018,750, Oct. 29. Various structural and operative details.

Air-cooling and -dehumidifying apparatus. Jesse E. Rasmussen. U. S. 2,018,804, Oct. 29. Various structural and operative details.

Air-conditioning and -cooling system. Matthew M. Lawler (to Cooling and Air Conditioning Corp.). U. S. 2,018,453, Oct. 22. Various structural and operative features.

Air-conditioning system. Lawrence A. Philipp (to Kelvator Corp.). U. S. 2,019,091, Oct. 29. Various details of app. and operation.

Air-conditioning system. Kemper P. Brace and Norman L. Rowe, 3rd (to Air Conditioning Systems, Inc.). U. S. 2,019,291, Oct. 29. Various details of app. and operation.

Air-conditioning apparatus. Harold F. Lathrop (to General Elec. Co.) U. S. 2,019,351, Oct. 29. Structural details.

Apparatus for cleaning air. Ralph L. Layte (to Motor Improvements, Inc.). U. S. 2,018,074, Oct. 22. Structural details.

Mixing-apparatus. American Machine & Foundry Co. Brit. 431,946, July 16, 1935. A mixing blade or beater comprises a central shank and a heart-shaped head, the shank being disposed in the re-entrant angle.

Apparatus for mixing a liquid and a gas as in regenerat-

ing spent sodium plumbite solutions. Frederick W. Stone and James N. Garrison. U. S. 2,019,325, Oct. 29. Structural, mech. and operative details.

Apparatus (with a pump and injectors, etc.) suitable for mixing oil, water and air for combustion, etc. Merle E. Nantiz. U. S. 2,017,867, Oct. 22. Structural and operative details.

Apparatus for mixing and blending dry batches of materials such as pigments, oxides, flour, cosmetics, etc. Frank J. Muench and John L. Muench (to General Machine Co.). U. S. 2,018,082, Oct. 22. Various structural, mech. and operative details.

Furnaces. Akt. Ges. Brown, Boveri & Cie. Brit. 430,870, June 26, 1935. In a furnace, more particularly a crematory furnace, in which difficultly combustible gases of combustion are burnt in secondary combustion channels and then pass through a recuperator, the channels are sep. from the recuperator and are arranged beneath an ash-collecting plate of the primary combustion chamber.

Furnaces. Victor Paschius. Ger. 618,168, Sept. 3, 1935 (Cl. 18c. 11.10). Furnaces in which materials are heated by traveling gases are provided with exchangeable hollow fillers, the no. , size and shape of which are varied in accordance with the gas velocity in such a manner as to obtain a max. heating effect. No sp. methods of procedure are indicated.

Muffle furnaces. Gibbons Brothers Ltd. and Wm. E. Gibbons. Brit. 431,871, July 17, 1935. In a furnace having side combustion chambers divided from the muffle by substantially vertical walls, the lower parts of the latter are formed of refractory material of greater heat cond. than that employed in the construction of the upper parts.

Rotary furnaces. Metallgesellschaft A.-G. Brit. 432,465, July 26, 1935. In a rotary furnace, particularly for chem. and metallurgical operations, e. g., the roasting of ores, a quantity of material equal to at least half the daily throughput is continuously maintained therein, preferably by employing baffle rings of suitable dimensions.

Rotary furnaces. Metallgesellschaft A.-G. Brit. 432,476, July 26, 1935. Divided on 432,465 (preceding abstr.). The gas outlet of a rotary furnace comprises a central valve-controlled passage, by which the gases issue directly from the end of the furnace, and an annular channel connected by piping with other off-takes along the length of the furnace and preferably situated in the 3rd 6th of the length starting from the upper end.

Furnace walls and linings. Sterling Metals Ltd. and Douglas J. Gascoigne. Brit. 432,512, July 29, 1935. The joints between the lining bricks and side walls of a high-temp. melting-furnace, e. g., a cupola, are protected from the corrosive action of the slag by using outwardly bulging bricks.

Rotary-furnace linings. Wm. Scott. Brit. 432,213, July 23, 1935. A rotary melting furnace has an inner lining of refractory brick laid on rammed gaseous which, for at least the main body portion of the furnace, is about half the thickness of the brick.

Heat exchanger (plate type). Treplin & Co. Nachf. Komm.-Ges. Ger. 619,450, Sept. 10, 1935 (Cl. 17f. 12.03).

Heat-exchange apparatus (with tubes and headers). Walter T. Wells (to Technicraft Engineering Corp.) U. S. 2,018,163, Oct. 22. Structural details.

Heat-exchange apparatus (of the shell and tube type) suitable for condensers, etc. Everett N. Sieder (to Foster Wheeler Corp.). U. S. 2,018,037, Oct. 22. Structural details.

Ball mills. Otto Liebeck. Brit. 430,471, June 19, 1935. A loose crushing element for a ball mill is of streamline or drop shape in order to prevent the formation of air eddies behind the falling element. Pins may be formed on the pointed end of the drop. An air current may be directed by nozzles in the direction of the falling bodies.

Ball mills for wet grinding. International Combustion Ltd. and James C. Farrant. Brit. 432,089, July 19, 1935. Preliminarily crushed material and  $H_2O$  are supplied to an auxiliary tank alongside the feed tank of the mill, the

auxiliary tank being provided with an overflow at the upper part and with an opening at the lower part through which  $H_2O$  and material pass into the feed tank.

Fusion apparatus for making "chemical asbestos" from lime, alumina, magnesite and silica, etc. Charles C. Whittier. U. S. 2,018,478, Oct. 22. Various structural and operative details.

"Batchmeter" for timing operations such as those of concrete mixers. Rudolph I. Lindow (to Chain Belt Co.). U. S. 2,018,964, Oct. 29. Various structural, mech. and operative details.

Apparatus for filling tins containing tobacco, foods, etc., with inert gas heavier than air at atmospheric pressure. Carl Loehle. U. S. 2,019,422, Oct. 29. Structural and operative details.

Apparatus for irradiating liquids such as milk, etc. with ultraviolet rays. Henning A. Trebler and Carl J. Larsen (to Hanovia Chemical and Mfg. Co.). U. S. 2,018,332, Oct. 22. Various structural, mech. and operative details.

Apparatus for condensing milk, juices of fruits, vegetables or beef or for distilling liquors, oils, etc. Frederick J. Allen. U. S. 2,018,049, Oct. 22. Various structural and operative details.

Pressure-actuated flow meters. Electroflo Meters Co. Ltd. and Burton Duglison. Brit. 430,970, June 27, 1935. The construction in a flow meter is formed by a

tapering element arranged within the conduit and the element is formed with 1 or more float wells.

Machines for moistening pulverulent material, e. g., cement raw material with water or roasted zinc blende with sulfuric acid. Mikael Vogel-Jorgensen. Brit. 431,352, July 5, 1935.

Counter-balanced sheet-metal covers, applicable to the lips of metal-pouring ladles. Bulpitt & Sons Ltd. and Walter H. Bulpitt. Brit. 431,247, July 3, 1935.

Hydraulic presses for thin materials, such as for vulcanizing rubber floor coverings, etc. John Shaw and Sons (Salford) Ltd. and John Shaw. Brit. 430,999, June 28, 1935.

Measuring-valve stopper for bottles. Alfred Berendt. U. S. 2,017,839, Oct. 22. Structural and operative details.

Use of solid absorbents such as calcium chloride or lithium chloride in refrigerating apparatus. Wulff B. Normell. U. S. 2,019,356, Oct. 29. App. and various details of operation are described.

Air-cooled condenser suitable for refrigerating systems. Christian Steenstrup (to General Elec. Co.). U. S. 2,019,324, Oct. 29. Structural details.

Apparatus for producing acetylene under constant pressure. Albert Mortreux. Fr. 784,306, July 22, 1935.

Acetylene generator. Egon Mandl. Ger. 618,391, Sept. 9, 1935 (Cl. 266 47 03).

## 2—GENERAL AND PHYSICAL CHEMISTRY

FREDERICK L. BROWNE

Frank Kenneth Cameron. Wilder D. Bancroft. *Ind. Eng. Chem., News Ed.* 13, 425 (1935).—A brief biography with portrait. E. J. C.

Wilber Dwight Engle. Alfred C. Nelson. *Ind. Eng. Chem., News Ed.* 13, 442-3 (1935).—A brief biography with portrait. E. J. C.

Carl von Linde and his work. R. Plank. *Z. ges. Kolloid-Ind.* 42, 162-8 (1935).—Includes a list of his publications. F. D. Rossini

C. v. Linde's contributions to the Society of German Engineers and the German Museum. C. Mattschow. *Z. ges. Kolloid-Ind.* 42, 169-70 (1935). F. D. Rossini

Professor Carl von Linde and his coworkers. Ernst Mönch. *Z. ges. Kolloid-Ind.* 42, 170-2 (1935). F. D. R.

Two letters from Linde to Professor Klein, Göttingen. Anon. *Z. ges. Kolloid-Ind.* 42, 172-4 (1935). F. D. R.

Our first great chemical enterprises—George T. Lewis. Anon. *Chem. Industries* 37, 325-30 (1935). E. H.

Hendrik Sjoerd van Klooster. F. M. Jaeger. *Chem. Weekblad* 32, 562-3 (1935).—Biography with portrait and bibliography. E. H.

Scheele. G. Dahl. *Svensk Farm. Tidskr.* 39, 365-9, 405-7, 421-4, 437-8 (1935).—This pertains to the literature on Carl W. Scheele, including addnl. items to A. E. Nordenskiöld's bibliography. A. R. R.

Hugh Charles Herbert Candy. Wm. Wright. *Analyst* 60, 730-1 (1935).—Obituary. W. T. H.

Percy Arthur William Sell. C. E. Sage. *Analyst* 60, 731 (1935).—Obituary. W. T. H.

Arthur Dehon Little. Avery A. Ashdown. *Science* 82, 362-3 (1935).—Obituary. E. H.

Nikolai Aleksandrovich Kolosovskii. L. N. Nikolaev. *J. Gen. Chem.* (U. S. S. R.) 5, 1029-36 (1935).—An obituary with portrait. A list of his scientific work is included. E. J. C.

Charles Thomas Kingzett. Anon. *Analyst* 60, 649 (1935).—Obituary. W. T. H.

Reminiscence of the late Dr. Stanislaw Tollacko. Anon. *Przemysł Chem.* 19, 133-6 (1935).—Biography with portrait and bibliography. A. C. Zachlin

Edward Wight Washburn. W. A. Noyes. *Trans. Illinois State Acad. Sci.* 28, No. 1, 39-40 (1935).—A memoir. J. H. Reedy

Chemistry in the Buchem. J. R. Partington. *Chem-*

*istry & Industry* 1935, 884-6.—Tabulated data give a summary of analyses of various objects such as dishes, cup, shovel, clamps and solder taken from the excavations in the Buchem, in Egypt. The solder on a flute corresponds in composition with the best-quality modern solder. J. S. Hicks

Progress of chemistry in China during the last twenty years. Chao-Lun Tseng. *Science (China)* 19, 1514-54 (1935). Cf. C. A. 29, 6115.—A brief, but authentic and all-inclusive review (in Chinese) of the progress of chemistry in China during the last 20 years, including the following topics: (1) early Chinese chemistry; (2) adoption of modern chemistry from the Western nations; (3) the Science Soc. of China and the beginnings of chem. research in China; (4) causes for rapid advance in chem. research within the last few years; (5) phases of chem. research now carried out in China; (6) institutions of chem. research; (7) progress in chem. education; (8) Chinese chem. nomenclature; (9) chem. societies; (10) chem. publications in China and Chinese chem. abstracts and (11) progress of chem. industries in China. C. L. Tseng

Treasures in the library of the Hahnemann Medical College. Joseph S. Hepburn and Rowland Ricketts. *Hospital Tidings* 44, No. 2, 5-17 (1935).—An account of the Hering Collection of the works of Paracelsus, the chem. treatises of Christian Friedrich Samuel Hahnemann and other rare texts of chem. interest. J. S. Hepburn

The way forward in chemistry. Wm. Albert Noyes. *Science* 82, 357-61 (1935). E. H.

The chair of chemistry in the University of Edinburgh in the XVIIIth and XIXth centuries. John C. Mackenzie. *J. Chem. Education* 12, 503-11 (1935). E. H.

Development of the curriculum in college chemistry. Bruce W. Merwin. *J. Chem. Education* 12, 541-3 (1935). E. H.

The contribution of laboratory work to general education. H. I. Schlesinger. *J. Chem. Education* 12, 524-8 (1935). E. H.

The relative instructional values of four methods of correcting objective tests in high-school chemistry. Vernon C. Lingren. *Science Education* 19, 123-7 (1935).—A study of 4 methods of correcting objective tests leads to the conclusion that teachers may use successfully some method in which the pupils correct their own objective

tests, provided the instructor accompanies this correction with explanations and class discussion. W. H. B. Method of recording laboratory notes in high-school chemistry. L. W. Applegarth *Science Education* 19, 107-112(1935) W. H. Boynton

A consumer approach to science teaching. G. P. Deyoe *Science Education* 19, 95-103(1935). W. H. B.

How could a national organization coordinate the activities of existing science teachers' associations? Philip G. Johnson *Science Education* 19, 105-7(1935).—A no of suggestions are given W. H. Boynton

The measurement of scientific attitudes Ira C. Davis *Science Education* 19, 117-22(1935). W. H. B.

Recent advances in science physics L. P. Bates. *Science Progress* 30, 283-8(1935).—A review of recent work on (1) the value of  $e$ , and (2) the masses of the light elements Joseph S. Hepburn

Variation in the composition of the atmosphere with altitude R. L. D. Clark. *School Sci. Rev* 17, 8-21(1935) O. Reinmuth

Determination of the parachor E. J. Williams *School Sci. Rev* 17, 22-25(1935).—Elementary expt with sample data from students' notebooks O. R.

The electric moments of some organic compounds J. N. Pearce and Luther F. Berhenke *J. Phys. Chem* 39, 1005-10(1935); cf. *C. A.* 29, 3504<sup>1</sup>.—The dielec. consts and  $d$  of dil solns were measured at 25°. The total polarization and the dipole moments are calcd. together with the spacial orientation of the group moments in the substituted benzaldehydes and in triethanolamine. The moments of diethanolamine and monoethanolamine calcd. from theoretical considerations agree well with the values detd. experimentally. The mol polarization, mol refractivity and elec. moment are:

Compd	P	M.R.	$\mu \times 10^{18}$ e.s.u.
<i>p</i> -Bromobenzaldehyde	134 1	35 03	2 19
<i>p</i> -Hydroxybenzaldehyde	396 0	33 34	4 10
<i>p</i> -Methoxybenzaldehyde	345 0	37 05	3 85
<i>p</i> -Tolualdehyde	262 0	30 43	3 30
2-Ethylhexanol-1	103 6	40 45	1 74
$\alpha$ -Ethylcaproaldehyde	183 3	39 19	2 04
Triethanolamine	301 0	37 82	3 57
Diethanolamine	189 9	26 93	2 81
Monoethanolamine	122 0	16 22	2 27

F. L. Browne

The adsorption of light and heavy hydrogen in relation to the hydrogenation of ethylene II R. Klar *Z. physik. Chem.* A174, 1-14(1935), cf. *C. A.* 29, 1383<sup>1</sup>.—The velocity was measured of the hydrogenation of ethylene with light and heavy H on an Fe catalyst between 0° and 175°. The velocity is lower with heavy H up to 100°; at higher temp (measurements up to 175°) it is higher with heavy H. The velocity shows a max. at 125° with light H and at 150° with heavy H. By plotting the ratio of the velocities against the temp, a max. is reached at 55°. To det. this max. more accurately measurements were made between 30° and 75° with a second catalyst (also Fe from oxalate). The max. was found at 53°. The heat of activation of the hydrogenation with light H was calcd. as 10.0 kg.-cal. and with heavy H it was 8.0 kg.-cal. at low temp and 12.5 kg.-cal. at high temp. A difference of 1800 cal. in activation energy was found between 80° and 120° while on Ni between 0° and 30° it was 880 cal. The heat of activation of ethylene was calcd. as 12.5 kg.-cal. R. H. Baechler

Electrical streaming potential with turbulent flow II. Reichardt *Z. physik. Chem.* A174, 15-21(1935).—From considerations of the boundary layer it follows that flow in the double layer remains a viscous flow during turbulence within a tube and that therefore the Helmholtz equation holds for streaming potential in turbulent flow. The ratio of streaming potential to difference in pressure is const. regardless of the form of the flow R. H. Baechler

Methods of observing electrophoresis of dyes, bacteria, blood corpuscles, etc., with Cellophane as a semiconductor Hans H. Waelchli *Kolloid-Z.* 73, 36-9(1935).—The

sign and relative magnitudes of electrophoresis can be detd. microscopically at high magnifications by observation of a drop of soln. suspended from a Cellophane strip. Elec. contact with the drop is made through the Cellophane which has been soaked in water to increase its cond. A modification is described for macroscopic demonstration of the electrophoresis of dyes. O. T. Q.

Viscosity and plasticity of disperse systems. VI. The effect of temperature and electrolytes on the plastic properties of kaolin M. Volarovich and D. Tolstol. *Kolloid-Z.* 73, 92-6(1935); cf. *C. A.* 29, 7741<sup>1</sup>.—In the temp. interval 10-57° the resistance to flow  $\theta$  of the kaolin water mixt. (58 1% anhyd. Gluehowsky kaolin) is nearly independent of temp., while its Bingham viscosity  $\eta$  (reciprocal of Bingham's coeff. of mobility) decreases less rapidly than the viscosity of water. Small amts of NaOH (0.01-1.0% on basis of anhyd. kaolin) reduce both  $\theta$  and  $\eta$  markedly. Larger amts of NaCl (1-7%) decrease  $\theta$ , but increase  $\eta$ . However, with increasing concn of both electrolytes, the plasticity (defined as  $\theta/\eta$ ) decreases. The reduction of  $\eta$  by NaOH is attributed to increased mobility of the clay particles as a result of peptization, that of  $\theta$  to the lubricating effect of the adsorption layer. The NaCl exerts both a coagulating and a dehydrating tendency, the former having the preponderating effect on  $\eta$ , which therefore increases, the dehydration causes  $\theta$  to decrease. VII. Plastic flow in an apparatus with cylinders capable of lengthwise displacement D. Tolstol. *Ibid.* 96-101.—An improved form of Pochettino's coaxial-cylinder app. (*C. A.* 11, 2080) is described for use in detg. the plasticity of concd., flowing clay suspensions. It gave data for resistance to flow agreeing with those obtained in a rotating-cylinder app. Bingham's equation of plastic flow was tested at very low rates of flow (kaolin-glucose-glycerol mixts.); the calcd. rates of flow were systematically higher than the exptl. Oscar T. Quimby

Viscosity of electrolytes in aqueous solutions and lyotropic numbers J. H. C. Merckel *Kolloid-Z.* 73, 67-75(1935).—At concn. of 0.5 N or lower the viscosity of solns. of a series of salts of a certain cation (or anion) is a linear function of the lyotropic nos. (cf. Buchner, *C. A.* 29, 7156<sup>1</sup>) of the anions (or cations). In a given series (e.g., the Na salts of univalent anions) one obtains a family of straight lines varying in slope with concn. but all intersecting at a relative viscosity of 1.000. An electrolyte at this point (e.g., KCl) gives aq. solns. with a relative viscosity of nearly 1.000 regardless of concn. unless the concn. exceeds 0.5 N. The slope at a given concn. is nearly independent of the alkali metal ion used with a series of univalent anions; this is not true for corresponding series of alk. earth salts. In either case a simple method of calcg. viscosities within 0.2% is available. The relative viscosities of 0.2 and 0.5 N solns. of the bromides and iodides of Mg, Ca, Sr and Ba were detd. at 25°. Oscar T. Quimby

Diffuse dispersion of light in white nonmetallic sols G. P. Luchinskii *Kolloid-Z.* 73, 30-42(1935); cf. *C. A.* 29, 2421<sup>1</sup>.—Application of Lambert's law to the light transmitted by colloidal solns. is complicated because of the change in light quality and the continuous variation of the absorption coeff. The scattered portion is decompd. more or less sharply into a spectrum. The dispersion coeff. for AgCl (75 mg/l.) and roua (155 mg/l.) hydrosols is inversely proportional to  $\lambda^3$  ( $\lambda$  = wave length). The true absorption coeff. is a more complicated function of  $\lambda$ . Distribution curves are given for transmitted and scattered light vs.  $\lambda$ . O. T. Q.

Determination of the particle size of silicic acid in a silicic acid-glycerol sol I. Erbe *Kolloid-Z.* 73, 1-14(1935).—The sol investigated was a com. product having the compn.  $\text{SiO}_2$  4.1,  $\text{H}_2\text{O}$  15.1 and glycerol 80.8%. Its  $\text{H}_2\text{O}$  content was varied by drying with  $\text{P}_2\text{O}_5$  or  $\text{dilu}$  with  $\text{H}_2\text{O}$ . Most of the particles had diams. between 40 and 50  $\mu$ . Of the methods used to det. particle size (diffusion, centrifuging, ultrafiltration, ultramicroscopic) diffusion into agar gels gave the best results and showed the heterodispersity to be small. Diffusion through porous

glass plates gave less reliable results and so did all of the other methods. A modified Keng method (colorimetric) for det.  $\text{SiO}_2$  in quantities down to 0.005-0.01 mg. is described. A few d. and vacuum measurements are given.

**Production of mercury sols by reduction.** E. Sauer and Dora Steiner. *Kolloid-Z.* 73, 42-4 (1933).—Hg sols with negatively charged particles can be prep'd. by reduction of  $\text{HgCl}_2$  but not  $\text{Hg}(\text{NO}_3)_2$ ,  $\text{Hg}(\text{ClO}_4)_2$  or  $\text{Hg}(\text{CN})_2$  with hydrazine in the presence of gum arabic and  $\text{NaOH}$ . Such sols can be obtained only when an intermediate, colloidal product of negative charge (e. g.,  $\text{Hg}_2\text{O}$ ) can be formed.  $\text{Hg}_2\text{NO}_3$  but not  $\text{Hg}_2\text{ClO}_4$  as well as  $\text{Hg}_2\text{Cl}_2$  can be used if the reduction is first carried out in  $\text{HCl}$  soln., where colloidal  $\text{Hg}_2\text{Cl}_2$  forms, and finally in  $\text{NaOH}$  soln., where the  $\text{Hg}_2\text{Cl}_2$  is converted to  $\text{Hg}_2\text{O}$  and then reduced to  $\text{Hg}$ .

**Chemical reactions between colloids.** II. Mercury and sulfur. E. Sauer and Dora Steiner. *Kolloid-Z.* 73, 45-7 (1933). of preceding abstr. and C. A. 29, 7497.—Mixing  $\text{Hg}$  sol with  $\text{S}$  sol produced a sol dark gray by reflected and redish brown by transmitted light. The product showed less tendency to sediment than the reactants. Coagulation with electrolyte, excess of excess  $\text{S}$  with  $\text{Na}_2\text{SO}_4$  soln. and analysis of the product demonstrated that  $\text{HgS}$  had been formed. The reaction is less rapid than that between sols of  $\text{Cu}$  and  $\text{S}$ , requiring  $1/4$  to 1 hr. for substantial completion.

**Manganese ethoxide. Production and hydrolysis.** B. Kandelaki, I. Setalvili and I. Tavtadze. *Kolloid-Z.* 73, 47-9 (1933).—Prep'd. from  $\text{MnO}_2$  was prep'd. by the reaction of  $\text{MnCl}_2$  and  $\text{NaOEt}$  in abs.  $\text{EtOH}$  at ordinary temp. The dark brown  $\text{Mn}(\text{OEt})_3$  soln. was poured off the insol.  $\text{NaCl}$ . After several days or weeks, violet crystals of  $\text{Mn}(\text{OEt})_3$  sep'd., which could be purified by reprecip. from abs.  $\text{EtOH}$ . The soln. of  $\text{Mn}(\text{OEt})_3$  in abs.  $\text{EtOH}$  is approx. 1% at room temp. Addition of  $\text{EtOH}$  solns. of  $\text{Mn}(\text{OEt})_3$  dropwise to redist.  $\text{H}_2\text{O}$  gave reproducible brown hydroxols whose stability varied with concn.

**The effect of ultrasonic waves on the colloidal solubility of metal hydrides.** I. Naumov, S. S. and S. S. Watanabe. *Kolloid-Z.* 73, 50-7 (1933).— $\text{Fe}(\text{OH})_3$  made from an. solns. of  $\text{FeCl}_3$  and  $\text{NH}_3$  and washed free of  $\text{Cl}$  and  $\text{NH}_4$  ions, can be dispersed permanently by ultrasonic waves (450/100 per sec.) without the addition of a peptizer unless too large an excess of  $\text{NH}_4\text{OH}$  (3-1) times the theoretical amt. was used in its prep'n. Ultrasonic waves greatly increase the peptization in the presence of small amts. of  $\text{HCl}$  (0.1-0.5 millimolar). Sols peptized by the ultrasonic method are much more readily coagulated than sols prep'd. by the usual methods.

**Influence of gases on silver sols.** T. Ivan Taylor and Wm. H. Cone. *J. Phys. Chem.* 39, 997-1001 (1935).—The effects of gases,  $\text{O}_2$ ,  $\text{N}_2$ ,  $\text{C}_2\text{H}_6$ ,  $\text{CH}_4$ , on Ag sols were det'd. by measuring pptn. values, cataphoretic velocities, conductivities and color changes. Gases have a stabilizing effect on sols that are on the border line of stability and apparently contain secondary particles. Agitation with gases produces a uniform distribution of stability was giving an apparently homogeneous sol. When the secondary particles are broken up the adsorbed gas apparently prevents their re-formation. No increase in the av. charge of the particles was observed.

**The stability of silver iodide.** Yu. N. Gorkhovskii and I. R. Protas. *Z. physik. Chem.* A174, 122-34 (1935).—A study of the intensity of light scattered by AgI sols of a concn. between  $5 \times 10^{-2}$  and  $5 \times 10^{-3} M$  led to conclusions concerning the stability of the disperse phase. The relation of stability to concn. of the excess ion was studied. The crystal lattice of one of the ions was observed. The p. values of the isoelectric point was det'd. ( $5 \times 10^{-2}$  to  $5 \times 10^{-3} M$  excess  $\text{Ag}^+$  as well as the p. values of max. stability that appeared on each side of the isoelectric point ( $5 \times 10^{-2}$  to  $5 \times 10^{-3} M$   $\text{Ag}^+$  and  $2 \times 10^{-2}$  to  $10^{-2} M$   $\text{I}^-$ ). The effect of elec-

trolytes with cations of different valences upon the stability of sols with excess amts. of  $\text{Ag}^+$  or  $\text{I}^-$  was studied. The potentials were measured. The  $\text{Th}^{4+}$  ion can charge AgI positively. The data were explained from the standpoint of adsorption.

**Surface salting-out by electrolytes.** I. Surface salting-out and dielectric constant. V. K. Semchenko and E. A. Davydovskaya. *Kolloid-Z.* 73, 24-30 (1933).—See C. A. 29, 7707. II. The dependence of surface salting-out upon temperature. V. K. Semchenko and A. F. Grushova. *Izv. VUZ*—See C. A. 29, 7723.

**The upper stability limit of (water) drops upon impact.** S. V. Gorbachev and V. M. Nefedova. *Kolloid-Z.* 73, 14-20 (1933).—When a moving water drop (1-1.5 mm. diam.) collides with another of the same size at rest, the 1 may coalesce or recoil with no apparent change, depending on 1. the angle  $\alpha$  between the line joining their centers at the instant of the impact and the direction of the moving drop and 2. the velocities of the moving drop. For a given  $\alpha$  and values of 2 in the range 0.1-1.9 m. sec. coalescence will occur until 2 exceeds a certain value.

**The lower stability limit of water drops upon impact.** S. V. Gorbachev and E. R. Mironov. *Kolloid-Z.* 73, 20-4 (1933). of preceding abstr.—V. sufficiently low relative velocity 2 colliding water drops will not coalesce, but the limiting value is not sharply defined. For drops of 1 mm. diam. at a relative velocity of  $\sim 4$  cm. sec. about half of the drops coalesce.  $\alpha = 0^\circ$ .

**The coagulation of coprecipitates of microcolloids by electrolytes.** I. Iorg. Popkov. *Kolloid-Z.* 73, 53-4 (1933).—A 2% soln. of electrodialyzed microcolloids (11.5%  $\text{Na}_2\text{CO}_3$  was treated with  $\text{FeCl}_3$  and  $\text{ZnCl}_2$ . With  $\text{FeCl}_3$  the soln. remained clear until a concn. of 30% was reached, where a slight turbidity was observed. Coagulation and slow sedimentation of the ppt. took place at a concn. of 40%  $\text{FeCl}_3$  (over 11 N). The ppt. had the appearance of a swollen gel, did not redissolve in  $\text{MnCO}_3$  and had a N content of only 7.4%. The coagulation is attributed to action of the products of hydrolysis of the  $\text{FeCl}_3$ . With  $\text{ZnCl}_2$  a concn. of about 4% (5 N) was required for coagulation, but the resulting ppt. could be redispersed by  $\text{MnCO}_3$ , indicating less destruction.

**The effect of water and alkali on the coagulation of egg white.** Joseph L. Dornmeyer. *Kolloid-Z.* 73, 7-10 (1933).—The effect of alkali ( $\text{NaOH}$ ,  $\text{KOH}$  and  $\text{NH}_4\text{OH}$ ) and heat on the sol-gel transition of egg white was studied qualitatively by use of the white of fresh eggs both diff. and centrif. with water. An increase in alkali concn. retards the hydrolysis of the protein and is accompanied by a part of the alkali protomate. An increase in the hydrolysis and coagulation of the acid protomate parallels a decrease in alkali concn., provided that the total amt. of water present is insufficient to dissolve the reaction product.

**Investigations of some properties of gold.** N. A. Yankin, D. N. Gwilt, J. D. Vennart and C. L. Pappas. *Kolloid-Z.* 73, 7-10 (1933).—A colloidal acetate gel in benzol sol. and the following hydrophils were studied: stannous phosphate,  $\text{Ta}$  phosphate, manganous acetate, silicic acid, zinc and gelatin. Various properties were measured and tabulated including gelatin time, turbidimetry, elasticity, etc., velocity of sedimentation, osmotic, loss of liquid on heating and light absorption. Brief comments are given on each property.

**Amplification of an empirical criterion to the Debye effect in the estimation of molecular weights of proteins by osmotic-pressure measurements.** David R. Briggs. *J. Phys. Chem.* 39, 984-997 (1935). of C. A. 29, 4638.—The empirical relation,  $E_{90} = (P_0 - P_1 - P_2) / (P_0 - P_1) - P_2$ , a constant, holds for protein solns. Simultaneous of the osmotic pressure  $P_0$ , the coll. osmotic pressure  $P_1$ , the osmotic pressure  $P_2$  on the basis of empirical data and of dissolvable  $\text{Mn}$ ,  $P_3$ , the coll. osmotic pressure of protein of mol. wt.  $M$ ,  $E = 39.1 (9m - P_0) / (P_0 - P_1) - P_2$  the molar ratio potential,  $a^*$  equivs. of diffusible an. derived from emul-



mass of the protein,  $[w]$ , concn. of all ions, bearing the same charge as the protein, in the colloid-soln. soln., i. e., concn. of foreign salt inside the membrane,  $\pi$ , a const. for a given colloid. This relation differs from the one previously found for gum arabic by the inclusion of  $F_p$ , which is negligible when the micelles are as large as those of gum arabic solns. By means of this relation the mol. wts. of proteins can be detd. in dil. solns. and in solns. in which the  $\pi$  is not that of the isoelect. point. Mol. wts. of 35,000, 60,000 and 93,000 ( $\approx$  about 15%) were found for crys'd egg albumin, uncrysd. serum albumin, and crys'd horse serum albumin, resp. F. L. R.

Remark on the work of E. Elsd and Th. Schachowsky concerning the effect of various metal compounds on gelatin A. Kuntzel *Kolloid-Z.* 73, 102-4 (1935) — E. and S. (C. A. 29, 5392) used a different definition of tanning than K. (C. A. 29, 3180). The two works agree essentially when this is taken into account.

Oscar T. Quimby

The solubilities of alkali chlorides and their mutual effect on their solubilities in liquid ammonia. G. Patschke and C. Tanne. *Z. physik. Chem.* A174, 133-55 (1935) — The system  $KCl-NH_3$  was studied in a temp. range of  $-80^\circ$  to  $+50^\circ$ . The solv. of  $KCl$  in liquid  $NH_3$  is small ( $m$ ,  $\approx 0.252^\circ$  at  $-77.2^\circ$ ) and decreases with increasing temp. Crystal compds. with  $NH_3$  do not form  $KCl$  crystallites in cubes which are largest when crys'd rapidly. The solv. of  $KCl$  in  $NH_3$  is almost doubled in the presence of  $NH_4Cl$  or  $NaCl$ . The system  $NaCl-NH_4Cl-NH_3$  was studied also. The solv. of  $NaCl$  is at first increased by the addn. of  $NH_4Cl$  but later depressed by large amts. of the latter. The solv. of  $NH_4Cl$  is slightly depressed by  $NaCl$ . These phenomena are explained on the basis of the electrostatic theory of Scherer (C. A. 25, 5821). The solv. curve for  $NH_4Cl$  by Kendall and Davison (C. A. 14, 2311) was confirmed.  $NaCl$  crystallizes in octahedra from  $NH_3$  which contains  $KCl$  or  $NH_4Cl$ . Practical applications of these findings are indicated. R. H. Baechler

The activity of the ions of hydrochloric acid. Zoltán Szabo. *Z. physik. Chem.* A174, 22-32 (1935) — The detn. of the ionic activities can be made with the aid of the  $e$  m f. of liquid chains with transport in cases in which the diffusion potential is known sufficiently accurately. The latter can be detd. as follows: let  $f_1$  and  $f_2$  represent ionic activity coeffs. at concns.  $m_1$  and  $m_2$ ,  $n$  and  $n_2$  the corresponding mean activity coeffs. and  $E$  the diffusion potential. The detd.  $e$  m f.  $E_d$  of the  $HCl$  concn. chain becomes  $(RT/F) \ln (f_1 m_1 / f_2 m_2) + e = E_d$ . A fictive  $e$  m f.  $E_s$  is detd. by the equation  $RT/F \ln n_1 m_1 / n_2 m_2 = E_s$ . The values  $E_d - E_s$  form a linear function of  $\log m$  up to a concn. of 0.2 M. The slope of  $E_d - E_s = F (\log m)$  is exactly that of  $E = (E_d + E_s)/2 = f (\log m)$  (where  $E_d$  is the  $e$  m f. of the cation chain and  $E_s$  the  $e$  m f. of the anion chain) an equation which must give the diffusion potential at least at great diln. Values

thus obtained for the diffusion potentials of the solns. of  $HCl$  of different concns. deviate about 3% from those calcd. by Henderson. The ionic activity coeffs. up to 0.2 M are equal to each other and to the mean activity coeffs. R. H. Baechler

Dependence of diffusion potential upon concentration. Zoltán Szabo. *Z. physik. Chem.* A174, 33-40 (1935) — The dependence upon concn. of various diffusion potentials was studied with the help of liquid chains with 2 identical electrodes, e. g.,  $Hg/HgCl$ , a mol.  $HCl/x$  mol.  $HCl$ , a mol.  $KCl/a$  mol.  $HCl$ ,  $HgCl/Hg$ . Between solns. of different concns. of a single electrolyte the diffusion potential is independent of the structure of the transition layer, const. and reproducible. The principle of superposition holds. The transition layer must possess a cylindrical symmetry to make attainable const. and reproducible diffusion potentials between solns. of different electrolytes. The sum of the diffusion potentials in the above-named chain proved to be a linear function of the log of the  $HCl$  concn. up to 0.5 M. R. H. Baechler

The equilibrium ratio of solid liquid in three-component systems in which congruent-melting two-component compounds exist. II. Konstanty Iljynkowski and Maria Smyt. *Z. physik. Chem.* A174, 60-72 (1935); cf. C. A. 29, 5725. — The systems: 1. salicylic acid-urea-acetanilide and 2. urea- $\beta$ -naphthol-acetanilide were studied. The effect of the third component on the 2-component compd. was studied by the method of thermal analysis and with the following results: System I contains the congruent-melting additive compd. salicylic acid-urea and contains 2 eutectic points namely: (1) 54.0% acetanilide 15.75% urea, 30.25% salicylic acid, temp.  $78.5^\circ$ ; (2) 61.3% acetanilide, 3.0% urea, 35.7% salicylic acid, temp.  $73.2^\circ$ . System II contains 2 congruent-melting 2-component compds., urea- $\beta$ -naphthol and  $\beta$ -naphthol-acetanilide as well as 2 eutectic points and a peritectic point. (1) eutectic at  $75.5^\circ$ ,  $\beta$ -naphthol, 3.0% urea, 31.5% acetamide and temp.  $50.5^\circ$ ; (2) peritectic at  $56.5^\circ$ ,  $\beta$ -naphthol, 12.5% urea, 31.0% acetamide and temp.  $53.4^\circ$  and (3) eutectic at  $58.0^\circ$ ,  $\beta$ -naphthol, 14.0 urea, 45.0 acetamide and temp.  $43.5^\circ$ . The course of crys'tn. in 3-component systems with peritectics is discussed with reference to phase diagrams. R. H. B.

The heat of dilution of nitric acid. G. Becker and W. A. Roth. *Z. physik. Chem.* A174, 104-14 (1935) — The heat of diln. of  $HNO_3$ , 4.5  $H_2O$  to  $HNO_3$ , 380  $H_2O$  at  $20^\circ$  was measured with 25 thermocouples. The heat of diln. plays practically no part; in bomb calorimetry one can use the const. value of 14.85 cal./millimol.  $HNO_3$ . The course of the heat of diln. is complicated and involves 3 distinct effects. R. H. Baechler

Handbook of Chemistry and Physics. 20th ed., edited by Chas. D. Hodgman. Cleveland: Chem. Rubber Pub. Co. 1931 pp. \$6, student ed. \$3.

### 5—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

W. ALBERT NOYES, JR.

Elements of the quantum theory. VI The hydrogen atom. Saul Dushman. *J. Chem. Education* 12, 524-39 (1935), cf. C. A. 29, 7171.

Potential barrier. K. C. Kar. *Current Sci.* 4, 93-4 (1935) — A criticism of Gamow's use (cf. C. A. 25, 5835) of the uncertainty principle of Heisenberg in his development of the idea of a potential barrier in the nucleus.

Recent advances in science: astronomy. R. W. Wrigley. *Science Progress* 30, 278-82 (1935) — A review of recent work on the chem. compn. of the atoms of the planets. Joseph S. Hepburn

Quantum mechanics investigation of the cohesive forces of metallic copper. K. Fuchs. *Proc. Roy. Soc. (London)* A151, 685-692 (1935) — The energy of the 4s electrons in metallic Cu is calcd. by an extension of the

method of Wigner and Seitz (C. A. 29, 349); the "exchange" energy between the 4s electrons and the closed shells is taken into account. The binding energy of the metal obtained was of the right order of magnitude, but, to calc. the correct compressibility, the interaction between the ions in the lattice, on account of the overlapping of the third shells with each other, must be considered. This is calcd. approx. by a statistical method. It is shown that for Cu the face-centered structure has a lower energy than the body-centered, but for Na the calcns. give almost equal energies for the 2 structures. E. R. Rushton

Determination of the effective temperature of some stars by the spectrophotometric method. M. Martin Lörén. *Annales soc. espagn. fis. quim.* 33, 603-26 (1935). — The color-temps. of several stars were detd. by measuring photo-

metrically the relation between the energies  $W_1$  and  $W_2$ , which a star emits in 2 wave lengths,  $\lambda_1$  and  $\lambda_2$ . To eliminate different sensitivities of the plate to each wave length, on each plate there were photographed simultaneously the spectrum of the star and that of the pot. crater of a C arc, which gives a temp. of 3750°K. The influence of atm. absorption was detd. in each observation by photographing the star at 2 different zenithal distances.

I. M. Symmes

The standardization of photoelectric cells for measurement of visible light. H. H. Poole and W. R. G. Atkins. *Trans. Roy. Soc. (London)* A235, 1-27 (1935).—Nineteen photoelec. cells, representative of the vacuum emission and rectifier types, have been standardized in light from the following sources: open solid-C arc, vacuum substandard filament lamp at 2360°K., artificial "mean noon sunlight" derived from the filament lamp by use of filters. The K and Na cells maintained const. sensitivity for more than 5 years and exhibit rectilinear proportionality between illumination and current up to full summer daylight. The Se and other rectifier cells exhibit a nonlinear illumination/current relationship. The perpendicular illuminations to daylight required to give currents of 1 microamp. were found to be 40,700 meter-candles (mc) for K, 2590 mc for K (thin film); 3710 mc for Na, 3700 mc for Cs (thin film), 16.4 mc for Se. The intrinsic sensitivities of the cell surfaces are, in microamps per lumen: 0.015 to 0.41 for Na, 0.21 for K, 1.78 for K (thin film); 3.81 for Cs, 12.12 for Se, 49.3 to 51.4 for Cu<sub>2</sub>O. C. C. Kieck

Shot effects of secondary electron currents. Lucy J. Hayner. *Physica* 6, 323-33 (1935).—The shot effects of secondary electron currents from plates of triode tubes were measured at 10<sup>3</sup> cycles. Calc. of electron charge from the shot effects agrees with Millikan's value within 1%. The av. no. of secondaries per primary electron,  $\bar{n}$ , ranges from 1 to 4.5. The variation of  $\bar{n}$  with energy of primaries agrees with the theory. The shot effects of both grid and plate currents were measured and Campbell's expressions applying to plate currents were extended to grid currents. From the difference in grid and plate effects it is concluded that secondary emission and primary impact are simultaneous within 10<sup>-8</sup> sec. C. E. P. J.

Photoelectric emission and the chemistry of surfaces. Cyrias Ouellet. *Naturaliste canadien* 62, 271-81 (1935); cf. C. A. 28, 3651<sup>1</sup>; 29, 2911<sup>4</sup>.—An explanation of the fundamental principles of the subject with a somewhat more detailed description of some of the results obtained by O. in expts. carried out with a photoelec. meter.

A. Papineau-Couture

Electron diffraction and surface structure. G. I. Finch, A. G. Quarrell and H. Wilman. *Trans. Faraday Soc.* 31, 1051-80 (1935); cf. C. A. 29, 2143<sup>1</sup>.—Two types of cold-cathode equipment are described, one for high-precision work and one for less precise work involving detection of cryst. phases and similar studies. A feature of both of these cameras is a beam trap wherein mol. rays and electrons of low energy and light are caught. The monochromatic electron beam is focused magnetically. A large no. of methods of prep. specimens are detailed. These methods include: flashing, foil-thinning, cathodic sputtering, colloid pptn., chem. deposition, electrodeposition for transmission metallic samples, distn., vapor deposition, crystn. from solvents, casting, gas etching, slag-sliming and crystal cleavage for transmission metalloid and compd. specimens; and technic for reflection specimens. A section on the interpretation of electron-diffraction patterns is included. The appearance of extra or forbidden rings in diffraction patterns of metals is ascribed to the interstitial soln. of gases in the metallic lattice. Some 80 diffraction patterns are reproduced and their interpretation is discussed. These patterns are made from Au foil, Al foil, Al<sub>2</sub>O<sub>3</sub>, Au, Pt and Cr electrodeposited films, colloidal Ag, Au and Pt, and flashed Zn deposits. It was found that Cr electrodeposited on Cu had a much smaller crystal size than that deposited on Ni, Fe, Sn or Bi. It has recently been found that a 30-sec. electrodeposition of Zn at 0.1 amp./sq. dm. sufficed to form a permanent cryst.

layer on cryst. Cu, but that a 3-minute deposition on polished Cu was necessary for the same effect.

H. A. Smith

A note on the reflection of cathode rays from a crystal surface. Shizuo Miyake. *Sci. Papers Inst. Phys. Chem. Research (Tokyo)* 27, 250-51 (1935).—The position and breadth of the reflection spectrum were obtained from an exact soln. of the wave equation for electrons for a teeth-valley potential field. The validity of the simple kinematical theory (cf. Lashkarev, *Z. Physik* 86, 797 (1933), 89, 820 (1934)) was confirmed. The results agree closely with expt.

Victor Hicks

Apparatus for electron diffraction at high voltages. G. P. Thomson. *Trans. Faraday Soc.* 31, 1049-51 (1935), cf. C. A. 29, 3593<sup>1</sup>.—An adaptation of the Thomson-Fraser type of electron diffraction camera has been made where two-stage acceleration of electrons from a gaseous discharge is utilized. The advantages of this type of source for high-voltage electrons over a hot-cathode source are: (1) elimination of most of the high-voltage x-rays and (2) the production of monochromatic electrons without large auxiliary app. The equipment works well from 45 to 85 kv. Rectified high-voltage secondary current employing the Cockcroft doubling principle is used as the source of power. All structural and operational details are given.

H. A. Smith

Secondary effects of cosmic ultraradiation. H. Geiger. *Ergebnisse exakt Naturw.* 14, 42-78 (1935)—A review.

C. D. West

Artificial nuclear transformations. R. Fleischmann and W. Bothe. *Ergebnisse exakt Naturw.* 14, 1-41 (1935).—This review brings up to date the previous accounts of F. and B. (C. A. 29, 997<sup>4</sup>) and of Kirchner (C. A. 29, 997<sup>9</sup>).

C. D. West

The transformation of the elements through impacts with hydrogen nuclei. F. Kirchner. *Chem.-Ztg.* 59, 713-10 (1935).—A review.

S. Bradford Stone

Measurement of the ranges of the residues of some of the lighter elements from bombardment with high-velocity protons. Hugo Neuert. *Physik. Z.* 36, 629-42 (1935); cf. C. A. 29, 7178<sup>3</sup>.—Protons with energies up to 200,000 v. were generated by Wien's acceleration method and the range and frequencies of the particles produced were studied by the cloud-chamber method of Kirchner (C. A. 28, 707<sup>9</sup>). For B, a continuous distribution frequency, with a broad and strong max. at 22 mm. and a small and weak, but decidedly marked max. at 44 mm. was observed, in agreement with previous results (C. A. 29, 2440<sup>4</sup>). For Be, a uniform range of 7.5 mm. was found for the residue particles. A new detn. of the range for Li in a chamber filled with H<sub>2</sub> showed that, instead of the range groups of 12, 9 and 7 mm. previously found, only 2 sharply defined ranges of 12 and 8.2 mm. are present. No nuclear residues for F were found at voltages up to 200,000.

E. R. Rushton

Detection of artificial radioactivity in a photographic emulsion. Tokio Takuchi and Takeshi Inai. *Proc. Phys.-Math. Soc. Japan* 17, 319-20 (1935).—Tracks due to  $\alpha$ - and induced radioactive particles formed by neutron bombardment are found in photographic emulsions.

E. O. Wieg

Investigations on neutrons. G. E. Monod-Herzen. *Ann. phys.* [11], 4, 137-201 (1935), cf. C. A. 29, 6498<sup>4</sup>.—Cloud chamber studies were made of the trajectories in H produced by Po-Be neutrons. The neutron-proton collisions appear to be like those between elastic spheres. A statistical study of the trajectories, long and short, shows the neutron source to emit a group of slow neutrons of 0.125  $\times 10^6$  e. v. energy in addn. to the fast neutrons of 4.5 and 7.8  $\times 10^6$  e. v. The former suffer considerable diffusion when passing through matter, while the latter do not. The role played by the neutron in nuclear structures is discussed and a simple relation is found between the nuclear mass defect and the mass no.

Morris Muskat

Absence of appreciable  $\gamma$ -ray emission in collisions of rapid neutrons with protons. Thérèse Gruet-Meyer. *Compt. rend.* 201, 658-60 (1935).—With 2 Al Geiger-Müller counters arranged according to the Rossi method

in parallel and a Po + Be source (40 millicurie) between them at about 10 cm from each, the no. of coincidences with the source bare was 7.2/min., and the same when a screen of borax was interposed, but if the source was surrounded by paraffin, 11.1 and 6.2, resp. Conclusion: The increased no. of coincidences when the source was surrounded with paraffin (without the borax screen) is due to the enhanced action on the Al shells of the counters of the neutrons slowed down by the H of the paraffin (cf. Amaldi, *et al.*, *C. A.* 29, 4237). This may explain the observations of Lea (*C. A.* 28, 1921, 29, 6198). C. A. S.

Radioelements produced by neutrons. Pierre Preiswerk and Hans v. Halban, *Proc. Complut. rend.* 201, 722-4 (1935).—When Tl is irradiated with Rn + Be (800 millicuries), in addn to the activity of 97 min (I) (cf. MacLennan, *et al.*, *C. A.* 29, 3594) one of  $4 \pm 0.5$  min (II) was observed. This is shown chemically not to be an isotope of Au, Hg or Pb. The amt. of both I and II is increased if the neutrons are slowed down by paraffin. It is suggested that both are isotopes of Tl,  $^{204}\text{Tl}$  and  $^{205}\text{Tl}$ , formed by capture of a neutron, and passing to  $^{204}\text{Pb}$  and  $^{205}\text{Pb}$  by emission of a  $\beta$ -ray. Sosnowski's results with Bi (*C. A.* 29, 3228) were not confirmed with 1200 millicuries of Rn + Be or 300 of Rn + B. When P was irradiated with slowed neutrons from Rn + Be in addn to activities of 3 and 145 min., one of  $15 \pm 1.5$  days was observed, attributed to formation of  $^{31}\text{P}$  by capture of a neutron. C. A. S.

Emission of fast particles. K. C. Kar, *Current Sci.* 4, 154-5 (1935).—The calcd. value for the radius of the core of a radioactive element is about  $10^{-14}$  whik the energy of emitted  $\alpha$ -particles indicates that they become free at distances of  $10^{-12}$  to  $10^{-13}$  cm. K. suggests that the region between  $10^{-13}$  and  $10^{-12}$  cm., instead of being filled with electrically neutral particles, is really packed with large nos. of  $\alpha$ - and  $\beta$  particles, such that the net charge is zero. When an  $\alpha$  particle is emitted by the core, it penetrates this "neutral shell"—really a series of thin shells—and eventually ejects one of its  $\alpha$  particles. This makes the rate of disintegration in reality the rate at which particles are ejected from the thin shells. W. W. S.

Determination of the amount of radon in the water of Wen-Chuan. Western Hills. Da-Tchang Tchong and Gung Tsong Yung, *Bull. Natl. Acad. Peiping*, 6, No. 2, 35-7 (1935).—The water of Wen-Chuan (hot spring), Western Hills, near Peiping, contains only 0.5 to 1 cman ( $10^{-12}$  curie per l) of radon. C. L. Tseng.

Indium isotopes and their nuclear spin. B. Venkatesachar and L. Sibaiya, *Proc. Indian Acad. Sci.* 2A, 203-7, *Nature* 136, 437.—The hyperfine structure patterns of some of the significant  $\alpha$  lines of Ir have been photographed. A hollow-cathode tube described (*C. A.* 29, 7795) is used as a source. The results indicate that there are 2 isotopes with nuclear spins  $1/2$ ,  $h/2\pi$  and  $3/2$ ,  $h/2\pi$ . A consideration of the known facts regarding the occurrence of isotopes of different mass nos. in the various elements has led to the inference that the mass nos. of the 2 Ir isotopes are 191 and 193, with a relative abundance of nearly 1:2, the isotope with the higher mass no. having the higher nuclear spin. Harold Gershinowitz.

Sudden changes of energy experienced by electrons of great energy. Louis Leprince Rungt, *Compt. rend.* 201, 712-14 (1935), cf. *C. A.* 29, 4258.—With the same arrangement, save that the radiation was filtered through 15 cm. Pb, trajectories of electrons totaling 75 m. were photographed. Of the tracks so examined 4 are considered to indicate production of pairs of electrons and 15 nuclear collisions. The energy losses indicate a no. of collisions about 10 times the theoretical. With a Pb screen, 0.1 mm. thick in the chamber (the length of the path through it is assumed to average double its thickness), examn of 60 tracks indicates losses of energy due to nuclear collisions 10 times more numerous than foreseen by theory. In both cases about  $1/10$  of the electrons were pos., and the energies of these favor the hypothesis that they result from the formation of pairs of electrons by materialization of photons from Th C'. C. A. Silberrad.

Investigations on counting tubes with alkali cathodes. Walter Christoph, *Ann. Physik* 23, 747-60 (1935).—

The possibility of prepg. Geiger-Muller counters for the visible spectrum was investigated. By making only a small part of the cylinder sensitive, counters were obtained which did not differ from the usual ones in the dark effect and in the counting range independent of voltage. It was not possible to stabilize their behavior. Whether an alkali-electrode photo-counter with a counting range independent of voltage is obtained or not appears to be independent of the construction. Victor Hicks.

The L emission spectrum of argon. M. Račlovský and V. Dolejšek, *Nature* 136, 643 (1935).—By careful regulation of pressure in a special ionic tube and the spectrograph the L emission spectrum of A as well as lines of Ca, Na and Si are obtained. The following values are given:

	$\lambda$	$\nu/R$	$\nu/R$ calcd.
L	561	16.2	16.29
L	567	16.1	16.13

New measurements and observation of nondiagram  $L_{\alpha}$  lines of mercury, platinum and tungsten. Yvette Cauchois, *Compt. rend.* 201, 721-2 (1935), cf. *C. A.* 29, 3911<sup>14</sup> and following abstract.—Two Hg lines already measured (cf. *C. A.* 29, 3911<sup>14</sup>) are identified as satellite lines,  $L_{\alpha}''$  and  $L_{\alpha}'$ , and 2 others of each of W and Pt, newly observed, as  $L_{\alpha}'$  and  $L_{\alpha}''$  (cf. Richtmyer, *C. A.* 28, 707<sup>6</sup>). C. A. Silberrad.

Fine structure of the  $L_{III}$  absorption edge of rare earths. V. Dolejšek and M. Hvilar, *Compt. rend.* 201, 100-2 (1935).—The fine structures of the  $L_{III}$  absorption edges of Hf, Pr, Nd, Sm, Gd and Yb were detd. They are similar among themselves and show dependence both on the crystal and chem. properties and on the at. no. (cf. Kromig, *C. A.* 26, 4247). C. A. Silberrad.

New observations of L emission spectrum of platinum. Yvette Cauchois, *Compt. rend.* 201, 598-600 (1935), cf. *C. A.* 29, 3911<sup>14</sup>.—By a slightly modified method 32 lines in the L spectrum of Pt were measured and classified. Previous results are substantially confirmed, but 13 new lines (8 nondiagram and 4 forbidden) were discovered, and 3 (1 nondiagram) shown to be doublets. C. A. S.

X-ray interferences in a monocrystal cathode. W. Kossel and H. Voges, *Ann. Physik* 23, 677-704 (1935), cf. *C. A.* 29, 4210<sup>14</sup>.—Interferences were obtained from x-rays excited by cathode rays striking the diffracting monocrystal. The positions of the interferences can be described as reflections of plane waves by lattice planes, all expected reflections occur. The relative positions of all the reflections are functions of the ratio of wave length to lattice const., this ratio can be calcd. from measurements of the film only. The depth of the contributing reflection planes can be estd. from the penetration of the cathode rays into the lattice, the estd. penetration agrees with that calcd. from the  $\mu$  for x-rays. With Cu the resoln. closely approaches that for the ideal crystal. When the diffraction angle  $\leq 25^\circ$ , the lines are weaker than the background on one side, but stronger on the other, an elementary explanation is given in terms of the coherence of the reflected ray with a ray parallel thereto directly from the excited atom, a complete explanation in terms of the optical reciprocity of x-ray reflections is indicated. The fluorescent x-rays from monocrystals (with an appendix concerning electron diffraction). M. v. Laue, *Ibid.* 703-46.—The optical reciprocity law in combination with the dynamical theory of x-ray interference gives the explanation of the expt. on the preferred direction of emission in a monocrystal. The *Aschroth lines* are the analogy thereto for electron waves. Victor Hicks.

New focusing method in spectrography of x-rays. A. Kunzl, *Compt. rend.* 201, 656-8 (1935).—The method combines that of Hamos (*C. A.* 27, 5002) in which the reflecting surface of the curved crystal forms part of a cylindrical surface of which the axis passes through the source of x-rays, in place of that of Cauchois (*C. A.* 27, 464), with that of Bragg and de Broglie. By its means  $M_{\alpha}$  and  $M_{\beta}$  of Ta oxide were detd. as 6764 and 6901 X. U., resp., the differences between observed and calcd. values

of  $r/R$  thence deduced are similar to those observed by Siegbahn for W (C. A. 25, 4179). C. A. Silberrad

Paschen-Back effect. Pierre Jacquinot and Tsai Belling. *Compt. rend.* 201, 778-9 (1935).—The Paschen-Back effect of lines 5780-60 and 5770 was measured by means of the Bellevue magnet with supplementary coils (cf. C. A. 28, 4280) with a field of 65,800 gauss (which gave a displacement of the central component of 5790 of 0.2 Å.). In both cases dissymmetry is shown and the results do not agree with calcn. based on Houston's theory. C. A. Silberrad

Effect of electric field on absorption spectrum of sodium. Ny Tu-de and Weng Wen-lo. *Compt. rend.* 201, 710-18 (1935); cf. C. A. 29, 5347.—With the same arrangement the effect of fields up to 2800 v./cm. on Na vapor has been examined. The results confirm and extend those of Segré (C. A. 29, 2416), and closely resemble those already obtained for Rb and Cs, allowance being made for the change in the Rydberg const. Absorption bands due to molecules bound by van der Waal forces were observed (cf. Kubn. C. A. 26, 8830). C. A. Silberrad

Wave-length shifts of the spectral lines of tin due to the change of pressure. Toshiko Yuasa. *Science Repts. Tokyo Bunriku Daigaku* 2, 267-77 (1935).—By use of an expl. procedure similar to that (C. A. 29, 447) employed for Sb, observations have been made on the change in appearance and wave length of lines of Sn I, Sn II and Sn III effected by increasing pressure, up to a few cm. Hg, of the emitting vapor. The shifts noted for Sn II and Sn III lines are attributed to the Stark effect resulting from the electric field set up by the ionized atoms. These shifts when correlated with the term designations of the lines increased with increasing inner quantum no. rather than with the total quantum no. Lines of Sn I were broadened with increasing pressure but not displaced because the field is either weak or absent owing to the paucity of ions. C. C. Kiees

New terms in the spectrum of Fe II. J. C. Dollbe. *Proc. Roy. Soc. (London)* A151, 703-20 (1935).—Wave lengths of about 2000 lines of Fe II, observed as electrode lines in the spectrum of an Fe arc, have been measured on high-dispersion spectrograms. These, and other, data have served to extend the term analysis of Fe II by bringing into the list of known terms many new doublet, quartet and sextet terms. The new terms are those required by at. theory and account for more than 100 previously unclassified lines, making the total for which term combinations are now known about 1250. All the Fe II terms are presented in tables together with the electron configurations to which they belong. C. C. Kiees

The intensities of the sodium lines in the sun's spectrum. G. Righini. *Z. Astrophys.* 10, 314-32 (1935).—The intensities of 22 Na I lines, appearing in the sun's spectrum, were measured by a method of photographic photometry. The observed equiv. breadths of the lines were found to follow the course given theoretically on the assumption that they are affected only by damping and Doppler effects, that the temp. is 5000°, and that a single coeff. of damping may be used for the various multiplets. C. C. Kiees

Intensity measurements of the 2P- $\pi$ D series of magnesium in the sun's spectrum. M. Minnart and Jean Genard. *Z. Astrophys.* 10, 877-81 (1935).—The equiv. breadths of the Mg I lines 2P- $\pi$ D in the solar spectrum were measured and from these data their oscill. strengths were derived. These were found to be near const. or to increase with the series no.,  $n$ . This improbable result cannot be accounted for by any of the causes known to affect the widths of lines. C. C. Kiees

Analysis of the spectrum of freely ionized zinc. Z. N. T. S. Subbaraya. *Proc. Ind. Acad. Sci.* 24, 115-18 (1935); cf. C. A. 29, 5747. Harold Gershinowitz

Effect of periodic variation in concentration of neutral atoms in an alternating current sodium-neon lamp. Willem Usterhoeven and Cornelis Vurburg. *Compt. rend.* 201, 647-9 (1935); cf. C. A. 29, 2487.—Oscillograms of a Na-Ne lamp fed by a c. e. (50~) for current,  $I_p$ , p. d. of terminals,  $V$ , and intensity of yellow light,  $I$ , show the

curves for  $V$  and  $I$  to be much deformed, only that for  $I$  being approx. sinusoidal (cf. Pound, C. A. 28, 5347). The deformations are traced to variations of the concn. of Na atoms and ions in the mixed Ne and Na vapor. C. A. Silberrad

Variations in the spectrum of Nova Herculis 1934 from January 10, to March 12, 1935. H. Ohlkr. *Z. Astrophys.* 10, 523-38 (1935).—On Jan. 10, 1935, the emission lines of H (Balmer series) and the spark lines of some of the metals were the outstanding features of the spectrum of Nova Herculis. By Mar. 12, the bright bands of He I, C I, O I, O II and N II had become the conspicuous features of the spectrum. C. C. Kiees

Possible variations of the profiles of the hydrogen lines in the sun's spectrum as dependent on the fluctuations in the sun's ultraviolet radiation. H. K. Kharadse. *Z. Astrophys.* 10, 539-43 (1935).—An indirect method for detecting variations in the ultraviolet solar radiation, which cannot be measured directly, would be to observe the variations in the widths of Fraunhofer lines at different phases of the sun's activity. Such observations, made for several lines in the solar spectrum, indicate that for the Balmer lines  $H\gamma$  and  $H\delta$  changes do occur in their profiles that coincide with the max. of solar activity. C. C. Kiees

Oxygen in the sun's chromosphere. T. Royds. *Nature* 136, 666-7 (1935).—Photographs show the infrared triplet of O at  $\lambda$  7771, 7774 and 7755 as emission lines in the sun's chromosphere. Oxygen is thus a normal and probably abundant constituent of the chromosphere. H. O. Wieg

Heat of dissociation of the hydrogen molecule,  $H_2$ , determined from the rotational structure of the continuous absorption at the long wave-length limit of 850 Å. The absorption spectrum of hydrogen. I. H. Neulter. *Z. Physik (Chem.)* B29, 315-27 (1935); cf. C. A. 29, 1316.—Absorption spectra were obtained for para- $H_2$  and ordinary  $H_2$  (ortho- +  $\frac{1}{2}$  para- $H_2$ ) in a vacuum spectrograph with pressures up to 0.05 mm. and absorption paths up to 2 m. The results show that for the rotational levels,  $K' = 0, 1$  and 2, continuous absorption begins at sharply different wave lengths. This indicates that for higher rotational energies, a smaller quantity of energy is needed to dissociate the mol. into a normal and an excited H atom. The result obtained for the heat of dissociation of H $_2$  mol. is  $102.72 \pm 0.02$  kcal. This is compared with earlier results, both expl. and theoretical. G. M. Murphy

Ultraviolet absorption of oxygen. Levi Herman and Renée Herman. *Compt. rend.* 201, 714-16 (1935); cf. C. A. 28, 4980.—By the same arrangement the 2 systems of absorption bands of O between 2400 and 2800 Å., hitherto regarded as independent, have been observed simultaneously, the predominance of one or other depending solely on the pressure. It is suggested that the Herzberg band may be in reality 2 branches, P and R, the lines of which are superposed near the head. C. A. S.

New emission spectrum of sulfur in photographic infrared. Maurice Desirant and Jules Duchesne. *Compt. rend.* 201, 507-8 (1935).—The new system extends from  $\nu = 13,521$  to 14,148, and is given by  $\nu = 13,521 + 806\epsilon - 4\epsilon^2 - 3\epsilon^3 + 2\epsilon^4$ , with  $\epsilon' = 0.3$  and  $\epsilon'' = 0.4$ . It is attributed to  $S_2$ . C. A. Silberrad

The  $\lambda$  4502 band of NH. R. Winstanley Iunt, R. W. D. Pearce and P. C. W. Smith. *Proc. Roy. Soc. (London)* A151, 662-9 (1935).—In the light from a hollow-cathode discharge in streaming  $NH_3$  a new band of NH, predicted theoretically, was observed at  $\lambda$  4502 Å. This band, photographed with high dispersion, was found to consist of single P, Q and R branches, of which the Q branch is the strongest. The analysis shows that it represents a  $3\pi - 12$  transition, the new rotational term differences for the  $3\pi$  state agreeing with those already known from the NH band at  $\lambda$  440 Å. ( $3\pi - 12$ ). The rotational constants, have been calcn. also for the new  $12^+$  state the discovery of which completes the list of stable states of the NH mol. resulting from combination of the  $4S$ ,  $2P$  and  $3P$  states of the N atom with the normal state of the H atom. C. C. Kiees

The continuous absorption spectrum of hydrogen bromide. C. F. Goodeve and A. W. C. Taylor. *Proc. Roy. Soc. (London)* A152, 221-30(1935).—With absorption cells ranging in length from 1 to 3355 cm. the intensity of the light transmitted by HBr gas at various pressures was measured in the spectral range from 2350 to 1800 Å. by a photographic process. From the observed extinction coefficients and the known characteristic function of the ground state of the mol an approx course for the upper potential energy curve has been derived, which indicates that the mol dissociates into 2 atoms each in its normal state.

C. C. Kiess

$\beta$  Bands of boron monoxide. A. Elliott. *Proc. Acad. Sci. Amsterdam* 38, 730-8(1935).—Measurements were made of the doublet septa of lines in the bands of BO. For K values below 53 the resoln. was too small to permit measurements. The observed septa in the  $\beta$ -bands are thought to be due to doubling of the  $\gamma$  level. If the doublet septa in this level is given by the expression:  $F_1(K + 1/2) - F_1(K - 1/2) = \gamma(K + 1/2)$ , then the value of  $\gamma$  calculated from the results is a little less than 0.02 cm.<sup>-1</sup>.

C. E. P. Jeffreys

Determination of atmospheric ozone. Comparison of spectrographic and chemical methods. Alexandre Dauvillier. *Compt. rend.* 201, 679-80(1935).—Numerous simultaneous detns of atm. O<sub>3</sub> were made during the winter 1934-5 at Abisko (Sweden) by the spectrographic (cf. Chalange, *et al.*, *C. A.* 28, 66337) (I), and chem. (cf. C. A. 29, 124) (II) methods. I gave an av. of 4.1 mg/100 cu. m. with max and min 6.4 and 1.5, II an av. of 3.3 with max. and min. 6.6 and 1.0. C. A. Silberrad

Reduced thickness of atmospheric ozone in polar winter. Daniel Barbier, Daniel Chalange and Etienne Vassy. *Compt. rend.* 201, 787-9(1935), cf. C. A. 28, 4980. —Measurements of the O<sub>3</sub> content of the atm. by the stellar spectrum method during the winter 1931-5 at Abisko (Sweden) showed reduced thicknesses of 1.55-3.45 mm, the amt. being low (under 2 mm.) when the origin of the air was in low latitudes, and high (often above 3 mm.) when of Arctic origin. C. A. Silberrad

Spectra of SeO and SeO<sub>2</sub>. R. K. Asundi, J. Jan Khan and R. Samuel. *Nature* 136, 612-3(1935).—The emission band of SeO consists of a long  $\nu'$  =  $\bar{\nu}$  progression expressed by  $\nu' = 55,453 - (882\nu' - 6\nu'^2)$  with  $\nu_0 = 33,107$  cm.<sup>-1</sup> and  $\omega' = 910$  cm.<sup>-1</sup>, giving the energy of disson. as 4.17 e.v. for unexcited mols. The excited frequency  $\omega' = 513$  cm.<sup>-1</sup>. SeO<sub>2</sub> has 2 vibration modes,  $\omega'_1 = 901$ ,  $\omega'_2 = 603$ , and those of the antisymmetric valence vibration  $\omega'_3 = 1189$  and  $\omega'_4 = 790$  cm.<sup>-1</sup>. The origin of the system is at 32,500. The close energy correspondence indicates strong localization of the bonds. G. M. E.

Infrared absorption of hydrogen peroxide. Alfredo Maione. *Nuovo cimento* 12, 358-60(1935).—The spectrum of a soln of 3% H<sub>2</sub>O<sub>2</sub> shows that the intensity of H<sub>2</sub>O bands at 1  $\mu$  and 2  $\mu$  is increased while their position remains unchanged. The bands at 3  $\mu$  and 4  $\mu$  are considerably altered, the latter being replaced by a doublet with max. at 4.4  $\mu$  and 4.85  $\mu$ . J. B. Austin

Absorption spectra of the halides of some elements of the second group. CdCl<sub>2</sub>, CdBr<sub>2</sub>, CdI<sub>2</sub>, ZnCl<sub>2</sub>, ZnBr<sub>2</sub>, and SnCl<sub>4</sub>. C. M. Bhaskar Rao and M. Karim. *Current Sci.* 4, 97(1935).—The regions of absorption maxima of the spectra of the above salts were obtained by plotting the values of log K against wave length. Results are tabulated. Helen S. Hopfield

Absorption spectrum of selenium dioxide. Léon Bloch, Lucienne Bloch and Choong Shin-Piaw. *Compt. rend.* 201, 654-5(1935).—Absorption begins in the ultra-violet at 200 Å as a series of bands degraded toward the red (I), with max. near 2700 Å. and 200-300 Å. in width, as temp. rises absorption increases and at 250 Å. is almost continuous from 2400 to 3200 Å. At 290 Å. a second similar but more diffuse system (II) appears with center about 4000 Å., which increases with temp. in similar fashion until at 315 Å. it extends from 3700 to 4300 Å., becoming almost continuous at 360 Å. At 430 Å. the 2 systems unite. In I there are 2 periodicities, 250 and 650 cm.<sup>-1</sup>, in II

only one, 200 cm.<sup>-1</sup> (Cf. Evans, C. A. 24, 3136.)

C. A. Silberrad

Absorption of light and double bonds. I. Problem and methods. Karl W. Hausser, Richard Kuhn, Alexander Smakula and Karl H. Kreuchen. *Z. physik. Chem.* B29, 363-70(1935).—Absorption measurements were made in a series of polyene dyes with photoelectric photometers, spectral photometers and with grating spectroscopes. The results of all 3 methods are in good agreement although the spectroscopic method shows the greatest deviations. The direction of the errors in this case depends on the position of neighboring bands and the magnitude of the errors is due to the width of the bands. II. Polyene aldehydes and polyene carboxylic acids. Karl W. Hausser, Richard Kuhn, Alexander Smakula and Max Hoffer. *Ibid.* 371-7.—Absorption was measured in hexane and abs. EtOH soln for Me-(CH=CH)<sub>n</sub>-CHO with  $n = 1, 2, 3$  and Me-(CH=CH)<sub>n</sub>-COOH with  $n = 1, 2, 3, 4$ . The height of the absorption bands and the strength of absorption are linear functions of  $n$ . The displacement of the bands toward longer wave lengths was smaller with increasing  $n$ , as is found in other homologous series. III. Investigation in the furane series. Karl W. Hausser, Richard Kuhn, Alexander Smakula and Adam Deutsch. *Ibid.* 378-83.—Absorption measurements in alc. and hexane soln were compared for aldehydes.



4 (CH=CH)<sub>n</sub>-CHO with  $n = 0, 1, 2, 3$  and for acids



5 (CH=CH)<sub>n</sub>-COOH with  $n = 0, 1, 2, 3$ . The place

of the long wave length absorption bands is very similar to that in aliphatic systems with 2 conjugated double bonds. This is also true for dicarboxylic acids as furan- $\alpha,\alpha'$ -dicarboxylic acid and muconic acid. The height of the bands is less than that of the corresponding aliphatic compds. The difference in the heights for the acids is approx. independent of  $n$ . IV. Diphenylpolyenes. Karl W. Hausser, Richard Kuhn and Alexander Smakula. *Ibid.* 384-9.—Absorption measurements were made on alc. and C<sub>6</sub>H<sub>6</sub> solns of Ph-(CH=CH)<sub>n</sub>-Ph with  $n = 1, 2, 3, 4, 5, 6, 7$ . The height of the principal absorption band and the strength of the band are nearly linear functions of  $n$ . The position of the absorption max. changes also with increasing  $n$ , as in other series. The differences found in frequency between the alc. and C<sub>6</sub>H<sub>6</sub> soln are independent of  $n$ . V. The absorption of compounds with conjugate double bonds of carbon at low temperature. Karl W. Hausser, Richard Kuhn and Georg Seitz. *Ibid.* 391-410.—Absorption spectra were obtained at -190° and between 3000 and 5000 Å. for (I) diphenylpolyenes, Ph-(CH=CH)<sub>n</sub>-Ph,  $n = 1, 2, 3, 4, 5, 6, 7$ , (II) polyene carboxylic acids, Me-(CH=CH)<sub>n</sub>-CO<sub>2</sub>H,  $n = 3, 4, 5$ , (III) lycopene, Me<sub>2</sub>-X<sub>1</sub>X<sub>2</sub>-Me<sub>2</sub>, where X = C=CH-CH<sub>2</sub>CH<sub>2</sub>- and Y = (-MeC=CH-CH=CH-), (IV) diphenylacetylenes, Ph-C≡C-Ph and Ph-C≡C-C≡C-Ph and (V) mesoporphyrin. For I, II and III, the position of the absorption bands is given by  $\nu = (\nu_0 + 37.0l + 47.1m) \times 10^{13}$  sec.<sup>-1</sup>,  $l, m = 0, 1, 2, \dots$ . For (I), there are other shorter wave-length bands that are similar to the biphenyl bands and of about the same intensity for all members of the series. They have been correlated with vibrations of the Ph groups in the mol. I or (IV), the observed bands agree with those of C<sub>6</sub>H<sub>6</sub> and are ascribed to the C triple bond. For (V) the strongest bands correspond with those of the double-bond C in aliphatic polyenes. VI. The fluorescence of diphenylpolyenes. Karl Wilhelm Hausser, Richard Kuhn and Ernst Kuhn. *Ibid.* 417-54.—The fluorescence bands of (I) are displaced with increasing  $n$  more than the corresponding absorption bands and the intensity becomes less. The dependence of the fluorescence on concn., solvent, temp. and state of

aggregation was investigated. The results are in agreement with the theory of quenched collisions, with collisions of the second kind more effective in the quenching than collisions with the solvent mol. At  $-190^\circ$ , the position of the bands is given by  $\nu = \nu_0 - 1550\tau - 1160\tau^2$  cm $^{-1}$  where  $\nu_0$  is the position of the band of shortest wave length and  $\tau$ ,  $\tau^2 = 0, 1, 2, \dots$ . The bands at 1550 and 1160 cm $^{-1}$  are also found in the Raman effect and correspond with C double-bond vibrations. The spectra are independent of the wave length of the exciting light and are a mirror image of the absorption spectra, the symmetry being very nearly the same with respect to position but differing in intensity. A few further compds were investigated, (II) with  $n = 5$  and isomethylloxin, showing almost identical fluorescent spectra.

G. M. Murphy

Ultraviolet absorption spectra of  $\alpha$ -ethylenic nitriles A. Castille and C. Ruppel. *Bull. soc. chim. Belg.* 44, 351-75 (1935).—Measurements have been made on hexane solns. of the cis and trans forms of nitriles of the crotonic series, RCH=CHCN. Values for  $\lambda$ ,  $1/\lambda$ ,  $\nu$ , and  $\log \epsilon$  are given for the following nitriles:  $\alpha$ -pentene,  $\alpha$ -hexene,  $\alpha$ -heptene,  $\alpha$ -octene,  $\alpha$ -nonene,  $\alpha$ -decene,  $\alpha$ -undecene, and  $\alpha$ -dodecene. Contrary to other homologous series contg. only 1 functional group, in this series the absorption varies with the mol wt. With the exception of  $\alpha$ -pentene nitrile the trans forms showed more pronounced bands than the cis variety.

W. J. Peterson

Absorption spectra of amines and amides in the near infrared, their use in determining amphoteric ions, influence of neighboring groups. Marie Freyrann and Paul Rumpf. *Compt. rend.* 201, 606-81 (1935), cf. C. A. 29, 3235 $^7$ .—The band near 1.04  $\mu$  characteristic of tricoordinated N does not appear in (NH $_3$ ), 2HCl aq or NH $_3$  (OH) HCl aq, but does so in (NH $_3$ ), 2HCl aq and NH $_3$  (OH) aq. Similarly it is absent in aq. solns. of glycocoll or taurine, but present in solns. of the Na and Li salts, resp., indicating that the neutral solns. contain the amphoteric ions  $^+NH_3CH_2CO_2^-$  and  $^+NH_3CH_2CH_2SO_3^-$ , but the salts the anions  $NH_2CH_2CO_2^-$  and  $NH_2CH_2CH_2SO_3^-$ . Similarly as regards  $\beta$ -toluenesulfonamide. Aq. soln. of urea shows a band at 1.0081  $\mu$ , but a concn soln. of the nitrate shows none. Connections are traced between the exact position of the band and the nature of the adjoining portion of the mol.; thus  $\lambda$  is increased by solubility of (OH), reduced by that of CO.

C. A. Silberrad

Raman spectra of oleum. Jean Chédin. *Compt. rend.* 201, 724-6 (1935).—The Raman spectra of solns. of SO $_2$  in H $_2$ SO $_4$  contg. up to 70% SO $_2$  shows the H $_2$ SO $_4$  lines below and their absence above about 45% SO $_2$ , and with further increase in the percentage of SO $_2$  the gradual appearance of lines attributable thereto. Conclusion. On adding SO $_2$  to H $_2$ SO $_4$  it first forms H $_2$ SO $_4$  (45% SO $_2$ ) with a Raman spectrum contg. lines 300, 327, 450, 735, 960 and 1250 cm $^{-1}$ ; any excess SO $_2$  above 45% dissolves as such in H $_2$ SO $_4$ , giving rise to lines at 239, 535, 688 and 1075 of increasing distinctness as its amt. increases.

C. A. S.

Absorption spectra of colloidal metallic and organic solutions and absorption of metallic films. Adolfo T. Williams. *Compt. rend.* 201, 665-7 (1935); cf. C. A. 26, 5436; 27, 1274.—The absorption bands and coeffs. of absorption of each of variously prepd. colloidal solns. of Ag, Au and Pt have been detd. with results in general agreement with those previously obtained. The absorption spectra of true and colloidal solns. of gamboge are very similar, only the coeff. of absorption changing by reason of lateral diffusion in the colloidal soln.

C. A. Silberrad

Infrared absorption spectra of plant and animal tissue and of various other substances. R. Stair and W. W. Coblenz. *J. Research Natl. Bur. Standards* 15, 295-316 (1935) (Research Paper No. 830); cf. C. A. 28, 1926 $^7$ .—Infrared absorption spectra of 15  $\mu$  of rubber, styrene, indene, polystyrene, polyindene, Cellophane, onion skin, translucent membrane of pith pokeweed, the seed septum of moonwort, and the seed wing of the cotton tree, dried specimens of chitin (the outer integument of insects), pith of feathers, air bladder of fish, bat's wings, film of

dried egg albumin, com. gelatin, lining of egg shell, wing of a dragon fly, polyvinyl acetate and polyvinyl chloroacetate resins, Glyprial resin, shellac, paraffin oil, sperm oil, linseed oil, CCl $_4$ , C $_2$ H $_5$ Cl, PhEt, PhCl,  $\alpha$ -C $_2$ H $_5$ Cl, AcOEt, PrDr, BuPr and C $_2$ H $_5$ Cl, were obtained with a mirror spectrometer, vacuum thermopile, and galvanometer. Percentage transmission is plotted against wave length. Considered as a whole, the proteins examd. exhibit a characteristic spectrum (with individual differences) as previously observed in groups of chem. compds., such as the carbonates, alics. and fatty acids. Gelatin shows characteristic absorption maxima at 3.0, 3.43, 4.2, 4.6, 5.9, 6.3, 6.8, 7.3, 8.1, 9.2, 11.0 and 12.2  $\mu$ . Some of these maxima have been identified with CH $_2$  and OH groups. Strong absorption at 6-8  $\mu$  is noted in hydrocarbons modified by NO $_2$  groups.

W. J. Peterson

The behavior of the infrared spectra of some aqueous salt solutions. Maria Battista. *Nuovo cimento* 12, 342-7 (1935).—The spectra of solns. of CuSO $_4$  (I), FeSO $_4$  (II) and MnSO $_4$  (III) have been detd. between 1  $\mu$  and 5.5  $\mu$ . The presence of I and II increases the intensity of all the H $_2$ O bands. The positions of the bands at 1.5  $\mu$  and 2  $\mu$  are not changed but the max. of the bands at 3  $\mu$  and 4  $\mu$  are shifted toward the red. III has a very complex effect, causing an increase in intensity for the band at 2  $\mu$  and a decrease for the bands at 3  $\mu$  and 4  $\mu$ . J. B. A.

A bibliography of the Raman effect. III. S. C. Sarkar and Dwijesh Chakravarty. *Indian J. Physics* 9, 553-622 (1935), cf. C. A. 27, 2381. E. H.

Depolarization of the light scattered by heavy water. S. Bhagavantam. *Current Sci.* 4, 94 (1935).—Heavy water supplied as 99.5% pure was examd. with sunlight focused by a long focal length lens as incident radiation. The depolarization of the transversely scattered light was found to be 0.04, as compared with 0.06 for ordinary water. This shows that the heavy water mol., similar to the ordinary water mol., has only a feeble optical anisotropy.

Helen S. Hopfield

Raman spectrum of heavy water. Edmond Bauer and Michel Magat. *Compt. rend.* 201, 667-9 (1935); cf. C. A. 28, 6635 $^1$ .—Examd. with Hg 4358 the Raman spectrum of D $_2$ O contains lines at 170, 350,  $\sim$  500, 1207, 2381 and 2501 cm $^{-1}$ , corresponding with those of H $_2$ O at 170, 500, 700, 1659, 3221 and 3435, resp.; 2380-2501 is a doublet, not a triplet. 170 and 176 are vibration bands, 350-500 and 500-700 are attributed to libration of the mol. around 2 of their axes of inertia.

C. A. Silberrad

Raman spectra of iodic acid and the alkaline iodates as solids and solutions. C. S. Venkateswaran. *Proc. Indian Acad. Sci.* 2A, 119-22 (1935).—The Raman spectra of the iodic acid as a function of concn. and of the iodates of Li, Na and K in the state of the solid and solns. have been investigated. The crystal of iodic acid yields 5 intense lines and a no. of weak and sharp lines, which are replaced by intense and broad bands in soln. The spectra of the solns. for a very wide range of concns. varying from 18 N to 0.15 N show an anomalous behavior regarding intensity and frequency shifts. From a qual. study of the solid and solns., evidence has been obtained for the progressive disson. of the acid. The results show that the disson. is incomplete even at concns. of 0.5 N. Suggestions have also been put forward that the acid is polymerized in the solns. to an appreciable extent, the polymerization decreasing with diln. The spectra of the iodates give in general lines which have been identified with the oscillations of the pyramidal form of the mols. of the AX $_3$  type. K biiodate crystal has a spectrum somewhat similar to that of iodic acid. The presence of a new line in its spectrum indicates the existence of H $IO_4^-$  ions. The soln. of this salt behaves as if it were only a mixt. of HIO $_3$  and KIO $_3$ .

Harold Gershinowitz

Raman spectra of isoprene, dipentene and ocimene. P. S. Srinivasan. *Proc. Indian Acad. Sci.* 2A, 105-12 (1935).—The Raman and the infrared spectra are compared and the results are interpreted in terms of the structures of the mols. The frequencies found for isoprene and ocimene lend support to the chem. structure assigned

to these compds. Although dipentene possesses a cyclic structure, it does not give the characteristic frequencies given by benzene or toluene. This is attributed to the presence of a puckered, rather than plane, ring.

Harold Gershnowitz

**Convergence error in depolarization measurements** R. Ananthkrishnan *Proc Indian Acad Sci* 2A, 133-42 (1935).—The scattering of light in an interference field is discussed, and it is shown that for the simple case of 2 parallel plane-polarized intersecting beams, the depolarization at the interference maxima gives the correct value, while at the minima the depolarization ratio is reversed. The av. of the depolarization taken over the whole field is higher than the correct value. The treatment is extended to the cases in which a lens covered with a square aperture, and with a circular aperture, resp., is used to conc. the light on the scattering medium. It is shown that the observed values of the depolarization would deviate from the genuine values by a correction factor which involves the square of the angle of convergence. It is pointed out that the same results follow by treating the incident beam as a bundle of incoherent rays. Some consequences of the theoretical results are discussed and the necessity for the perfection of the optical parts used in depolarization work is emphasized. Exptl. results are given which illustrate the points discussed in the paper.

H. G.

**Redetermination of the depolarization of light scattering in gases and vapors** R. Ananthkrishnan *Proc Indian Acad Sci* 2A, 153-60 (1935), cf. C. A. 28, 7933<sup>1</sup> and preceding abstr.—It is pointed out that the existing depolarization data of gases and vapors are gravely defective, and a redet. of the values has been made with improved exptl. technic. The results obtained are strikingly different from those of previous workers, and afford at the same time a natural explanation of many of the existing anomalies. The genuine depolarization of methane appears to be only of the order of 0.3%, which is in all probability to be attributed to the highly depolarized vibrational Raman lines. The depolarization shows a steady increase as one goes to the higher members of the homologous series, but the values in all cases are much smaller than they were hitherto assumed to be. Cyclopropane shows a very small depolarization in conformity with the known small depolarization of cyclohexane, while propylene shows a high value as would be expected from the presence of the double bond in it. MeCl is more anisotropic than FCl, and the depolarization factor of the former is much higher than that of CH<sub>4</sub>. CCl<sub>4</sub> shows an extremely small depolarization of 0.15%, which is discussed in detail. It is tentatively concluded that the depolarization of A is nil. The depolarization of H<sub>2</sub>S is only 0.3%. An explanation is offered for the low depolarization of the hydride mols. in general.

H. G.

**Active nitrogen of long duration, law of decay and of increased brightness on compression** Lord Rayleigh *Proc Roy Soc (London)* A151, 567-84 (1935).—The catalytic activity of the walls was reduced to a min. when they were wetted with H<sub>2</sub>SO<sub>4</sub> or H<sub>3</sub>PO<sub>4</sub>. With H<sub>3</sub>PO<sub>4</sub>, in a large bulb, the glow remains visible for 6 hrs. or more, and the reaction is probably of the sec. order. When *Apezon* oil is used, the reaction is apparently of the first order. Photometric measurements of the luminosity made while the gas is being compressed show that the intrinsic brightness varies as the square of the concn. of the active material. This indicates a bimol. reaction, in which the excess of neutral N<sub>2</sub> mols. takes no part, confirming the observation that the time of decay at const. vol. between 2 standard intensities is, at low pressures, almost independent of the pressure. At higher pressures, some secondary cause for the action of neutral gas, comparable to the

action of the walls, perhaps, must be sought.

E. R. Rushton

**Action of hydrogen bromide on the nitrogen afterglow** W. H. Rodush and M. L. Spealman *J. Am. Chem. Soc.* 57, 1891-2 (1935).—The presence of HBr or Br intensifies certain bands in the N afterglow while certain other bands are weakened. It appears to be the Br atom rather than the HBr mol. which is responsible for the effects.

M. McMahon

**Influence of light on the stability of bleaching solutions.** J. P. A. Tuenter *Chem. Weekblad*, 32, 429-30 (1935).—The amt. of active Cl<sub>2</sub> in bleaching solns. decreases after 70 days' exposure to daylight in filled white glass bottles 48-61%, in half-filled bottles 63-76% and in half-filled brown bottles 11-29%.

B. J. C. v. d. H.

**A photochemical reaction of chlorophyll** Emil Baur *Helv. Chim. Acta* 18, 1157-60 (1935).—The following systems mixed with photographic collodion were exposed to sunlight: (1) chlorophyll, CO<sub>2</sub> water, (2) chlorophyll, methylene blue, air-free, CO<sub>2</sub> free water, and (3) chlorophyll, methylene blue, CO<sub>2</sub> water. In (1) no CH<sub>2</sub>O was found; in (2) and (3) 0.5 mg. was found. The results suggest that chlorophyll in the presence of oxidation-reduction systems such as methylene blue or ferric salts converts carbonic acid into O and carbohydrates. Since reaction occurs in CO<sub>2</sub>-free water the COOH groups of chlorophyll itself must supply the CO<sub>2</sub>. The exptl. amt. of HCHO found is in agreement with this hypothesis.

E. O. W.

**Behavior of iodostarin in light** Emil Baur, *Helv. Chim. Acta* 18, 1149-56 (1935).—A CHCl<sub>3</sub> soln. of iodostarin (6,7-di-*ortho*-6-octadecenoic acid) undergoes decompn. in light from the sun, a Hg vapor lamp or an Osram lamp forming I<sub>2</sub> and tartaric acid (6-octadecenoic acid). The reaction is reversible and has been measured from both sides, at room temp. the equl. follows the mass-action law. Both the decompn. and formation of iodostarin are I-sensitized reactions, the rate in each case is proportional to the light intensity.

E. O. W.

**Further investigation of the decompn. of flavins by light** P. Karrer and Hans P. Meerwein *Helv. Chim. Acta* 18, 1126-30 (1935); cf. C. A. 29, 3605<sup>4</sup>, 4678<sup>1</sup>.—The decompn. by sunlight of a flavin contg. a tert. OH group, *o* (2'-hydroxy-2' methyl-1'-propyl)isoxaloxazine (I), shows that it is not necessary that dehydrogenation occur at the 2'-position of the hydroxy side chain, as previously suggested. However, the rate of decompn. is much slower with I than with flavins contg. a primary or a secondary OH group. As with the other flavins photolysis proceeds more rapidly in aq. MeOH than in water.

E. O. W.

**Spectrum analysis** F. Twyman *Nature* 136, 609 (1935).—It is suggested that "spectrochem analysis" be used to denote the analysis of substances by their spectra, leaving "spectrum analysis" to indicate the analysis of the structure of a spectrum.

E. O. W.

**Variation of detonation spectra with nature of surrounding gas** (Michel Lévy, Muraour) 24 Raman spectrum of the esters of some derivatives of  $\beta$  ketobutyric acid (Milonc) 10

**Light polarizers** Alvin M. Marks *Tr* 784,487, July 22, 1935. A transparent polarizer is made by suspending a transparent isotropic support in a soln. of an optically active substance and evap. the solvent so that a cryst. layer is formed on the support.

**Activating substances** Rafal Products Radium Berne snc anon *Swiss* 171,897, Apr. 16, 1935 (Cl. 116/). App. for submitting solid or liquid substances to the action of Ra is described.

#### 4-ELECTROCHEMISTRY

COLIN G. FINK

**Electrofiltration in electrolysis of gels** Jean Swynedauw *Compt. rend.* 201, 821-3 (1935); cf. C. A. 28,

5704<sup>1</sup>.—The phenomena of cathodic and anodic swelling previously described can be summarized in the statement

that if in any region the direction of the gradient of ionic concn. and of electrofiltration are the same, swelling occurs, if opposite, depression. Isoelec. depression is also shown to be due to electrofiltration. C. A. Silberrad

**Theory and practice of chromium plating.** II Mechanism of chromium deposition. N. D. Biryukov, S. Makareva and A. A. Timokhin *Korrosion u Metall-schutz* 11, 193-201 (1935) — See C. A. 29, 1717

**Throwing power of electrolytic baths.** Albert Portevin and Michel Cymboliste *Compt. rend.* 201, 810-21 (1935), cf. C. A. 29, 6510<sup>1</sup>.—With the spiral arrangement previously described the throwing power (II) has been plotted against current density,  $D$  ( $= 0.5$  A./sq. dm.), for ordinary baths for Cd and Zn (cyanide), Sn ( $\text{Na}_2\text{SnO}_3$ ), Ni and Cu (sulfate), and Cr ( $\text{CrO}_3$ ) for varying temps. ( $T$ ), and concns. ( $c$ ). As  $D$  increases II diminishes except for Cd and Cr,  $c$  increase affects the yield of Cu and Ni but little, increases it for Cr and diminishes it for Cd, Zn and Sn, II decreases with rise in  $T$  for Cu, Cd and Zn, and Ni in a coned bath, but in general varies little for Cr; with increase in  $c$  it increases for Ni up to a limit, and changes little for Cu and Cr, increase in cond. of the bath may cause an increase or decrease in II. C. A. Silberrad

**Electrometric control of displacement reactions.** Suzanne Veil *Compt. rend.* 201, 855-7 (1935), cf. C. A. 29, 5359<sup>1</sup>.—Electrodes of Pt and Zn previously connected to an electrometer were inserted, the Pt was placed first in aq.  $\text{CuSO}_4$  (e. g., 0.2 N) and the e. m. f. developed on inserting the Zn electrode plotted against time. The e. m. f. first measured (1.19 v.) decreases to a min. (0.98 v.) after about 0.5 hr., and then rises to about 1.18 v. after 2 hrs., remains approx. steady for 1 hr., and then becomes irregular. (Results vary with form of electrodes and concn. of bath.) It is considered that the phenomena point to the chemical being merely accessory to the Volta effect, and that the initial decrease in e. m. f. is a secondary Volta effect caused by replacement of the Zn surface of the cathode by a Cu one. C. A. Silberrad

The current flow between a small (point) glow cathode and a very large (infinite) anode in glow discharges with varying gases. F. Keller. *Z. Physik* 97, 8-33 (1935).—In the rare gases, diffusion streaming and gradient flow can be distinguished. The diffusion stream takes place when the electron density in front of the cathode is sufficient to make up for the fall in concn. This current streaming obeys the theoretical law. With too small an electron density, gradient current flow is observed. Measurements are made on  $\text{N}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{O}_2$  and  $\text{H}_2$ . S. T.

**Reactivity of carbonaceous materials in the electric arc.** M. S. Maksimenko and Ya. M. Markovskii. *Metallurg* 10, No. 2, 81-7 (1935); cf. C. A. 29, 7183<sup>1</sup>.—In an atm. of N the loss in wt. of the C electrodes is 4-5 times greater than in A because of the formation of  $(\text{CN})_2$ ,  $\text{C}_2\text{H}_2$  is formed in a H atm. and CO in a  $\text{CO}_2$  atm. The quantity of these gases formed varies with the type of C electrode; those of petroleum coke are more reactive than graphite electrodes. H. W. Rathmann

**Nitrogen in arc-furnace steel and deoxidation by carbide slag.** Isao Hayashi. *Tetsu-u-Hagane* 21, 707-22 (1935).—The high N in elec. steel is due to  $\text{CaCN}_2$  produced in carbide slag by the reaction between  $\text{CaC}_2$  and  $\text{N}_2$  in the furnace. The reaction between  $\text{CaCN}_2$  in the slag and the metal bath is assumed to be  $\text{CaCN}_2 + 2(\text{FeO}, \text{MnO}) \rightarrow \text{CaO} + 2(\text{Fe}, \text{Mn}) + \text{CO} + \text{N}_2$ ;  $\text{CaCN}_2 + \text{CO} \rightarrow \text{CaO} + 2\text{C} + \text{N}_2$ . The  $\text{N}_2$  liberated goes into the bath. The greater the N content of the bath, the more nearly complete is deoxidation; and the lower the temp. of the bath, the more  $\text{CaCN}_2$  is produced in slag.

**The reaction mechanism at a graphite anode.** V. Sihvonen. *Suomen Kemistilehti* 8B, 35 (1935) (in German). E. E. Jukkola

**Organic depolarizers.** W. H. Hunter and L. F. Stone. *J. Phys. Chem.* 39, 1133-47 (1935).—The single potentials of quinone, ferro-ferric, m-nitroaniline, azobenzene-sulfonic acid,  $\text{H}^+$  and quinone-quinhydrone against cathodes of Pt, Au, Ag, Ni and Sn; 3,3'-diaminoazobenzene and

1 quinhydrone against Pt; and  $\text{Sn}^{++}$  against Sn were measured in a special cell (diagram of app. given) while a const. current of 50 milliamperes was passed through. The magnitude of the increased positiveness obtained with any one depolarizer depends on the work function of the cathode, i. e., the work required to remove electrons, and on the "electron affinity" of the depolarizer, i. e., its ability to reduce the potential of the electrode to its normal potential against its own ions. With a given depolarizer the values of the single electrode potentials become more neg. and the work function increases in the order of metals given above. The first 5 depolarizers are arranged in the order of decreasing positiveness against Au and are practically the same for the other cathodes. The relative values of electron affinity must increase in the same order. On the assumption that all cations present in a soln. may be considered as depolarizers, "overvoltage" of any depolarizer as well as of  $\text{H}^+$  is considered the result of electron affinity and other effects such as adsorption of H into the metal and gas films. Janet E. Austin

**Chemical detection of the use of azides in the preparation of electron tubes.** H. Fritz *Mikrochemie* 18, 162-8 (1935).—In a previous paper, C. A. 29, 5374<sup>1</sup>, it was pointed out that the chem. examn. of the mirror deposits in electron tubes can give practical indications concerning the method used in making the tubes. In this paper it is shown that the detn. of the total N content of the mirror deposits gives pos. proof as to whether azides were used in the manuf. of the tubes. The tubes which have been made without the use of azides contain only about 1-2% of N per tube. When the N content of the mirror deposits is distinctly higher, it is certain that azides, usually  $\text{BaN}_3$ , have been used. The exact quantity cannot be detd. in this way but some idea as to whether little or much azide has been used can be shown. W. T. H.

**Elec. furnace for heat-testing refractory products** (Fr. pat. 784,105) 19. **Elec. furnaces for melting glass** (U. S. pat. 2,018,883-4) 19.

**Electric batteries.** La compagnie générale de piler Wonder Fr. 784,413, July 22, 1935. Anodes for batteries of the Zn-NH<sub>4</sub>Cl type are composed of pure electrolytic Zn alloyed with Pb which is preferably 0.75-0.9% of the wt. of Zn.

**Galvanic battery.** Henry W. Brownson and Richard Chadwick (to Imperial Chemical Industries Ltd.). U. S. 2,018,942, Oct. 29. A homogeneous, malleable alloy of Zn with less than 0.5% of Hg is used for the manuf. of pressed cup-shaped battery containers.

**Galvanic battery.** "Petrus" Chemische Fabrik A.-G. Fr. 784,181, July 22, 1935. In a battery depolarized by air the liquid electrolyte contains a substance capable of swelling, e. g., starch products, in amt. such that the liquid is not sufficient to cause complete swelling. The pos. electrode is surrounded by an impregnated paper which is so slowly permeable to liquid that during the swelling operation no liquid reaches the electrode.

**Storage batteries.** Albert Strasser and Walter Germann. Swiss 175,782, July 1, 1935 (Cl. 109). To the active mass for Pb accumulators is added c. p. metallic Si, preferably in the colloidal form.

**Electric cell of the Lalande type.** Martin L. Martus and Edmund H. Becker. U. S. 2,018,563, Oct. 22. A battery with a Zn anode is used with a caustic alkali electrolyte soln. contg. about 1% of an added Mg salt such as  $\text{MgCl}_2$ ,  $\text{MgCO}_3$ , or Mg phosphate, and about 2% of an added reducing agent such as Na formate and hydroquinone.

**Electrolytic condenser.** Samuel I. Cole (to Aerovox Corp.). U. S. 2,018,480, Oct. 22. Structural features.

**Electrolytic condenser.** Werner Herrmann and Erich Evers (to Siemens & Halske, A.-G.). U. S. 2,018,522, Oct. 22. In producing an electrolytic condenser, a spacing strip of fibrous material such as paper or cloth is spirally wound on at least one thin metallic ribbon so as completely to enclose the ribbon, and the wound ribbon is



moved through an electrolyte and afterward is wound on a core

Electrical conductor Henri Bienfait and Willem L. C. van Zwet (to Radio Corp. of America). U. S. 2,018,343, Oct. 22. A solid nonconducting core or core formed of material not freed from oxides or other metal compds is provided with a coating comprising finely divided particles of metal such as Zn coated with Ag and mixed with an org. binding agent.

Electrical resistances Steatit-Magnesia A.-G. Fr. 784,325, July 22, 1935. Conductive materials, such as charcoal, graphite, metal powder or metal oxides, in a fine state of division, are suspended in etherified compds of cellulose to which softening agents, resins, waxes or filling materials may be added.

Electrolytic deposition of metals Bauindustrie A.-G. (Bieg). Fr. 784,574, July 22, 1935

Electroplated master phonograph records. Alexander G. Russell (to Bell Telephone Laboratories, Inc.). U. S. 2,018,471, Oct. 22. Various structural and mfg. details

Electrolysis Peintal soc. anon. Swiss 176,639, July 1, 1935 (Cl. 44a). A bath for the electrolytic production of an oxide layer on Al or Al alloys contains a sulfonic acid of an aromatic hydrocarbon such as benzene-, naphthalene-, or anthracene-monosulfonic acid, or a mixt of these. Examples are given

Electrolytic apparatus "Elin" A.-G. fur elektrische Industrie. Ger. 618,062, Sept. 2, 1935 (Cl. 48a 14). See Austrian 140,522 (C. A. 29, 3611)

Electrolytic apparatus for making thin sheet metal. Anaconda Copper Mining Co. Ger. 618,213, Sept. 4, 1935 (Cl. 48a 9). See Brit. 419,764 (C. A. 29, 2853)

Electrolytic cell suitable for producing oxygen and hydrogen. Andrew C. Brown (to Compressed Industrial Gases, Inc.). U. S. 2,018,703, Oct. 29. Structural details

Anodic oxidation of aluminum and its alloys Aluminum Colors, Inc. Ger. 618,195, Sept. 3, 1935 (Cl. 48a 16). See Fr. 758,545 (C. A. 28, 3007)

Silicon carbide Henry P. Kirschner (to Carborundum Co.) U. S. 2,018,133, Oct. 22. A small proportion of buckwheat hulls or the like is added to a charge of sand, C and salt for producing SiC in an elec. furnace.

Protecting metals against corrosion Societe d'exploitation des cables electriques, systeme Berthoud, Borel et Cie., and James Borel Swiss 175,038, Apr. 16, 1935 (Cl. 44d). Metals are protected against electrolytic corrosion by covering them with a noncorrosive coating in direct elec. contact with the metal and capable of conveying electricity from the metal by a flow of electrons. In the example the metal is coated with bitumen or fiber contg. free particles of C

Mixture for liberating alkali metal. Percy L. Spencer (to Raytheon Mfg. Co.) U. S. 2,018,815, Oct. 29. A mixt. suitable for supplying alkali metal in elec. discharge devices comprises a stable alkali metal compd. such as CsCl together with Cd

Induction furnaces. Ivar Rennerfelt. Brit. 432,712, July 29, 1935. A furnace with an open magnetic core having pole pieces adjacent to the hearth is provided with 1 or more arcing electrodes arranged in the upper part of the furnace. Cooling tubes for the core are provided.

Coreless induction furnace operated with three phase current Heraeus Vacuumschmelze A.-G. and Wilhelm Rohm. Ger. 616,900, Aug. 7, 1935 (Cl. 21a. 18 03). Addn. to 572,445 (C. A. 27, 2833).

Electric combustion furnace for organic chemical analysis Rudolf Engeland. Ger. 618,466, Sept. 10, 1935 (Cl. 42i 3 02)

Electrically heated salt bath furnace Siemens-Schuckertwerke A.-G. (Johann Schnepf, inventor). Ger. 618,262, Sept. 4, 1935 (Cl. 21a. 15 01). Preliminary fusion of the salt is effected by a heating element resembling an elec. immersion heater, which is embedded in the salt.

Apparatus for ionizing air or other gases Rudolf Auerbach (to General Elec. Co.). U. S. 2,019,333, Oct. 29. Structural and operative details

Apparatus for generating negative ions Earle W.

Ballentine (to Solar Industries, Inc.). U. S. 2,018,434, Oct. 22. An app. for generating neg. ions substantially free from pos. ions in air at atm. pressure and suitable for treating air of houses comprises a metal anode which may be formed of sheet metal, a resistance wire of Ni-Cr alloy adapted to serve as a hot cathode, and a source of a. c. by which the wire is energized and the anode given a potential difference. Structural details are described

Obtaining coherent masses from powdered materials The British Thomson-Houston Co. Ltd. Brit. 431,509, July 9, 1935. A mold for mfg. cemented carbides by the application of heat and pressure comprises a relatively heavy W or Mo tube supporting, by a shoulder, a hollow C tube within which the charge is compressed by plungers. The assembled mold is placed between C blocks and clamped between H<sub>2</sub>O-cooled electrodes, the current between which heats the metal tube, which acts as a heat reservoir and reinforcing member for the C tube.

Apparatus for decomposing or vaporizing liquids by means of a submerged electric arc. Kurt Wappler. Ger. 618,161, Sept. 3, 1935 (Cl. 12a. 4).

Noncontact making relays. Erich Habann. Brit. 431,861, July 10, 1935. In app. for generating or amplifying elec. oscillations with the aid of finely divided materials, e. g., oxides, having a neg. resistance characteristic, the oxide, etc., is present as a coating on cores that are good conductors of heat and electricity. Thus, Pt pellets of about 0.1 mm. diam. and coated with V<sub>2</sub>O<sub>5</sub> are mixed with similar pellets coated with Mn<sub>2</sub>O<sub>3</sub> in the proportions already known for mixts. of the powd. oxides. The metal of the oxide may be used as core and, in case a mixed oxide coating is desired, e. g., ZnO and CuO, the core may be a suitable alloy, e. g., of Zn and Cu. In an alternative arrangement, Pt wires are coated with the desired oxide, e. g., by spraying, drawing through the molten oxide, electrolysis, e. g., of vanadates or zincates, or, in the case of ZnO, by sublimation or by coating the wire electrolytically with the appropriate metal and then oxidizing

Electrodes for rectifiers Harold B. Conant. U. S. 2,017,842, Oct. 22. A Cu plate is heated in a furnace having a temp. of 1000° until a coating of an O compd. is formed on it, then cooled to about 600°, quenched in a bath of heavy mineral oil having a temp. of at least 70°, and then cooled to room temp. in a bath of relatively light mineral oil.

Electric fuse. Vernon Hope. U. S. 2,018,556, Oct. 22. A fusible element comprises a metal of low m. p. embedded in an arc-quenching powder such as marble dust and boric acid is held in contact with the surface of the metal to cause the metal to run when it is melted by an overload current.

Discharge apparatus N. V. Philips' Gloeilampenfabriek. Brit. 431,923, July 17, 1935. An artificially cooled Hg-vapor discharge tube having a gas-filling has an internal diam. of less than 3.5 mm. and the Hg-vapor pressure is so high that the potential drop in the discharge path exceeds 150 v. per sq. cm. Cd or Zn may be added to the Hg in the form of amalgams. In 1 form, the lamp is constituted by a cylinder of quartz of internal diam. 2 mm. and wall thickness 2 mm., the W leads being surrounded by insulating material and sealed into the lamp through intermediate glass members composed of SiO<sub>2</sub> 83.3, B<sub>2</sub>O<sub>3</sub> 8.4, Al<sub>2</sub>O<sub>3</sub> 2.4 and CaO 0.4% sealed to the quartz.

Electron discharge tube containing a gas such as neon, argon or helium Charles J. Kayko (to Sparks-Withington Co.). U. S. 2,018,174, Oct. 22. Various structural details

Electron emission elements Chester H. Braselton (to Sirian Lamp Co.). U. S. 2,018,993, Oct. 29. A pasty mixt. is formed contg. BaCO<sub>3</sub>, SrCO<sub>3</sub>, BaCl<sub>2</sub> and dist. water, the paste is spread upon a supporting member such as a wire and the assembly is heated in the presence of O to convert the BaCl<sub>2</sub> to BaO and then at a higher temp. to convert the BaCO<sub>3</sub> to BaO

Light-sensitive cells N. V. Philips' Gloeilampenfabriek. Brit. 431,746, July 15, 1935. A photoelec. device comprises a base plate of metal, e. g., brass, or of C,

which carries a photoelec. semiconductive layer of Se, Cu iodide, Cu<sub>2</sub>O or Mo sulfide which is sep'd. by a thin barrier layer of artificial resin from a metallic conductive electrode, e. g., a translucent Au film, the boundary between the Se and barrier layers being within reach of the light rays. The resin may be applied to the Se layer as a soln. of a PhOH- or cresol-CH<sub>3</sub>O resin in alc. and, after the evapn. of the solvent, the resin is further hardened by heating to about 200°. Alternatively, if the barrier layer is made of polystyrene, which is sol in CCl<sub>4</sub>, no subsequent heating after evapn. of the solvent is necessary.

**Suppression layer cathodes for photocells.** Franz Rother. U. S. 2,019,088, Oct. 29. In producing suppression layer cathodes with a Cu base and an oxide coating, the base is coated with a thin layer of another metal such as Au which is not of the alkali group and then heated to cause the Cu to diffuse into and form an alloy with the other metal and to effect oxidation of at least part of the Cu so diffused to form an oxide coating over the alloy. Cf. C. A. 29, 20324.

**"Clean-up" material suitable for use in thermionic valves.** John D. McQuade (to Kemet Laboratories Co.). U. S. 2,018,965, Oct. 29. A mixt. of comminuted Cu about 25-50% is used with comminuted silver-colored active "clean-up" material such as a Ba-Sr-Mg alloy and serves to render the material more readily seen, by giving color contrast.

**Thermionic cathodes.** Siemens Electric Lamps & Supplies Ltd., Percy D. Oakley and John N. Aldington. Brit. 431,810, July 16, 1935. In a high-pressure, metal-vapor discharge lamp, the emissive electrode consists of a core of emissive material surrounded by a helix of Ta.

**Glowing cathode.** Akt.-Ges. Brown, Boveri & Cie. Swiss 174,419, Apr. 1, 1935 (Cl. 112). A discharge tube cathode made of graphite coated with a metal of m. p. about 2000°, e. g., Mo or W, is described.

**Cold cathode electric discharge lamp containing gas such as neon and argon.** Franz Rother (to Rodalite Co.). U. S. 2,018,974, Oct. 29. At least 2 gas components such as Ne and Ar are used for producing described color effects. Cf. C. A. 29, 4134.

**Discharge lamps.** The General Electric Co. Ltd., Victor J. Francis and John W. Ryde. Brit. 431,651, July 12, 1935. In a high-pressure metal-vapor lamp having a double-walled envelope, blackening of the walls of the inner discharge tube is prevented by providing a gaseous or vapor filling in the space between the walls; the filling may be O, H, air, H<sub>2</sub>O vapor, S vapor, CO<sub>2</sub> or N<sub>2</sub>O in specified amts. The glass that may be used for the tube has the compn., SiO<sub>2</sub> 54, CaO 15, B<sub>2</sub>O<sub>3</sub> 8, Al<sub>2</sub>O<sub>3</sub> 21, Na<sub>2</sub>O 1 and BaO 0.5 parts.

**Positive column gaseous discharge lamp containing neon.** Leo L. Beck (to Claude Neon Lights, Inc.). U. S. 2,018,620, Oct. 22. Various details are described of a lamp contg. Ne together with 0.07-1.25% of A, Kr or Xe.

**Electric illuminating lamp for operation on alternating current and containing a rare gas such as neon and mercury.** Jean M. E. de Beaufort and Anton Lederer (in part to Ernest A. Lederer). U. S. 2,018,347, Oct. 22. Various details.

**Electric incandescent lamp.** Mary R. Andrews (to General Elec. Co.). U. S. 2,019,331, Oct. 29. A sep. heated body of carbon is placed adjacent to but out of direct contact with a lighting body contg. carbide of Ta, Hf, Zr or Cr.

**Electric incandescent lamp filaments.** Samuel Ruben (to Sirian Lamp Co.). U. S. 2,018,470, Oct. 22. Metal wire filaments such as those of W or Mo are coated with a mixt. formed from a compn. of Th oxide about 10% and Ce oxide about 1 part through which about 25% of H oxide is distributed. Cf. C. A. 29, 4144.

## 5—PHOTOGRAPHY

F. R. BULLOCK

**Recent developments in color photography.** Gustav Grote. *Phot. Korr.* 70, 149-64(1934), cl. C. A. 28, 7170<sup>1</sup>.—A summary.

**The flash exposure.** W. B. Hislop. *Process Engraver's Monthly* 42, 265-6(1935).—The flash exposure in half-tone screen work, besides overcoming the inertia of the emulsion in the shadows, improves the gradation of the reproduction and the printing of the shadow dots.

**A photographic development effect.** P. Odilo Hofmann. *Physik. Z.* 36, 650(1935).—It is shown that 2 images made at different light intensity levels, but which ultimately will be of equal d., develop at unequal rates in the initial stages of development. The image made at the lower intensity level appears first. J. H. Webb.

**Acceleration of development by thiocarbamides.** Lippman-Cramer. *Phot. Korr.* 70, 145-8(1934).—When almost unripened (such as lantern) plates are bathed for 1.5 min. in a 0.001% soln. of thionamine or thiocarbamide before being developed in hydroquinone, a remarkable acceleration, without any production of fog, is seen in the early stages of development. High-speed plates show, however, considerable fog, in addition, to a similar acceleration of development. Acceleration can also be seen, prior to the well-known appearance of "pseudosolarization," on high-speed plates when thionamine or thiocarbamide in comparatively large amt. (e. g., 0.025%) is merely added to the developer. This pseudosolarization has not been found, with any one of 7 brands of plates tested, to give a min. d. as low as would be required for a practically useful process.

**Research on fine-grain development.** Georg Schwarz. *Kino-technik* 16, 73-8(1935).—A study of the properties of a no. of different developers leads to the conclusion that very little difference exists for equal d. and  $\gamma$ . Prog-

ress in reducing graininess depends largely on the photographic material.

**Fine-grain developers.** Lippman-Cramer. *Phot. Korr.* 71, 40-50(1935).—Many recent investigations have verified L.-C.'s contention that, at equal values of  $\gamma$ , the so-called fine-grain developers show no advantage over normal developers. In a recent paper comparing certain fine-grain developers with dild. M.-Q. developers, Schwarz (preceding abstr.) found that the only difference was a longer exhaustion life for the fine-grain developers. L.-C. points out that this is to be expected because of the much higher concn. of developing agents.

**Do emulsion and developer alone determine fineness of grain?** Ph. Strauss. *Phot. Ind.* 33, 721-3(1935).—Much stress is laid on the developer in published articles on the subject. S. points out that factors such as the time of development, time of exposure, quality of the exposing light, etc., should not be overlooked. In making enlargements, the optics of the projection machine, and the contrast and surface of the paper used, are important in detg. the graininess in the print. C. F. M.

**Different factors determining the behavior of a photographic developer.** M. C. F. Heukers. *Chem. Weekblad* 32, 328-9(1935).—A short review of recent work on the influence of different factors, especially pn, on the behavior of developers (C. A. 27, 6260; Reinders, C. A. 28, 6377). Conclusions: Hydroquinone is 5 times more sensitive to pn changes than metol. Hydroquinone develops from pn 9.5 on, metol from pn = 7 on. At pn 10.5 to 11 both substances give equal ds. At lower pn both increase the induction period, metol less than hydroquinone. If errors due to pn as well as to the induction period are eliminated the blackening d. is a linear function of developer concn. The influence of KBr is considerably more at slightly lower pn; it causes increased induction time for hydroquinone. In metol-hydroquinone mixts.

at  $pH = 9$  practically all action during short developing periods is due to metal, at  $pH 8.8$  the hydroquinone can as well be omitted. The most effective mixt. is 1 part metal to 3-5 parts hydroquinone. B. J. C. van der H.

Sensitizing photogravure tissue. Alois Heigl and Karl Stötzer. *Reproduktion* 6, 159-61(1935).—Various phot. control sets are discussed in connection with the  $K_2Cr_2O_7$  bath. While for C transparencies sensitized tissue may be kept under refrigeration for months, this is not considered applicable to photogravure. A C transfer may show only slight evidence of scum on glass or celluloid, and still form a heavy scum on Cu. A. M.

Influence of water on the sensitivity of photographic emulsions. André Charriou and Suzanne Valette. *Bull. soc. franç. phot.* 22, 156-8(1935).—See C. A. 29, 4276<sup>5</sup>. P. W. Vittum

Desensitizing properties of iron nitrosulfide. Jacques Duclaux. *Science ind. phot.* 6, 321-2(1935).— $NaFe(NO)_2$  (prepn given) is a sol black compd possessing marked desensitizing properties. Details are given, the best results being attained by long immersion in a soln of the compd. A tendency to stain is diminished by adding alc. to the bath. Unlike some dye desensitizers, the compd shows no sign of sensitizing action at high diln. Fogged plates may be cleared by treatment with the compd followed by exposure to infrared radiation.

L. G. S. Brooker

Characteristic surface for the Villard effect. III. Hans Arens. *Z. wiss. Phot.* 34, 125-35(1935); cf. C. A. 26, 3740, 28, 7182<sup>1</sup>.—In continuation of the previous expts with a low-speed, non-color-sensitized film emulsion, 2 other emulsions, 1 of which is of large grain, have now been used and A's earlier results have been qualitatively confirmed. It is considered that the results are in agreement also with those of Wood, Schaum, Volmer and Langerbanns, although the present investigation is more extensive, since it includes the independent variation in intensity and time of the white-light exposure.

E. R. Bullock

The concept of inert gelatin. A Stegmann. *Phot. Ind.* 33, 298-9(1935).—S defines inert gelatin as a gelatin which is almost entirely free from sensitizing substances. He considers that ripening sensitizers would be better termed ripening accelerators, since when added to a restrained gelatin they exert a desensitizing action on continued ripening.

E. R. Bullock

Restraining substances in gelatin, and the age of the animal. A Stegmann. *Phot. Ind.* 33, 276(1935).—Calf gelatin is richer in restraining substances than is the corresponding gelatin from cattle. Putrefaction in the luming operation is also of influence, this renders the restraining substances more readily removable. E. R. B.

Structure of negative layers. P. Wiegler. *Schweiz. Phot.-Ztg.* 37, 93-5(1935).—The graininess of a negative depends not only on the structure of the original emulsion but also on the developer and the degree of development. For fine grain development, W suggests the following: metol (elon) 16 g,  $Na_2SO_4$  75-100 g,  $H_2O$  1 l., this soln to be dild. for use with an equal vol. of either  $H_2O$  or 1% borax soln.

E. R. Bullock

The Agfa step color chart. Martin Biltz. *Phot. Ind.* 33, 746-52(1935).—This chart, introduced in 1931, has been used in numerous exptl. investigations of emulsion coatings and light filters. A description is given of its optical characteristics, including hue, reflection, brightness, etc.; and the precision of measurements afforded by the chart.

C. E. Meulendyke

Recent processes for the reproduction of multicolor subtractive pictures (Gasparcolor process). Béla Gáspár. *Z. wiss. Phot.* 34, 119-24(1935).—A discussion of the relative merits of additive and subtractive processes is followed by a review of previously proposed theoretical multilayer films, in which 3 emulsion layers, each carrying a dye or dye-forming substance, are coated in superposition. Each layer is sensitized to light of a color complementary to the final color of the layer. Their imperfections are discussed. The dye-forming sub-

stances, i. e., certain esters of vat-dye leuco bases, while satisfactory, are difficult to control in processing. Incorporating actual dyes in the emulsion layer offers the advantage of advance detn. of both shade and concn. of the dye. Here certain polyazo dyes are used. Sensitization of the new material is not complementary, but displaced toward the long-wave end of the spectrum, i. e., the yellow layer is green-sensitive, the magenta layer, red-sensitive, and the blue-green layer, sensitive to infrared to take advantage of the addnl. transmission of the dyes in this region. The entire production and processing of the Gasparcolor material now in use are described.

H. A. Kurtzner

Detection of artificial radioactivity in a photographic emulsion (Takéuchi, Inai) 3

Photographic developer. Charles W. Bennett (to Photo-Cast, Inc.). U. S. 2,018,657, Oct. 29. Borax and acetone are used in developers such as those contg. pyrocatechol, etc. They stabilize the developer against atm. oxidation although it is operative at temps. as low as 0°.

Photographic development baths. I. G. Farbenind. A.-G. Ger. 616,890, Aug. 7, 1935 (Cl. 57b 13 01). Addn. to 612,492 (C. A. 29, 4688<sup>5</sup>). The chlorobenzotriazole used according to Ger. 612,492 is replaced by a deriv. thereof, e. g., a methylchlorobenzotriazole. Cf. C. A. 29, 4685<sup>5</sup>.

Photographic emulsions. B. Kankelwitz. Ger. 618,354, Sept. 6, 1935 (Cl. 57b 8 01). Mixts. of Hg salts and Au salts are added to photographic emulsions, to eliminate gray and yellow fog.

Tanned images. Kalle & Co. A.-G. Fr. 784,190, July 22, 1935. Collodion films sensitized by aromatic nitro derivs. which acquire tanning properties under the action of light (Mg 1-nitronaphthalene-8-sulfonate, o-nitrobenzyl chloride, Na 4-nitronaphthalene-1,8-dicarboxylate, and Na 1-nitro-8-methylnaphthalene-4-sulfonate) are exposed to light under a negative and then treated in known manner to obtain tanned images. Cf. C. A. 29, 7842<sup>1</sup>.

Combined sound record and colored picture films. Béla Gáspár. Brit. 432,464, July 26, 1935. The metal deposit images (both sound record and pictures) are produced in 1 or more layers of a single or multi-layer photographic material that has color-forming substances incorporated in the said layer or layers, either prior to or subsequent to the formation of the metallic images, the layer(s) being afterward so treated as to produce a colored sound record and colored pictures at the metal image or nonimage points. The color-forming substances comprise any substance which, when incorporated in the photographic layer, can be converted by oxidation, reduction, diazotization, etc., into a dye or pigment and which permits of selective destruction of the color in the required manner, they may include a substance which is itself colored but which is convertible into a dye of a different color to form the finished sound image. Various methods for carrying out the invention are described.

Thus, the dye-forming substance may be converted by a developer into a dye, at the points of the developed Ag, as described in Brit. 2562/1913 (C. A. 8, 2657<sup>1</sup>); by this method a blue image may be produced in a layer contg.  $\alpha$ -naphthol by using a developer contg. diethyl-p-phenylenediamine hydrochloride, followed by the use of Farmer's reducer to remove the Ag. Or the oxidation method described in Brit. 379,679 (C. A. 27, 2892<sup>1</sup>) may be used. Preferably, however, a method is used in which the formation of the dye is independent of the Ag image, the latter being effective only for the selective destruction of the dye or dye-forming substance, among several examples, Zn ferriammonium, diffusely incorporated in a photographic layer, preferably before the formation of the metallic sound and picture images, is developed by ferrous oxalate, a Prussian blue color being formed which is subsequently converted into reversed sound and picture images by a bath contg. thiocarbamide and HCl. Ni dimethylglyoxime or  $ZnCrO_4$  may alternatively be used for producing

red and yellow images, resp. A leuco base, which is subsequently oxidized to form the dye, may be incorporated in the layer as a finely divided deposit, e. g., a salt of phosphotungstic, molybdic or tannic acid. The diffusely formed dye is then destroyed at the image or nonimage portions, e. g., as described in Brit. 205,718 (C. A. 28, 559), 397,139 (C. A. 28, 3014), 397,188 (C. A. 28, 3015) and 377,192 (C. A. 28, 3015); or the dye-forming substance may be first destroyed locally and the remainder then converted into the dye by further treatment. Cf. C. A. 29, 61.

Carrier for the recording of sound oscillations or picture impulses. Cornelis J. Dippel (to N. V. Philips' Gloeilampenfabrieken) U. S. 2,019,215, Oct. 29. A cutting liver contains gelatin and a sulfonated castor oil

Photographic papers. Paul Dau. Ger. 618,353, Sept. 6, 1935 (Cl. 57b. 2 02). In the manuf. of highly lustrous photographic papers with the aid of aq. emulsions of wax, paraffin wax, shellac or like materials, more stable products are obtained by using emulsions which have been prepd. by mech. disintegration without the addn. of chem. emulsifying agents.

Multi-color positive photographs or cinematographic pictures. John E. Thornton U. S. 2,018,104, Oct. 22. Numerous operative details are described.

Printing colored pictures. Anton Jasmitz. Ger. 616,542, Aug. 6, 1935 (Cl. 57d 9). Addn. to 585,202 (C. A. 28, 1202). Details are given of a modified process of the kind described in Ger. 585,202 in which dyes not contg. salts are used. Cf. C. A. 29, 429.

## 6—INORGANIC CHEMISTRY

A. R. MIDDLETON

Bromine oxide,  $\text{Br}_2\text{O}$ . W. Brenschede and H. J. Schumacher. Z. physik. Chem. B29, 356-8 (1935).—The reaction between  $\text{H}_2\text{SO}_4$  and  $\text{Br}_2$  in  $\text{CCl}_4$  soln. results in a product contg. more than 40%  $\text{Br}_2\text{O}$ . Methods of prep. and analysis are given. It decomposes in light according to the reactions:  $\text{Br}_2\text{O} \rightarrow \text{Br}_2 + \frac{1}{2}\text{O}_2$ ,  $\text{Br}_2\text{O} + \text{CCl}_4 \rightarrow \text{COCl}_2 + \text{Br}_2 + \text{Cl}_2$  and at room temp. in the dark in a few days according to the same reactions. G. M. M.

Lower oxides and sulfates of iodine. Rama K. Bahl and James R. Partington. J. Chem. Soc. 1935, 1258-63.— $\text{I}_2$  in  $\text{HNO}_3$  (d. 1.4) does not give  $\text{I}_2\text{O}_5$ . Action of  $\text{HNO}_3$  (d. 1.5) on  $\text{I}_2$  gives hygroscopic yellow  $\text{I}_2\text{O}_5$ . Hot concd.  $\text{H}_2\text{SO}_4$  reacts with  $\text{HNO}_3$  to give yellow, granular, nonhygroscopic  $\text{I}_2\text{O}_5$  which heated above  $85^\circ$  forms  $\text{I}_2\text{O}_5$  (cf. Muir, C. A. 3, 1849). An intermediate product is an equimol. mixt. of  $\text{I}_2\text{O}_5$ ,  $\text{H}_2\text{SO}_4$  and  $\text{I}_2\text{O}_5$ ,  $\text{H}_2\text{SO}_4$  (cf. Millon, Ann. chim. phys. 12, 330, 345, 353 (1844); J. prakt. Chem. 34, 321 (1845)). Adding  $\text{I}_2$  to  $\text{HNO}_3$  in hot concd.  $\text{H}_2\text{SO}_4$  or heating  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  until  $\text{I}_2$  is evolved gives  $\text{I}_2\text{O}_5$ ,  $\text{H}_2\text{SO}_4$  with a small amt. of  $\text{I}_2\text{O}_5$ ,  $\text{H}_2\text{SO}_4$ .  $\text{I}_2\text{O}_5$ ,  $\text{SO}_3$ , 1/2  $\text{H}_2\text{O}$  (Christen, Comp. rend. 123, 814 (1896)) could not be produced. Oxonized  $\text{O}_2$  contg. 8-10%  $\text{O}_3$  gives yellow, granular, hygroscopic  $\text{I}_2\text{O}_5$  from  $\text{I}_2$  vapors. On heating above  $85^\circ$   $\text{I}_2\text{O}_5$  is formed. Foster Dee Snell

Amorphous and crystallized oxide hydrates and oxides XXIII. The formation of ozone by the oxidation of ferric hydroxides and ferric oxides. The existence of iron peroxide compounds. Alfons Krause, E. Kemnitz, F. Wyzinski and J. Sawicki. Ber. 68B, 1734-43 (1935); cf. C. A. 29, 7753.—The dependence of  $\text{O}_3$  evolution on concn. of the reactants in the system  $\text{K}_2\text{S}_2\text{O}_8$ - $\text{H}_2\text{SO}_4$  was studied. Under conditions where no  $\text{O}_3$  was evolved, addn. of ferric hydroxides or sulfate caused  $\text{O}_3$  evolution. Orthoferric hydroxide,  $\gamma\text{-Fe}(\text{OH})_3$ , and  $\alpha\text{-Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$  showed individual effects, depending on the strength of acid and their resp. rates of soln. Too slow soln. lowered rate of the  $\text{O}_3$  formation; too rapid soln. (high concn. in the soln.) caused decompn. of the  $\text{O}_3$ .  $\text{O}_3$  formation through a peroxide of the type,  $\text{Fe-O-O-SO}_3\text{H}$ , is suggested. L. E. Steiner

Combinations of basic oxides and metalloids. M. Iemachians and D. Samir. Bull. soc. chim. [5], 2, 1709-16 (1935).—See C. A. 29, 3249.

Bromine salts of trivalent Iridium. Madeleine Delapine-Tard. Ann. chim. [11], 4, 282-91 (1935).— $\text{Ir}(\text{OH})_3$  and  $\text{HBr}$  when heated on a water bath yield  $\text{Ir}^{++}$  and  $\text{Br}_2$ . Upon evapn. a black mass results. Dissolve, filter and evap. again when the black or brownish bromide seps. easily. To  $\text{Ir}(\text{OH})_3$  add an excess of  $\text{HBr}$ , evap. and a black amorphous mass contg.  $\text{Ir}$  34.85 and  $\text{Br}$  57.0% results, which corresponds approx. to  $\text{IrBr}_3 \cdot \text{H}_2\text{O}$ . This compd. was used in prep.  $\text{K}_2(\text{IrBr}_6) \cdot 4\text{H}_2\text{O}$ ,  $\text{Ir}(\text{H}_2\text{O})_6\text{Br}_3$ ,  $\text{K}_2\text{Ir}_2\text{Br}_9$  was prep. by dissolving  $\text{IrBr}_3$  and  $\text{KBr}$  in  $\text{H}_2\text{O}$ . Upon evapn.  $\text{HBr}$  is expelled and the salt is taken up with  $\text{H}_2\text{O}$ , filtered and when evapd. octahedral crystals are deposited. When pptd. by alc. needles are formed of compn.  $\text{H}_2\text{O}$  4.65%,  $\text{Ir}$  27.4,  $\text{KBr}$  33.6 and volatile  $\text{Br}$

33.5. To prep.  $\text{RbIrBr}_6 \cdot \text{H}_2\text{O}$  use a large excess of  $\text{RbBr}$ . 1 or  $\text{Ir}(\text{H}_2\text{O})_6\text{Br}_3$  pour a soln. of 5 g.  $\text{Na}_2(\text{IrBr}_6) \cdot 4\text{H}_2\text{O}$  into a soln. of 5 g.  $\text{RbBr}$  in 20 g.  $\text{H}_2\text{O}$  with const. stirring. A granular ppt. results after filtering and drying in the air, found  $\text{Ir}$  24.6%,  $\text{RbBr}$  21.8, volatile  $\text{Br}$  30.4.  $\text{Cs}_2\text{IrBr}_6 \cdot \text{H}_2\text{O}$ ,  $\text{Cs}_2\text{Ir}_2\text{Br}_9 \cdot 2\text{H}_2\text{O}$ ,  $\text{AgIr}(\text{OH})_3$  and  $\text{AgIr}(\text{OH})_2\text{Cl}$  were prep. H. E. Messmore

Preparation of cuprous chloride. Charles B. Dewitt. Chemist Analyst 24, No. 4, 15 (1935).—Mix 80 g. of powd.  $\text{CuO}$  and 64 g. of powd.  $\text{Cu}$  in a dry flask, add 200 cc. of concd.  $\text{HCl}$  while shaking constantly. If the solid salt is desired, use a slight excess of  $\text{CuO}$  and from the soln. ppt. the solid by adding 500 cc. of water. Allow to settle, decant off the mother liquor and wash the ppt. with glacial  $\text{AcOH}$  until free from  $\text{Cu}^{++}$  and then with alc. and finally ether. W. T. H.

Hydrates of monocalcium silicate. Jacques Lefol. Comp. rend. 201, 669-72 (1935).—Application to the ppt. formed by  $\text{CaCl}_2$  and  $\text{Na}_2$  silicate dried over  $\text{CaCl}_2$  (of compn. 1.2  $\text{CaO} \cdot \text{SiO}_2 \cdot 2\text{H}_2\text{O}$ , the 0.2 excess  $\text{CaO}$  being adsorbed) of the static method and that of dehydration at increasing temp. (cf. C. A. 28, 722; 29, 1025) indicates the existence in addn. to that with 2.5  $\text{H}_2\text{O}$  of hydrates contg. 1 and 2  $\text{H}_2\text{O}$ . C. A. Silberrad

The reaction between potassium permanganate and oxalic acid. O. M. Lidwell and R. P. Bell. J. Chem. Soc. 1935, 1503-5.—Contrary to Launer (C. A. 26, 452) the reaction rate at const.  $\text{pH}$  is essentially independent of the  $\text{Ca}^{++}$  concn. The presence of the complex  $(\text{Mn} \cdot (\text{C}_2\text{O}_4)_2)$  is confirmed by the decompn. of  $\text{K}_2\text{Mn}(\text{C}_2\text{O}_4)_3 \cdot 2\text{H}_2\text{O}$  in acid solns. at a rate corresponding to the reaction of  $\text{KMnO}_4$  and  $\text{H}_2\text{C}_2\text{O}_4$  over ranges where reaction is slow. A neutral soln. of the salt is more stable than the  $\text{KMnO}_4 \cdot \text{H}_2\text{C}_2\text{O}_4$  mixt. and suggests that  $\text{H}^+$  is involved in the reaction which detrs. the rate of oxidation. F. D. S.

Electrometric and analytical evidence for the composition of precipitated basic copper perchlorate. Ralph A. Beebe and Seymour Goldwasser. J. Phys. Chem. 39, 1075-8 (1935); cf. C. A. 24, 3190; 26, 607.— $\text{Cu}(\text{ClO}_4)_2$  solns. were prepd. (A) from a soln. of  $\text{CuSO}_4$  by adding a slight excess of  $\text{BaCl}_2$ , centrifuging and adding a small weighed excess of  $\text{HClO}_4$  to prevent hydrolysis and (B) from  $\text{HClO}_4$  in excess with basic  $\text{CuCO}_3$ . When solns. 0.1 N or above were titrated electrometrically with a quinhydrone electrode, 1.714 mols. of carbonate-free  $\text{NaOH}$  was required for both direct and delayed titrations indicating the formation of a 1.6 basic salt,  $\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{CuO} \cdot \text{H}_2\text{O}$ . This was confirmed by the analysis of the ppt. which showed a ratio of mols. of  $\text{Cu}$  to  $\text{ClO}_4^-$  to be 6.01. 2. No satisfactory theory for the structure of this salt has yet been advanced. Janet E. Austin

Data on the system  $\text{BeSO}_4 \cdot \text{H}_2\text{O}$  and  $\text{Be}_2\text{O} \cdot \text{H}_2\text{SO}_4$ . L. Schreiner and A. Sieverts. Z. anorg. allgem. Chem. 224, 167-72 (1935).—The binary system  $\text{BeSO}_4 \cdot \text{H}_2\text{O}$  and the ternary system  $\text{BeSO}_4 \cdot \text{H}_2\text{O}$  were studied from  $-1^\circ$  to  $110^\circ$ . Method of prep. and testing are given. The eutectic for  $\text{BeSO}_4 \cdot \text{H}_2\text{O}$  is at  $-18^\circ$  and

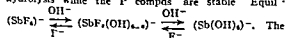
the solid phase is found as ice and the tetrahydrate. Isohexahydrate was observed under any conditions. Isotherms of the ternary system were made for 0°, 25°, 50° and 75° where up to 65%  $\text{H}_2\text{SO}_4$  the solid phase is the tetrahydrate while above 65% it is thought anhyd  $\text{BeSO}_4$  exists.

Raymond H Lambert

**Formation of mix-crystals with tin monoxide.** Erich Havel. *Monatsh.* 66, 197-200 (1935); cf. *C. A.* 27, 1838. —Mix-crystals of  $\text{SnO}$  with oxides of Pb, Mn, Fe, Zn and Ca were prepd by pptn from soln. The oxides of Co, Ni, Mg, Sr and Ba are not taken up by the  $\text{SnO}$  lattice. The colors produced by the mix-crystals are described. Those contg Cd, Fe and Mn were found to give x-ray powder-diagrams almost identical with those for pure  $\text{SnO}$ , whereas those contg Pb give a somewhat altered diagram, depending on the quantity of Pb. E. R. R.

**Some substances analogous to graphite.** IV. Riccardo Cusa and Francesco Bellino. *Cazz chim. ital.* 65, 461-4 (1935), cf. B, C, A 27, 241. —A better yield of tetraodofuran (I) than that by the method of prepn. already described (*C. A.* 21, 2686) can be had by agitating at 50-60° a mixt of aq pyromucic acid and aq  $\text{Hg}(\text{OAc})_2$  (4 mols) (acidified with  $\text{AcOH}$ ) until pptn is complete, treating the tetramercurofuran tetraacetate with I in aq KI, and purification of the pptn with  $\text{Me}_2\text{CO}$ . In I expt., the product crystd from  $\text{C}_6\text{H}_6$  gave a compd  $\text{C}_6\text{O}_I$ , i. e., the analysis of a tetraodofuran, which m 253°, and which on recrystn from  $\text{Me}_2\text{CO}$  was transformed into I. The decompn of I to a graphite was carried out by the method used in earlier expts. The product obtained at 300° was heated in N at higher temps. The following data give the temp., the percentages of C, of I, of ash and of O of the 6 products: 300°, 32.62, 56.69, 0.325, 10.37; 400°, 63.61, 7.88, 0.55, 28.06; 500°, 78.25, 0, 1.46, 20.39; 600°, 87.35, 0, 0.50, 12.15; 700°, 91.90, 0, 0.56, 7.54; 910°, 96.83, 0, 0.55, 2.60. The compn of the particular graphite (II) from heating at 500° approaches  $(\text{C}_6\text{O})_n$ . The O in the 910° product is probably still present in the bridge form. All the products have high adsorptive powers, e.g., whereas the adsorptive power of furan graphite (prepd at 500°) for  $\text{HgCl}_2$  is 111.8%, that of II is 283.6%. C. C. Davis

**The salts of  $\text{HSbF}_6$ .** Willy Lange and Konstantin Askitopoulos. *Z. anorg. allgem. Chem.* 223, 369-81 (1935). —Soly of  $\text{NaSbF}_6$  in  $\text{H}_2\text{O}$  at 20° is 128g per l.  $d_{20}^{20} = 3.379$ , easily sol in  $\text{CH}_3\text{OH}$ ,  $\text{C}_2\text{H}_5\text{OH}$ ,  $(\text{CH}_3)_2\text{CO}$ . It decomposes without melting. Soly of  $\text{KSbF}_6$  in  $\text{H}_2\text{O}$  at 20° is 1022 g per l.  $d_{20}^{20} = 3.34$ . It begins to sinter at 288° and decompose. Soly. of  $\text{NH}_4\text{SbF}_6$  in  $\text{H}_2\text{O}$  at 0° is 1187 g per l.  $d_{20}^{20} = 3.205$ . It sinters at 315° with decompn. Soly of  $\text{RbSbF}_6$  in  $\text{H}_2\text{O}$  at 18° is 502.5 g per l.  $d_{20}^{20} = 3.955$ . It sinters at 280° and m 340° without much decompn. Soly of  $(\text{CH}_3)_4\text{NSbF}_6$  in  $\text{H}_2\text{O}$  at 0° is 117.05 g per l.  $d_{20}^{20} = 2.078$ . It decomposes at about 350°. Solubilities in  $\text{H}_2\text{O}$  diphenyl-4,4'-bis-diazonium-antimony hexafluoride,  $\text{C}_6\text{H}_5\text{N}_2\text{O}_2\text{SbF}_6$ , at 0° = 106.5 g/l., styrylene antimony hexafluoride,  $\text{C}_6\text{H}_5\text{N}_2\text{O}_2\text{SbF}_6$ ,  $\text{H}_2\text{O}$  at 28° = 5.125 g/l., bromantimony hexafluoride,  $\text{C}_6\text{H}_5\text{N}_2\text{O}_2\text{SbF}_6$ ,  $\text{H}_2\text{O}$  at 28° = 2.545 g/l.; cocaine antimony hexafluoride  $\text{C}_6\text{H}_5\text{N}_2\text{O}_2\text{SbF}_6$ , at 27° = 12.20 g per l.  $\text{C}_6\text{H}_5\text{N}_2\text{O}_2\text{SbF}_6$ ,  $\text{H}_2\text{O}$  decomposes at 256°.  $\text{C}_6\text{H}_5\text{N}_2\text{O}_2\text{SbF}_6$ ,  $\text{H}_2\text{O}$  at 253°:  $\text{C}_6\text{H}_5\text{N}_2\text{O}_2\text{SbF}_6$ , m 178.6° without decompn. Soly of  $\text{NaPF}_6$ ,  $\text{H}_2\text{O}$  in  $\text{H}_2\text{O}$  at 0° is 1032 g per l. of anhyd. salt;  $d_{20}^{20} = 2.369$ . The alkali salts in water produce an acid soln. due to hydrolysis while the F compds are stable. Equil-



The differences in properties of P hexafluorides and Sb hexafluorides due to difference in ion sizes are discussed.

Arthur A. Vernon

**Magnetochemistry.** II. Complex platinum compounds. J. A. Christiansen and R. W. Asmusen. *Kgl. Danske Vidensk. Selskab Math.-fys. Medd.* 13, No. 11, 16 pp (1935); cf. *C. A.* 28, 4950. —The Guoy app, previously used for the study of complex Rh compds (cf

*C. A.* 28, 4950), was used to measure the susceptibilities of 23 complex org. Pt compds. contg. bivalent Pt as a central ion and 4 or 6 coordinated groups. The measurements indicate that, without exception, all of these compds. are diamagnetic, contrary to the predictions of Bose's rule. The implications of this in connection with Pauling's theory are discussed. The values for  $\chi$  lie in the range -0.472 to -0.292  $\times 10^{-4}$ . W. W. Stiller

**The trichlorides of iridium.** III. Aquo dipyrindines. Marcel Delépine. *Ann. chim.* [11], 4, 271-81 (1935). — See *C. A.* 29, 3933.

**Bromodipyrindine derivatives of iridium.** Madeleine Delépine-Tard. *Ann. chim.* [11], 4, 292-300 (1935). — See *C. A.* 29, 3934.

**The constitution, optical activity and photochemical behavior of platinum complexes.** I. I. Lifschitz and W. Froentjes. *Z. anorg. allgem. Chem.* 224, 173-93 (1935). —  $\beta$ -Dichlorobis(MeEt sulfide)Pt[(MeEtS)<sub>2</sub>](PtCl)<sub>2</sub>, m. 127° (cf. *C. A.* 24, 5658). The  $\alpha$ -isomer is prepd. by heating the  $\beta$ -compd. to 130° for 30 min. and crystallizing from petr. ether in the absence of light, pale orange needles, m. 63°.  $\alpha$ - and  $\beta$ -Dichlorobis-di-Et sulfide Pt, ClPt-(EtS)<sub>2</sub>, also were prepd. (cf. *C. A.* 24, 2960).  $\alpha$ -S-Ethyl- $\alpha$ -thiolactic acid platinum, Pt(CH<sub>3</sub>CH(SO)<sub>2</sub>COO)<sub>2</sub>, (I) is prepd from MeCH(SO)<sub>2</sub>CO<sub>2</sub>H and K<sub>2</sub>PtCl<sub>6</sub> in a KOH soln. The greenish white compd, recrystd from 1 alc-benzene soln, m. 208-9° (decompn). The  $\beta$ -form (II) is prepd. from the  $\alpha$ -on exposure to the quartz Hg lamp or better to direct sunlight in aq soln. The crystals are yellowish green, m. 184-9°.  $\alpha$ -Dichlorobis-S-ethyl- $\alpha$ -thiolactic acid platinum (III), m. 149-50°, crystallizes from a soln of I in cold 10-12 N HCl. The  $\beta$ -isomer (IV), m. 163-4°, is formed by heating I in 5 N HCl. The optically active isomers of I and II are prepd. in the same manner as the inactive salts, with care to avoid racemization. The active  $\alpha$ -compd. m. 205-6°. The  $\beta$ -, obtained from the  $\alpha$ -by exposure to the quartz Hg arc, m. 180-2°. The optically active form of III m. 140-2°, that of IV m. 170-1°. The active forms readily undergo racemization and mutarotation. There probably occurs a hydration to [(H<sub>2</sub>O)Pt d-[ethylthiolactic acid]] without splitting of the spirite type complex. The polarimetric behavior of  $\alpha$ - and  $\beta$ -forms is that of the Werner-Ramberg formula necessary for cis-trans compd. formation without the spiran structure. The results add weight to the validity of the Wardlaw hypothesis. R. H. L.

**Complex compounds of cobalt, nickel and copper with amides and imides.** Magneto-chemical studies. L. Cambi and Tremolada. *Gazz. chim. ital.* 65, 322-38 (1935). —The paper contains the exptl part of a discussion of the magnetic behavior and constitution of complexes by C. (cf. *C. A.* 29, 6838), and deals with the various classes of Cu, Ni and Co complexes derived from amides and imides which give the buret reaction. The following data give the magnetic susceptibilities (at 294° (abs) in Weiss magnetons)  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2$ , 8H<sub>2</sub>O (I), 16.50, the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 2\text{H}_2\text{O}$  (II), by dehydration of I at 105°, 15.45; the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 2\text{C}_6\text{H}_5\text{N} \cdot 2\text{H}_2\text{O}$  (III), prepd by letting II stand in  $\text{C}_6\text{H}_5\text{N}$ , 10.70,  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 3(\text{H}_3\text{NCH}_2\text{CH}_2\text{NH}_2) \cdot 2\text{H}_2\text{O}$  (IV), 15.75, the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 3\text{F}(\text{OH})$  (V), from evapn *in vacuo* of aq-alc K phthalimide and  $\text{Ni}(\text{OAc})_2$ , 16.60; the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 3(\text{H}_3\text{NCH}_2\text{CH}_2\text{NH}_2) \cdot 2\text{H}_2\text{O}$  (VI), from V and  $\text{H}_3\text{NCH}_2\text{CH}_2\text{NH}_2$  in EtOH, violet, 18.20,  $[\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2] \cdot \text{Cl}_2 \cdot 2\text{H}_2\text{O}$  (VII), 14.95, Ni acetateacetate,  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2$  (VIII), 17.1,  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 2\text{MeNH}_2$  (IX), 2.40,  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 2\text{F}(\text{OH}) \cdot 2\text{H}_2\text{O}$  (X), 2.40,  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 2\text{C}_6\text{H}_5\text{NH}_2$  (XI), 4.70, the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot \text{C}_6\text{H}_5\text{N} \cdot 4\text{H}_2\text{O}$  (XII), from succinimide (XIII) and piperazine, 3.40, the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot \text{C}_6\text{H}_5\text{N} \cdot \text{H}_2\text{O}$  (XIV), from XIII and putrescine, 3.70, the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot \text{C}_6\text{H}_5\text{N} \cdot \text{H}_2\text{O}$  (XV), from XIII and cadaverine, 4.80, the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 2\text{C}_6\text{H}_5\text{NH}_2 \cdot 7\text{H}_2\text{O}$  (XVI), from hydantoin,  $\text{Ni}(\text{OAc})_2$  and  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{NH}_2$ , yellow, 3.20, the compd  $\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2 \cdot 2\text{H}_2\text{O}$  (XVII), from dicyanodiamidine (XVIII), 3.20; the compd  $[\text{Ni}(\text{C}_6\text{H}_5\text{O}_2\text{N})_2] \cdot \text{K} \cdot 4\text{H}_2\text{O}$  (XIX), from alc. XIII,  $\text{Ni}(\text{OAc})_2$  and KOF, yellow, hydrolyzes in water to I, 1.40, the

compd.  $[\text{Ni}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2 \cdot 12\text{H}_2\text{O}$  (XX), prepd. from 1 violet, 0.82; the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2 \cdot 6\text{H}_2\text{O}$  (XXXIV), from oxamide,  $\text{CuCl}_2$  and  $\text{KOH}$ , rose-violet, 8.89; the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2$  (XXXV), from malonamide,  $\text{CuCl}_2$  and  $\text{KOH}$ , rose-violet, 10.00; the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2 \cdot \text{H}_2\text{O}$  (XXXVI), from biuret,  $\text{CuCl}_2$  and  $\text{KOH}$ , 8.80, the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4] \cdot 2\text{H}_2\text{O}$  (XXXVII), from XVII, 8.90, the compd.  $[\text{Co}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2$  (XXXVIII), from alc. barbitol,  $\text{Co}(\text{OAc})_2$  and  $\text{KOH}$ , violet, 23.00, the compd.  $[\text{Co}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2$  (XXXIX), from phenobarbital,  $\text{Co}(\text{OAc})_2$  and  $\text{KOH}$ , violet-red, 22.40, the compd.  $[\text{Co}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{H}_2\text{K}$  (XL), from oxamide,  $\text{Co}(\text{OAc})_2$  and excess of  $\text{KOH}$  at high concn., bright red, 14.40 XXXVIII, XXXIX and XL are unstable, absorb moisture and decompose by oxidation. I, IV, VII, IX, X, XI and XIV were prepd by Tschugaeff (*Ber* 39, 3181(1906)). Compds similar to XIV and XV, but of indefinite compn because of polymerization, were also prepd by T. I to VIII and XXIX to XXXVII are paramagnetic, whereas IX to XII, XIV to XVII and XIX to XXVIII are diamagnetic. Based on the magnetic susceptibilities, the constitutions of the various complex compds, and the corresponding structures of the amides and imides from which they are formed, are discussed C. C. D.

compd.  $[\text{Ni}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2 \cdot 12\text{H}_2\text{O}$  (XX), prepd. from 1 violet, 0.82; the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2 \cdot 6\text{H}_2\text{O}$  (XXXIV), from oxamide,  $\text{CuCl}_2$  and  $\text{KOH}$ , rose-violet, 8.89; the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2$  (XXXV), from malonamide,  $\text{CuCl}_2$  and  $\text{KOH}$ , rose-violet, 10.00; the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2 \cdot \text{H}_2\text{O}$  (XXXVI), from biuret,  $\text{CuCl}_2$  and  $\text{KOH}$ , 8.80, the compd.  $[\text{Cu}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4] \cdot 2\text{H}_2\text{O}$  (XXXVII), from XVII, 8.90, the compd.  $[\text{Co}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2$  (XXXVIII), from alc. barbitol,  $\text{Co}(\text{OAc})_2$  and  $\text{KOH}$ , violet, 23.00, the compd.  $[\text{Co}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{K}_2$  (XXXIX), from phenobarbital,  $\text{Co}(\text{OAc})_2$  and  $\text{KOH}$ , violet-red, 22.40, the compd.  $[\text{Co}(\text{C}_4\text{H}_9\text{O}_2\text{N})_4]\text{H}_2\text{K}$  (XL), from oxamide,  $\text{Co}(\text{OAc})_2$  and excess of  $\text{KOH}$  at high concn., bright red, 14.40 XXXVIII, XXXIX and XL are unstable, absorb moisture and decompose by oxidation. I, IV, VII, IX, X, XI and XIV were prepd by Tschugaeff (*Ber* 39, 3181(1906)). Compds similar to XIV and XV, but of indefinite compn because of polymerization, were also prepd by T. I to VIII and XXIX to XXXVII are paramagnetic, whereas IX to XII, XIV to XVII and XIX to XXVIII are diamagnetic. Based on the magnetic susceptibilities, the constitutions of the various complex compds, and the corresponding structures of the amides and imides from which they are formed, are discussed C. C. D.

## 7—ANALYTICAL CHEMISTRY

W. T. HALL

Polarographic methods in analytical chemistry I Theoretical introduction II J Antweiler *Z anal Chem* 102, 385-05(1935). Cf Heyrovsky, *C A* 19, 2975, 26, 3444; Kemula, *C A* 26, 9, Majer, *C A* 27, 2902

The sulfur content of illuminating gas as a source of error in analytical work A Ievins *Z anal Chem* 102, 412-18(1935).—Analytical data are given which show that ppts. of  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{MgFe}_2\text{O}_4$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{NiO}$ ,  $\text{CuO}$ ,  $\text{ZnO}$  and  $\text{KCl}$  often weigh too much after ignition because of the S present in the gas used for heating

Little-known qualitative inorganic reactions Lucjan Szynajder, *Przemysl Chem*, 19, 13-18(1935).—Unfamiliar qual. reactions are given for Ag, Pb, Bi, Cu, As, Sb, Sn, Zn, Mn, Cr,  $\text{I}^{++}$ ,  $\text{Ni}^{++}$ ,  $\text{Co}^{++}$ , Al, Ca, Mg, Na,  $\text{SO}_4^{--}$ ,  $\text{SO}_3^{--}$ ,  $\text{BO}_3^{--}$ ,  $\text{I}^-$ ,  $\text{CN}^-$ ,  $\text{CNS}^-$ . References are given to original articles in the literature A. C. Zachin

Chemical analysis by hydrogenation. H ter Meulen. *Bull. soc. chim* [5], 2, 1692-14(1935).—Gauthier (*C A* 29, 36274) has studied some of the methods of ter Meulen (*C A* 23, 5875; 28, 26157) who now makes crit. comments on the work.

Quantitative analysis of solutions by spectrographic means. O. S. Dufendach, F. H. Wiley and J. S. Owens. *Ind. Eng. Chem., Anal. Ed.* 7, 410-13(1935).—An uncondensed spark is passed between a suitably chosen solid electrode and the soln. to be analyzed. The resulting spectrum is photographed and from measurements of the relative intensities of certain spectral lines, the analysis is made. Na, K, Mg and Ca have been detd. in this way in concns. corresponding to their occurrence in human urine. The results are within about 2% of the truth.

Improvements in methods of dealing with small quantities of liquids and precipitates. Beverly L. Clarke and H. W. Hermance. *Mikrochemie* 18, 289-08(1935).—Convenient capillary siphon pipets, a steam-jacketed capsule for evapn., the use of closed tubes for pptn. with  $\text{H}_2\text{S}$ , use of  $\text{AcEt}$  in place of  $\text{H}_2\text{S}$ , and a micro-distg. app., are described and illustrated. These improvements result from work in the Bell Telephone Labs. in N. Y.

Notes on the Pregl method of microanalysis. Josef Unterzaucher. *Mikrochemie* 18, 312-15(1935).—The manipulation before every analysis of a hydrocarbon is amplified by introducing a glass stopcock in the tubing that leads to the Mariotte bottle. After releasing the pressure from Carus tubes in the usual way, all danger of contami-

nating the contents with glass splinters is avoided by sealing the tubes again and then heating with a 2.3 cm. long blast flame in such a way that the tube can be bent back and opened just below the constricted part

Microchemical notes XII I. Rosenthaler *Mikrochemie* 19, 17-22(1935). Cf *C A* 28, 6385.—Under the microscope the more rapid reaction of red  $\text{HgO}$  as compared with yellow  $\text{HgO}$  can be shown with  $\text{NH}_4$ ,  $\text{NaHSO}_4$  or  $\text{KHSO}_4$ . Typical crystals of vanillin compd. can be obtained with  $\text{Ba}(\text{OH})_2$ ,  $\text{Ca}(\text{OH})_2$ ,  $\text{Pb}(\text{OAc})_2$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{NaOH}$ ,  $\text{NaOH}$  in  $\text{MeOH}$ , Zwickler's pyridine-Cu reagent, acid added to the alk. soln., and Br +  $\text{HBr}$ . Many phenols, such as phenol itself, o-cresol, m-cresol, p-cresol, vanillin, thymol, guaiacol, o-naphthol, p-naphthol, pyrocatechol, resorcinol, quinone, orcinol, phloroglucinol and pyrogallol, as well as apomorphine and acetoacetic ester, give characteristic ppts. with p-diazotraniline. Phloroglucinol and pyrocatechol, in addn. to those mentioned in a previous paper, also give cryst. ppts. with Zwickler's Cu-pyridine reagent. Protosol, the trivial name for 2',4'-diamino-4-azobenzene-sulfonamide, gives characteristic crystals with the K salt of tetranitro-diamino-Co, o-antraquinonesulfonic acid,  $\text{AgNO}_3$ ,  $\text{Hg}(\text{NO}_3)_2$  and  $\text{Cu}(\text{OAc})_2$

New procedures for carrying out electrolytic spot tests. H. Fritz *Mikrochemie* 19, 16-18(1935). Cf *C A* 24, 310.—A theoretical discussion of some possibilities with or without an outside source of elec. current, and with stationary or rotating electrodes.

Notes on the J. Lawrence Smith fusion. Mark O. Lamar, Wallace M. Hazel and Wm. J. O'Leary. *Ind. Eng. Chem., Anal. Ed.* 7, 429-31(1935).—Difficulties in getting accurate results in the detn. of Na and K are likely to result from insufficient grinding of the sample, incomplete mixing with the  $\text{CaCO}_3$  used, use of too low an ignition temp. and failure to correct for the small  $\text{Hg}$  content of the  $\text{NaCl} + \text{KCl}$  obtained.

Use of cupferron in spectral analysis Oskar Baudisch *Archiv. Chem. Mineral. Geol.* 12B, No. 8, 6 pp.(1935).—In spectral analysis it is often desirable to obtain spectra free from the Fe lines. To remove Fe from samples such as, e. g., a mineral water, it is advantageous to add  $\text{HCl}$  and cupferron reagent. Cu is removed at the same time.

pn indicators. Chas. E. Mullin. *Textile Colorist* 57, 518-20, 589-92, 664-70(1935).—A rather complete alphabetical compilation, with cross indexing of dyestuffs used in colorimetric pn detns., with Colour Index and Schultz nos., common and chem. names, formula, color and form,

$pH$  range, color changes, soly., prepn. of indicator solns., concn. generally recommended in  $pH$  detns. and titrations, comments, recommendations and suggestions, and references, where possible. It is of interest to dyers as indicating the  $pH$  of possible color change of dyed goods.

Chas. L. Mullin

Theory of neutralization indicators. Maria Mercedes Rodriguez Rego. *Anales asoc. quim. farm. Uruguay* 38, 3-36(1935).—A general review. C. E. P. Jeffreys.

The use of styryl dyestuffs in qualitative microanalysis. P. Krumholz and E. Krumholz. *Mikrochemie* 19, 47-54(1935).—Heterocyclic bases which contain a  $CH_2$  group in the  $\alpha$ -position to the trivalent N atom can condense with aromatic aldehydes to form styryl derivs. The condensation products thus formed are yellow but yield red or violet salts as a result of tautomerism. If such a styryl dyestuff forms a difficultly sol. double salt with a heavy metal halide, the dyestuff will behave as a univalent base and yield red salts even when formed in a yellow soln. In this way very sensitive tests can be made for Ag, Au, Pd, Pt, Cu, Hg, Sn, Cd, Co, Fe, Bi and Mn.

W. T. H.

Dipicrylamine as a micro-reagent for potassium, rubidium and cesium. C. J. van Nieuwenburg and T. van der Hoeft. *Mikrochemie* 18, 175-8(1935).—Poluektov, C. A. 28, 2642<sup>3</sup>, has advocated the use of dipicrylamine in  $Na_2CO_3$  soln. as a spot test for K. The reagent is equally useful for the identification of K, Rb and Cs under the microscope, as the crystals produced by the reagent are characteristic.

W. T. H.

New macro- and micro gravimetric method for determining copper. G. Spacu and C. G. Macarovici. *Z. anal. Chem.* 102, 350-2(1935).—In previous papers (C. A. 17, 1772, 18, 2300, 19, 3444) it has been shown that sensitive tests for  $Cu^{++}$  can be made by forming deep blue, flocculent ppt. such as  $[CuBz_2(SCN)_2]$ ,  $[CuBz_2I_2]$  and  $[CuTi_2(SCN)_2]$ , which are complex compounds similar to the ammoniates and in these formulas Bz and Ti represent resp. benzidine and toldine. Since the pptn. is quant the ppt. can be filtered off, ignited to  $CuO$  and weighed. To the aq. soln. of Cu salt contg. not more than 10 mg. of Cu in 50-75 cc. of water, add 40-50 cc. of 1%  $NH_4CNS$  soln. and follow this with 10-20 cc. of a 2% soln. of either benzidine or toldine in alc. The thiocyanate should correspond to 4-5 times the theoretical value but the benzidine should be used in only slight excess. Filter off the blue ppt., wash thoroughly with water, dry, ignite and weigh the residual  $CuO$ . The results are excellent. For the microdetn. of approx. 5 mg. of Cu, add 20-25 cc. of the thiocyanate to 20-25 cc. of soln. and use only 4-6 cc. of the benzidine soln. After filtering, the color of the filtrate will darken as a result of oxidation, but this does no harm.

W. T. H.

Application of controlled potential to microchemical electrolytic analysis. A. J. Lindsey and H. J. S. Sand. *Analyst* 60, 739-44(1935).—The micromethod described in this paper is not only suitable for detg. Cu, Ag, Cd, Hg, Pb, Ni and Co but also for sepg. Bi from Pb and Cu from Sn and other metals, this cannot be accomplished with the electrodes recommended by Pregl and without control of the cathode potential. Suitable app. is shown in a series of 5 drawings and the method of working is described. The inner, or working, electrode is the ordinary Pregl cathode with the beads removed. Instead of a central wire anode, as recommended by P., the anode is the outside electrode and is made of thin gauze. By using this large electrode, it is possible to keep the p. d. due to ohmic resistance in the electrolyte small and if suitable depolarizers are used it is possible to maintain the variation of anode potential within narrow limits.

W. T. H.

The micro-electrolytic determination of bismuth and lead and their separation by graded potential. A. J. Lindsey. *Analyst* 60, 744-6(1935).—*Detn. of Bi*.—To the soln. contg. not more than 6 mg. of Bi, add 1 ml. of concd.  $HNO_3$ , 2 drops of 50% hydrazine hydrate soln. and sufficient water to make 12 ml. Heat to 70-75° and electrolyze in the app. described in the preceding abstr. with

a p. d. between the electrodes of 0.8 v. while stirring with  $N_2$ . The current falls during the electrolysis from 80 to about 10 milliamperes. After 10 min. wash down the tube with a fine jet of water and increase the p. d. to 0.9 v. After 3 more min., cool, rapidly replace the electrolysis tube with a shorter tube contg. water, dip the electrodes in alc. and finally in ether, and dry 1 min. *To det. Pb*.—Add, to the soln. contg. not over 6 mg. of Pb, 2 ml. of concd.  $HNO_3$  and dil. to 12 ml. Heat nearly to boiling with a microburner and maintain this temp. during the entire electrolysis while stirring with  $N_2$ . Use a p. d. between the electrodes of 1.0 v. but this is not crit. and higher potentials can be used. After 7 min. wash down the sides of the vessel and in another 3 min. terminate the electrolysis as described above. The results are about 0.02 mg. high with 4 mg. of Pb. *To sep. Pb and Bi*.—Add to the soln. contg. not more than 6 mg. of either metal, 1 ml. of concd.  $HNO_3$ , 2 drops of 50% hydrazine hydrate soln. and water to make about 12 ml. Electrolyze at 60-70°, with  $N_2$  stirring and an anode-cathode potential of 0.8 v. After 10 min., when the current will have fallen from 70 to 10 milliamperes, wash down the sides of the tube and electrolyze another 2 min. with a p. d. of 0.9 v. Remove and weigh each electrode as described above.

W. T. H.

Determination of aluminum in nickel-chromium and nickel chromium-iron alloys. A survey of available methods. Fred P. Peters. *Chemist Analyst* 24, No. 4, 4-10(1935).—The removal of Fe, Ni, Co, Cr, Cu, etc., by electrolysis with a cathode of Hg is recommended. When this is not convenient, the use of cupellron is recommended to ppt. Fe and heating with  $HClO_4$  to oxidize  $Cr^{+++}$ .

W. T. H.

Determination of small quantities of antimony in zinc alloys. Frank W. Scott. *Chemist Analyst* 24, No. 4, 16-17(1935).—To 5 g. of spelter add 50 ml. of  $H_2O$  water, and very slowly, 10 ml. of concd.  $HNO_3$ . Next add 50 ml. of concd.  $HCl$  and boil the resulting soln. until it becomes sirupy. Neutralize with concd.  $KOH$  until  $Zn(OH)_2$  ppt. Add 20 ml. of concd.  $HCl$  and sat. with  $H_2S$ . Wash the ppt. formed with 1.2 N  $HCl$  which is satd. with  $H_2S$ . Dissolve the ppt. in 50 cc. concd.  $HCl$  + a little  $KClO_4$ . Evap. to 10 ml. after filtering. Add 50 ml. of 10% tartaric acid soln., and add the mixt. slowly to 150 ml. of a soln. contg. 60 g.  $Na_2S$  and 40 g.  $NaOH$  per l. Filter and wash with very dil.  $Na_2S$  soln. Make the filtrate slightly acid with  $HCl$  and again sat. with  $H_2S$ . Filter off the sulfides of As, Sb and Sn and wash with 1.2 N  $HCl$  which is satd. with  $H_2S$ . Treat the filter and ppt. with concd.  $HCl$  and  $KClO_4$ , dil. to 100 ml. and filter, wash the residue well. Evap. to 50 cc., add 2 g.  $KI$  and evap. to 25 ml. to volatilize  $AsCl_3$ . Neutralize with  $KOH$ , add 1 g. of tartaric acid (at least 20 times the wt. of Sb present) and oxidize with  $H_2O_2$  (10 ml. of 3% soln. is usually sufficient). Boil to decompose or expel the greater part of the excess peroxide, cool slightly and add 2.5 g. of oxalic acid crystals. Boil 10 min. to remove  $CO_2$ , dil. to 100 ml. and sat. with  $H_2S$ . After 15 min. dil. to 250 ml. with boiling hot water and introduce  $H_2S$  for another 15 min. Filter through a filtering crucible, wash with  $H_2S$  water, alc.,  $CS_2$  and alc., dry at 105° and weigh as  $Sb_2S_3$  (cf. F. W. Clarke, *Chem. News* 21, 124, 1. Henz, *Z. anorg. Chem.* 37, 18).

W. T. H.

Direct titration of barium salts with potassium chromate in the presence of rosolic acid as indicator. Application of the method to the analysis of sulfates and in particular to the determination of sulfur in pyrite and in slags. A. V. Vinogradov. *Ann. chim. anal. chim. appl.* 17, 285-88 (1935); cf. C. A. 28, 4333<sup>1</sup>, 4334<sup>1</sup>.

W. T. H.

Microdetermination of bismuth as bismuthyl iodide. Friedrich Hecht and Richard Reinherz. *Z. anal. Chem.* 103, 283 8(1935).—Streibinger and Zins, C. A. 22, 12<sup>18</sup>, 1926, described a micro-method for detg. Bi as  $BiOI$ . The method has been studied anew and some slight modifications were suggested. To the  $Bi(NO_3)_3$  soln. contg. not more than 0.01 g. of  $HNO_3$  add 0.1 N  $KI$  soln. drop wise until the soln. above the black  $BiI_3$  shows a yellowish tint. Dil. with water to 8 cc. and heat for a few min. on

the boiling water bath to about 70°. The ppt. will change to light red, lustrous BiOI. Add 0.25 cc. more of the KI soln. for each 0.5 mg. of Bi; present and a drop of methyl red indicator soln. Add 1% NH<sub>4</sub> soln. until the indicator begins to change from red to yellow. Heat 1–2 min on the gently boiling water bath, allow the ppt to settle, filter through sintered glass, wash 3 times with hot water, dry at 105° and weigh. The results are not very accurate.

W. T. H.

**Determination of small amounts of boron by means of quinalizarin.** G. Stanley Smith. *Analyst* 60, 735–39 (1935).—Feigl and Krumholz (C A 24, 2400) made use of the fact that quinalizarin, as well as other hydroxyanthraquinones, yields highly colored solns in concd H<sub>2</sub>SO<sub>4</sub> and on the addn of H<sub>2</sub>BO<sub>3</sub> a marked change in color takes place which can be used for identification purposes. About 0.002% of B can be detected by means of a soln of quinalizarin in 93% H<sub>2</sub>SO<sub>4</sub>. Suitable concns of H<sub>2</sub>SO<sub>4</sub> for the colorimetric detn. are obtained by dilg 9 vols and 4 vols, resp., of concd H<sub>2</sub>SO<sub>4</sub> with 1 vol of water. The latter soln is much less sensitive but is better for detg. larger quantities of B. The reagent is prep'd by dissolving 10 mg. of quinalizarin in 100 ml of H<sub>2</sub>SO<sub>4</sub> (9.1). Suitable standards are obtained from a soln obtained by dissolving 5 mg. of H<sub>2</sub>BO<sub>3</sub> per l of H<sub>2</sub>SO<sub>4</sub> of the above concn. The method can be used for detg. 0.005 to 0.25 mg. of H<sub>2</sub>BO<sub>3</sub>. To det. B in Al-Si alloys, treat 0.5 g. of sample with 20 ml of 10% NaOH in a large N<sub>2</sub> crucible. After the initial attack subsides, evaporate nearly to dryness, add 20 ml of water, boil and pour the resulting soln. into 30 ml of 60% H<sub>2</sub>SO<sub>4</sub>. Make up to exactly 100 ml. Place 1 ml. of this soln and of the standard in sep. small comparison tubes, add 8 ml of concd H<sub>2</sub>SO<sub>4</sub>, mix, cool and add 0.5 ml of 0.01% alizarin soln. Compare the colors.

W. T. H.

**The oxalates of calcium, strontium and magnesium.** J. Haslam. *Analyst* 60, 668–72 (1935).—Goy (C A 8, 478) showed that CaC<sub>2</sub>O<sub>4</sub>·H<sub>2</sub>O is formed from boiling solns and can be dried at 100–105°. Dick (C A 23, 4634) recommended drying by washing the ppt with alc and ether but Moser and von Zombory (C A 24, 4725) obtained results 1.6–3% too high by D's method. The expts. here described show that there is a slight pos. error but only in the neighborhood of 1 part in 1000. They recommend, however, titrating the ppt with KMnO<sub>4</sub>. Expts. with oxalates of Sr, Ba and Mg show that they are not well suited for the accurate detn. of Sr, Ba or Mg but when these ions are present, they are likely to cause error in the Ca detn.

W. T. H.

**Microdetermination of cadmium with 8-hydroxyquinoline.** P. Wenger, Ch. Cimerman and M. Wyszewska. *Mikrochemie* 16, 182–4 (1935).—To 2 cc of neutral or faintly acidic soln. contg. 1–3 mg. of Cd in a micro beaker, add 1 drop of Merck's universal indicator, 1 drop of 3% Na<sub>2</sub>CO<sub>3</sub> soln. and 2–3 drops of 3% AcOH to dissolve any ppt. produced by the soda. Add 6–10 drops of 40% NaOAc soln. to make the indicator show pH = 6–7. Heat the buffered soln. to about 90° and introduce dropwise 3 times the theoretical quantity of 2% oxine soln. in alc. Stir, heat to boiling and allow the ppt. to settle during 15 min. Filter, wash with hot water and with two 1-cc. portions of cold water. Dry at 120–130° and weigh. Of the 6 analyses reported the greatest error was 0.007 mg. Cd with a sample contg. 1.5 mg.

W. T. H.

**Application of the spectrograph to the determination of carbon in steel.** F. H. Finery and Harold S. Booth. *Ind. Eng. Chem., Anal. Ed* 7, 419–21 (1935).—The apertic spark does not have sufficient potential to show small or large variations in the C content of steel. The nonmetallic constituents can be detd. spectrographically by the exploded wire, the vacuum spark or by the elec. furnace arc method.

W. T. H.

**Direct, simultaneous microdetermination of carbon, hydrogen and oxygen.** II. Analysis of pure compounds containing carbon, hydrogen, oxygen and sulfur. W. R. Kurner. *Ind. Eng. Chem., Anal. Ed* 7, 363 (1935); cf. C. A. 28, 6394.—The purpose of this investigation was to extend the procedure for the simultaneous detn. of C,

H and O to compds. which also contain S, to det. the ultimate fate of S present in different kinds of compds., and to study the mechanism of the reaction between oxides of S and the various absorbents which are present in the combustion tube filling. It was found that SO<sub>2</sub> or SO<sub>3</sub> is the primary product of the combustion and it is almost entirely the latter that is present in the final combustion product. The SO<sub>3</sub> is absorbed by the metallic Ag placed in the tube and retained as Ag<sub>2</sub>SO<sub>4</sub>. III. Analysis of pure compounds containing carbon, hydrogen, oxygen and nitrogen. *Ibid* 366–8.—As a result of numerous expts. the hypothesis is advanced that in the thermal decomposition of substances contg. N, simple products are first formed which are eventually converted into only N<sub>2</sub> and NO<sub>2</sub>. The ratio formed appears to be a function of the manner in which the N is linked in the original org. compd. For amines and amides the ratio of N<sub>2</sub>:NO<sub>2</sub> = 74:26, for nitriles, nitro and heterocyclic N compds. the ratio is 41:59. The O values obtained by the method described are within 0.3% of the truth and the errors are largely a result of errors in the detn. of H and C.

W. T. H.

**Modified method for the determination of cobalt by the use of α-nitroso-β-naphthol.** Archibald Craig and Louis Culdroff. *Chemist Analyst* 24, No. 4, 10–14 (1935).—Evap. the combined filtrates from 2 basic acetate pptns of Fe+++ to 200 cc, add 5 cc of concd HCl and 50 cc of glacial AcOH. Heat nearly to boiling and add gradually 300 cc of hot reagent (75 cc of 50% AcOH for each g. of solid reagent) for not over 0.1 g. of Co. Keep hot for 2 hrs., filter, wash 10 times with 12% HCl and 10 times with hot water, dry and ignite to Co<sub>2</sub>O<sub>3</sub>, the results are satisfactory.

W. T. H.

**Spectrophotometric determination of copper in ores and mattes.** J. P. Mehlig. *Ind. Eng. Chem., Anal. Ed* 7, 387–9 (1935).—The spectrophotometric method for detg. Cu which depends upon the measurement by a spectrophotometer of the transmittancy of light of a given wavelength through an ammoniacal soln. of Cu does not require a series of color standards, eliminates the matching of color shades by the eye and gives results which are more dependable than those obtained by the conventional colorimetric procedure. The use of a light filter or color screen is not necessary. It is claimed that the results obtained are just as good as those obtained by the iodide method and the procedure is quicker and easier.

W. T. H.

**Determination of gold with the aid of carbon monoxide.** Rodica N. Costeanu. *Z. anal. Chem.* 102, 336–8 (1935).—By the action of concd H<sub>2</sub>SO<sub>4</sub> on HCO<sub>2</sub>H, CO is produced. The gas is passed through a tube contg. glass beads covered with P<sub>2</sub>O<sub>5</sub> and then comes in contact with 4 strips of filter paper which have been moistened with HAuCl<sub>4</sub> solns of known Au content and with 4 other strips of filter paper which have been moistened with a soln. prep'd. by dissolving the sample in aqua regia. The excess CO as it leaves the app. is absorbed in ammoniacal CuCl<sub>2</sub> soln. By comparison of the color of the deposited Au with that of the standards, the colorimetric estn. is made.

W. T. H.

**Volumetric method for the estimation of lead.** Sachindra Nath Roy. *J. Indian Chem. Soc.* 12, 584–5 (1935).—Nearly neutral solns. contg. 0.2–2.0 g. of dissolved Pb can be detd. with an accuracy of about 0.5% by titrating with standard K<sub>2</sub>SO<sub>4</sub> with fluorescein as an external indicator.

W. T. H.

**Rapid electrometric method for determining manganese in tungsten steel.** N. Ya. Khlopun. *Z. anal. Chem.* 102, 263–70 (1935); cf. C. A. 29, 2473.—To 0.2 g. of steel in a 400-cc. beaker add 3 cc. of water, and 25 cc. of 9 N H<sub>2</sub>SO<sub>4</sub>. Heat to start the chem. reaction. After the first, stormy evolution of gas has ceased, add 5–7 drops of 6 N HNO<sub>3</sub> and later, when the steel has all dissolved, add more HNO<sub>3</sub> and heat until the dark brown color of the soln. becomes yellowish. Boil to expel nitrous fumes, add 40 cc. of hot water and 10 cc. of 1.7% AgNO<sub>3</sub>. Pay no attention to a turbidity of WO<sub>3</sub>. Now add 3 cc. of 33% (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> soln. and allow to stand in a warm place (not over 60°) until the color of the soln. shows that some MnO<sub>4</sub><sup>-</sup> has been formed (about 5 min.). Cool to room temp., wash down the sides of the beaker and dil. to 200



cc. Add 3 cc. of 5% NaCl soln. to reduce  $\text{MnO}_4^-$  and at once proceed to the electrometric titration with arsenite soln. As electrodes Pt/W or Pt/Carborundum (graphite) proved satisfactory. With a little experience the end point is easy to find and the method is said to be more rapid than any electrometric method for detg. Mn hitherto proposed. Data are given to show that the accuracy is satisfactory. W. T. H.

**Bismuthate method for manganese.** II. Bartholow Park. *Ind. Eng. Chem., Anal. Ed.* 7, 427 (1935); cf. C. A. 20, 2171 and Kolthoff, C. A. 29, 5770<sup>2</sup>.—II, after filtering off the excess  $\text{NaBiO}_3$ , a measured excess of Na arsenite soln. contg. 3 drops of 0.01 M  $\text{OsO}_4$  is added, the  $\text{MnO}_4^-$  will be reduced quantitatively to  $\text{Mn}^{++}$  and in the titration with  $\text{KMnO}_4$  to an electrometric end point the same quant. reduction of the  $\text{MnO}_4^-$  takes place. W. T. H.

**Colorimetric determination of manganese in the presence of titanium.** Geo. J. Hough. *Ind. Eng. Chem., Anal. Ed.* 7, 408-9 (1935).—When Mn is detd. colorimetrically in soils or rocks the persulfate method is unsatisfactory. When 0.05% of Mn is present and 2-12% Ti, often no  $\text{MnO}_4^-$  color is obtained unless considerable persulfate and Ag soln. is present and even then the color develops slowly.  $\text{KIO}_4$  or  $\text{NaBiO}_3$  should be used as oxidizing agent. Attempts to remove Ti by evapn. with  $\text{HCl}$  were futile. W. T. H.

**Colorimetric determination of molybdenum.** Loren C. Hurd and Harry O. Allen. *Ind. Eng. Chem., Anal. Ed.* 7, 396-8 (1935).—In the colorimetric detn. of Mo, the  $\text{HCl}$  concn. should be about 5.0%, the  $\text{KCNS}$  about 0.6% and the  $\text{SnCl}_4$  above 0.1% by wt. Under some conditions  $\text{H}_2\text{SO}_4$  has an appreciable effect upon the color. The colored compd. should be extd. with ether 5 min. after adding the reagents. Cyclohexanol can be used in place of ether but Bu acetate is not as suitable. W. T. H.

**Rapid determination of nickel and chromium in presence of manganese in ferrous alloys containing more than 1% carbon.** Tadeusz Cichocki. *Przemysł Chem.* 19, 1-2 (1935).—The sample is dissolved in  $\text{HClO}_4$  and the resulting  $\text{Cr}_2\text{O}_7^{2-}$  titrated with a soln. of Mohr's salt with diphenylamine in concd.  $\text{H}_2\text{SO}_4$  as indicator. Another sample is dissolved in the same way and the Ni detd. with dimethylglyoxime. A. C. Zachlin.

**Microanalytical determination of certain metals of the platinum group in simple and complex salts.** Julius Meyer and Karl Hoehne. *Mikrochemie* 19, 64-71 (1935), cf. C. A. 29, 2471<sup>1</sup>. By igniting complex salts, such as phenylpyridine-hexachloroplatinate, in the Pregl microcup the residual Pt weighed almost exactly the theoretical value, 17-30 mg. of complex salt and 4-8 mg. of Pt were obtained. Loss of Pt, as found by Pregl, only occurred when insufficient  $\text{H}_2$  to form  $\text{HCl}$  was present. Similar treatment of Rh compds. gave high results unless the final product was heated in  $\text{H}_2$ . Contrary to the experience of Palmer (*Z. anorg. Chem.* 10, 321 (1893); 13, 211 (1897)) who worked with larger quantities of material, heating in  $\text{H}_2$  gave a metal which could be weighed in the air with satisfactory accuracy. With Ru it was found necessary to heat in  $\text{H}_2$  and cool in  $\text{CO}_2$  to get the proper wt. of pure metal. With Os compds. there was always some loss of Os by volatilization. Pd salts are decompd. and reduced by  $\text{H}_2$  even in the cold. By heating in  $\text{H}_2$  and cooling in  $\text{CO}_2$ , correct results can be obtained. With halides of Au there is always some loss by volatilization. W. T. H.

**Determination of small quantities of selenium in sulfur.** Geo. G. Marvin and Walter C. Schumb. *Ind. Eng. Chem., Anal. Ed.* 7, 423-5 (1935).—From 10 to 20 g. of the sample is burned in a current of  $\text{O}_2$  and the Se removed from the products of combustion by a plug of asbestos in the front end of the combustion tube. The asbestos pad is treated with  $\text{HNO}_3$  to dissolve the Se and any Fe present is removed after pptn. with  $\text{NH}_4\text{OH}$ . The filtrate is made acid with  $\text{HNO}_3$ , any  $\text{HNO}_3$  present removed by adding urea, the selenium acid is reduced with KI and the liberated  $\text{I}_2$  titrated with  $\text{Na}_2\text{S}_2\text{O}_3$ . As little as 0.001% of Se can be detd. by this method. W. T. H.

**Electrolytic determination of silver.** A. Friedrich and

S. Rapoport. *Mikrochemie* 18, 227-34 (1935).—The electrolysis takes place in a total vol. of about 7 cc. to which a little concd.  $\text{H}_2\text{SO}_4$  and 1 cc. of 20% tartaric acid have been added. If an ag. soln. is to be analyzed, the addn. of a few drops of the acid is sufficient but if a wet oxidation has been accomplished, the acid soln. should be reduced to less than 1 cc. At the start, the p.d. between the terminals is 1.3-1.4 v. but after 15 min. it is increased to 1.8 v. and kept so for half an hr. Before breaking the circuit, it is necessary to replace the electrolyte with water. In detg. up to 3.6 mg. of Ag the largest error was 0.003 mg. W. T. H.

**Electrometric determination of thallium.** Winifred R. A. Hollens and James F. Spencer. *Analyst* 60, 672-6 (1935).—The method proposed consists in converting the Tl to the trivalent state, adding KI to the acid soln. and titrating the liberated  $\text{I}_2$  with either arsenite or thiosulfate soln. The end point can be detd. satisfactorily by means of the electrode system recommended by Fouk and Bawden (C. A. 20, 3144). Both reducing agents give reproducible end points down to 0.002 N solns. Presence of Zn and Fe does no harm but Cu behaves like Tl. W. T. H.

**Analysis of tin plate.** Walmet and Mathieu. *Ann. chim. anal. chim. appl.* 17, 280 (1935).—Instead of stripping off the plate, dissolving in acid and analyzing the resulting soln. for Sn and Pb it is recommended to take a piece of the metal as anode, suspend it in 12% NaOH soln. and electrolyze at 75° with a Cu wire as cathode. After the electrolysis, the cathode is dissolved in  $\text{HNO}_3$  and in a second electrolysis the Pb is deposited in the usual way upon a Pt anode as  $\text{PbO}_2$  and the electrolyte is filtered, leaving residual  $\text{SnO}_2$  which can be weighed. W. T. H.

**The use of induced precipitation for the detection of small quantities of titanium and zirconium.** F. Fregl and E. Rajmann. *Mikrochemie* 19, 60-63 (1935).—A very dil. Ti or Zr soln. (1:50,000) will give no ppt. with  $\text{H}_2\text{AsO}_4$  but if the other element is present pptn. occurs even in extreme dilns. such that other reagents fail to give the typical tests. Thus to test for Ti, take 10 cc. of the soln. which is about N in  $\text{HCl}$  and add a drop of 1% Zr soln. and 10 drops of 20%  $\text{H}_2\text{AsO}_4$  soln. Remove the ppt. with the aid of the centrifuge and wash until all colored ions are removed. Take a little of the ppt., add a drop of concd.  $\text{H}_2\text{SO}_4$  and dissolve by heating in a bath of boiling water. Cool and add a drop of  $\text{H}_2\text{O}_2$ ; a yellow color will be obtained if 1% of Ti is present. Similarly to test for Zr, take 10 cc. of the soln. which is about 2 N in  $\text{HCl}$ , add 12 drops of acid, 4% Ti soln. and 1 cc. of  $\text{H}_2\text{AsO}_4$  soln., boil 1-2 min., filter and wash with the aid of the centrifuge. Moisten the centrifugate with 1 drop of a mixt. of 10 parts concd.  $\text{H}_2\text{SO}_4$ , 1 part 30%  $\text{H}_2\text{O}_2$  and 10 parts water and dissolve by heating. Test the soln. for Zr with alkyl azoarsenic acid test paper. In this way 1.25-γ of Zr can be detected in 10 cc. of soln.; in other words the diln. is  $1.8 \times 10^4$ . With  $\text{H}_2\text{AsO}_4$ , Zr alone gives a barely visible ppt. at a diln. of  $1:10^4$ . W. T. H.

**Determination of vanadium in the field.** V. A. Zilbermuntz and K. P. Florenzu. *Mikrochemie* 18, 154-8 (1935).—Tananaev and Patschenko (C. A. 24, 567) showed that a most sensitive test for V consists in treating a soln. contg.  $\text{HVO}_3$  with aniline +  $\text{HCl}$ , the V is reduced and a blue compd. is formed. The test can be utilized for a rapid colorimetric detn. of V which is sufficiently accurate for field tests. W. T. H.

**Spectrographic microdetermination of zinc.** Preliminary note. Lewis H. Rogers. *Ind. Eng. Chem., Anal. Ed.* 7, 421-2 (1935).—A spectrographic method for detg. Zn in plant materials contg. 0.005-0.1% Zn is described. Zn is used as an internal standard. Fe when present in quantities exceeding 1% interferes. W. T. H.

**Determination of chlorine in organic compounds.** I. A. rapid lamp method. Wm. Marias Malisoff. *Ind. Eng. Chem., Anal. Ed.* 7, 428 (1935).—Dissolve the org. compd. in a combustible solvent to make a soln. contg. about 0.2% Cl. Burn in the standard lamp and titrate the  $\text{HCl}$  formed. W. T. H.

**Simplified method of S. L. Penfield for the determina-**

tion of fluorine in phosphorites and apatites. S. N. Rozanov. *Z. anal. Chem.* 102, 328-36 (1935).—See C. A. 29, 1032<sup>a</sup>.

Determination of oxygen and nitrogen in steel. Improvements in the vacuum-fusion method. John Chipman and M. G. Fontana. *Ind. Eng. Chem., Anal. Ed.* 7, 391-5 (1935).—Improvements in the technique and changes in the construction of the necessary app. have resulted in a vacuum-fusion app. that yields better results. The accuracy has been increased and the length of time required shortened. The O of  $Al_2O_3$  formed in the decarburization of molten steel by Al can be recovered within 1% of the truth. W. T. H.

Estimation of nitrogen by fumeless digestion. I. Y. V. Narayanayya and V. Subrahmanyan. *Proc. Indian Acad. Sci.* 2B, 213-35 (1935).—See C. A. 29, 4504<sup>a</sup>. W. T. H.

Semi-micro-Kjeldahl determination of nitro and azo nitrogen. Robert A. Harste. *Ind. Eng. Chem., Anal. Ed.* 7, 432-3 (1935).—Take sufficient sample to yield 2-5 mg of N in a 100-cc., dry Kjeldahl flask, contg. 300 g. of pure dextrose, 1-1.5 g. of  $K_2SO_4$ , about 20 mg. of blue vitriol and a little well-washed aluminum (gran. size 14). Add 4 ml. of concd.  $H_2SO_4$  and digest near the b. p. until the soln. is clear and then for an addnl. 15 min. To the cooled soln., add 35 ml. of water and 12 ml. of 50% NaOH. Distill as usual and receive the distillate in 25 ml. of 0.02 N HCl. Finally titrate the excess acid with 0.02 N  $Ba(OH)_2$  with a mixt. of methyl red and methylene blue as indicator. W. T. H.

Detection of small quantities of phosphorus and of phosphine. Ludwig Wolf, Werner Dusing and Andreas Martos. *Mikrochemie* 18, 185-92 (1935).—The method is based on the formation of black Ag phosphate when the vapors contg. P or  $PH_3$  are passed into an ammoniacal  $AgNO_3$  soln. The vapors are passed in an intermittent current through a capillary tube which rests in a beaker contg. the reagent. The deposit is thus formed in the capillary and from the amt. of deposit the quantity of P present can be estd. W. T. H.

Benzidine acetate in the determination of bromides by silver. F. Burrell. *Anales soc. españ. fis. quim.* 33, 692-5 (1935), cf. C. A. 26, 420<sup>a</sup>.—The method of detg. I<sup>-</sup> by titration with  $AgNO_3$  with  $Cu(NO_3)_2$  + benzidine acetate as absorption indicator, is applicable to the detn. of Br<sup>-</sup>. The method is accurate in 0.02 N solns. of Br but it is not applicable to the detn. of Cl<sup>-</sup>. E. M. Symmes.

Analytical determination of chlorides with adsorption indicators. F. H. C. Kelly. *Australian Chem. Inst. J. & Proc.* 2, 250-4 (1935).—Chiefly a review of previous work. W. T. H.

Titration of iodides in the presence or absence of chlorides and bromides with iodos starch as indicator. Eugen Chirnogai. *Z. anal. Chem.* 102, 339-42 (1935).—Koltzoff (C. A. 11, 2868, 15, 3053) has shown how iodos starch can be used as an indicator in the titration of  $I^-$  with  $Ag^+$ . However, added  $KIO_3$  or a little  $I_2$  to the soln. and the improvement in the procedure which is now suggested is to add a little  $Fe^{+++}$  to the soln. instead. For each 10 cc. of approx. 0.1 N iodide add 1-2 drops of satd.  $Fe(SO_4)_3$  soln., shake and add 2 cc. of 0.4% starch soln. Titrate with 0.1 N  $AgNO_3$  soln. until the blue iodos starch changes to the characteristic yellow color of  $AgI$ . The iodos starch turns first from blue to green, then to orange yellow and finally to the yellow end point. When  $Cl^-$  is present, the titration can take place in a soln. to which  $NaOAc$  has been added in place of  $(NH_4)_2CO_3$  as suggested by K. Then when the iodos starch end point has been obtained, the  $AgI$  ppt. can be removed by filtration and  $Cl^-$  detd. by the Volhard method. The results thus obtained appear to be more accurate than those obtained by the procedure of K. W. T. H.

Volumetric determination of nitrites by means of ceric sulfate solution. H. Bennett and H. F. Hartwood. *Analyst* 60, 677-80 (1935).—Sol. nitrites can be titrated with  $Ce(SO_4)_3$  soln. with eriochrome as indicator. For detg. small amts. of K in soils the ppt. of  $K_2Co(NO_3)_6$  can

be dissolved and the nitrite titrated with  $Ce(SO_4)_3$  instead of  $KMnO_4$  as is common practice. W. T. H.

Potentiometric determination of phosphate. I. A. Atanasiu and A. I. Velulescu. *Z. anal. Chem.* 102, 344-50 (1935).—The titrations described in this paper were carried out with  $Pt/Ni$ ,  $Pt/calomel$  and  $Pt/Hg$  electrodes but the best results were obtained with  $Pt/Ni$ . Irrespective of whether a monobasic, dibasic or tribasic alkali phosphate soln. is titrated with  $UO_2(OAc)_2$  soln., the ppt. is always  $HUO_2PO_4$ . The temp. of the soln. should be 60-70° and sufficient  $AcOH$  should be added to make  $pH = 5-5.6$ . The presence of 0.5-1.0% of hydroquinone is necessary. The titration must take place slowly. The presence of alkali or  $NH_4$  salts does not affect the end point. W. T. H.

Colorimetric determination of orthophosphate in the presence of metaphosphate and pyrophosphate. Kazimierz Boratynski. *Z. anal. Chem.* 102, 421-8 (1935).—The colorimetric method of Lohmann and Jendrasik (C. A. 21, 1206) slightly modified is recommended. Instead of heating to 37°, it is recommended to heat 10 min. at 25° and allow to cool 5 min. As reagent for the reduction of the phosphomolybdic acid either 1,2,4-aminonaphtholsulfonic acid (eikonogen) or p-methylaminophenol sulfate (photo-rex) can be used. The test is not given by metaphosphate or pyrophosphates but when much of the latter is present more molybdic acid is required. W. T. H.

Rapid gravimetric method for the analysis of selenates. Raluca Ripan-Tilici. *Z. anal. Chem.* 102, 343-4 (1935), cf. C. A. 29, 7222<sup>a</sup>.—To det. Se, dil the soln. until it is approx. 0.01 M, heat to boiling and introduce dropwise 0.5 M  $Pb(NO_3)_2$  soln. until about 0.1 cc. in excess is present. Boil 2 min. while stirring, stop heating, add sufficient  $FIOH$  to make the soln. contain 30-35% alc. and allow to stand 4-5 hrs. with occasional stirring during the first hr. Filter, wash by decantation with 30% alc. and finally wash in the crucible with 3 cc. of concd. alc. and 1 cc. of ether. Allow to stand a few min. in a vacuum desiccator and weigh as  $PbSeO_4$ . If care is taken to avoid adding too much reagent, the results are excellent but the ppt. has a high degree of ad-orptive power. W. T. H.

Analysis of small quantities of gas by means of the usual microanalytical apparatus. Wm. F. Bruce. *Mikrochemie* 18, 201-5 (1935).—In connection with work on the metabolism of bacteria, small quantities of gas were collected from culture media, the  $CO_2$  was detd. in the usual way and then the unabsorbed gas was heated with air and the resulting  $CO_2$  and  $H_2O$  were weighed. Slight changes in the Pregl combustion train were made. W. T. H.

Determination of carbon dioxide in gas mixtures by means of  $pH$  measurements. Yrjö Kauko and Julius Carlberg. *Z. anal. Chem.* 102, 393-407 (1935); cf. Kauko, C. A. 28, 3377<sup>a</sup>.—Further details concerning the method are given, particularly with respect to lab. practice. W. T. H.

The determination of carbon dioxide in air with a photo-colorimeter. Bytchun and Laska. *Chem. Listy* 29, 201-2 (1935).—A known vol. of air previously washed with water was passed through a reagent in a glass cell standing between the photocell of a Se cell colorimeter and a light source. The most satisfactory reagent was water contg.  $1 \times 10^{-4}$  g. mol.  $NaHCO_3$  and  $2 \times 10^{-4}$  g. mol. dibromothymolsulfothalen per l. The equl. between the color tint and the partial pressure of  $CO_2$  in atm. ( $P$ ) is given by the equation  $b/P = 3.94 - 0.85 \log P$  (18°), where  $b/P$  represents the  $pH$  detd. colorimetrically and not corrected for the electrolytic content of the soln. The const. 3.94 was detd. experimentally and is a function of the temp. and concn. of the substances in soln. The equation was correct for 0.03-0.30%  $CO_2$  in air by vol. The temp., pressure, water content, and vol. of the drawn air have no influence upon the results providing that a precision of not greater than 0.01  $pH$  is sought. Theoretically, the reagent ought to serve for an unlimited no. of detns.; actually, it must be changed in frequent intervals. Soot, tobacco smoke,  $SO_2$ ,  $H_2S$  and  $NH_3$  in the air are detri-

mental to the reagent, but all of these substances are removed by scrubbing with water. With a suitable app. the air can be analyzed for  $\text{CO}_2$  in 3 min with an accuracy of 2-3% based on the quantity of  $\text{CO}_2$  present. For temp. other than  $18^\circ$  the vol. of  $\text{CO}_2$  has to be corrected by increasing the b.p.n. 0.004 for every  $1^\circ$  rise in temp.

Frank Maresh

The determination of carbon dioxide in carbonates and in baking powders. I. Vojit. *Chem. Listy* 29, 185-7 (1935), cf. C. A. 29, 5040<sup>3</sup>.—The use of  $\text{H}_3\text{PO}_4$  to liberate  $\text{CO}_2$  is advocated.

Frank Maresh

A test for hydrogen peroxide with diphenylcarbazide. L. N. Lapin. *Z. anal. Chem.* 102, 418-20 (1935).—The test for  $\text{H}_2\text{O}_2$  which depends upon the formation of blue, ether-sol. perchromic acid can be made much more sensitive with the aid of diphenylcarbazide which reacts with the perchromic acid and as a result a reddish violet color is imparted to the ether in cases where the color of the blue perchromic acid is hardly visible. Make the soln. to be tested acid with 2-3 drops of 20%  $\text{H}_2\text{SO}_4$  by vol., add 3-4 cc. of freshly prep'd abs. ether and a few drops of 0.01 N  $\text{K}_2\text{Cr}_2\text{O}_7$ . Shake and look for a blue color in the ether layer. Then add cautiously to the sep'd ether 2 drops of reagent prep'd. by heating a few crystals of diphenylcarbazide with 0.5 cc. of 96% alc. by vol. and adding, when cold, 5 cc. of abs. ether. As little as 5  $\gamma$  of  $\text{H}_2\text{O}_2$  in 5-10 cc. of liquid can be detected. W. T. H.

Color test for ammonia with hypobromite and thymol. P. Arne Hansen. *Z. anal. Chem.* 102, 279 (1935).—The test recommended by Lapin and Hein (C. A. 29, 78<sup>3</sup>) is not new but was proposed by Hansen (C. A. 24, 3032) and has been used by others. W. T. H.

Some applications of thermomagnetic analysis in the study of oxides of iron. Georges Chaudron. *Bull. soc. chim. Belg.* 44, 339-50 (1935).—Thermomagnetic analysis of any of the oxides of Fe is accomplished by tracing the curve of variation of magnetization as a function of temp. A new app. is described which permits heating of the sample in the open air. Curves are recorded, photographically. It is possible with the procedure described to det. the nature of the constituents of a mixt. of iron oxides. W. J. Peterson

Determination of potassium bitartrate in commercial calcium tartrate. L. Gadais and J. Gadais. *Ann. chim. anal. chim. appl.* 17, 290 (1935).—The proposed method consists in boiling 10 g. of the sample with 150 cc. of water and 3 g. of  $\text{CaCO}_3$ . The soln. is made up to a definite vol. and from an aliquot part of the filtered soln.  $\text{KHC}_2\text{H}_3\text{O}_4$  is obtained by evapn. nearly to dryness, adding  $\text{AcOH}$ . The results obtained are low partly because some of the tartrate originally present as  $\text{KHC}_2\text{H}_3\text{O}_4$  is converted into less sol.  $\text{CaC}_2\text{H}_3\text{O}_4$ . No analytical data are given. W. T. H.

Estimation of small quantities of magnesium sulfate. Max Moller and Gunver Schlegel. *Mikrochemie* 18, 159-61 (1935).—Emrich, C. A. 28, 65<sup>3</sup>, has shown how  $\text{MgSO}_4$  can be treated with  $\text{Ba(OH)}_2$  in excess, the washed ppt. of  $\text{Mg(OH)}_2$  treated with  $\text{H}_2\text{SO}_4$  and then, after removing the excess acid, a new ppt. of  $\text{Mg(OH)}_2$  and  $\text{BaSO}_4$  obtained as before and the process repeated until finally an easily weighable quantity is obtained. The suitable techn. for converting 0.05 mg. of  $\text{MgSO}_4$  into a ppt. of  $\text{BaSO}_4$  weighing over 1 mg. is explained. In the series of expts. described, the greatest error was less than 10% of the truth. W. T. H.

Quick method for silica. R. E. Stephenson. *Chemist Analyst* 24, No. 4, 14-15 (1935).—Heating the dried sample with HF and  $\text{H}_2\text{SO}_4$  and detg. the  $\text{SiO}_2$  by loss in weight are again recommended. W. T. H.

Studies concerning the relation between sensitivity and molecular weight (weighting effect). P. Krumholz and H. Watzel. *Mikrochemie* 19, 65-68 (1935).—The weighting effect in the salt formation of HBil, with heterocyclic bases was studied. Bi salts with iodide and org. bases in the presence of acid give difficultly sol. yellow ppts. of the general formula HBil, base; such compds. can be used for the detection of Bi or for the detection of the base in question. Studies with quinoline and thiazole bases show that

the sensitivity of the test increases with the mol. wt. of the base. On the other hand, the decrease in the sol. product of the salts is out of all proportion to the increase in the mol. wt. of the salt formed. Thus the picoline salt has the mol. wt. = 684 and the quinaldine salt = 734 whereas the soly. of the first salt is about 25 times as great as that of the second. The effect of making a quaternary compd. also increases the sensitivity but it seems highly improbable that here merely a weighting effect comes into play. W. T. H.

Rapid determination of moisture in small quantities of substance. J. Lrdos. *Mikrochemie* 18, 250-60 (1935).—In tech. analysis, water is very commonly detd. by adding to the weighed sample some org. liquid which does not mix with water and has a slightly higher b. p. The sample + liquid is then heated and the 2 immiscible liquids are collected where they distil off, the vol. of  $\text{H}_2\text{O}$  is then read (cf. Schlapfer, C. A. 8, 1656). The same method is applicable to the study of medicinal and biol. substances. When proper corrections are made, the results are nearer the truth than is the case when  $\text{H}_2\text{O}$  is detd. by loss in wt. A suitable app. is shown and the method of carrying out the analysis explained. W. T. H.

Volumetric determination of evaporation rates. L. A. Wetlaufer and J. B. Gregor. *Ind. Eng. Chem., Anal. Ed.* 7, 290-3 (1935).—Rates of evapn. of org. solvents and thinners were detd. by blowing dry air over the samples contained in standard graduated oil centrifuge tubes with tapered bottoms and held in a horizontal position. At definite time intervals the tubes were brought to the vertical position and after 1 min. the vol. was read. Duplication of results were secured to within  $\pm 3\%$ . A considerable saving in time and labor over gravimetric methods can be secured by the use of this evaporimeter. J. W. Shipley

The yellow color of hydrochloric acid containing selenium. II. The behavior of hydrochloric acid containing selenium in the cold and on dilution with water. Friedrich Ullrich and Hugo Ditz. *Z. anorg. allgem. Chem.* 224, 213-24 (1935), cf. C. A. 29, 1361<sup>1</sup>.—The Se content of the yellow solns. was detd. at decreasing temps. (lowest temp.  $-22.5^\circ$ ) and in various concns. of HCl. The results indicate that the initial pptn. of elementary Se (Tyndall effect) depends upon both the temp. and the concn. of  $\text{SnCl}_4$ . The lower the temp. the less the amt. of  $\text{SnCl}_4$  necessary to cause the initial pptn. of elementary Se. L. W. Roth

Elimination of phosphoric acid in microchemical qualitative analysis. S. Ginsburg and M. H. Pringheim. *Bull. soc. chim.* [5], 2, 1694-7 (1935).—To the hot  $\text{AcOH}$  soln. add some aged, colloidal Fe hydroxide and all the  $\text{H}_3\text{PO}_4$  will be pptd. W. T. H.

Notes on microsilicate analysis. I. Determination of silicic acid. Karl Schoklitsch. *Mikrochemie* 18, 144-53 (1935).—The method of Galfayan and Tarayan (C. A. 28, 1302<sup>2</sup>) was applied to the detn. of  $\text{SiO}_2$  in basalt, granite, omphacite (pyroxene) and hornblende-feldspar; remarkably accurate results were obtained in the analysis of samples weighing 7-14 mg. The dried sample was heated with HF and  $\text{H}_2\text{SO}_4$  and then with  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ . The resid. was heated for an hr. at about  $750^\circ$  with a weighed amt. of  $\text{NaPO}_3$  and from the final wt. the wt. of volatilized  $\text{SiO}_2$  obtained. It was necessary to correct for slight volatilization of the  $\text{NaPO}_3$ . W. T. H.

The determination of very small quantities of hydrocyanic acid. Applications in plant physiology and toxicology. M. T. François and N. Lafitte. *Bull. soc. chim. bel.* 17, 1088-96 (1935).—One cc. of the slightly acidified soln. to be analyzed is placed in a test tube and the tube closed with a paraffined cork from which is suspended a dampened strip of Na picrate paper prep'd. according to the method of Guignard (C. A. 2, 865). After standing 24 hrs. at room temp. the color of the paper is compared with that of control strips exposed in the same manner to solns. containing known quantities of HCN. As little as 0.002 g. HCN per l. can be detd. In the presence of starch or  $\text{NH}_3$  and its compds. the values obtained are a little too low. EtOH, castor oil and liver and thymus tissues do not interfere.

heart tissue fires part of the HCN and causes considerable error.

L. E. Gilson

Determination of small quantities of mercury in leafy vegetables by means of diphenylthiocarbazon (dithizone) W. O. Winkler. *J. Assoc. Official Agr. Chem.* 18, 638-44 (1935).—The method described requires no elaborate app., is not time-consuming and yet gives consistently accurate results (to within 0.005 mg.) in detg. 0.005 to 0.02 mg. of Hg. It is based on the following principles: (1) When a dil. acid soln. contg. Hg and other metals is shaken with a  $\text{CCl}_4$  or  $\text{CHCl}_3$  soln. of dithizone, the normal green color of the dithizone soln. changes to a bright orange yellow because of formation of a sol. org. Hg complex (1 mg. Hg reacts with 2.6 mg. of dithizone). The yellow color persists as long as the Hg is in excess, when sufficient dithizone is added to react with all the Hg, any excess of reagent turns the soln. green or red or reddish violet, depending on whether traces of Cu are present in the mixt. (2) Cu in excessive concn. must be removed before the titration of the Hg, the sepn. being accomplished by adding to the soln. some KI in presence of which the Cu may be extd. with dithizone, the Hg being left in soln. Pt metals are practically the only ones that interfere. (3) The Hg cannot be extd. or titrated with dithizone in acid soln. when iodides are present, but it can be extd. in ammoniacal soln.; it can be extd. from an acid soln. contg. iodides by the use of Na diethylthiocarbamate and  $\text{CHCl}_3$  as extractant. The method is essentially as follows: Destroy org. matter by gently refluxing a suitable sample (150-200 g. in the case of lettuce) with 50 cc.  $\text{HNO}_3$  and 300 cc.  $\text{H}_2\text{O}$  followed by oxidation with  $\text{KMnO}_4$  and addn. of  $\text{H}_2\text{O}_2$  to complete soln. of  $\text{MnO}_2$ , add 0.5 g. crysd.  $\text{NH}_4\text{OH}$  sulfate or chloride; if Sb is present add 15 cc. of 10% tartaric acid soln. previously extd. several times with dithizone soln. (0.05 mg. per cc. in  $\text{CCl}_4$  and  $\text{CHCl}_3$ ) to free it from Hg, to concn. the Hg ext. with successive portions of dithizone soln. till the ext. is green or reddish; if Cu is present in large quantities, shake the combined dithizone exts. with  $\text{H}_2\text{O}$  contg. a few drops of 1 + 1  $\text{H}_2\text{SO}_4$ , a few crystals of KI and a few drops of 5% Na arsenite soln. to prevent liberation of free I, the Cu remaining in the ext. and the Hg being transferred to the aq. phase; titrate by one of the following procedures: (1) make the soln. ammoniacal and titrate directly with dithizone (0.0125 mg. per cc. in  $\text{CCl}_4$ ) to a red end point (it is preferable first to make an approx. titration), liberate the Hg in an acid iodide soln. and repeat the titration accurately; (2) ext. the Hg from the acid soln. contg. iodides by adding 2 cc. of 1% Na diethylthiocarbamate soln. and using several 10-cc. portions of  $\text{CHCl}_3$ , then oxidize the exts. and titrate with dithizone. A. P. C.

Detection of organic compounds by means of spot tests X. F. Feigl and O. Frehden. *Mikrochemie* 18, 272-6 (1935); cf. C. A. 29, 6385<sup>1</sup>.—Place a fragment of the sample in a micro test tube and melt it together with a little diphenylamine over a free flame. Take up the melt in a drop of EtOH; if aniline blue is formed, it is proof that an oxalate was present. Carry out a blank test at the same time.

W. T. H.

Determination of selenium in organic matter. K. T. Williams and H. W. Lakin. *Ind. Eng. Chem., Anal. Ed.* 7, 409-10 (1935).—The method described by Robinson, Dudley, Williams and Byers (C. A. 28, 5363<sup>1</sup>) has given good results in several thousand detns. but is tedious and there is danger of losing some Se by spattering. To det. Se in vegetation, first grind the sample to pass through a 2-mm. sieve. Take 10 g. and digest with 50 cc. of concd.  $\text{H}_2\text{SO}_4$  and 100 cc. of concd.  $\text{HNO}_3$  in a 600-cc. beaker. Stir with a thermometer and keep the temp. below 100° until frothing has ceased and then raise the temp. to 120°. Heat until there is no further evolution of  $\text{NO}_2$ . Cool, transfer to an all-glass distr. flask, add 100 cc. of HBr and 1 cc. of  $\text{Br}_2$  and collect 75 cc. of distillate. From this point continue as in the other procedure. W. T. H.

Volumetric modification of the Pregl halogen micro-combustion method for organic iodine. Paul L. Kirk and Kenneth Dod. *Mikrochemie* 18, 179-81 (1935).—The volumetric method of Kendall (C. A. 5, 3551; 6, 843;

1901; 8, 3665) can be used to advantage in connection with Pregl's halide combustion method. Excellent results were obtained with 3-10 mg. samples of 5 typical org. compds. contg. I. W. T. H.

Determination of protein nitrogen. Accelerating the Kjeldahl-Gunning digestion by addition of phosphates. H. W. Gerritz and J. L. St. John. *Ind. Eng. Chem., Anal. Ed.* 7, 380-3 (1935).—The substitution of 10 g. of anhyd.  $\text{K}_2\text{HPO}_4$  or 12 g. of the trihydrate crystals for 10/16 of the  $\text{Na}_2\text{SO}_4$  or  $\text{K}_2\text{SO}_4$  used in the digestion of the samples shortened the required time to 25 min. or less. For the analysis of "mixed feeds" it is recommended to digest 25 min. or less over the Bunsen flame. When heated on a preheated 700-w. elec. plate, 9 min. proved sufficient. Samples of dried blood, fish meal, soybean meal and dried skim milk required less than 15 min. over grid burners. Low results were obtained when all the alkali sulfate was replaced by phosphate. The addn. of Fe to the catalysts and of  $\text{K}_2\text{S}_2\text{O}_8$  is unnecessary. W. T. H.

Titrimetric determination of organic substances by oxidation with chromic acid H. C. S. Sreethala. *Z. anal. Chem.* 102, 321-8 (1935); cf. C. A. 29, 4683<sup>1</sup>, 6565<sup>1</sup>.—von Fellenberg (C. A. 21, 3829) succeeded in detg. C in org. compds. by oxidizing them to  $\text{H}_2\text{O}$  and  $\text{CO}_2$  with  $\text{CrO}_3$ , but some of the results obtained varied about 10%. The expts. described in this paper were undertaken to det. whether the inaccurate results were due to faulty procedure and whether it were possible to modify the procedure so that a complete oxidation of the C will take place. As a result of expts. with benzoic acid, mannitol, sucrose, malonic acid, methanol, succinic acid, glutaric acid and adipic acid, the following procedure is recommended: Take of the org. substance an amt. equiv. to about 1 milli-equiv. of  $\text{K}_2\text{Cr}_2\text{O}_7$ . While cooling with ice, add slowly 2 cc. of ice-cold  $\text{H}_2\text{SO}_4$  and 10 cc. of 0.23 N  $\text{K}_2\text{Cr}_2\text{O}_7$  in 84%  $\text{H}_2\text{SO}_4$ . Heat 15 min. or longer if necessary, in boiling water. Cool, transfer to a 250-ml. flask contg. 40 cc. of water. To the mixt. add 20 cc. of 10 N KOH, dil. to about 150 cc. and cool to room temp. Then add 25 cc. of 0.4 N KI soln. from a buret, mix well, let stand 1 min. and titrate the liberated I with 0.1 N  $\text{Na}_2\text{S}_2\text{O}_3$ . In this way 5-10 mg. of substance can be detd. with an accuracy of 0.005 to 0.025 mg. W. T. H.

Color reactions of tartaric, citric and ascorbic acids. Otto Fürth and Heinz Herrmann. *Biochem. Z.* 280, 448-57 (1935).—On warming with pyridine and  $\text{Ac}_2\text{O}$  tartaric acid gives an emerald green, citric acid a carmine red and ascorbic acid a violet-red color reaction, while other dicarboxylic acids give either a brown color or none at all. The pigments formed are high-mol. condensation products, sensitive to  $\text{O}_2$  and sol. in pyridine or acetone. Under the influence of the  $\text{Ac}_2\text{O}$  a chromogen is produced from the dicarboxylic acids, which is converted to the pigment by the pyridine. The pigments from citric and ascorbic acids were obtained in the form of beautiful rosette-shaped crystals which had the compn.  $\text{C}_6\text{H}_4\text{O}_8$  and  $\text{C}_6\text{H}_4\text{O}_8$ , i. e., of an acetylated dihydroxytartaric and ascorbic anhydride, resp. The color reaction is extremely sensitive and the presence of even 1  $\gamma$  of the dicarboxylic acid can be detected by its yellow fluorescence in the ultraviolet light of a Zeiss fluorescent lamp. S. Morpurgo.

A semi-micromethod for determining oxalic acid and calcium by the use of a permanganate titration and an indicator. Miloš Kminek. *Litv. Cukrov. 53*, 496-500 (1935).—From 1 to 40 mg. of oxalic acid was dissolved in hot water and pptd. as  $\text{CaC}_2\text{O}_4$  in a buffered  $\text{AcOH}$  soln. The ppt. was dissolved in 20%  $\text{HNO}_3$  contg. 1% of urea, treated with a few drops of  $\text{Mn}(\text{NO}_3)_2$  soln. and slightly overtitrated with 0.005 N  $\text{KMnO}_4$ . The soln. was cooled and treated with 10 cc. of 0.0012 N  $\text{Fe}^{++}$  soln. and the excess of this reagent detd. by a second titration with  $\text{KMnO}_4$ , to an end point with eriochrome. A similar procedure served for detg. Ca. The results were accurate to within about 1% of the truth. Frank Maresh.

Microscopy of the amino acids and their compounds I. Phosphotungstates and phosphomolybdates. Berlingame Bullock and Paul L. Kirk. *Mikrochemie* 18, 129-36 (1935).—Nearly all of the known amino acids were

studied and those ppts which could be obtained by treatment with phosphotungstic and phosphomolybdic acids were studied under the microscope and the results described with illustrations. II. Picrates and flavanates B. L. Crosby and P. L. Kirk. *Ibid* 137-43.—The wide use of picric and flavanic acids in the sepn. and isolation of the amino acids makes it desirable to know the microscopic appearance and properties of the resulting ppts. In this paper are reported the results of successful attempts to prep. cryst. compds. of nearly all the known amino acids with picric and flavonic acids. Photomicrographs are shown for representative compds. of each series.

W. T. H.

Microdetection of volatile amines, particularly monomethylamine, in the presence of ammonia A. v. Wacek and H. Löffler. *Mikrochemie* 18, 277-82(1935).—The method of Klein and Steiner (*C. A.* 22, 4577) has been modified so that a direct sepn. from considerable  $\text{NH}_3$  can be accomplished in the micro oven and the detection of volatile amines accomplished in a single drop of the fluid. At the bottom of a gas micro-oven which is 10-15 mm. high and of 15 mm. diam., place 0.03-0.06 cc. of the neutral or faintly acidic soln. and add sufficient yellow  $\text{HgO}$  to make the surface of the liquid appear dry. Then add a few drops of 5% soda and of a 5%  $\text{NaCl}$  soln. Cover the heating chamber with a watch glass which carries on its bottom surface a drop of water to which has been added a few crystals of a nitronaphthol (usually 2,4-dinitro-1-naphthol). After 8-12 hrs. the drop with the reagent will be dry. Now exam. the product on the cover glass under the polarizing microscope. In this first fraction will be found, first of all, the trimethylamine nitronaphtholate together with excess reagent. After the identification of the trimethylamine, if it is present, add a few drops of 2%  $\text{NaOH}$  to the sample and proceed as before with a new cover glass and reagent. Sometimes it is well to add 8%  $\text{NaOH}$  and carry out the expt. a 3rd time. The isomeric test is considerably more sensitive than that described above, as it permits the detection of 0.5  $\mu$  in 1 cc., whereas the above test may not indicate less than 10  $\mu$  in the most unfavorable cases. It should be used in doubtful cases.

W. T. H.

Tests on the von Fellenberg method of determining sugar by titration Th. v. Fellenberg and Paul Demont. *Mitt. Lebensm. Hyg.* 26, 168-82(1935).—The method (*C. A.* 14, 3618) is reviewed in the light of recent experiences and a new table is given showing the values of 0.5-30.0 cc. in 0.1 cc. increments of 0.1  $N$   $\text{I}_2$  soln. in terms of glucose, invert sugar, sucrose, lactose hydrate and maltose hydrate.

W. T. H.

Determination of glucose in the presence of disaccharides with Barfoed's reagent Th. v. Fellenberg. *Mitt. Lebensm. Hyg.* 26, 182-92(1935).—The effects of variations in the concn. of the  $\text{Cu}$  soln. and the manner and length of boiling are shown and a table is given for the glucose values of  $\text{I}$  soln. obtained by the following method of analysis: Reagents (1) Dissolve 25 g. of pure  $\text{Cu}$  ( $\text{OAc}$ ),  $\text{H}_2\text{O}$  in hot water contg. 3 cc. of glacial  $\text{AcOH}$  and dil. to 1 l. after filtering if necessary. (2) Dissolve 30 g. of Rochelle salt in water and dil. to 100 cc.; the soln. should be prepd. frequently as mold develops rapidly. Use the acid  $\text{NaCl}$ ,  $\text{I}_2$  and  $\text{Na}_2\text{S}_2\text{O}_3$  solns. as in the method of v. Fellenberg. Place 60 cc. of the above  $\text{Cu}$  soln. in a 200-cc. Erlenmeyer flask contg. some boiling stones. Add 20 cc. of the sugar soln., heat to boiling and keep at this temp. for 5 min., without violent boiling which will cause the loss of too much  $\text{AcOH}$ . Cool, add 100 cc. of the acid  $\text{NaCl}$  soln. to dissolve the pptd.  $\text{Cu}_2\text{O}$ , add 5 cc. of tartaric soln. and introduce  $\text{NaHCO}_3$  in small portions until a residue remains undissolved. Titrate with  $\text{I}_2$  soln. The application of the method to the analysis of ext. of malt is described.

W. T. H.

Tests on the gravimetric determination of maltose by the method of Wein C. Zach. *Mitt. Lebensm. Hyg.* 26, 192-5(1935).—Krause has studied the volumetric detn. of maltose on the same sample as that used by Z. The procedure of Wein (*Z. anal. Chem.* 26, 255(1887)) was changed slightly as follows: 25 cc. of the maltose soln.

was added to 50 cc. of Fehling soln. and 25 cc. of water. After heating to boiling again, the boiling was continued 4 min. As a result of numerous analyses a table is given showing the maltose hydrate content corresponding to wts. of  $\text{Cu}_2\text{O}$  ranging from 10 to 350 mg. for increments of 0.1 mg.

W. T. H.

Determination of hexamethylenetetramine, E. Schulek and Wilhelmine Gervay. *Z. anal. Chem.* 102, 271-4(1935).—Schulte (*C. A.* 29, 3939) had some difficulty in carrying out the simple procedure recommended by Schulek and Gervay (*C. A.* 27, 1934). In the light of this criticism further expts. were made and results obtained showing that good results are obtained if certain simple precautions are taken. The hydrolysis must take place at 100° and if a strongly boiling water bath is not available a free flame should be used. A glass-stoppered Erlenmeyer flask should be used with a thin-walled, broad bottom.

W. T. H.

Determination of urethan also in the presence of aminopyrine, theobromine, caffeine and derivatives of carbamide E. Schulek and Wilhelmine Gervay. *Z. anal. Chem.* 102, 275-9(1935).—The app. used is the same as that recommended by Schulek and Vastagh (*C. A.* 25, 3332) and by Schulek and Kerényi (*C. A.* 26, 4276, 4412). Dil. the medicinal prepn. to make it contain about 10 mg. ethylurethan in 5 cc. To this quantity of soln. in the 100-cc. Kjeldahl flask add 2 cc. of concd.  $\text{H}_2\text{SO}_4$  and boil vigorously under reflux condensation for 1 hr. Remove the condenser, add 2 drops of methyl red indicator soln., some rough pumice powder and 30-40 cc. of water. Rinse out the condenser and dry the ground glass connection. Distil into a 100-cc. Erlenmeyer flask contg. 10 cc. of 0.02  $N$   $\text{H}_2\text{SO}_4$ . While boiling the soln. in the flask, slowly add "boiled out" 10%  $\text{NaOH}$  until the methyl red changes to yellow and the soln. contains 2-3 drops of  $\text{NaOH}$  in excess. After distg. off 30-35 cc. of liquid, remove the receiver, boil off  $\text{CO}_2$  and titrate the excess  $\text{H}_2\text{SO}_4$  with 0.02  $N$   $\text{NaOH}$ . Numerous results show that the method is good.

W. T. H.

Curves for use in the colorimetric estimation of carotene W. S. Ferguson. *Analyst* 60, 680-3(1935).—The colorimetric detn. of carotene is usually based on a comparison with the color of  $\text{K}_2\text{Cr}_2\text{O}_7$  soln. In this paper, a curve is given showing the mg. of carotene corresponding to yellow units observed in a Lovibond tintometer and another curve showing the carotene values corresponding to readings obtained with a Klett colorimeter and dichromate solns. Very pure carotene was used in prep. these curves and the analytical data are given so that chemists can construct their own curves.

W. T. H.

Method for the analysis of technical  $\alpha$ -nitronaphthalene B. P. Fedorov and A. A. Spruskov. *Z. anal. Chem.* 103, 28-38(1935).—See *C. A.* 29, 2189.

W. T. H.

Volumetric determination of camphor by the hydroxylamine method Robert Vandoni and Gerard Desseigne. *Bull. soc. chim.* [5], 2, 1685-91(1935).—In previous methods, the ketone has been allowed to react in an alk. soln. with an excess of  $\text{NH}_4\text{OH}$   $\text{HCl}$  and the excess was detd. by a double titration. In the method here proposed, a direct titration is made of the  $\text{HCl}$  liberated as a result of the formation of the oxime, bromophenol blue is used as the indicator. For the neutralization at the start of the analysis  $\text{Na}_2\text{CO}_3$ ,  $\text{CaCO}_3$  or  $\text{NaOH}$  in alc. can be used. Thus with  $\text{CaCO}_3$  the procedure is as follows: Into a 100-cc. flask introduce 50 cc. of 2  $N$   $\text{NH}_4\text{OH}$   $\text{HCl}$ , 0.2 cc. of the indicator soln. and bring to the neutral tint. Add exactly 1 g. of pure  $\text{CaCO}_3$  and 1-2 g. of the substance. Boil on the water bath with reflux condensation and finally cool to 30°. The soln. should remain alk. to the indicator. Add a measured vol. of  $\text{HCl}$  until all the  $\text{CaCO}_3$  is dissolved and finally titrate with  $N$   $\text{NaOH}$  soln. The results are accurate to within about 1% of the truth. The procedure is applicable to the analysis of other ketones such as camphor and menthone.

W. T. H.

A comparative color test for coumarin and melibiotic acid in *Melilotus* species J. S. Clayton and R. K. Larmour. *Can. J. Research* 13, C, 89-100(1935).—The coupling reaction between phenolic bodies and diazotized  $p$ -nitroaniline

in alk. soln. is shown to be of value in the detection and estn. of the coumarin and melilotic acid content of small quantities of sweet clover. The intensity of crimson color developed on treatment of pure coumarin solns. is shown to be proportional to the concn. of coumarin and to be adaptable to colorimetric measurements. For practical purposes it is shown that this reaction can be used for estn. of coumarin and melilotic acid together. It is

possible to obtain the crimson color directly with plant exts. contg. coumarin or melilotic acid, and a tentative method enabling comparisons to be made among small amts. of plant material is outlined. A comparison between results obtained by the colorimetric method outlined and by that of Obermayer shows that the latter method is open to several serious errors in the estn. of coumarin.

J. W. Shipley

## 8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND J. F. SCHLAIRER

**Pyrophyllite in San Diego County, California.** L. M. Richard. *Bull. Am. Ceram. Soc.* 14, 353 (1935).—There is a very large deposit, probably suitable for refractories. A typical analysis is:  $\text{SiO}_2$  66.80,  $\text{Al}_2\text{O}_3$  28.20,  $\text{Fe}_2\text{O}_3$  0.10,  $\text{MgO}$  trace,  $\text{CaO}$  trace,  $\text{K}_2\text{O}$  trace,  $\text{TiO}_2$  0.02,  $\text{MnO}$  0.04,  $\text{S}$  0.06,  $\text{H}_2\text{O}$  5.00 and  $\text{Na}_2\text{O}$  0.05%.

**Mapping Michigan for geologists.** Talbert Abrams. *Eng. Mining J.* 136, 564-5 (1935).—Over 12,000 miles have been photographed from the air for the study of Au, Fe and nonmetallic mineral deposits. W. H. B.

**The nickel-copper occurrence in the Bushveld Igneous complex west of the Pilansberg.** C. M. Schwellnus. Union S. Africa, Dept. Mines, *Geol. Series Bull.* No. 3, 36 pp. (1935).—The source, grade, treatment and uses of these ores are discussed. Alden H. Emery

**Some geological aspects of recent research on coal.** H. C. A. Hickling. *Colliery Guardian* 151, 423-5, 471-2 (1935).—An address. G. G.

**Geology of southern Saskatchewan.** F. J. Fraser, F. H. McLearn, L. S. Russell, P. S. Warren and R. T. D. Wickenden. Can. Dept. Mines, *Geol. Survey Mem.* No. 176, 137 pp. (1935).—The occurrences of coal in southern Saskatchewan are described and 25 analyses given. The petrographic and mech. analyses of over 1000 samples of sediments are summarized; the heavy mineral assemblages of the various formations are described. The gray, feldspathic sands in the Eastend area probably represent the undecomposed sediments; the sandy clays are derived by prolonged weathering and leaching. A. H. E.

**Scotland: The tertiary volcanic districts.** J. E. Lichy. *Geol. Survey Gt. Brit.* 1935, 115 pp.—The compn. and mutual relations of the intrusive and extrusive volcanic and plutonic rocks are discussed. Barite is worked in Arran; small deposits of diatomite occur in Mull and northern Skye; oolitic limy ironstone, mainly chamosite, with some Fe oxides and siderite forms a bed, worked during the War, averaging 8 ft. thick in the Upper Lias of Raasay, and immediately above it some 8-10 ft. of shale yielding 12-12.8 gallons and 6.2-7.4 lb.  $(\text{NH}_4)_2\text{SO}_4$  per ton. A little coal occurs in Arran, and lignite in Skye and Mull. Sapphires, of no value as gems, are found in Mull and Ardnamurchan. C. A. Silberrad

**The South of Scotland [geology].** J. Pringle. *Geol. Survey Gt. Brit.* 1935, 97 pp.—At Leadhills argenteiferous galena, hematite, chalcopyrite, antimony, pyrolusite, sphalerite and alluvial Au have been found and some are still worked; at Wanlockhead galena, hematite and sphalerite. Monazite and arsenical pyrite occur at Cairnmore of Fleet; barite at Auchencrain (Kirkcudbrightshire); Cu carbonate in the Priestlaw granite, and coal is or was worked at Canonbie and Sanquhar. There are several S and chalybeate springs in the area. C. A. Silberrad

**The Welsh Borderland [geology].** R. W. Pocock and P. H. Whitehead. *Geol. Survey Gt. Brit.* 1935, 84 pp.—Barite occurs near Pontesbury (5 m. S. W. of Shrewsbury) in veins and bunches, usually iron-stained and of platy habit, often encrusted with bitumen and associated with small quantities of malachite, chalcocite, azurite and bornite. Galena and sphalerite associated with barite, fluorite and pyrite are found near Shelve (5 m. further S. W.). Coal is still worked south and west of Shrewsbury, but at Pitchford (6 m. S. S. E. of Shrewsbury) a black bitumen occurs from which the medicinal prepn.

Bedton's British Oil, was formerly made. The only occurrence in England and Wales of monchiquite is at Golden Hill between Chepstow and Usk. C. A. S.

**North Wales [geology].** Bernard Smith and T. Neville George. *Geol. Survey Gt. Brit.* 1935, 92 pp.—The most important economic products at present are the slates of Penrhyn and the coal and breccia of Denbigh and Flint, but numerous metalliferous deposits are or have been worked. In the Cambrian area of Carnarvon and Merioneth oolitic and psilolitic Fe (hematite, pyrite, etc.), and (elsewhere) ores of Cu, Fe, Zn, As, Sb, Pb as sulfides, and a little Au. Galena, sometimes argenteiferous, sphalerite and chalcopyrite, sometimes associated with barite and fluorite, occur in the Silurian and Carboniferous areas, especially at Parys and Halkyn Mountains. C. A. S.

**Northern England [geology].** T. Eastwood. *Geol. Survey Gt. Brit.* 1935, 76 pp.—Besides coal the economic products of this region are numerous. Hematite, especially in large masses resulting from metasomatic replacement of limestone; Cu ores, formerly worked near Keswick; argenteiferous galena associated with sphalerite, at many places, wolframite and scheelite in Cumberland, and, in amounts too small to be of commercial importance, ores of Co, Sb, Bi, Au, Mn and Mo. In all cases there are many different mineral species. Barite and witherite are associated with the galena, and in some cases are found in close juxtaposition with coal; the barite is often associated with fluorite. Gypsum occurs in the Vale of Eden, and anhydrite in massive form at Billingham; while salt is frequently associated with both. Diatomite is found near Kendal, and graphite was worked near Keswick. There are numerous examples of pneumatolytic and thermal action. C. A. Silberrad

**The Grampian Highlands [geology].** H. H. Read. *Geol. Survey Gt. Brit.* 1935, 81 pp.—In this area there are, or have been, worked: ores of Mn at Tomentoul, in Islay, and at Dalroy (Nairn), the last containing up to 60-70%  $\text{MnO}_2$ ; of Fe at Tomentoul, Dalroy, Ardrilly (Banff) and Stonehaven; of Pb and Zn (galena and sphalerite often carrying Ag and Au, and associated with pyrite and chalcopyrite) in Islay and Argyll, at Tyndrum (Perth), Aberdeen, Strathspey and Lossiemouth; of Cu (usually chalcopyrite) in Islay and around Loch Fyne, (Cu-Ni ore, and elsewhere in the neighborhood nickeliferous pyrrhotite) near Inverary, while chrysocolla and malachite occur elsewhere. Barite, graphite, diatomite, talc and asbestos are also found, and a feldspar containing 8.90%  $\text{K}_2\text{O}$  and 2.48%  $\text{Na}_2\text{O}$ . There is much metamorphism due to intrusive rocks. C. A. S.

**The geology of Ceylon.** J. S. Coates. *Ceylon J. Sci.* B19, 101-87 (1935).—The mineralogical constituents of Ceylon rocks are discussed and data are given on the occurrences of economic minerals such as graphite, precious stones, mica, thoranite, ilmenite, monazite and zircon sands. K. D. Jacob

**The porosity of Ceylon rocks.** J. S. Coates. *Ceylon J. Sci.* B19, 189-91 (1935).—The porosity was in the approx. ascending order: garnetiferous khondalite, dolerite, charnockite, khondalite, biotite-gneiss, pink granitoid gneiss, leptynite and pink granulate. The values for nearly all samples of charnockite were within the range for Aberdeen granite and Stonehaven granite from Great Britain. With the exception of the leptynite and pink granulate, the rocks were, for all practical purposes,

impervious to water A method for the detm of the porosity of rocks is outlined K. D. Jacob  
Notes on analysis of igneous rocks Amílcar Mário de Jesus *Rev chim pura applicada* 7, 115-19(1932) — de J considers several ways of saving time in rock analysis

such as a single evapn. for  $\text{SiO}_2$ , and a shorter  $\text{K}_2\text{SO}_4$  fusion (2 hrs) in an open crucible for  $\text{Al} + \text{Fe}$  C. D. W.

Petrographic studies of American coals (Thuesen Sprunk) 21

## 9—METALLURGY AND METALLOGRAPHY

D. J. DFMOREST, OSCAR F. HARDER AND RICHARD RIMACI

The nonferrous mining and metallurgical industry in Spain. E. Jimeno and I. R. Morral. *Metals & Alloys* 6, 317, 321(1935) D. S.

The role of patents in modern metallurgy. A. W. Deller. *Metals & Alloys* 6, 303-6(1935) D. S.

The physical chemistry of flotation. VI. The adsorption of amines by sulfide minerals. Plisc Evelyn Wark and Jan Wm Wark. *J. Phys. Chem.* 39, 1021-30 (1935), cf. C. A. 29, 6374<sup>1</sup> —The max contact angle at the line of triple contact, air-solid- $\text{H}_2\text{O}$  is independent of the particular amine chosen, being within a few degrees of  $60^\circ$  for all amines. The amine induces a more or less permanent effect on the mineral surface, probably because of adsorption. Of the minerals tested, the Cu minerals respond most readily and pyrite responds least readily to amines; activated sphalerite closely resembles chalcopyrite in its response. With hexylamine, sphalerite can be floated away from galena. Of the amines tested, the quaternary ammonium salts are most readily adsorbed. The higher the homolog, the lower the necessary concn., i. e., the ethylamines are more effective than the methylamines, the propylamines than the ethylamines, etc. The primary amines are the least active, the secondary amines are more active, and the tertiary amines are still more active. Three cyclic amines—*aniline*,  $\alpha$ -naphthylamine and piperidine—are of about the same order of activity as isoamylamine, but tribenzylamine is much more active. Contact tests closely parallel actual flotation tests carried out in identical solns. In general, the amines are more readily adsorbed from acid than from alkali solns.

Further tests in flotation of free gold—effect of amyl xanthate and pulp  $\text{pH}$  on recovery is studied—acid wash to remove chemical films has beneficial result. Tests should be run with checks. John W. Johns. *Eng. Mining J.* 136, 498-9(1935), cf. Lange, C. A. 29, 2484<sup>1</sup> —Results are shown of a series of flotation tests showing the effect of Am xanthate and the  $\text{pH}$  of the pulp on the recovery of placer Au. Very low Au recovery resulted when K Am xanthate and terpineol were used alone, but marked improvement was evident when the  $\text{pH}$  was kept between 7.7 and 8.2 by addns of  $\text{H}_2\text{SO}_4$  or  $\text{NaOH}$  as required to keep between these limits. W. H. B.

Milling practice for small gold mines. C. S. Parsons. *Trans. Can. Inst. Mining Met.* 1935 (in *Can. Mining Met. Bull.* No. 282) 437-64 —There are 3 types of Au mills commonly used at small mines: (1) amalgamation and gravity concn., (2) amalgamation and flotation, and (3) cyanidation. Costs of a small mill will be \$6000-\$20000 per ton of ore treated per day. Operating costs (without taxes, depletion or depreciation) are \$7-\$12. The general design and layout of small mills are discussed in detail. The flow sheets of 8 small mills are given and discussed. Alden H. Emery

Manitoba's newest gold producer—A brief description of Gold's Lake Gold Mines, Ltd. J. P. de Wet. *Can. Mining J.* 56, 474-6(1935) W. H. Boynton

Southern gold at Hog Mountain. N. O. Johnson. *Eng. Mining J.* 136, 505-10(1935) —Progress in mining and milling with standard methods at the leading enterprise in the Appalachian Mountains is indicated. Recovery and increase in mill capacity are expected upon completion of the installation of finer crushing equipment, and the production of a low-grade primary concentrate (10:1 ratio of concn.) which will be reground and re-floated in a sep ball mill and flotation circuit. A cyanide plant will be installed at a later date. W. H. B.

Cam and motor Gold Mine, Southern Rhodesia. Digby V. Burnett. *Mining Mag.* 53, 201-13(1935), cf. *Ibid* 34, 73 85(1926) —At the surface the lodes appear as quartz veins, below the first level they assume the character of lode 4, comprising parallel quartz stringers with stibnite, arsenopyrite and pyrite intimately mixed with a gang of quartz, Al and Ca silicates, and Mg carbonates. Wet crushing in Nissen stamps, concn and roasting constitute the present metallurgical practice. Originally, gravity concentrates and oil flotation concentrates were mixed together before roasting, but because of the finely divided nature and chem. compn of the latter a very poor extn was obtained with roasting and subsequent cyanidation. In consequence of this and the disappearance in depth of the Sb content of the ore, it was considered advisable to abandon flotation and resort to gravity concn. on sliming tables. The total tonnage of concentrates from all sources is 11% of the tonnage milled. After crushing and a rough mech. concn. the ore is treated on special reconc. tables, the richest cut-off passes to an amalgamating barrel. Some 71% of the total output is recovered by amalgamation. The concentrates after roasting are water-washed and leached for 14 days with 0.15% KCN soln. The residues after roasting for 10 hrs at temps up to  $740^\circ$  are sent to a dump and weathered for 4 months, after which they are retreated by grinding and cyanidation. Tonnages and recoveries are tabulated. In 1935 an ore of value 35 81 s is treated with an extn. of 84% A. W. P.

Statistical microscopic study of ores and mill products from the Anxox plant of the Granby Consolidated Mining, Smelting & Power Co., Ltd., Anxox, British Columbia. R. F. Head, Arthur L. Crawford, F. E. Thackwell and A. Lee Christensen. *Bur. Mines, Rept. Investigations* No. 3290, 18 pp (1935) —Except for an insignificant amt. of covellite, the entire Cu content is present as chalcopyrite, the mill heads (classifier overflow) contain quartz 27.00, chlorite 12.50, phlogopite mica 5.00, feldspar (chiefly secondary albite) 3.00, calcite 3.00, bronzite, actinolite, tremolite, uraltite, etc., 5.79, pyrite 18.37, pyrrhotite 17.88, magnetite, goethite, etc., 3.05, and chalcopyrite 3.41%. Assocn. of the Cu and Fe minerals is very close. The ore is ground to liberate most of the Cu and to free the rest from gang and large particles of Fe sulfides capable of being floated in a low-grade concentrate that is reground and re-floated. An alk. of 0.11 lb  $\text{CaO}$  per ton of soln is maintained in the primary cells and 0.02 lb in the regrid. Reagent consumption is neutral cresote oil 0.10, K 1 t xanthate 0.05, pine oil 0.06, hydrated lime 2.00, NaCN 0.023 lb per ton of ore. The flow sheet is given. The best concentrate is made at 150-mesh, in sizes smaller than 150 mesh free pyrite and pyrrhotite float in increasing quantities. Greater difficulty is experienced in rejecting pyrrhotite than pyrite. Magnetite rejection is fairly good and open to little improvement, since 50% is locked with chalcopyrite. Locked chalcopyrite is negligible at -400 mesh. In the tails 64.9% of the chalcopyrite is free. Data are given showing the amt. of pyrite, pyrrhotite, magnetite and chalcopyrite in each size of the classifier overflow, in the primary Sub-A concentrate, in the middling feed, in primary middling, in middling concentrate, in the scavenger concentrate, in primary Sub-A tails, in middling tails, and in general tails. Alden H. Emery

Stamp milling and amalgamation practice at Golden-ville, N. S. L. H. Henderson. *Trans. Can. Inst.*

*Mining Met.* 1935 (in *Can. Mining Met. Bull.* No. 283), 473-4; cf. *C. A.* 29, 6183<sup>1</sup>.—Discussion. A. H. E.

Copper mining and smelting in Germany. O. Bertoya. *Metalurgia* 12, 165-8 (1935).—A description of the ore deposit, mining, smelting and refining at Mansfeld plant at Harz mountain. J. L. Gregg

Purification of gallium by fractional crystallization. James I. Hoffman and Bourdon F. Scribner. *J. Research Natl. Bur. Standards* 15, 203-9 (1935) (*Research Paper* No. 823); cf. *C. A.* 29, 6991.—When Ga contg. (as impurities) small amts of Sb, Bi, Cr, Co, Ch, Cu, Au, In, Fe, Pb, Mn, Hg, Mo, Ni, Os, Pd, Pt, Rh, Ru, Ag, Ti, Sn, V and Zn is subjected to fractional crystn of the metal, all the impurities named tend to conc. in the cryst. portion, with the following exceptions. Ag, Hg, In, Pb and Sn are concd. in the molten residue. Cu and Ti remain about equally divided between crystals and residue, Zn is dissolved by the HCl under which the crystn takes place and is entirely eliminated. It is shown that the sepn from Fe or Pt in excess of 0.001%, from In or Pb in excess of 0.01% or from Sn in excess of 0.02% by fractional crystn. of the metal is impractical. W. J. Peterson

Manganese vein on Gowland Mountain, Albert County, N. B. W. J. Wright. *Can. Mining Met. Bull.* No. 283, 575 (1935); cf. *C. A.* 29, 6183<sup>1</sup>.—Discussion. A. H. E.

Concentration of titaniferous ores at Taberg, Sweden, with and without heat treatment. Gust G. Bring and P. G. Kihlstedt. *Jernkontoret Ann.* 119, 303-42 (1935).—The ore consists of a magnetite network with interlamellar growths of ilmenite parallel to the octahedral surfaces. These lamellae have a thickness of 0.1-5  $\mu$ , so mech. sepn. is impossible. Moreover the gang (chiefly olivine) clings tenaciously to the ore by a rim of tough hornblende. By heating to 1000° the hornblende becomes friable and crushing to free minerals is facilitated. The magnetite attached to the hornblende, however, makes the gang magnetic. By mixing with a little coal the magnetization can be slightly prevented. At 1150° the ore begins to give off gas, but at 1100° the ore begins to soften and is quite homogeneous. A high degree of concn. cannot be attained. H. C. Duns

Recovering zinc and vanadium at the Rhodesia Broken Hill plant. T. R. Pickard. *Eng. Mining J.* 136, 459-93 (1935).—Concn., acid leaching and electrolysis are employed at this plant. Flowsheets are given for gravimetric concn. of V ore, for leaching of V concentrates, for Cu and P, for removal of Cu and P, and for Zn-ore leaching. Al cathodes are employed; the Zn is stripped by native laborers, piled on cars and taken to the furnace room. W. H. Boynton

Direct determination of concentration of zinc vapor in thermal reduction of zinc oxide. Octave Doué-Hénault and Claude Decroly. *Compt. rend.* 201, 736-8 (1935).—A preliminary note describing an arrangement for detg. the concn. of Zn vapor (a) immediately above the crucible and (b) in the condenser, and giving as examples of the results obtainable for (a) 19.4-19.8%, and for (b) 3.4-3.8%, the temp. of the charge varying from 920° to 1075° and of the furnace from 988° to 1125° (cf. Maier, *C. A.* 24, 5644).

The uses and occurrences of chrome. Gordon H. Chambers. *S. African Mining Eng. J.* 46, Pt. I, 708-9 (1935).—General. Alden H. Emery

Blast-furnace slag. A valuable by-product. James Watson. *Iron & Steel Ind.* 8, 457-61; 9, 12-13 (1935).

A study of blast-furnace slag. L. Rodríguez Pire and J. Garrido. *Anal. soc. españ. fis. quim.* 33, 709-12 (1935).—A crystal from blast-furnace slag was examined optically and by x-rays, showing  $m(110)p(001)$ , period  $7.8 \times 10^{-8}$  for a prism edge. Chem. analysis gave  $SiO_2:CaMgO:Al_2O_3 = 6:10:1$ . E. M. Symmes

Design and construction of hot blast stoves. Albert Mohr, Jr., and Fred Wille. *Blast Furnace Steel Plant* 23, 689-94, 702 (1935).—See *C. A.* 29, 5701<sup>1</sup>. E. H.

Melting nonferrous alloys in a cupola-type furnace. W. C. Alvin. *Trans. Am. Foundrymen's Assoc.* 6,

737-44 (1935).—The necessary changes are described for adapting an ordinary Fe-melting cupola to one for melting nonferrous alloys. Metal-fuel ratios, losses, the advantages and disadvantages are detailed for this type of melting. Downs School

Continuous pouring of Ford cylinder blocks. E. F. Conc. *Metals & Alloys* 6, 299-303 (1935).—From 6000 to 6500 Ford cylinder blocks as well as some 1300 tons of other castings are poured daily by taking metal from a blast furnace (producing standard foundry Fe) and pricing it in a 400-ton mixer with a definite quantity (about 60%) of cast Fe from a battery of cupolas (cupola charge contains 15% steel scrap), after mixing the metal is transferred to 20-ton elec. furnaces for 20-30 min. where it is superheated to 1445° and from which the metal flows at intervals into a specially designed air furnace fired with pulverized coal which acts as a heating reservoir for the pouring operations. The metal from the air furnace flows into a movable pouring truck or ladle so synchronized in its longitudinal and rotating motions that it receives metal continuously and discharges it intermittently to the molds as they travel on the mold conveyor reel. D. S.

Casting by pressure. P. Roux. *Méaux* 10, 139-54 (1935).—General principles and methods of casting by pressure are discussed. G. T. Motok

Finishing zinc and aluminum die-castings. Herbert Chase. *Machinery* 42, 120-2, 181-4 (1935). cf. *C. A.* 29, 4316<sup>1</sup>.—The relative merits of different methods designed to finish Zn and Al die-castings for pleasing appearance and resistance to corrosion are discussed. Low-cost procedures for cleaning, buffing, plating and polishing are described. Plating-bath compns. for nickel plating zinc and aluminum die-castings are given together with a recommended procedure for etching prior to plating. Methods of applying enamel, lacquer and varnish coatings are discussed. A. L. Kave

Recrystallization and grain growth in cold-worked polycrystalline metals. L. W. Eastwood, Arthur E. Bousm and C. T. Eddy. *Am. Inst. Mining Met. Engrs., Inst. Metals Div., Tech. Pub.* No. 644, 19 pp. (1935).—Complete quant. data are presented on the grain sizes of cold-drawn and cold rolled  $\alpha$ -brass just after the complete recrystn. and after the coalescence produced by annealing. Several new principles are deduced, which may disprove many current misconceptions. The most important of these principles are: (1) The grain size just after complete recrystn. of  $\alpha$  brass is dependent only upon the compn. and history of the material prior to deformation and upon the degree of deformation. (2) All grain growth obeys the same laws, germination as such being generally nonexistent since there is no discontinuity in the size of new grains produced. (3) Abnormality of grain growth in  $\alpha$ -brass is not produced by temp. gradients. (4) The large grains produced by annealing metals contg. low degrees of strain are due to the large grains formed upon recrystn. rather than to the absorption of grains by "germinant grains" produced by a "critically strained" metal at the "germinant temp." (5) Annealed cold-worked metals that do not undergo an allotrophic transformation have coarser grains with higher annealing temp. since rapid heating through the so-called "germinant temp." to a higher temp. produces a coarser grain rather than a finer one. C. L. Mantell

Structure of polished metal surfaces. C. S. Lees. *Trans. Faraday Soc.* 31, 1102-6 (1935).—Specimens of Cu and Au were polished in 3 ways: (1) by hand on a pitch lap with water and rouge, (2) under different loads on wet rouged flannel on a rotating disk, and (3) on flannel and a com. liquid polish. Electron-diffraction photographs were made of these surfaces alternately with electrolytic etching. The Cu was etched with a soln. of 4 g. of  $Na_2SO_4$  per l. The Au was etched with 5% KCN soln. In each of these metals there is an amorphous layer on the surface which is sepd. from the underlying undisturbed metal by a layer of fine crystals oriented so that the (110) planes are approx. parallel to the surface. It is suggested that this preferred orientation is due to deformation caused by pressure normal to the surface,



The thicknesses of these 2 layers are influenced by time of polishing, pressure used and polishing agent employed. On Cu the 2 layers together are 200-1000 Å deep. The amorphous layer is 2-40 Å deep and the oriented layer 150-500 Å deep when method (1) or (2), resp., is employed for polishing. Au has a much thicker oriented layer, 10,000 Å or over. It is suggested that Cu is polished mainly by abrasive action of the rouge while Au is polished largely by the pressure applied to the sample. H. A. S.

**Metallic cementation VI. Cementation by means of silicon powder.** Tsutomu Kase. *Kinzoku-no-Kenkyu*, 12, 397-410 (1935), cf. C. A. 29, 7900\*—By means of com. Si powder the cementation of Si into Fe, Ni and Cu at various temps between 500° and 1200° was investigated. For the surface layer of the cemented specimens, the d-pth of penetration was measured, microscopic examn and chem. analysis were carried out, and the oxidation at high temp and corrosion by  $H_2SO_4$ ,  $HNO_3$  and HCl were studied. Si diffuses into the metals above noted at a temp over 500°, and the rate of diffusion increases as the temp rises. The rate of diffusion of Si into Fe increases abruptly at the  $A_1$  point of Fe. The relation between the increase of wt. of the specimen ( $\Delta W$ ) or the d-pth of penetration ( $P$ ), and the abs. temp. of the cementation ( $T$ ) is given by an exponential function  $\Delta W$  (or  $P$ ) =  $ae^{-b/T}$ . The relation between the  $\Delta W$  or  $P$  and the length of time for cementation ( $t$ ) is given by the similar function  $\Delta W$  (or  $P$ ) =  $ae^{at}$ , where  $a$  and  $b$  are different constants in each case. Though the cemented surfaces of Fe and Ni are brittle, they resist atm. oxidation at high temp and they are not easily attacked by dil.  $H_2SO_4$ . The surface of Cu cemented by Si tends to become porous and decrease the wt. of the specimens. Tomoo Sato.

**Surface hardening for bearing purposes.** Christopher H. Bierbaum. *Machinery* 42, 204-6 (1935)—Pairs of bearing surfaces with desirable properties are made by mating a case-hardened steel surface with one of hard or chilled bronze. Best results are obtained when hardness of case gradually blends into that of softer core. Sudden transitions in hardness giving bearing surfaces susceptible to cracking or checking should be avoided. Optimum conditions are given for the use of chromium plate and nitrided hard cases as bearing surfaces. A. L. Kaye.

**Heat treatment by forced convection.** W. A. Darrah. *Metal Progress* 28, No. 5, 55-60 (1935)—Hot gases from oil or gas combustion are forced around work in the furnace. Greater uniformity and economy in heat treatment are claimed. Good results are obtained up to 1500-1600° F. W. A. Mudge.

**Certainty of results as the basis for the manufacture of high test gray iron.** E. Piwowarsky. *Trans. Am. Foundrymen's Assoc.* 6, 707-26 (1935)—Tensile, transverse and shear strength, hardness and carbide content were detd. on cast Fe test pieces cut from hollow box-shaped castings poured from melts containing various amts of Si, P, Ni, Cr, Mo, W and Cu. D. S.

**Scaling of iron and other metals.** Kurt Fischbeck and Franz Salzer. *Metallwirtschaft* 14, 733-9, 753-8 (1935)—The work of other authors on the scaling and oxidation of Fe, given in 42 references, is reviewed at some length. Scale formed above 570° on Fe consists of several layers. Next to the Fe is "oxoferrite," which is Fe with O in solid soln., then follow  $FeO$ ,  $Fe_2O_3$  and  $Fe_3O_4$ . At lower temps.  $FeO$  breaks up into oxoferrite and  $Fe_2O_3$ . The compn and thickness of the different layers and the speed with which they form depend on the compn of the gas in contact with the metal, its pressure and velocity, and on the temp and the resistance to reaction and diffusion. Equations giving the relation of these factors under various conditions are developed. The rate of scaling of Fe increases until the  $A_1$  transformation point is reached, where a sudden drop occurs, after which the rate increases again. Scaling of Fe can be reduced by the addn of 10% Al, 15% Cr or Al and Cr. The authors themselves investigated the oxidation of pure Fe wire in  $CO_2$ ,  $H_2O$ ,  $NO$ ,  $SO_2$  and  $O_2$  and of Mn in lump form in

$O_2$  at various temps. The increase in weight is plotted against time and the initial reaction velocity against temp. The latter curves for Fe have a sharp break at the  $A_1$  point in the case of each gas except  $SO_2$ . The oxidation in  $SO_2$  was so rapid that it was not carried above 850°. The curve for Mn has a much smaller break at the transformation point than the Fe curves. The scale from a heavily oxidized Fe wire was removed by filing off thin layers, and each layer was analyzed for Fe content and some were examd by x-rays. About 95% of the scale consisted of  $FeO$ , the balance of the higher oxides. C. E. Macfarlane.

**Effect of high temperatures on the strength of soft and medium carbon steels under static and dynamic stressing.** K. Uhlemann. *Metallwirtschaft* 14, 773-8, 795-8 (1935)—Static tensile, impact and endurance tests with smooth and notched test bars, were made at 20°, 200°, 300°, 400° and 500° on 5 steels. A was a 0.12 C steel, B 0.50 C normalized, C 0.50 C heat treated, D 0.60 C quenched and drawn, E 0.70 C, 0.27 Ni, 0.06 Cr, 0.12 W heat-treated. All the steels showed the characteristic blue brittleness at 200-300°, as evidenced by increased tensile strength and lowered elongation and reduction in area. The yield point did not increase in this range. The endurance limit increased similarly, but reached its max at higher temps. For steel B the max was between 400° and 500°, for the others between 300° and 400°. At 500° the endurance limit was lower than at 20°, with the exception of steel B. For steels A to D the increase in tensile strength was up to 25%, the increase in endurance limit slightly higher, from 18 to 27%. With steel E the increase in tensile strength above room temp was slight, the endurance limit dropped 7% and then rose slightly but did not regain its 20° value. The impact strength dropped at 400° to 500°. No relation was found between impact and endurance properties. The surface oxidation which occurred at 400° and 500° did not lower the endurance limit in the same manner as salt-water corrosion. The notched bars for endurance tests had a 45° notch, 3.5 mm deep. The ratio of notched to smooth endurance limit at 20° was close to 0.5 and did not change much at elevated temps. It was slightly higher for steel A than the others, and for steel C dropped to 0.36 at 500°. Some tests were also made with a 0.25-mm deep notch. The ratio was higher and varied more with C content, 0.8 for A, 0.66 for B, 0.62 for D, and 0.56 for E. Complete data are given in tabulations and in graphs. Thirteen references. C. E. Macfarlane.

**Shafts of 1040 steel hardened from controlled atmosphere.** S. K. Olver. *Metal Progress* 28, No. 4, 4-10 (1935), cf. C. A. 29, 7252\*—Air-natural gas mixts are introduced with the work into a horizontal continuous furnace and heated to 1550°F. The work progresses against rising temp. Arrangements are made for vertical quenching. Hardness may be controlled to 58-60 Rockwell C. W. A. Mudge.

**Case hardening and case hardening steels.** Thomas W. Hardy. *Iron & Steel Can.* 18, 51-3, 70-4 (1935)—A review. E. H.

**Case-hardening nickel-chromium steel ingots.** Seiy Nishigori. *Tetsu-to-Hagane* 12, 722-6 (1935).—Non-metallic inclusions in steel ingots consist of C 0.13-0.18, Ni 3.30-4.18 and Cr 0.89-1.15% were nearly spherical and 0.12-0.25 mm in diam. The no. of inclusions was max at the center of the ingot and min at the "transcrystallization zone," of which the thickness was about 40 mm. The inclusions were shown microscopically to be iron oxide, not silicate nor sulfide. Tomoo Sato.

**Tool steels.** André Michel. *Métaux* 10, 3-21, 83-100 (1935)—A comprehensive survey of tool steels, their treatment and characteristics. Seven references.

**Diffusion of hydrogen through mild steel sheet and timplite.** T. N. Morris. *Dept. Sci. Ind. Research Rept. Food Investigation Board* 1934, 186 (1937)—See C. A. 29, 2494\*.

**High manganese steel.** L. Sanderson. *Metallurgia*

12, 171-2, 176(1935).—Properties and uses of Hadfield steel are discussed. J. L. Gregg

Transformation points of nickel steels. Marius Sauvageot and Edmond Rousseau *Compt. rend.* 201, 611-13 (1935).—Dilatometric curves for a steel (Ni 6, C 0.62, Cr 0.5, Mo 0.42) heated for 1 hr. at 650-775° and slowly (3 hrs.) cooled show that the transformation point observed by Andrew and Dickie (cf. C. A. 21, 3336) occurs below 0° when heating is to about 650°, but at a higher temp. (but becoming less marked) as the max temp. is higher. It gradually disappears as Ar' and Ar'' appear. That the phenomenon is essentially due to Ni is confirmed by similar curves being obtained for steels contg. 11 and 20% Ni (C 0.10-0.15%). It is due to segregation of the Ni, possibly as FeNi (34.15% Ni). Mn appears to behave similarly. C. A. Silberrad

Comparison of nickel and nickel-chromium steels with ordinary carbon steels. A. Leblanc *Mémoires* 10, 131-4 (1935).—A general comparison of phys. properties of steels based on mech. tests. G. T. Motok

Aluminum additions in modern commercial steels. H. W. McQuaid *Metal Progress* 28, No. 6, 33-7 (1935)

Phosphorus as an alloying element in steel. H. W. Gillett *Metals & Alloys* 6, 280-3, 307-10 (1935)

Aluminum and its alloys—with special reference to transportation. N. Warren Waterhouse *J. Inst. Engrs Australia* 7, 295-304 (1935).—Methods for the production of Al and its alloys are outlined and their value is indicated. Applications in aircraft, pleasure craft and railway and highway transportation are pointed out. W. H. H.

The abnormal phenomenon of cast copper-rich copper-silicon alloys during heating. Takumi Takedani and Saburo Katori *J. Chem. Soc. Japan* 56, 1058-64 (1935).—The abnormal thermal expansion which takes place in heating the cast Cu-Si alloy contg. 3.0-7.5% Si at 200-800° has been studied. The phenomenon is attributed to the homogenization of the  $\alpha$ -phase, and the soln. of the  $\beta$ -phase into the  $\alpha$ -phase as solid soln. T. Katsuragi

The homogenization of cast structures in copper-rich copper-silicon alloys. Takumi Takedani. *J. Chem. Soc. Japan* 56, 1064-71 (1935); cf. preceding abstr. With the increase of Si content the temp. needed to cause homogenization of the  $\alpha$ -phase becomes lower and the tendency of the  $\beta$ -phase to remain undissolved by heating predominates. T. Katsuragi

The constitution of alloys of copper, aluminum and silicon. I. The equilibrium diagrams of three binary systems. Chiyo Hisatsune. *Tetsu-to-Hagane* 21, 726-42 (1935).—By means of a differential method of thermal analysis, elec. resistance method and microscopic examn., the equil. diagram of the binary alloy of the Cu-Al system was investigated, and a new diagram was proposed. The liquidus of this system consists of 8 branches corresponding to the crystn. of  $\alpha$ ,  $\beta$ ,  $\gamma$ ,  $\delta$ ,  $\epsilon$ ,  $\zeta$ ,  $\eta$ , and  $\theta$ . All  $\delta$  and  $\zeta$  phases form solid solns. in some range of compn.  $\epsilon$  and  $\eta$  phases show the polymorphic transformations, that is,  $\epsilon_1 \rightleftharpoons \epsilon_2$  and  $\eta_1 \rightleftharpoons \eta_2$ , resp. The  $\theta$  phase crystallizes directly from the melts and forms solid soln. in a narrow range. Tomo-o Sato

Magnetic hard iron-nickel-copper alloys. Preliminary report. H. Neumann. *Metallwirtschaft* 14, 778-9 (1935).—The existence of Fe-Ni-Cu alloys with high magnetic and low mech. hardness, which was recently reported by Dhal, Pfaffenberger and Schwartz (C. A. 29, 7257\*) is confirmed. Higher values for coercive force, for a slightly different compn., than the max. reported by the other authors were found, also a different effect of heat treatment. C. E. Macfarlane

A new bearing metal—a lead-base alloy. C. H. Hack. *Metal Progress* 28, No. 5, 61-4 (1935).—A Pb bearing alloy contg. 2.5% Ca, Sn and other hardening elements has high strength at temps. near melting and is used for high-speed gas engines. W. A. Mudge

Alloys of lithium with mercury and (with) indium.

C. Grube and W. Wolf. *Z. Elektrochem.* 41, 675-81 (1935).—Heating and cooling curves have been made of 67 alloys of Li with Hg. The Li-Hg system as constructed from these data is (the compn. of the alloy is stated in weight% Li) 0-0.6 pure Hg, 0.6-18.0 LiHg, 18.0-29.0 LiHg, 29.0-70.0 solid solns. of LiHg, 70.0-72.0 LiHg, 72.0-91.0 LiHg, 91.0-92.0 LiHg, 92.0-100.0 solid solns. of Li. One eutectic point lies at 0.6% Li and -42° and another at 92% Li and 160°. LiHg and LiHg in 375° and 690°, resp. Li are fusible without decomn. The upper existence limit of the other compds. are for LiHg, 164°, Li-Hg, 375°, LiHg, 340° and LiHg, 235°. All compds. except LiHg are formed perfectly. In the Li system only 1 compd., LiHg, is formed which melts without decomn. at 625°. LiHg forms with Li a continuous series of solid solns. while with In it forms only a limited solid soln. The system between 0 and 10% Li requires more work for clarification. The compds. of these systems fit into the generalization regarding alkali metal compds. of Biltz and Zintl and their co-workers. H. A. Smith

The equilibrium diagram of the silver-rich silver-aluminum alloys, with a note on the nature of the transformations. Ichiji Obinata and Masami Hagiya. *Kinzo-ko-Kenkyu*, 12, 419-29 (1935).—The equil. diagram of the Ag-Al system in the range 0-11% Al has been revised from the results of x-ray analysis at ordinary and high temps., elec. resistance methods and microscopic study. In the alloys contg. 5.6 to 8.2% Al a eutectoid and a metatectoid-reaction take place, the temps. of which are 615° and 420°, resp. The metatectoid reaction,  $\alpha + \gamma \rightleftharpoons \alpha_2\beta$ , can easily be suppressed by water-quenching of the alloy. The eutectoid reaction,  $\beta \rightleftharpoons \alpha + \gamma$ , is but little suppressed by the same treatment. The alloys quenched from the  $\beta$ -field consist of the  $\gamma$ -phase, supersatd. with Ag, showing röntgenographically the diffraction lines belonging to the hexagonal close-packed lattice. In the quenched hypoeutectoid alloys an anomalous increase of elec. resistance was observed at about 200° in heating. This was attributed to the formation of a compd. Ag<sub>3</sub>Al. The crystal structure of the  $\beta$ -phase has been detd. by means of x-ray analysis at high temp. This phase was of a body-centered cubic lattice, the parameter of which was found to be 3.295 Å at 700° (Al = 7.93%). Tomo-o Sato

Preparation and physical properties of amalgams of silver, tin and zinc. Kazimierz Duzko. *Przemysł Chem.* 19, 10-13 (1935); cf. C. A. 29, 2859\*.—The 2 amalgams with highest Ag content were prepd. by mixing Hg with electrolytic Ag. All others were prepd. by the electrolytic method—depositing each metal from its anode through a soln. of its salt and to the Hg cathode. Single potentials

Metal in amalgam	%	Brinell hardness	Density	Resistivity Ohm cm 38°	$E_{Ag}^{+}$ 18°	$E_{Zn}^{+}$ 23°
Ag	47.02	103.00	11.09	0.042 $\times 10^{-4}$	0.4191	0.8200
Ag	32.67	111.00	14.25	0.045 $\times 10^{-4}$	0.3493	0.8336
Ag	32.32	18.50	13.38	0.047 $\times 10^{-4}$	0.7083	0.8206
Ag	27.47	20.00	11.26	0.093 $\times 10^{-4}$	0.6512	0.8410
Ag	21.25	8.7	13.40	0.103 $\times 10^{-4}$	0.6374	0.8357
Ag	19.69	8.00	7.95	0.084 $\times 10^{-4}$	0.6350	0.8139
Ag	19.43	7.40	8.04	0.081 $\times 10^{-4}$	0.7125	0.8119
Ag	12.09	0.42	8.36	0.078 $\times 10^{-4}$	0.2230	0.8119
Zn	42.55	15.20	10.54	0.069 $\times 10^{-4}$	0.7538	0.7155
Zn	32.23	13.40	11.34	0.124 $\times 10^{-4}$	0.7696	0.7203
Zn	31.65	13.30	11.43	0.119 $\times 10^{-4}$	0.7510	0.7392

were measured against a H<sub>2</sub> electrode. After prepn. the amalgams were pressed under a pressure of 2500 kg. Amalgams of Ag and Sn form chem. compds., while those of Zn form only an ordinary mixt. This is in agreement with results of Preston (C. A. 26, 411) and Stenbeck (C. A. 27, 6233). A. C. Zachlin

An investigation of the zinc-rich portion of the system iron-zinc. E. C. Truesdale, R. L. Wilcox and J. L. Rodda. *Am. Inst. Mining Met. Engrs., Inst. Metals Div., Tech. Pub. No. 651*, 37 pp. (1935).—New values for the soly. of Arceio iron in high-purity Zn were obtained by 3 direct sampling methods at temps. between 425° and 875°. These data are lower than all previously published

values, obtained from primary cooling-curve arrests. The solid soly of Fe in Zn was investigated by microscopical, elec-cond, x-ray and magnetic-susceptibility methods, but only the microscope gave useful results. The eutectic temp was detd from differential cooling-curve arrests. The 2 peritectic temps were relocated at new high values by cooling curves, but especially by heating curves, which were more accurate. The Zn-rich portion of the system has thus been fixed as follows. Solid soly of Fe in Zn, between 0.0009 and 0.0023%, m p of Zn 419.45°, eutectic temp not less than 419.40 ± 0.05°; lower peritectic 782 ± 1°, upper peritectic 782 ± 1°, soly of Fe in molten Zn 0.018% at 419.4° (the eutectic), 3.0% at 672°, 7.4% at 782°, 9.2% at 875°. All previous data bearing on the Fe-Zn system are critically reviewed, and the equil. diagram is brought up to date.

C. L. Mantell

Isomorphism of the ternary compounds  $Mg_2Zn_3Al_4$  and  $Mg_2CuAl_4$ . F. Laves, K. Lohberg and H. Witte. *Metallwirtschaft* 14, 793-4 (1935).— $Mg_2Zn_3Al_4$  was prep'd. by melting the 3 metals together in the theoretical proportions, and a homogeneous melt was obtained.  $Mg_2CuAl_4$  could not be made by melting.  $CuAl_3$  and  $Mg$  in powder form were pressed under 20,000 atm. and heated 60 hrs. at 500° to alloy them. Twelve alloys of various compns. were made and exam'd microscopically and by x-rays. Only the alloy corresponding to the formula  $Mg_2Zn_3Al_4$  showed lines corresponding to a cubic lattice.  $Mg_2Zn_3Al_4$  is also cubic. Both compds contain approx. the same at % of Mg, 37, and correspond to the formula  $Mg_2X_3Al_4$ , where X may be Cu, Zn or Al. The av. radius of the X atom in both compds. is about the same, 1.39 Å. and 1.41 Å. The lattice const.,  $a$ , for  $Mg_2Zn_3Al_4$  is 14.16 Å. and for  $Mg_2CuAl_4$  14.25 Å. C. E. Macfarlane

Electrical conductivity of rust. Gunther Cohn. *Z. Elektrochem.* 41, 660-4 (1935).—For the local-element theory of corrosion the cond. of rust must be appreciable and such detns. are thus valuable. The conds. of rusts (1) formed at 50° for 21 years on the inside of a cast iron condenser tube, (2) formed for many years on a garden bench near the sea coast, (3) formed for 206 years on a cast iron common submerged in sea water and (4) formed on an old Viking wrought iron sword in a dry place for 1000 years were det'd. The compns. of the rusts in percentage of  $Fe_2O_3$ ,  $FeO$ ,  $H_2O$ , total C and free C were, resp., for (1) 20.6, 41.4, 10.6, 7.9, 5.8, for (2) 50.3, 37.1, 9.2, 2.9, 1.5, for (3) 41.3, 23.1, 13.4, 8.8, 6.1; and for (4) 77.1, 7.2, 12.5, 1.2, 0.0. The total C and free C in the original iron in percentage were, resp., for (1) 3.0, 2.8, for (2) 2.9, 1.5, for (3) 2.7, and for (4) 0.25, 0.0. The specific conductivities for these samples of rust were, 19.7, 0.3, 28.0 and 0.8 mho per cm. The rust consisted of mixts. of a  $FeO(OH)$  and  $Fe_2O_3$  with graphite and carbide except for (3) where the crystal size was so small that sep. x-ray diffraction lines could not be distinguished, except for graphite. These cond. values are high because of networks of graphite and carbide in the rust. These highly conducting networks are responsible, through their cond., for the corrosion-accelerating power of rust. The depolarizing action is effective through this action of the graphite and carbide. The action of the C in the rust is similar to the action of graphite originally mixed with the Ni oxide used in the alk. or Leclanche cell. The elec. conds. of 2 types of Acheson graphite with 0.44 and 0.08% ash and porosities of 29.8 and 31.4%, resp., were det'd. at 20° as 866 and 1420 mhos per cm. H. A. Smith

Corrosion resisting materials in chemical industry. Evert Norlin, et al. *Medd. Sterings Ind* 18, 68-108 (1935).—An introductory address followed by discussions.

A. R. R.

Corrosion of metals and alloys in apiezon oil. Gunji Shinoda. *Proc. Phys. Math. Soc. Japan* 17, 267-8 (1935).—Low-C steel, Cu, Al, Sn, Pb, Duralumin, 12%-Sn bronze, 40%-Zn brass, Constantan, Ni and Sn-solder were exposed for 1 month to the oil at 170-5°, at a pressure of  $10^{-2}$ — $10^{-4}$  mm. Al was most satisfactory on account of the lack of effect on both metal and oil. Steel

is best for the outer case. Solder cannot be used near the heater. Gregg M. Evans

The corrosion of magnesium alloys. IV. The corrosion of ternary alloys of magnesium. Susumu Morioka. *Kinsoku-no-Kenkyu* 12, 322-50 (1935). cf. C. A. 28, 5796f.—Ternary alloys of Mg contg. Bi and Ca or Sb. 14 series of ternary alloys of Mg contg. 1-8% of 2 of the alloying elements, Zn, Cd, Sn, Pb, Al and Sb, except for Sb (0.3-3.0%), and those contg. Si and Mn or Co were subjected to the corrosion tests for 0.1 N NaCl soln. The ternary alloys of the systems, Mg-Zn-Sn, Mg-Zn-Cd, Mg-Sn-Cd, Mg-Sn-Pb, Mg-Zn-Sb and Mg-Sn-Sb were very resistant to corrosion, especially in the range of

Zn	1-3%	Sn	2-8%
Zn	3-6%	Cd	1-6% (Zn + Cd less than 10%)
Sn	more than 3%	Cd	1-3%
Sn	more than 4%	Pb	1-6%
Zn	1-6%	Sb	0.3-0.6%
Sn	2-3%	Sb	less than 0.8%

Tomo-o Sato

Removal of corrosion layer. B. B. Getzow. *Zavodskaya Lab* 4, 353 (1935).—Steel or cast Fe is treated first with hot 50% HCl and then with a freshly prep'd. mixt. of 1 g  $As_2O_3$  in 100 cc of conc'd HCl with 2.5 g.  $SnCl_2$  and 5 cc  $CH_3O$  soln. in 250 cc  $H_2O$ . Chas. Blanc

Welding. E. Jimeno, A. Modolell and F. R. Morral. *Anales soc. espan. fis. quim.* 33, 696-708 (1935).—A general survey of welding gives the outstanding phys. and chem. problems, and a macro- and micrographic study of 3 samples of welds shows the structure and hardness in the various regions. X-ray photographs show the defects. E. M. Symmes

The corrosive action on metals of solutions of ammonium nitrate in liquid ammonia. I. M. Libinson, I. I. Kuskikh and A. S. Morozova. *J. Chem. Ind. (Moscow)* 12, 590-7 (1935).—Al is not corroded by conc'd.  $NH_4$  solns. of  $NH_4NO_3$ , Pb and Sn are slightly corroded, Fe is strongly corroded and Zn is completely dissolved. The presence of  $H_2O$  in the soln. increases the corrosion of Fe, but when  $H_2O$  is entirely removed, the vapor pressure of the  $NH_4$  becomes too high for practical use. The mechanism of the corrosion is discussed. Oxidation of Fe surfaces by such compds as  $K_2CrO_4$  or coating them with Bakelite lacquers protects the surface. Prevention of disconn. of the  $NH_4NO_3$  by addn. of  $CaCl_2$  or better,  $NH_4CO_3NH_3$ , also prevents corrosion. The partial pressure of  $NH_3$  over solns. contg. 70-80%  $NH_4NO_3$ , at 20-6° and 739-47 mm. varies from 220 to 450 mm. H. M. Leicester

Al cans for fruits (Morris, Bryan) 12. Corrosion of steel and tinplate by exsts. of fruits (Morris) 12. Machines for moistening pulverulent material [roasted Zn blend] (Brit. pat. 431,352) 1. Protective metals against corrosion (Swiss pat. 175,038) 4. Coating surfaces (Brit. pat. 431,562) 20

Concentration of ores by flotation. Royal S. Handy. U. S. 2,019,306, Oct. 29. In the treatment of a finely divided ore such as complex Pb-Zn ore, natural colloidal matter is substantially completely removed by adding a deflocculating agent such as Na silicate substantially completely to release the ore from any depressing action of such matter in the subsequent pulp, water is added to the residual cryst. matter to form a deflocculated pulp free from any depressant, and this pulp is agitated and there is added to it an artificial colloidal material such as com. glue in such selective quantity as definitely to depress all the different kinds of ore particles contained in the pulp and to permit the depressed particles to be subsequently acted upon by respective conditioning agents for selective flotation.

Metal separation by selective chloridization. Jesse O. Betterton (to American Smelting and Refining Co.) U. S. 2,019,470, Oct. 29. In the sepn. of metals, such as Pb and Zn or Bi, by selective chloridization, corrosion of the app. employed in introducing Cl into the molten

metal bath is decreased by maintaining the temp. of the app. below the surface of the molten bath at a lower temp. than the surrounding molten metal by circulating a cooling medium through the app. out of contact with the Cl and molten bath. App. is described.

**Treatment of oxidized ores** Meyer Mineral Separation Co. Ger. 618,462, Sept. 9, 1935 (Cl. 40a. 2.60). See U. S. 1,898,018 (C. A. 27, 2659).

**Treatment of sulfide ores** Carl Goez Ger. 618,185, Sept. 3, 1935 (Cl. 40a. 11.50) Commuted sulfide ores are heated gradually with a solid, liquid or gaseous hydrocarbon material to a temp. somewhat above 1000° but substantially below the normal reduction temp. of the sulfide, e. g., below 1400° when CuS ore is treated. The treatment is effected in the absence of air but in the presence or absence of steam. The gases evolved in the process are returned to the furnace, with or without combustion gases or water gas. The treatment liberates the metals from the sulfides. Cf. C. A. 29, 439.

**Treatment of sulfide ores containing iron** Charles R. Kuzell Ger. 618,186, Sept. 3, 1935 (Cl. 40a. 12.70) See Brit. 394,690 (C. A. 28, 4514).

**Chlorination of sulfide ores** Raymond F. Bacon Ger. 618,183, Sept. 5, 1935 (Cl. 40a. 2.60) See Brit. 374,071 (C. A. 27, 4040).

**Multiple-chamber plant for reducing ores, etc., with gases** Maria Grütter (nee Pfeiffer) Ger. 614,953, June 22, 1935 (Cl. 40a. 11.50) Addn. to 602,278 (C. A. 29, 5799).

**Concentrating gold** Stanley Tucker and Minerals Separation Ltd. Brit. 432,046, July 10, 1935. Au is concd. by forming the ore in finely ground condition into a pulp with H<sub>2</sub>O in the presence of a mercurial reagent, e. g., H<sub>2</sub> or a Hg salt, e. g., Hg(CN)<sub>2</sub>, and free alkali cyanide, such that the surfaces of the Au particles present are modified by the reagent and then sepp. the Au from the ore by flm. or froth flotation. Hg, if used, is not used in amt. sufficient to effect amalgamation. A sulfidizing agent, e. g., an alkali polysulfide, alkali sulfide or S or an S deriv. of H<sub>2</sub>CO<sub>3</sub>, e. g., an alkali xanthate, a fat acid, e. g., oleic acid, and (or) a Na salt, e. g., NaCl, may be used in conjunction with the mercurial reagent.

**Amalgamator for working fine gold** Ulysses H. Nottingham. U. S. reissue 19,739, Oct. 22. A reissue of original pat. No. 1,961,428 (C. A. 28, 4692).

**Deoxidizing and purifying copper** Société d'électrochimie, d'électrometallurgie et des acides électriques d'Ugine. Brit. 430,520, June 20, 1935, and 430,569, June 26, 1935, divided on 430,520. These correspond to Fr. 770,362 (C. A. 29, 4439), 430,520 being restricted to the addn. of metals and 430,569 to the addn. of metal-  
loids. Cf. C. A. 28, 5031.

**Refining copper** Norddeutsche Affinerie. Brit. 430,563, June 20, 1935. Cement Cu, in non-briquetted form, is melted down and refined in an internally heated rotary-drum furnace. The Cu is so charged as to lie on the bottom and both side walls of the furnace and preferably contains 6-16% H<sub>2</sub>O as a result of drying and re moistening, or is preheated to between red heat and the m. p. in a drum or circular furnace provided with rabbling arms.

**Condensing magnesium vapor** Frank R. Kemmer (to American Magnesium Metals Corp.). U. S. 2,018,205, Oct. 22. The vapor is caused to impinge against a cooled surface provided with a film of liquid inert to Mg, such as kerosene, to condense the Mg to a solid state, and the surface is continuously moved to present new cooled areas and fresh liquid film to the vapors and maintain continuous condensation, while excluding air. App. is described, and U. S. 2,018,266 also relates to such app.

**Condensing magnesium vapors** Oesterreichisch Amerikanische Magnesit A.-G. Brit. 431,537, July 10, 1935. In the condensation of Mg vapors from gases consisting of or contg. CO, the vapors are quickly cooled by direct contact with a liquid indifferent to Mg and which protects the Mg from chem. reaction down to a temp. at which a reaction between the Mg and CO no longer takes place,

e. g., room temp. Cooling liquids, e. g., hydrocarbon oils, that wet the Mg powder, are used. App. is described.  
**Zinc** La Nouvelle Montagne Soc. anon. Ger. 618,463, Sept. 10, 1935 (Cl. 40a. 34.30). Compact Zn is recovered from powd. mixts. of Zn and ZnO by fusing the mixts. with NH<sub>4</sub>Cl in an amt. 1.5-2.5 times that required to convert the ZnO into ZnCl<sub>2</sub>. Layers of fused Zn and ZnCl<sub>2</sub> are obtained, and the excess of NH<sub>4</sub>Cl volatilizes. The treatment is particularly applicable to the powd. deposits which accumulate in the tubes of Zn distn. retorts.

**Zinc** Octave Dony-Hénault. Fr. 784,407, July 22, 1935. The sepp. of liquid Zn in the distn. of this metal is made complete by interposing a diaphragm of artificial carbon, of fine and regular texture, capable of allowing gases to pass but retaining the heavier metal vapor.

**Deoxidizing zinc** Metamine G. m. b. H. Ger. 618,464, Sept. 9, 1935 (Cl. 40a. 34.80) Fused com. Zn is agitated, at a temp. within its solidification range, with a metal having a higher m. p. than Zn and a greater affinity for O, e. g., Cu, Al or Mg. A chloride, sulfide or like compd. of such a metal may also be used.

**Compound metal articles** I. G. Farbenind. A.-G. (Franz Dufschmid, inventor) Ger. 618,063, Sept. 2, 1935 (Cl. 49. 5) Layers of different metal powders, e. g., layers of powd. Fe and Ni or layers of powd. Fe-Ni mixts. of different compn., are superimposed and sintered together, and the product is subjected to mech. treatment, e. g., compression or rolling, in a reducing atm. Numerous details are given, and various modifications are indicated. Thus, a layer of a metal oxide may be included, or one or more of the layers may be sintered separately before the other layers are applied.

**Furnace for reducing ores** Akt.-Ges. Brown, Boveri & Cie. Ger. 618,184, Sept. 5, 1935 (Cl. 40a. 11.50). Details are given of an internally heated furnace to which reducing gases are supplied and in which a no. of superimposed traveling bands support the ore.

**Metallurgical furnaces** Maurice H. V. Delot and Roland A. Pigal. Brit. 432,497, July 29, 1935. A smelting furnace, preferably of the rotary type, is heated by a burner that produces an annular flame with an inert cone, the burner being so directed that the flame is flattened by contact with the bath of molten metal.

**Metallurgical furnaces** Eduard Wecke Ger. 618,216, Sept. 4, 1935 (Cl. 18a. 11.01) Means is described for preventing the destruction of furnace arches and suspended roofs through thermal expansion.

**Rotary-tube furnace suitable for roasting pyrites, etc.** Carl P. Debuch (to American Lurgi Corp.). U. S. 2,019,397, Oct. 29. Structural, mech. and operative details.

**Rotary hearth furnace for production of lead from sulfide ores** Felix Freiherr von Schluppenbach (to American Lurgi Corp.). U. S. 2,018,242, Oct. 22. Various structural, mech. and operative details.

**Metal-heating furnaces** John Fallon. Brit. 430,833, June 26, 1935. In a continuous furnace for heating sheets or plates, having a roller-way conveyor comprising a series of rollers geared together for rotation at uniform speed, electrically operated means is provided for imparting, at intervals, a high-ejecting speed to a variable no. of rollers at the discharge end.

**Heat-treating furnaces having roller-way conveyors comprising rollers carrying work-supporting disks** Driver-Harris Co. Brit. 431,459, July 8, 1935. Divided on 429,781 (C. A. 29, 7130).

**Fusion furnace for metals** Paul L. C. Blanchard, Maurice H. V. Delot and Roland A. Pigal. Fr. 784,660, July 22, 1935. The flame is annular and composed of a layer of carbon particles in combustion directed on to the metal.

**Blast-furnace tuyères** Vereinigte Stahlwerke A.-G. (Theodor Richter and Karl Heintmann, inventors). Ger. 615,790, July 12, 1935 (Cl. 18a. 5). Addn. to 610,239 (C. A. 29, 3643).

Means for securing metal bands to blast-furnace shafts

Demag A-G Ger 618,274, Sept. 4, 1935 (Cl. 18a 4.01)

Cupola furnace fired with gaseous, liquid or pulverulent fuel Peter Marx Ger. 611,120, Sept. 20, 1935 (Cl. 31a 1.10)

Discharge device for furnaces for annealing sheet metal. Benno Schilde Maschinenbau-A-G Ger. 618,452, Sept. 9, 1935 (Cl. 18c 9.50)

Apparatus for charging furnaces with materials such as sheet steel in annealing boxes. Clarence L. Taylor (to Aetna Standard Engineering Co.). U. S. 2,019,102, Oct. 29. Mech. features.

Furnace for coating wire with molten metal as in coating wire with spelter Wm E. Weaver (to General Elec. Co.). U. S. 2,019,283, Oct. 29. Various structural and operative details.

Thermal treatment of metals and alloys. Gesenkschmiede Handelsges. m. b. H. Ger. 618,279, Sept. 6, 1935 (Cl. 18c 8.80). In treating metals or alloys in gas-fired or elec.-heated hearth furnaces having a heart of reducing material, scaling is prevented by repeatedly turning the metal or alloy so that the whole surface is brought into contact with the hearth.

Apparatus for bending and hardening leaf springs. Vulkanhammer - Maschinenfabrik Hans Geismann. Ger. 618,434, Sept. 9, 1935 (Cl. 18c 2.21).

Hardening and quenching continuous treads of car wheels Alfred Walcker (to American Steel Foundries). U. S. 2,019,281, Oct. 29. A plurality of streams of quenching fluid are directed against the tread so that each of the streams is disposed at an acute angle to an axial plane through the tread at the point of impact of the stream.

Pickling metals Henkel & Cie G m b H. Fr. 784,182, July 22, 1935. Comps. which contain at least one thiosulfuric acid ester group in the mol are added to pickling baths to reduce corrosion. Such comds include Na benzylthiosulfate,  $\text{OHCH}_2\text{CH}_2\text{SSO}_3\text{Na}$ ,  $\text{NaSO}_3\text{SCH}_2\text{COOR}$ ,  $\text{CH}_3\text{CH}(\text{SSO}_3\text{Na})\text{COOR}$  (where R is  $\text{C}_6\text{H}_5$ ,  $\text{C}_6\text{H}_5$ ,  $\text{C}_6\text{H}_5$ ,  $\text{C}_6\text{H}_5$ ,  $\text{C}_6\text{H}_5$ ),  $\text{EtSSO}_3\text{Na}$  and the Na salt of aryl and octyl-thiosulfuric acid ester.

Apparatus for pickling coiled strips of wire, etc. Stephen L. Williams (to Extruded Metal Products Co.). U. S. 2,019,296, Oct. 29. Structural and mech. features.

Hot-plating containers such as milk cans with material such as molten Zn Isaac J. B. Demers (to Superior Metal Products Co.). U. S. 2,018,951, Oct. 29. Various operative details are described.

Extensometer suitable for testing wire, sheet metal, etc. Constantine D. Timpolt (to Baldwin-Southwark Corp.). U. S. 2,017,876, Oct. 22. Various structural, mech. and operative details.

Apparatus for testing the tensile and compression strengths of materials such as metals Albert R. Arthus U. S. 2,018,593, Oct. 22. Structural, mech and operative details.

Apparatus for degreasing wire with volatile solvents. Clarence F. Dazley (to James H. Bell). U. S. 2,018,648, Oct. 22. Various structural, mech. and operative details.

Magnets Siemens & Halske A-G Brit 431,210, July 3, 1935. A permanent magnet carried on the end of a train to operate a relay over which the magnet travels is in the form of an ellipsoid of rotation and is made of a non-tensile steel alloy having a coercive force of over 300 Oersted, e. g., an alloy contg. Al 2-25, Ni 6-30% and the remainder Fe with or without additions of Co, Cr, W and (or) Mn. In an example, the alloy contains Al 10, Ni 30, Fe 50% and the remainder is Cr and Co.

"Industrial iron" production from iron sponge. Hermann Esser. U. S. 2,018,200, Oct. 22. Iron sponge contg. C and Fe or Fe in its original pulverulent form is pressed into a rough form, and the pressed material is further treated in an extrusion press which causes a considerable flow of the particles and friction between them which causes them to become further heated. The product may be formed into tubes or bars. App. is described.

Carbonizing iron and steel Ewald Hauns. Ger

618,029, Aug. 30, 1935 (Cl. 18c. 3.15). The material is treated in known manner with a gaseous or vaporized carbonizing agent until the rate of carbonization begins to slacken. The supply of carbonizing agent is then cut off, and the material is kept at a temp. near the lower critical temp. in an inert atm. or *in vacuo* until a sufficient proportion of C has diffused inward from the surface of the material. The carbonizing treatment is then resumed.

Steel Abdul M. Malik and Sardar M. K. Alvi Brit 431,260, July 1, 1935. Steel is manufd. from pig Fe contg. Si up to 1 and P of about 0.1-1.75% in basic converters without increasing the P content by using a preheated blast in the converter. If the Si content is higher than 1%, cold blast is used until the Si is reduced to 1% or less and then the preheated blast is used. The temp. of the blast varies from 200° to 900° according to the P and Si present. The tuyères are made of fireclay contg. 40%  $\text{Al}_2\text{O}_3$ .

Alloy-steel machine elements such as axles and toothed gears. Augustus B. Kinzel (to Electro Metallurgical Co.). U. S. 2,018,297, Oct. 22. Machine elements designed to withstand shock at temps. between about -20° and -80° and having at such temps an Izod impact strength of at least 10 foot lbs. are formed of an alloy steel contg. Fe together with Cr 0.25-3.25, Cu 1.25-0.25 and C not over 0.55%.

Overhead electric-transmission line. Frank P. Fowle and Frederick M. Crapo (to Indiana Steel & Wire Co.). U. S. 2,019,447, Oct. 29. A C-steel conductor is used contg. at least 0.25% of C, not more than 0.3% Mn and not more than 0.15% Si.

Overhead electric-transmission line. Frederick M. Crapo (to Indiana Steel & Wire Co.). U. S. 2,019,445, Oct. 29. A conductor is formed of C steel which contains less than 0.5% C, less than 0.3% Mn when the C exceeds 0.25% and which has been quenched from above its Ac<sub>1</sub> crit. temp. by a liquid medium which has a temp. below the m p of Pb.

Difficultly fusible alloy. Jean Mayor. Fr. 784,223, July 22, 1935. The constituents of the alloy, e. g., W carbide 85, Co 12 and Ni or Mo 3%, are heated in a closed mold under vacuum until the constituents are fused, whereby a pressure is exerted on the alloy until the moment of cooling. The constituents may be introduced in the form of powder or pieces.

Hard alloys Allgemeine Elektrizitäts-Ges. Ger. 618,125, Sept. 2, 1935 (Cl. 49b 17). See Brit. 385,629 (C. A. 27, 2769).

Aluminum alloys Vereinigte Leichtmetall Werke G m b H. Brit 431,842, July 16, 1935. Age-hardenable Al alloys contain Cu 3-5, Mg 1.3-1.4, Si 0.4-0.7 and Mn 1.25-1.45%.

Alloys of barium with aluminum. John E. McCarty and Donald W. Randolph (to General Motors Corp.). U. S. 2,018,143, Oct. 22. Ba provided with an oxidation-preventing coating such as pyroxylin lacquer is immersed in molten Al maintained at a temp. of about 700-750°, resultant slag is removed, another portion of Ba is similarly added, slag is again removed, and these operations are continued until the desired proportion of Ba has been added, the melt benz then held for a few min to insure thorough alloying and then poured into a suitable mold.

Aluminum magnesium alloys. I. G. Farbenindustrie A-G Brit. 431,054, June 24, 1935. Cold-worked Al-Mg alloys that contain at least 3% Mg, are free from any intentional addition of Si and may contain up to 1.5% of additional elements, e. g., Mn, Zn, Ca, are improved by subjection to annealing under such conditions of temp and duration as to preclude recrystallization. Cl. C. A. 28, 6957.

Separating the components of copper and silver alloys I. G. Farbenindustrie A-G Brit. 432,144, July 22, 1935. Alloys contg. 30-70% Cu with the remainder consisting substantially of Ag are treated with Cl<sub>2</sub> whereby, after the reaction has started, a fluid chloride melt is formed without further application of extraneous heat. The melt is poured into a solvent for CuCl<sub>2</sub> when dil. HCl and (or) a NaCl soln. is used, AgCl is recovered as

such, but by using an excess of  $\text{NH}_4\text{OH}$ , the Ag is liberated as metal and  $\text{CuCl}_2$  is formed and retained in soln. To control the temp. of reaction, neutral salts, e. g.,  $\text{NaCl}$ ,  $\text{CaCl}_2$ , that increase the soly. of  $\text{CuCl}_2$  in the solvent used may be incorporated in the reaction mixt.

**Hardening copper-zinc alloys.** Imperial Chemical Industries Ltd. Ger. 618,465, Sept. 9, 1935 (Cl. 404 1,600). See Brit. 390,177 (C. A. 28, 1653).

**Iron alloys.** Fried. Krupp A.-G. Brit. 431,248, July 3, 1935. Addn. to 423,900 (C. A. 29, 6064). Fe alloys capable of pptn. hardening and suitable for cutting and hot-working tools contain W 10-35 and (or) Mo 2-12, Co 25-50, C 0.1-1, Cr 3-15 and V, Ti and (or) Ta up to 10%. If there is only 1 of the last 3 metals, there is at least 6 times as much V as C or at least 4 times as much Ti as C or at least 6.5 times as much Ta as C. If there is more than 1, there is a corresponding total amt. in relation to the C. The alloys are heat-treated by cooling quickly from about 1000° and then reheating Cf. C. A. 29, 6763.

**Iron alloys, deoxidizing steel.** Société d'électrochimie, d'électrometallurgie et des aciéries électriques d'Ugine. Brit. 431,329, June 27, 1935. Fe alloys are made by violently intermixing a molten fluid slag other than a deoxidizing slag that has been exhausted by a violent intermixing with oxidized steel, the slag contg. 1 or more oxide compds. of alloying substances, with a molten ferrous metallic bath that reduces the said oxide compds., the violence of the intermixing being such that the slag is finely divided and dispersed in the metal, the slag being sepd. from the molten alloy after the turbulence has subsided. For producing Fe-Ni, Fe-Mo and Fe-Cu alloys, the ferrous bath itself acts as the reducing agent, the slag contg., e. g., Ni silicate,  $\text{CaMoO}_4$  or  $\text{CuO}$ , resp. Reducing agents, e. g., C,  $\text{CaC}_2$ , Al, Si, Mn, Fe-Ti, Ca, Ti, Al silicide, Ca silicide, may be added to the bath, e. g., for the reduction of Cr ores contained in the slag. For the production of alloys contg. reducing agents, e. g., Si, Al, Mn or Ti, excess of such agent is added to the ferrous bath, the excess being calcd. to correspond to the amt. of oxides to be reduced in the slag. The ferrous bath may be of deoxidized Fe, steel that has been deoxidized by violent intermixing with very fluid non-reducing slags capable of extg. the oxides dissolved therein, or Fe alloys, the alloying element or elements being that or those to be reduced from the slag or some other element or elements. Basic, neutral or acid slags may be used, e. g., slags contg.  $\text{SiO}_2$ ,  $\text{CaO}$ ,  $\text{Al}_2\text{O}_3$ , titanate acid,  $\text{MgO}$ , alkali, Fe oxide or Mn oxide. For example, a slag may contain  $\text{SiO}_2$ ,  $\text{CaO}$ ,  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$ ; in this case, by the use of Si-Al and Si-Ca as reducing agents, it is sufficient to add  $\text{MgO}$  to the slag to compensate for losses due to manipulation.

**Iron alloy.** Alloy Research Corp. Fr. 784,293, July 22, 1935. Fe alloys of high heat resistance contain Ni, Cr, Mo and Cu. Mn, Si and Al may also be present. Examples are: (1) Cr 18, Ni 8, Mo 2.8, Cu 2.8, C 0.1%, (2) Cr 18, Ni 8, Mo 3, Cu 2.5, W 2.5, C 0.1%, (3) Cr 18, Ni 8, Mo 1.5, Cu 1.5, Mn 3, C 0.1%, the rest being Fe in each case.

**Rustless iron alloys.** Alloy Research Corp. Brit. 431,469, July 1, 1935. An alloy of inherently fine grain structure comprises Cr 10-30, C 0.00-0.09, N 0.00-0.2 and Ni, Mo, W, V and (or) Cu 0-3%, the remainder being substantially Fe. Small amts. of Mn and Si may be present. Cf. C. A. 28, 5802.

**Magnetic iron alloys.** Swift, Levick & Sons Ltd., Geo. D. L. Horsburgh and Frederick W. Tetley. Brit. 431,660, July 12, 1935. Permanent magnets are made from Fe alloys contg. Al, Ni, Cu and Co, with or without other elements, e. g., Cr, the casting being rapidly cooled from 850-1350° and thereafter hardened at 550-725°. The alloys may contain Al 5-20, Ni 10-25, Cu 1-15 and Co 1-20%. The casting may be subjected to the above heat treatment directly or may be cooled before being heated to 850-1350°.

**Magnetic iron alloys.** Heihachi Kamura. Brit. 431,975, July 18, 1935. Addn. to 420,543 (C. A. 29, 3293).

Modifications of the alloys of 420,543 contain also 0.01-2.5% Si, with or without up to 1% Ti.

**Magnetic nickel-iron alloys; annealing.** Telegraph Construction & Maintenance Co. Ltd., Walter F. Randall and Frank H. Smith. Brit. 430,957, June 21, 1935. In the manuf. of magnetic Ni-Fe alloys contg. 30-60% Ni to which has been added at least 1 element of low solid soly., e. g., Ag, Be, Sb, Mg, Ca, Cu, the alloys are annealed at 900° or over to form a homogeneous solid soln., cooled sufficiently rapidly to obtain a state of supersat. of the added element or elements in solid soln., subjected to an amt. of cold working sufficient to reduce them to their final size, e. g., a reduction of thickness of 10-90%, and then soaked at over 250° but below 500° sufficiently long to produce a pptn. of some of the added element or elements. One or more freely sol. resistance elements, e. g., Cr, Mo, Mn, W, Al, Si, V, Co, may be added to the alloys. Cu may act as a pptg. element or as a freely sol. element especially when 1 of the other freely sol. elements is present. In an example, 98.5 parts of an alloy contg. Ni 40, Fe 60, Mn 0.5 and Cr 1.5 parts are melted with Ag 1.5 parts and the alloy cast, the casting is hot forged, annealed at 1100° for 30 min., cold rolled without intermediate annealings down to 0.01-in. strips and sheared; the cold-rolled strip is then soaked for 2 hrs. at 300°.

**Foils, wire, ribbons and tape of magnetic alloys.** Heraeus Vacuumschmelze A.-G. Brit. 427,295, Apr. 17, 1935. Magnetic alloys for use in elec. app. involving the use of small magnetic fields have an initial permeability of over 800 and are produced as tape, etc., by drawing or rolling to a thickness not exceeding 0.03 mm, the constancy of permeability under working conditions being thereby increased. The tape, etc., may be subjected to cold deformation after a preceding annealing. Alloys contg. Fe 50, 19, Ni 50, 76 and Cu 0, 5 are mentioned. In 431,511, July 5, 1935, divided on 427,205, such wires, etc., are incompletely annealed after a preceding cold working to improve the constancy of permeability under working conditions. The annealing consists of a heat treatment at such temp. and for such time as will modify the magnetic properties appreciably but not to the extent obtained by heating above the crit. temp.

**Removing alloying metals from lead and its alloys.** National Lead Co. Brit. 431,353, July 5, 1935. See U. S. 1,976,333 (C. A. 28, 7241).

**High-strength nickel-cobalt-iron-titanium alloys.** Geo. P. Hallwell (to Westinghouse Elec. & Mfg. Co.). U. S. 2,018,520, Oct. 22. Alloys which are suitable for engine valves or valve seats are formed of Ni 20-70, Co 60-10, Fe 5-50 and Ti 0.5-10% and are quenched from a temp. of 900° or higher and aged at 500-800° for at least a half hr.

**Sintered alloys of tungsten carbide and carbonitride with metals such as tungsten and cobalt.** Richard R. Walter. U. S. 2,018,752, Oct. 29. Alloys suitable for machining tools, etc., are formed from W carbide or carbonitride 60-80 and a metal such as W and Co.

**Journal brass for rail-vehicle axles.** Walter Peyerlinghaus. U. S. 2,018,417, Oct. 22. An alloy is used formed from Mn 1.5-7.0% together with a lead-bronze contg. Cu and 10-30% Pb.

**Coated centrifugally cast metal pipe.** Frederick C. Langenberg and Horace S. Hunt (to U. S. Pipe and Foundry Co.). U. S. 2,018,025, Oct. 22. An externally cooled centrifugal metallic pipe mold is coated with a finely divided dry coating material such as ferro-Si of which not more than 5% is retained on a 150-mesh screen and not less than 10% on a 200-mesh screen (various operative details being described).

**Coppering iron.** Ernst Stettler Sohn. Swiss 176,045, June 1, 1935 (Cl. 445). The Fe surface is cleaned, polished and treated with a coppering soln. consisting of an aq. soln. of  $\text{CuSO}_4$  and  $\text{HCl}$ . The coppered surface is then washed with an alk. liquid.

**Aluminum bronze powder.** Dale M. Boothman (to Aluminum Co. of America). U. S. 2,017,850, Oct. 22. Thin flat Al such as foil or sheet material is coated with material such as tallow, lard, oil or stearic acid to prevent

welding of overlapping pieces under rolling pressure, then thinned by rolling to break up the metal and form flakes. U. S. 2,017,551 relates to rolling Al coated with lubricant to form flakes in the form of a cake which is then broken up. App is described.

Treating aluminum surfaces to coat them. Herbert B. Wetherbee (10% to Benton H. Grant and 70% to Richard F. Grant) U. S. 2,018,694, Oct. 29. The surface is subjected to the action of an aq. alk. soln. of borax and boric acid (suitably at the b. p.).

Coating aluminum and its alloys. Peintal soc. anon. Swiss 175,365-7, May 16, 1935 (Cl. 444). Al and its alloys are given a firm corrosion-proof covering by first forming an oxide layer and then treating with a soln. of chlorinated rubber products (175,365). A corrosion-proof oxide coating is given to Al or its alloys by treatment with an aq. alk. bath at temps above 100° (175,366). Another method is to form an oxide coating on the Al or alloy and then apply a coating of aq. asphalt emulsion (175,367).

Coloring aluminum. Peintal soc. anon. Swiss 176,414, July 1, 1935 (Cl. 91). Colored patterns are formed on Al or Al-alloy surfaces by first producing an oxide layer on the surface, coloring this and then etching away this layer in the required pattern by an alk. reagent.

Coloring aluminum articles. James F. Leahy (to Atlas Tack Corp.) U. S. 2,019,229, Oct. 29. Articles such as buttons or eyelets are treated with a soln. of an oxidizing agent such as a hot dil. soln. of  $\text{Na}_2\text{Cr}_2\text{O}_7$  and  $\text{Na}_2\text{CO}_3$  to form an oxide coating and are then treated with an alk. soln. such as one of  $\text{Na}_2\text{CO}_3$  and having a pH of about 10-11, and are finally treated with a lake-forming dye to form an insol. color lake on the articles. Cl. C. A. 29, 43234.

Coloring aluminum objects. Peintal soc. anon. Swiss 176,415, July 1, 1935 (Cl. 91). Al objects are colored by oxidizing the surface, forming a pattern on this layer with a water repellent such as bees wax, and finally treating the surface with an aq. soln. of dye or coloring matter. The water repellent may be itself colored.

Coloring aluminum and its alloys. Siemens & Halske A.-G. (Alexander Jenny and Nikolai Budiloff, inventors) Ger. 615,692, July 9, 1935 (Cl. 484 3). Addn. to 607,012 (C. A. 29, 17664). A light-sensitive coating, produced on superficially oxidized Al or Al alloy as described in Ger. 607,012, is uniformly exposed to light and then converted into a colored coating by any of the usual photographic developing, fixing and toning processes.

Coloring the surface of aluminum and its alloys. Martin Tosterud (to Aluminum Colors, Inc.) U. S. 2,018,388, Oct. 22. An adsorbent oxide coating is formed on the surface and in this coating there are adsorbed coloring substances such as various metal compounds.

Producing colored protective coatings on zinc. Heinz Tschauer. Ger. 615,665, July 9, 1935, 615,815, July 12, 1935 and 615,911, July 15, 1935 (Cl. 484 3). Addn. to 613,024 (C. A. 29, 54117). In the process of Ger. 613,024, (1) the baths used are improved by adding 1-2% of  $\text{NH}_4\text{Cl}$  (615,665), (2) the heavy metal sulfate is replaced by other salts of heavy metals, and the  $\text{NH}_4\text{Cl}$  molybdate by other molybdates (615,815) and (3) the baths are regenerated after use by addn. of up to 3% of K bitartrate (615,911).

Rustproofing compositions. Eberhard Wurbs. Brit.

431,641, July 10, 1935. See Ger. 667,349 (C. A. 28, 46137).

Inhibiting the interior corrosion of sealed metallic containers such as cans containing foods. James E. McConkie (to American Can Co.). U. S. 2,018,682, Oct. 29. The inside of the container is provided with a coating such as a varnish or lacquer and which contains a Ca sulfite or  $\text{Na}_2\text{SO}_3$ .

Uniting corrosion-resisting plates or sheets such as those of stainless steel with an intervening plate or sheet of mild steel. Wallace C. Johnson and Alfred E. Maskrey (to Pylkrome Corp.). U. S. 2,018,725, Oct. 29. Various operative details are described.

Electric welding apparatus. Conrad L. Neffler (to Western Elec. Co.). U. S. 2,018,379, Oct. 22. App. is described in which the force compressing together materials to be welded is gradually reduced to zero as their fusion together is effected. U. S. 2,018,280 relates to welding Al or Cu rods or the like under specified pressure and c-d. conditions, and describes app.

Apparatus for welding silicon-steel sheets for magnetic uses. Ralph E. Asbury (to American Rolling Mill Co.). U. S. 2,017,933, Oct. 22. Mech. features.

Welding or fusing bar or rod. Walter S. Bingham. U. S. 2,018,116, Oct. 22. A rod is formed of an alloy of Fe contg. C, Mn, Si, P and S in such proportions that a weld deposit made with the rod on a larger mass of parent metal and air-cooled from the molten state has the cementite structure of hypoeutectic white cast iron and a brittleness less than that of such white cast iron.

Welding fluxes. The British Thomson-Houston Co. Ltd. Brit. 430,722, June 24, 1935. A flux for use in elec. arc welding and preferably used as a coating on an electrode comprises substantially equal parts of feldspar (I),  $\text{TiO}_2$  and liquid Na silicate. A preferred flux consists of 100,  $\text{TiO}_2$  100, low-C Fe-Mn 8, liquid Na silicate 100 and  $\text{H}_2\text{O}$  47 parts. A tape of cellulosic material, e. g., cotton, may be impregnated with the flux and folded longitudinally about an electrode core with its edges abutting 1 another and parallel to the axis of the core. A substantial layer of flux may be provided between the core and the impregnated tape.

Welding fluxes. The British Thomson-Houston Co. Ltd. Brit. 431,324, July 4, 1935. A flux for use in arc welding and preferably used as a coating on an electrode comprises approx. feldspar 1,  $\text{TiO}_2$  1, liquid Na silicate 2 parts and C equal to 0.5-2% of the total of the preceding ingredients. The preferred flux consists of feldspar 22.52,  $\text{TiO}_2$  22.52, liquid Na silicate 45.05, petroleum coke 0.9 and  $\text{H}_2\text{O}$  9.1 parts.

Solders; fluxes. Harold Turner and Johnson, Matthey and Co. Ltd. Brit. 431,584, July 11, 1935. A solder, particularly for use with rustless or stainless Fe or steel alloys consists of Ag 4-80, Mn 1-40 and Cu 10-70%. Up to about 1% of a deoxidizing agent, e. g., Al, Si, P, may be present. The solder may be used with a flux of a fluoride and  $\text{H}_3\text{BO}_3$  or a borate, e. g., powd. borax glass 60 and KF 40%.

Soldering strip for securing joints of metal such as electrical splices. Harold W. Burk (50% each to Ralph M. Burk and Ulla E. Guthrie) U. S. 2,018,117, Oct. 22. A sheet of metal such as Al is provided on one side with a layer of soldering material and on the opposite side with a layer of readily inflammable material and is sufficiently pliable to be bent to conform to the joint to which it is to be applied.

## 10-ORGANIC CHEMISTRY

CHAS. A. RUTILLER AND CLARENCE J. WEST

Aurochromes and resonance. C. R. Bury. *J. Am. Chem. Soc.* 57, 2115-17 (1935).—A general discussion of the role of resonance in the color of dyes. C. J. W.

Reaction between sulfur dioxide and olefins. III. Higher olefins and some limitations of the reaction. L. L. Ryden and C. S. Marvel. *J. Am. Chem. Soc.* 57,

2311-14 (1935); cf. C. A. 29, 72764.—Ten vols. of liquid olefin and 10 vols. liquid  $\text{SO}_2$  with 1 vol. 3%  $\text{H}_2\text{O}_2$  and 1 vol.  $(\text{CH}_3\text{O})_2$ , allowed to stand about 12 hrs., give the following polyulfones: *methylpropene*,  $(\text{C}_3\text{H}_5\text{SO}_2)_n$ , m. (Blac. Maquenne, decomposition)  $340^\circ$ , 80% yield; *2-pentene*, m.  $290-300^\circ$ , 75%; *1-pentene*, m.  $340^\circ$ , 80-90%, 1-

none, m. above 300°, 75–80%; 3-cyclohexylpropene, m. 330°, 75%; 3-methylcyclohexene, m. 270°, 5–10%.  $\text{Cl}_2\text{CCl}$  reacts with the sulfones to form trichloroacetates, indicating that they contain HO groups at the ends of the chains; from the percentage of Cl the mol. wt. have been calculated, but the expl. error is so large that it can be concluded only that the polysulfones have mol. wts. in the region of 100,000 to 200,000. The mol. wt. of propylene polysulfonamide is about 60,000. 2-Pentene polysulfone, on heating with NaOH, yields 2-pentene, dipentyl sulfone and  $\text{Na}_2\text{SO}_4$ . Methylpropene polysulfone gives large amts of methylpropene and  $\text{Na}_2\text{SO}_4$ . No other hydrolysis products were obtained. 1-Pentene polysulfone does not regenerate the olefin when heated with alkali. Undecylenyl alc yields a polysulfone, darkens at 330°, trichloroacetate. 1-Heptyne polysulfone m. 160–9°, 50–60% yield. No evidence of addn. of  $\text{SO}_2$  to typical olefins contg. Cl, CN, phenolic HO, aldehyde,  $\text{CO}_2\text{Et}$ ,  $\text{CO}_2\text{H}$  or salts of  $\text{CO}_2\text{H}$  could be obtained.

C. J. West

**Chemistry of the acetylenes** II. Pharmacological properties of the acetylenic linkage. G. Bryant Bachman. *J. Am. Chem. Soc.* 57, 2167–8 (1935). cf. C. A. 29, 724<sup>1</sup>.—*O*-Odenyl chloride,  $\text{C}_8\text{H}_{13}\text{ClO}$ , b<sub>m</sub> 109–11°, n<sub>D</sub> 1.4649, d<sub>4</sub> 0.9841. The following esters were prepd by adding a slight excess of acid chloride in an equal vol of  $\text{C}_6\text{H}_6$  to the NH<sub>2</sub> alc. in  $\text{CaH}_2$  and refluxing 1 hr.:  $\text{C}_8\text{H}_{13}\text{CO}_2(\text{CH}_2)_2\text{NEt}$  (I), b<sub>m</sub> 162–4°, n<sub>D</sub> 1.4390, d<sub>4</sub> 0.883,  $\text{C}_8\text{H}_{13}\text{CO}_2(\text{CH}_2)_3\text{NEt}$  (II), b<sub>m</sub> 178 81°, n<sub>D</sub> 1.4405, d<sub>4</sub> 0.888;  $\text{C}_8\text{H}_{13}\text{CO}_2(\text{CH}_2)_4\text{NEt}$  (III), b<sub>m</sub> 212–15°, n<sub>D</sub> 1.4451, d<sub>4</sub> 0.870;  $\text{C}_8\text{H}_{13}\text{CH}(\text{CHCO}_2\text{CH}_2)_2\text{NEt}$  (IV), b<sub>m</sub> 171–3°, n<sub>D</sub> 1.4560, d<sub>4</sub> 0.901;  $\text{C}_8\text{H}_{13}\text{CH}(\text{CHCO}_2\text{CH}_2)_3\text{NEt}$  (V), b<sub>m</sub> 182–6°, n<sub>D</sub> 1.4565, d<sub>4</sub> 0.901;  $\text{C}_8\text{H}_{13}\text{CH}(\text{CHCO}_2\text{CH}_2)_4\text{NEt}$  (VI), b<sub>m</sub> 217–20°, n<sub>D</sub> 1.4574, d<sub>4</sub> 0.888;  $\text{C}_8\text{H}_{13}\text{C}(\text{CO}_2\text{CH}_2)_2\text{NEt}$  (VII), b<sub>m</sub> 153–2°, n<sub>D</sub> 1.4723, d<sub>4</sub> 0.916;  $\text{C}_8\text{H}_{13}\text{C}(\text{CO}_2\text{CH}_2)_3\text{NEt}$  (VIII), b<sub>m</sub> 164–6°, n<sub>D</sub> 1.4739, d<sub>4</sub> 0.921;  $\text{C}_8\text{H}_{13}\text{C}(\text{CO}_2\text{CH}_2)_4\text{NEt}$  (IX), b<sub>m</sub> 183–5°, n<sub>D</sub> 1.4750, d<sub>4</sub> 0.900. The times in min. for anesthesia, sensory and motor, are: I, 4, 23 5; II, 4, 12 5; III, 3 5, 11 5; IV, 0 5, 13 5; V, 5, 27 5; VI, 4, 10; VII, 8, 24; VIII, 0, 22; IX, 4 5, 10; novocaine, 8, 15. Within the limits of the compds. described here it appears that the anesthetic power is directly proportional to the mol. wt. of the NH<sub>2</sub> alc. part of the mol. and inversely proportional to the degree of unsat. of the acid.

C. J. West

**Acetylene polymers and their derivatives.** XXXIII. The preparation and polymerization of oxyprenes. Harry R. Dykstra. *J. Am. Chem. Soc.* 57, 2255–9 (1935). cf. C. A. 29, 7938<sup>1</sup>.— $\beta$ -EIO(CH<sub>2</sub>)<sub>2</sub>Ac, HC(OEt), and HCl in abs. EtON, allowed to stand 4 days, give 62% of 1,3-trickorybutane (I), b<sub>m</sub> 73°, d<sub>4</sub> 0.8940, n<sub>D</sub> 1.4148, tri-Me analog, b<sub>m</sub> 61–3°, d<sub>4</sub> 0.8998, n<sub>D</sub> 1.4112. tri-Bu analog, b<sub>m</sub> 120°, d<sub>4</sub> 0.8745, n<sub>D</sub> 1.4310. I with  $\text{KH}_2\text{SO}_4$  at 150–40° gives 59% of ethoxybutadiene and 13% of diethoxybutene.  $\text{CH}_3\text{CH}(\text{OEt})\text{Me}$ , Me, b<sub>m</sub> 98–100°, d<sub>4</sub> 0.8756, n<sub>D</sub> 1.4038; Et, b<sub>m</sub> 69°, d<sub>4</sub> 0.8470, n<sub>D</sub> 1.4062; Bu, b<sub>m</sub> 79–80°, d<sub>4</sub> 0.8497, n<sub>D</sub> 1.4238; Et(Et), b<sub>m</sub> 81°, d<sub>4</sub> 0.8645, n<sub>D</sub> 1.5128.  $\text{ROCH}_2\text{CH}(\text{OEt})\text{Me}$ : Et, b<sub>m</sub> 101°, d<sub>4</sub> 0.8711, n<sub>D</sub> 1.4242; Bu, b<sub>m</sub> 91–3°, d<sub>4</sub> 0.8633, n<sub>D</sub> 1.4342.  $\text{CH}_3\text{CH}(\text{OR})\text{CH}_2\text{Me}$ , Me, b<sub>m</sub> 75°, d<sub>4</sub> 0.8281, n<sub>D</sub> 1.4422; Et, b<sub>m</sub> 96 5°, d<sub>4</sub> 0.8153, n<sub>D</sub> 1.4401; Bu, b<sub>m</sub> 65°, d<sub>4</sub> 0.8233, n<sub>D</sub> 1.4445. 2-Ethoxy-1,3-butadiene and BuOH at 93° give EtOH, BuOH and 3,3-dibutoxy-1-butene. 2-Ethoxy-1,3-butadiene (II) and EtOH at 100° for 24 hrs. give EtOCH<sub>2</sub>CH<sub>2</sub>C(OEt)Me and polymerized II. II and (CH<sub>3</sub>OH)<sub>2</sub> at 110° for 24 hrs. give EtOH and a compd., either  $\text{CH}_3\text{CH}(\text{OCH}_2\text{CH}_2\text{OH})\text{CH}_2\text{Me}$  or  $\text{CH}_3\text{CH}(\text{Me})\text{OCH}_2\text{CH}_2\text{Me}$ , b<sub>m</sub> 58°, which yields Me vinyl

ketone on hydrolysis: d<sub>4</sub> 0.9547, n<sub>D</sub> 1.4213. PhOH and PhSH add readily to II at room temp. With naphthoquinone II gives 2-ethoxyanthraquinone, m. 136°. Hydrolysis of II with dil. HCl yields Me vinyl ketone. Catalytic reduction of II is accompanied by the absorption of 0.17 mole of H<sub>2</sub>, giving 2-ethoxy-1- or 2-butene or a mixt. of the 2. Heating 40 g. of II at 130° for 13 days gives 20 g. of the dimer, b<sub>m</sub> 105–25°, and 17 g. of a solid polymer, with I as catalyst, the product is 65% of dimer

and trimer and the remainder a soft resin. 2-Butoxy-1,3-butadiene in ultraviolet light for 1 week gives 135% of a polymer, somewhat sticky and elastic. Heating I with acid catalysts gives 10–30% of 3,3-diethoxy-1-butene, hydrolyzed to Me vinyl ketone and reduced to EtC(OEt)Me (40% yield), b<sub>m</sub> 68–9°, d<sub>4</sub> 0.8423, n<sub>D</sub> 1.4012. EtO(CH<sub>2</sub>)<sub>2</sub>C(OEt)Me and Al<sub>2</sub>O<sub>3</sub> at 110–65° give 95% of 1,3-dithoxy-2-butene (I), b<sub>m</sub> 57°, d<sub>4</sub> 0.9024, n<sub>D</sub> 1.4089. As substitutes for rubber these polyoxyprenes are inferior to polychloroprene.

C. J. West

**Syntheses from natural gas hydrocarbons** II. Identity of monochlorides from simpler paraffins. H. B. Hass, E. T. McElree and Paul Weber. *Ind. Eng. Chem.* 27, 1190–5 (1935). cf. C. A. 25, 2689<sup>1</sup>.—Propane, n- and isobutane, and n- and isopentane were chlorinated photochemically at temps from –60° to 300° and in the dark up to 600° in app. providing for preheating the reactants and close temp. control. The products were sep'd by distn. and rigorously identified. For comparison, b. ps. of all the possible monochlorides and corresponding alcs. in the literature were critically exam'd and supplemented by data on compds. prep'd. by the authors by methods other than chlorination (table). A common source of error in b. ps. of the chlorides was found to be the presence of alcs. and olefin polymers, which were, however, easily removed by  $\text{H}_2\text{SO}_4$ . "Carbon skeleton rearrangements do not occur during either photochemical or thermal chlorinations if pyrolysis temperatures are avoided, every possible monochloride derivable without such rearrangement is always formed. This generalization extends to all polychlorides so far as this study has gone."

Arnold M. Collins

**Aliphatic substitution and the Walden inversion** I. E. D. Hughes, F. Juliusburger, S. Masterman, B. Topley and J. Weiss. *J. Chem. Soc.* 1935, 1525–9.—The velocity in MeCO of the substitution of the radioactive isotope of I for I in *sec*-C<sub>4</sub>H<sub>9</sub>I has been measured and also the velocity of racemization of the d-isoxide by NaI. The abs. rates of the 2 processes are the same within the expl. error of the measurements of the radioactivity (10%). The results confirm in the most direct way possible the causal connection between aliphatic substitution and optical inversion, in reactions of this type. For the racemization  $k \times 10^4$  was found to be: 10° 0.152, 19 85° 0.454, 29 85° 1.20, 39 8° 3.21.

C. J. West

**Chloroform-d (deuteriochloroform).** F. W. Breuer. *J. Am. Chem. Soc.* 57, 2236–7 (1935).—Chloroform-d, CDCl<sub>3</sub>, from Cl<sub>2</sub>CH(OD)<sub>2</sub> and DONa, b<sub>m</sub> 0.5° higher than CHCl<sub>3</sub>, the m. p. is –64.68° to –64.15°, the f. p. is –64.12° (values for CHCl<sub>3</sub>, –65.77° to 65.33°, –63.49°); d<sub>4</sub> 1.5004; n<sub>D</sub> 1.4450 (CHCl<sub>3</sub>, same). Assuming identical mol. vols. for CHCl<sub>3</sub> and CDCl<sub>3</sub>, the calcd. d. is 1.5013.

C. J. West

**Reaction of ethylene oxide with acetylenic Grignard reagents.** James P. Daney, Richard R. Vogt and J. A. Newland. *J. Am. Chem. Soc.* 57, 2327 (1935). cf. C. A. 29, 725<sup>1</sup>.—Two moles (193 g.) heptyne (I), transformed into the Grignard reagent and treated with (CH<sub>3</sub>)<sub>2</sub>O, gives 63% I, 93% HOCH<sub>2</sub>CH<sub>2</sub> and 60 g. 3-nonyl-1-ol; the Grignard reagent from I and PhC≡CH with (CH<sub>3</sub>)<sub>2</sub>O gives analogous products in substantially the same percentage yields.

C. J. West

**Preparation of dialkylmagnesium compounds from Grignard reagents** Arthur C. Cope. *J. Am. Chem. Soc.* 57, 2238–40 (1935).—The extent of the disproportionation  $2\text{RMgX} \rightleftharpoons \text{R}_2\text{Mg} + \text{MgX}_2$  depends upon the solvent employed and increases with increasing temps., as det'd. by the diastane pptn. method. The ppt. formed during the prepn. of the Grignard reagent from MeCl contains MgCl<sub>2</sub> etherate from the disproportionation of MeMgCl, so that the reagent contains 20% of Me<sub>2</sub>Mg in addn. to that formed by the disproportionation of the MeMgCl remaining in soln.

C. J. West

**The oxidation of dl-α-hydroxystearic acid and its significance as regards the structure of cerebromide acid** A reply to the paper of Levene and Yang. E. Klenk and P. Ditt. *J. Biol. Chem.* 111, 749–50 (1935). cf. C. A. 27, 2167; 28, 1016<sup>1</sup>, 6150<sup>1</sup>.—A reply to the paper



of Klenk and Ditt. P. A. Levene and P. S. Yang. *Ibid.* 751—Polemical. A. P. Lothrop

Preparation of ethylenimine from ethanalamine Henry Wenker. *J. Am. Chem. Soc.* 57, 2328 (1935).  $\text{HOC}_2\text{H}_4\text{NH}_2$  (810 g.) and 1020 g. of 96%  $\text{H}_2\text{SO}_4$ , heated rapidly over a free flame until charring begins at 250°, give 71%  $\text{CH}_2=\text{CH}_2$ ,  $\text{NH}_3$ ,  $\text{O}$ ,  $\text{SO}_2$ ,  $\text{O}$ , distn with 40%

NaOH gives 26.5% of  $\text{C}_2\text{H}_5\text{NH}_2$ , b. 55–56°. C. J. West

Preparation and properties of acetyl-d- and acetyl-l-methylcholine chloride Randolph T. Major and Howard T. Bonnett. *J. Am. Chem. Soc.* 57, 2125–6 (1935). *d*-Dimethylaminoisopropanol (I), b.m. 124–5–6°,  $[\alpha]_D^{25}$  –17.1°, is obtained by resolution of the *dl*-isomer with bromocamphorsulfonic acid in EtOAc; *d*-tartaric acid gives the *l*-isomer, b. 125°,  $[\alpha]_D^{25}$  –14.8°. I and MeI give *d*- $\beta$ -methylcholine iodide, m. 170–7°,  $[\alpha]_D^{25}$  +24.7°; the *l*-isomer, m. 176–7–5°,  $[\alpha]_D^{25}$  –24.7°. *d*- $\beta$ -methylcholine, m. 165–7°,  $[\alpha]_D^{25}$  +38.8°, *l*-isomer, m. 165–7°,  $[\alpha]_D^{25}$  –38.2°, *d*-acetyl- $\beta$ -methylcholine chloride (II), m. 200–1°,  $[\alpha]_D^{25}$  +41.9°, *l*-isomer (III), m. 201–2°,  $[\alpha]_D^{25}$  –41.3°. The muscarine-like action on the blood pressure of III is  $1/10$  that of the *dl*-isomer, that of II is somewhat greater than that of the *dl*-isomer. The action of III on the isolated intestine is considerably less and that of II is comparable to that of the *dl* form. C. J. West

The crystalline and gelatinous salts of phosphoglyceric acid C. Neuberg and W. Schuchardt. *Biochem. Z.* 280, 293–6 (1935), cf. *C. A.* 28, 7249°—Phosphoglyceric acid gives beautifully crystalline salts with Ba or Cd, but with Co, Zn, Mn, Fe<sup>++</sup> or UO, it yields glass-clear gelatinous compds. The present study deals with the salt formation of the synthetic racemic acids. The formation of these gelatinous compds. is less marked with racemic 3-phosphoglyceric acid than with the optically active acid. With racemic 2-glyceric acid phosphoric acid this tendency is so slight that a gelatinous salt was prepd only with UO, whereas with Cd an amorphous product was formed. The benzidine salt is as beautifully crystal as that with the isomeric 3-phosphoglyceric acid.

#### S. Morgulis

Action of hydrogen peroxide upon simple carbon compounds III. Glycolic acid H. Shupley Fry and Kenneth L. Miltstead. *J. Am. Chem. Soc.* 57, 2269–72 (1935), cf. *C. A.* 25, 2968°—Since the yields of  $\text{HCO}_2\text{H}$  to those of  $\text{H}_2$  were invariably greater than 2, which ratio, according to the equation  $2\text{HOCH}_2\text{CO}_2\text{H} + 3\text{H}_2\text{O}_2 \rightarrow \text{H}_2 + 2\text{HCO}_2\text{H} + 2\text{CO}_2 + 4\text{H}_2\text{O}$  (I), is 2, it follows that the reactions represented by this equation and  $\text{HOCH}_2\text{CO}_2\text{H} + 2\text{H}_2\text{O}_2 \rightarrow \text{HCO}_2\text{H} + \text{CO}_2 + 3\text{H}_2\text{O}$  (II) were concurrent. Secondary reactions are  $\text{HCO}_2\text{H} + \text{H}_2\text{O}_2 \rightarrow \text{CO}_2 + 2\text{H}_2\text{O}$  (III),  $2\text{H}_2\text{O}_2 \rightarrow 2\text{H}_2\text{O} + \text{O}_2$ . The extents of the occurrence of the reactions represented by I, II and III with increasing concns of  $\text{H}_2\text{O}_2$  are. II shows a tendency to decrease, I, small variations, and III a tendency to increase. The sums of the percentage quantities of the  $\text{H}_2\text{O}_2$  active in the several reactions (equations I, II and III) and the inactive or directly decompd.  $\text{H}_2\text{O}_2$  are, within the limits of exptl. error, practically 100% of the initial quantities of the  $\text{H}_2\text{O}_2$  employed in each exptl. run. The  $\text{HOCH}_2\text{CO}_2\text{H}$  can also be accounted for in practically 100%. The results serve to check quantitatively the accuracy not only of the exptl. method employed, but also of the equations derived for the 3 reactions assumed to occur and the extents of their occurrence, resp., when  $\text{H}_2\text{O}_2$  reacts with  $\text{HOCH}_2\text{CO}_2\text{H}$ . C. J. West

Configurational relationships of acids of the isopropyl and isobutyl series to those of the normal series. P. A. Levene and R. E. Marker. *J. Biol. Chem.* 111, 299–312 (1935)—Substances of the type  $\text{Me}(\text{CH}_2)_m\text{RCH}(\text{CH}_3)_X$ , where  $m$  was varied, holding  $n$  const., and  $n$  varied, were studied to det. the effect on rotation of substitution of a normal radical by a branched chain. Substitution of an iso-Pr for a normal Pr group produces a significant effect on the rotation both with respect to sign and numerical value. As compared with members of the normal Bu series, those of the iso-Bu series show differences in the direction of rotation only in the case

of 2-isobutyl-1-bromopentane, the rotatory dispersion curves of which are being studied. The rotations of the members of the iso-Bu series compared with those of the iso-Pr series show similarity in the direction of rotation in 3 cases and it is suggested that a difference in the partial rotation of 1 of the 2 significant constituents may exist. Removal of the iso-Pr group by a  $\text{CH}_3$  from the asym. center brings about a difference in the effect of this group on the rotations similar to that observed with the hydrocarbons and secondary carbinols contg. this group. Removal of an iso-Pr group by 2  $\text{CH}_3$  groups from the asym. center causes little change in the rotations from those of the corresponding normal Pr substances. By methods previously described (*C. A.* 29, 5827°) the following were prepd.: *l*- $\text{Me}_2\text{CHCHMeCO}_2\text{H}$  (I), b. 90°,  $[\text{M}]_D^{25}$  –21.9°; *d*- $\text{Me}_2\text{CHCHMeCO}_2\text{Et}$ , b. 140°,  $[\text{M}]_D^{25}$  0.866,  $[\text{M}]_D^{25}$  38.5°; *d*- $\text{Me}_2\text{CHCHMeCH}_2\text{OH}$ , b. 142°,  $[\text{M}]_D^{25}$  0.823,  $[\text{M}]_D^{25}$  6.3° (*Ac. deriv.*), b. 147°,  $[\text{M}]_D^{25}$  3.8°; *d*- $\text{Me}_2\text{CHCHMeCH}_2\text{Br}$ , b. 140°,  $[\text{M}]_D^{25}$  1.190,  $[\text{M}]_D^{25}$  30.6°; *d*- $\text{Me}_2\text{CHCHMeCH}_2\text{CO}_2\text{H}$ , b. 109°,  $[\text{M}]_D^{25}$  0.921,  $[\text{M}]_D^{25}$  16.6°; *d*- $\text{Me}_2\text{CHCHMeCH}_2\text{CO}_2\text{Et}$ , b. 165°,  $[\text{M}]_D^{25}$  0.869,  $[\text{M}]_D^{25}$  20.5°; *d*- $\text{Me}_2\text{CHCHMeCH}_2\text{CH}_2\text{OH}$ , b. 164°,  $[\text{M}]_D^{25}$  31.2°; *d*- $\text{Me}_2\text{CHCHMeCH}_2\text{CH}_2\text{Br}$ , b. 162°,  $[\text{M}]_D^{25}$  1.109,  $[\text{M}]_D^{25}$  56.6°; *Me\_2CHCHMeEt, b. 90°,  $[\text{M}]_D^{25}$  0.695,  $[\text{M}]_D^{25}$  28.3°; *Me\_2CHCHMe(CH\_2)\_2\text{CO}\_2\text{H}, b. 92°,  $[\text{M}]_D^{25}$  0.912,  $[\text{M}]_D^{25}$  1.4278,  $[\text{M}]_D^{25}$  –13.7°; *Me\_2CHCHMeCHMeCO}\_2\text{H}, b. 117°,  $[\text{M}]_D^{25}$  0.899,  $[\text{M}]_D^{25}$  –18.1°; *Me\_2CHCHMeCHMeCO}\_2\text{Et}, b. 164°,  $[\text{M}]_D^{25}$  0.855,  $[\text{M}]_D^{25}$  21.5°; *Me\_2CHCHMeCHMeCH}\_2\text{OH}, b. 157°,  $[\text{M}]_D^{25}$  0.816,  $[\text{M}]_D^{25}$  –10.8°; *Me\_2CHCHMeCHMeCH}\_2\text{Br}, b. 68°,  $[\text{M}]_D^{25}$  –7.5°; *Me\_2CHCHMeCHMeCH}\_2\text{CO}\_2\text{H} (II), b. 91°,  $[\text{M}]_D^{25}$  0.9012,  $[\text{M}]_D^{25}$  –1.1°. All values for  $d$  are for  $d^{25}$  unless otherwise stated; for  $[\text{M}]_D^{25}$  at 25° and are calc'd max. values, except those for I and II which are not max.*******

R. C. Elderfield

Synthesis of homocysteine. Wilbur I. Patterson and Vincent du Vigneaud. *J. Biol. Chem.* 111, 393–8 (1935)—Na (23 g.) was added to MeOH (150 cc) and followed by  $\text{PhCH}_2\text{SH}$  (124 g.). When the Na was dissolved,  $\text{Cl}(\text{CH}_2)_2\text{Cl}$  (I) (380 cc) was added, the mixt. shaken and the violent reaction checked by cooling in ice. After pouring into  $\text{H}_2\text{O}$ , sepd. the product and removing I at 100 mm., distn gave 71% of benzyl  $\beta$ -chloroethyl sulfide (II), b. 114–16°. To Na (46 g.) in abs EtOH (1 l.) was added ( $\text{EtO}_2\text{C})_2\text{CH}_2$  (480 g.), followed by II (373 g.). After refluxing 6 hrs., the mixt. was slowly added to KOH (560 g.) in 2 l. of 50% EtOH and boiled 1 hr. Removal of the alc., acidification with HCl, extrn. with Et<sub>2</sub>O, and crystn. from  $\text{C}_2\text{H}_5$  gave 75% of benzylthiolethylmalonic acid, m. 114–17°. To this (127 g.) in Et<sub>2</sub>O (1 l.), Br (26 g.) was slowly added at 0° during 15 min. The mixt. was poured into concd.  $\text{NH}_4\text{OH}$  (1300 cc). After 48 hrs., when all was in soln., excess  $\text{NH}_3$  was removed *in vacuo*, concd. HCl (200 cc) was added and the soln. heated 1 hr. Neutralization to litmus with  $\text{NH}_4\text{OH}$  gave *S*-benzylhomocysteine (III) (65 g.). To III (40 g.) in liquid  $\text{NH}_3$  (300 cc) was added, with stirring, Na in slight excess. After evapn. of the  $\text{NH}_3$ , the residue was treated with  $\text{H}_2\text{O}$ , and, after acidification to phenolphthalein with HCl, removal of unchanged III, aeration in the presence of  $\text{FeCl}_3$ , 75% of homocysteine (IV) was obtained. III with Na and BuOH gave 78% of IV.

R. C. Elderfield

Synthesis of crystalline cystinylglycine and benzylcystinylglycine and their isolation from glutathione Hubert S. Loring and Vincent du Vigneaud. *J. Biol. Chem.* 111, 385–92 (1935)—Dicarboxybenzoxycystinyl chloride coupled with glycine (3 mols.) in *N*-NaOH gave dicarboxybenzoxycystinylglycine (I), m. 182–3° (EtOAc), *Et ester*, m. 166°. To I (6.2 g.) in liquid  $\text{NH}_3$  (II) (50 cc) was added Na (1.7 g.) After appearance of the blue color, II was evapd.,  $\text{H}_2\text{O}$  added and the soln. neutralized with III and aerated. Acidification with HCl, concn. and addn. of EtOH pptd. cystinylglycine (III) (2.7 g.), m. 210° (Dennis base),  $[\alpha]_D^{25}$  –67.5° ( $\text{H}_2\text{O}$ ), –80.0° (*N*-HCl). Amorphous cystinylglycine (IV) was obtained by pptn. of the residue from evapn. of II obtained above with  $\text{H}_2\text{SO}_4$ . To III (0.76 g.) in II (15 cc) was added Na (0.3 g.) followed by  $\text{PhCH}_2\text{Cl}$  (0.6 g.). Evapn.

addn. of  $H_2O$ , extrn with  $Et_2O$  and acidification of the aq. layer gave 5-benzylcysteinylglycine, m. 160–7° ( $H_2O$ ). This was also prep. directly from I in 84% yield. IV (310 mg) was isolated from glutathione (1 g) after hydrolysis by the method of Kendall, Mason and McKenzie (C. A. 24, 57(10)). This was converted into the above cryst. compds. by the above methods. R. C. Elderfield

Catalytic hydrogenation of aldonic acid  $\delta$ - and  $\gamma$ -lactones and of the aldoses. J. W. B. Glaffield and G. Weber Schimpff. *J. Am. Chem. Soc.* 57, 2204–8 (1935).—Details are given of 44 expts on the catalytic reduction of various  $\gamma$ - and  $\delta$ -lactones and aldoses, yields of sugar, acid and alc. being reported. An increase in concn. of the lactone decreased the rate of reduction; a concn. of 0.2 M was satisfactory. Large quantities of catalyst favored the production of alc. and small quantities necessitated an increase in the time of hydrogenation, which also tended to increase the yield of alc. The time of reduction was reduced from 20 hrs to 30 min by increasing the rate of shaking from 120 to 350 cycles per min. Under conditions that resulted in an 80% yield of  $d$ -glucose with no alc from the  $\delta$ -lactone, there was obtained from the  $\gamma$ -lactone 23.4% sugar and 24% alc. Both lactones of  $d$ -mannonic acid are reduced very readily, while those of  $d$ -rhammonic acid were reduced slowly and gave relatively high yields of alc., the  $\gamma$ -lactone gives 3–4 times as much alc. as sugar, whereas the  $\delta$ -lactone gave approx. equal amounts of the 2 products. The aldoses give 63–80% of alcs. The original should be consulted for the details of the expts. C. J. West

Preparation of high molecular weight acryloins. V. L. Hansley. *J. Am. Chem. Soc.* 57, 2303–5 (1935).—The acryloins were prep. by the action of Na upon Me esters in  $CH_3I_2$ ; capronoin, m. 9° (acryloin, m. 119–20°); capryloin, m. 39°; nonyloin, m. 45°; capriroin, m. 51–2° (acryloin, m. 79–80°); lauroin, m. 61–2° (acryloin, m. 61–3°); myristoin, m. 71–2° (acryloin, m. 44–6°); palmitoin, m. 77–8°; stearoin, m. 82–3°. Catalytic reduction of the acryloins gave ( $RCO_2H$ ), with R as follows:  $C_{11}H_{23}$ , m. 123–4°;  $C_{12}H_{25}$ , m. 124°;  $C_{13}H_{27}$ , m. 125–6°;  $C_{14}H_{29}$ , m. 129–50°. Little if any polymerization resulted during the isolation of acryloins from acids of 8 or more C atoms. Oxidation of lauroin with  $Wgs$  soln. in  $CHCl_3$  gives a  $\gamma$ - $\alpha$ ,  $\beta$ -diketone, yellow, m. 71–1°.

Reaction of periodic acid with  $\alpha$ -ketols,  $\alpha$ -diketones and  $\alpha$ -ketonealdehydes. Percival W. Clutterbuck and Fritz Reuter. *J. Chem. Soc.* 1935, 1467–9.—With  $HIO_4$ ,  $AcCHO$  gives 1 mol. each of  $HCO_2H$  and  $AcOH$ ;  $Ac$  gives approx. 2 mols.  $AcOH$ ;  $AcCH(OH)Me$  gives  $AcH$  and  $AcOH$ , and  $BzCH(OH)Ph$  yields  $BzH$  and  $BzOH$ ;  $p-MeC_6H_4COCH(OH)Ph$  yields  $BzH$  and  $p-MeC_6H_4CO_2H$ ;  $HOCH_2COCH_2OH$  gives  $HCHO$  and  $HOCH_2CO_2H$ ; hydrated 3,5-dihydroxy-2-carboxybenzoyl ketone gives  $AcOH$  and 3,5-dihydroxyphthalic acid; 3,5-dihydroxy-2-carboxyphenylacetylcarbinol gives  $AcOH$  and 6-aldehydo-2,4-dihydroxybenzoic acid; benzofuroin gives  $BzH$  and pyromucic acid. The results show that simple glycols give no acid,  $\alpha$ -ketols give 1 equiv. and  $\alpha$ -diketones and  $\alpha$ -ketonealdehydes give 2 equivs. of acid; provided, therefore, that the reaction is complete or almost so, as indicated by the  $O$  absorption, the acid figure gives an indication of the type of compd. oxidized. Benzil reacts much more slowly than the above compds., absorption being only 80% of the theory after 2 weeks;  $BzOH$  is formed. C. J. West

The specific effect of zinc on the dismutation of methylglyoxal. C. Neuberg and M. Kobel. *Biochem. Z.* 280, 297–306 (1935).— $ZnCO_3$  as well as  $ZnO$  exerts a favorable effect on the dismutation of methylglyoxal, and promotes specifically its transformation into lactic acid. Whereas with  $CaCO_3$  very little lactic acid is formed, even a small amt. of the  $Zn$  salt yields a large amt. of lactic acid (up to 78% of the theoretical). S. Morghulis

Derivatives of glucuronic acid. VI. Preparation of  $\alpha$ -chloro- and  $\alpha$ -bromotriacetylglucuronic acid methyl ester, and the synthesis of  $\beta$ -glucuronides. Walther F. Goebel and Frank H. Babers. *J. Biol. Chem.* 111, 347–53

(1935); cf. C. A. 29, 6880°.—Me glucuronate (II) is prep. in improved yield by refluxing glucurone (III) (33 g) in abs. MeOH (400 cc) for 72 hrs. After concn., soln. of the residue in abs. EtOH (200 cc.), filtration of unchanged II, and acetylation of the syrupy residue, Me  $\beta$ -triacetylglucuronate (III) (10 g.) and the  $\alpha$ -deriv. (IV) (22 g.) were obtained. To III (10 g.) in  $CHCl_3$  (80 cc) was added  $TiCl_4$  (5 g.). After heating for 3 hrs. at 40–5° the mixt. was poured into ice- $H_2O$ . Concn. of the  $CHCl_3$  soln gave Me  $\alpha$ -triacetylglucuronate (8 g.), m. 99–100° ( $Li_2O$ ),  $[\alpha]_D^{25} 168.7^\circ$  ( $CHCl_3$ ). This (6 g) shaken with  $Ag_2CO_3$  (1.2 mols.) in abs. MeOH (50 cc) for 24 hrs. gave the  $\beta$ -Me glycoside of Me triacetylglucuronate (V) (3.4 g.), m. 149–50° ( $EtOH$ ),  $[\alpha]_D^{25} -28.9^\circ$  ( $CHCl_3$ ). In contrast to the previously reported isomer (C. A. 29, 6880°) V does not reduce Fehling soln., neutralizes 4 equivs. of NaOH on sapon., and shows no mutarotation in dioxane contg. 0.005 N  $HCl$ . Hence it is assigned a true glycosidic rather than an orthoacetate structure and probably has a  $\beta$ -configuration. In order to secure a substance suitable for the synthesis of glucuronides, Me  $\alpha$ -triacetylglucuronate (VI), m. 104–5° ( $Et_2O$ ),  $[\alpha]_D^{25} 198.0^\circ$  ( $CHCl_3$ ), was prep. from IV and  $HBr-AcOH$  by the method of Fischer. To VI (10 g) and  $p-N_2C_6H_4CH_3CO_2H$  (5 g.) in dry  $Et_2O$  (100 cc) was added  $Ag_2O$  (1.2 mols.) and the mixt. shaken 10 min. The triacetyl- $p$ -nitrobenzylglycoside of I (4.5 g.) crystd. and was extrd from  $Ag$  salts with  $CHCl_3$ . This was deacetylated with  $Ba(OH)_2$  and gave the  $\beta$ - $p$ -nitrobenzyl glycoside of I, m. 167–8° ( $EtOH$ ),  $[\alpha]_D^{25} -13.2^\circ$  ( $H_2O$ ). R. C. E.

The Raman spectrum of the esters of some derivatives of  $\beta$ -ketobutyric acid II. Mario Milone. *Gazz. chim. ital.* 65, 339–49 (1935), cf. C. A. 29, 3310°.—Raman spectra measurements were utilized to study the tautomeric keto-enolic equil. of Et esters of  $\beta$ -ketobutyric acid derivs., i. e.  $AcRR'CO_2Et$  compds. It was proved that the enolic form is present in large proportion when R is H and R' is Cl or Br, that it is present only in small proportion when R is H and R' is allyl or Bz, and that it is absent when R and R' are alkyl groups. The results indicate that the Raman effect is an excellent method for studying tautomeric enolic equil., not only in a qual. but in a quant. way. The intensity of the  $\delta$  1630  $cm^{-1}$  line appears to be strictly proportional to the percentage of the enolic form, and the percentage calcd. thus agrees well with the data of other authors with other methods (cf. Baker, *Tautomersm.*, London 1934, 58, C. A. 28, 2012°). The app. and technic were those used in the previous expts. The compds. measured included  $AcCH_3CH_2CO_2Et$ ,  $AcCH_2CO_2Et$ ,  $AcCHClCO_2Et$ ,  $AcCHBrCO_2Et$ ,  $AcCH(Ph)CO_2Et$ ,  $AcCH(Bz)CO_2Et$ , and  $BzCH_2CO_2Et$ , for which the Raman measurements are tabulated in detail. C. C. Davis

Reaction of ketenes with Grignard reagents. Charles D. Hurd, Ralph N. Jones and F. H. Munck. *J. Am. Chem. Soc.* 57, 2033–6 (1935).—Et ethylketenecarboxylate (I) was prep. by direct dehydration of  $EtCH(CO_2Et)CO_2H$  (prep. from  $CH_3(CO_2Et)$  with  $Et_3SO$  and  $NaOEt$ ) with  $P_2O_5$ . I reacts with  $RMgX$  with addn. preferably at the ketene  $CO$ , giving  $\beta$ -keto esters. No allene type was produced when the addn. product of I and  $EtMgBr$  was pyrolyzed,  $EtBr$  and an unsat. compd. being formed. I (0.06 mol., 8.5 g.), treated with 0.06 mol.  $PhMgBr$ , gave about 6 g. Et  $\alpha$ -benzoylbenzoate (II) and about 2 g.  $PhCO_2H$ . When 0.077 mol. (11 g.) of I was dropped into 0.2 mol.  $PhMgBr$ , there resulted 0.3 g.  $Ph_2$ , some Et and 2.5 g.  $Ph$   $\alpha$ -ethyl- $\beta$ , $\beta$ -diphenyl- $\gamma$ -ketone, m. 120–7°; with  $O_2$  this yielded  $Ph_2CO$ ,  $BzOH$  and  $EtCO_2H$ . The dimer of I and  $PhMgBr$  gave I.  $EtMgBr$  and I or its dimer gave principally Et  $\alpha$ -propionylbutyrate. C. J. West

Action of diacromethane on imides. II. Letterio Irrera. *Gazz. chim. ital.* 65, 464–6 (1935); cf. Labruto, C. A. 27, 3920°.—Glutarimide (I) and  $CH_3N_2$  (II) in  $Et_2O$ , let stand 24 hrs., evapd. and fractionally distd., yield  $N$ -methylglutaramide, b. 128–30°, prep. otherwise by Luleš and Smetšková (*Trav. chim. Tchec.* 2, 69

(1933), cf. *C. A.* 27, 2629) Likewise diphenimide (III) and II in Et<sub>2</sub>O on standing ppt. *N*-methylidiphenimide, m 108°, while the mother liquor yields very small quantities of an unidentified compd., m. 183°, and an unidentified compd., m. 150°. An unidentified methylphenimide was obtained by Wagerhoff (*Ann.* 252, 22) from Na diphenimide and MeI. C. C. Davis

**Mechanism of oxidation processes.** XLII. The degradation of citric acid. Heinrich Wieland and Robert Sonderhoff *Ann.* 520, 160-6(1933); cf. *C. A.* 29, 4320<sup>7</sup>—It has been shown previously (*C. A.* 27, 4872) that citric acid (I) is decomposed by yeast into 2 AcOH, HCO<sub>2</sub>H and CO<sub>2</sub>. Longer action of the yeast produces H<sub>2</sub>, which probably results from the decomposition of the HCO<sub>2</sub>H. It is probable that CO(CH<sub>2</sub>CO<sub>2</sub>H)<sub>2</sub> is an intermediate product in the decomposition of I, its reaction with yeast and that of CH<sub>2</sub>(CO<sub>2</sub>H)<sub>2</sub> are described. The aerobic dehydration of I is discussed. C. J. West

**Hydrogenation of carbon dioxide and a correction of the reported synthesis of urethans.** Mark W. Farlow and Homer Adkins. *J. Am. Chem. Soc.* 57, 2222-3 (1935), cf. *C. A.* 29, 725<sup>7</sup>—In an attempt to prep. *N*-pentamethylenesalicylamide through the reaction of Me salicylate and piperidine at 250° under H<sub>2</sub>, Wojcik and A. (*C. A.* 29, 725<sup>7</sup>) obtained a product which they reported as Ph *N*-pentamethylenurethane; it is now shown that the product is a mixt. of PhOH and formylpiperidine. The hydrogenation of CO<sub>2</sub> to HCO<sub>2</sub>H in the presence of an amine takes place at 80° or less over Raney Ni. The rate and temp. necessary for hydrogenation apparently varies with the structure of the amine as well as with the catalyst. At 250° sheet brass is an active catalyst. If the hydrogenation is carried out much above 100° the formate is dehydrated, giving the amide (formylamine). 2,2,6,6-Tetramethyl-1-hydroxypiperidine in EtOH with Ni at 100° for 6 hrs. gives 67% of the formate of the amine (47% after 3 hrs.), piperidine at 150° gives in 9 hrs. 76% of *N*-formylpiperidine, AmNH<sub>2</sub> at 150° in 5 hrs. gives 45% *N*-formylamine, b<sub>p</sub> 124-5°. 1-Phenyl-2-aminopropanol at 80° in 1 hr. gives 53% of the formate of the amine. *O*-Aminopropiophenone at 80° in 1 hr. gives the formate of the amine (its purity did not justify calcn. of the yield), *N*-butyl-2-methylpiperidine at 100° in 6 hrs. gives the formate of the amine. Piperidine in PhOH at 250° in 5 hrs. with brass gives 50% of *N*-formylpiperidine. Ph(CH<sub>2</sub>)<sub>3</sub>NH<sub>2</sub> in PhOH at 250° in 4 hrs. with brass gives 23% *N*-formyl deriv. AmNH<sub>2</sub> (in PhOH) at 250° in 5 hrs. with brass gives 33% of the *N*-formyl deriv. *N*-Formyl-*N*-phenylethylamine, b<sub>p</sub> 180-1°. *N,N'*-Diamylurea, m 88°. *N*-Formyl-*N*-phenylethylamine formate, m 128-9° (decolorn). 2,2,6,6-Tetramethyl-4-hydroxypiperidine formate, m. 207°. 1-Phenyl-2-aminopropanol formate, m 160° (decolorn). The possible mechanism of the reaction is discussed. C. J. West

**Separation of guanidine and methylguanidine by means of  $\beta$ -naphthalenesulfonyl chloride.** W. C. Hess and M. X. Sullivan. *J. Am. Chem. Soc.* 57, 2331-2(1935)—*Bis*( $\beta$ -naphthalenesulfonyl)guanidine, m 204-6°, results in 93% yield from 1.5 g. guanidine (I) carbonate and 1 cc. 5 *N* NaOH in 10 cc. H<sub>2</sub>O and 4.5 g. *Cu*-H<sub>2</sub>SO<sub>4</sub> (II) in 10 cc. Et<sub>2</sub>O, it contains 2 mols. H<sub>2</sub>O, at 24° 100 cc. H<sub>2</sub>O dissolves 9 mg. Methylguanidine (III) does not react under these conditions, but with 110 mg. III in H<sub>2</sub>O and 220 g. II in Et<sub>2</sub>O with 2.5 cc. 5 *N* NaOH,  $\beta$ -naphthalenesulfonylmethylguanidine, m 101-2°, seps in 65% yield. Creatine, creatinine, glycoamine and glycoxyamine do not react. From a mixt. of 50 mg. each of I and III, 90% of the I and 45% of III may be sepd. C. J. West

**Rearrangement of allyl ethers in the purine series,** with some remarks on the hydrogenation of allyl ethers. Ernst Bergmann and Heuz Hemhold. *J. Chem. Soc.* 1935, 1235-7—2,6-Dichloro-7-methylpurine (I) and CH<sub>3</sub>CHClCH<sub>2</sub>OH with Na, heated 3 hrs. at 100°, give 2,6-diallyloxy-7-methylpurine (II), m 111-12°. Catalytic reduction of II gives heteroxanthine (III). Heating II at 170° for 2 hrs. gives 1,2-diallyl-7-methylxanthine, m 227-8°, which does not give III on reduction. I and 2,4-hexan-4-ol with Na, heated 4 hrs. at 165° with shaking,

give 1,2-bis( $\alpha$ -methyl-2'-pentenyl)-7-methylxanthine, yellow, b<sub>p</sub> 216-22°, rearrangement occurring during distn. in *vacuo*; O<sub>2</sub> gives EtCHO and EtCO<sub>2</sub>H. Catalytic reduction of Ph allyl ether gives 75% PhOPr and 25% free PhOH. 2,6-Dipropoxy-7-methylpurine m 92°; this is not changed on treatment with II and Pd. C. J. West

**Constitution of the purine nucleosides.** III. Potentiometric determination of the dissociation constants of methylated xanthines. Alexander G. Ogston. *J. Chem. Soc.* 1935, 1876-9; cf. *C. A.* 29, 741<sup>7</sup>.—Values of *p*<sub>K</sub> are reported as follows, the 2 values being in H<sub>2</sub>O and 90% EtOH at 18°: xanthine 7.7, 9.3; 1-Me 7.7, 9.2; 3,7-di-Me 9.9, 11.3; 3-Me 8.5, 8.6; 1,3-di-Me 8.6, 8.7; 7-Me 8.5, 8.2; 1,7-di-Me 8.5, 8.7; 9-Me 6.3, 6.8; 1,9-di-Me 6.3, 6.6; xanthosine 6.0, 6.6. The basic functions in all cases are very weak; in terms of potential, the buffering produced was of the same order of magnitude as the reproducibility of the liquid junction potential, so no accurate estimate of their consts. could be made. The values show that substitution of the 1-position produces no change in the dissociation const. in most cases; it is probable that where the dissociation consts. of different xanthines have the same values, the structures are similar, in those having "Zwitter" ion structures the distribution of the charge in the mol. will have the controlling effect of the dissociation const., since this is largely detd. by the elec. work of removal of the proton and an identity of the values of the consts. argues an identity of charge distribution. Xanthosine is probably substituted in the 9-position. Structures of several of the above compds. are discussed. C. J. West

**Chemistry of the tetrose sugars.** I. A crystalline triacetate of *d*-threose from the degradation of strontium xylonate with hydrogen peroxide. Nomenclature in the tetrose group. Robert C. Hockett. *J. Am. Chem. Soc.* 57, 2260-4(1935)—Electrolytic reduction of xylone in NaBr-SrCO<sub>3</sub> soln gives 95% of Sr *d*-xylonate, with 5 mols. H<sub>2</sub>O, [α]<sub>D</sub><sup>20</sup> 13.2° (0.4268 g. in 25 cc. H<sub>2</sub>O); oxidation with H<sub>2</sub>O<sub>2</sub> and acetylation gives 15% of *d*-threose triacetate, m 117-18° (cor.), [α]<sub>D</sub><sup>20</sup> 35.55° (0.891 g. in 25 cc. CHCl<sub>3</sub>); the mol. wt. indicates that it is a monomer. MeONa in dry MeOH-CHCl<sub>3</sub> gives *d*-threose, with an equl. rotation of -12.3° (1.1288 g. in 50 cc. H<sub>2</sub>O); the sugar was characterized as *d*-threosazone, m 164-5°; dibenzylidene-*d*-threitol, m 218-22°, [α]<sub>D</sub><sup>20</sup> -78.2° (0.1800 g. in 25 cc. CHCl<sub>3</sub>), and triacetic *d*-threosone, m 197-8° (cor.), [α]<sub>D</sub><sup>20</sup> -36.8° (0.4420 g. in 25 cc. H<sub>2</sub>O). Oxidation gives *L*-tartaric acid. Certain errors in nomenclature in the tetrose group are corrected. II. The degradation of *d*-xylone by the method of Wohl.

The rotation of *d*-threose. *Ibid.* 2265-8—Acetylation of the reaction product of *d*-xylone and NH<sub>4</sub>OH HCl with MeONa gives 48% of tetraacetyl-*d*-xylonic nitrate, m 81-2° (m. ps. cor.), [α]<sub>D</sub><sup>20</sup> 50.3° (0.7615 g. in 25 cc. CHCl<sub>3</sub>); concd. NH<sub>4</sub>OH gives 78% of *d*-threosylacetamide (I), m 165-7°, [α]<sub>D</sub><sup>20</sup> -10.83° (0.5979 g. in 25 cc. H<sub>2</sub>O); *tri*-*d*-acetic, m 179-89°, [α]<sub>D</sub><sup>20</sup> 74.2° (0.3434 g. in 25 cc. CHCl<sub>3</sub>). Quant. hydrolysis of I with 0.1 *N* H<sub>2</sub>SO<sub>4</sub> at 100° shows a rate corresponding to *k* (mins. and decimal logs) = 0.0063 = 0.0013 and permits detn. of the equl. sp. rotation of *d*-threose in dil. acid as -12.5°, during hydrolysis the rotation is found to drift toward a small *L*-rotation. The formation of the tri-Ac deriv. indicates an aldehydo structure for I. The prepn. of *d*-erythrosazone and benzylidene-*d*-threitol from I hydrolyzates shows the identity of the sugar formed with that obtained by deacetylation of *d*-threose triacetate.

C. J. West  
***d*-Xylomethylolose and derivatives.** P. A. Levene and Jack Compton. *J. Biol. Chem.* 111, 325-33(1935); cf. *C. A.* 29, 2923<sup>4</sup>—A general method for obtaining pentomethyloloses from pentoses, consisting in the catalytic reduction of the  $\omega$ -halogen aldose deriv., to the corresponding aldomethylolose, is given. 5-Iodomethylolose-*d*-xylolose (I) was prepd. by the method of Levene and Raymond from 5-tosylmethylolose-*d*-xylolose. I (20 g.) in abs. MeOH (150 cc.) was reduced with Raney's Ni catalyst (II) with addn. of 10% NaOH soln (40 cc.). After 1 hr.

the filtrate from II was sated with  $\text{CO}_2$ , concd. and the residue extrd. with  $\text{Et}_2\text{O}$ , giving a quant. yield of *acetone-d-xylose* (III), m. 69–70° (l.i.O.-petr. ether), b. 80–77°,  $[\alpha]_D^{20}$   $-21.0^\circ$  ( $\text{H}_2\text{O}$ ),  $-18.2^\circ$  ( $\text{CHCl}_3$ ). This with  $\text{Ac}_2\text{O}$  in  $\text{C}_6\text{H}_5\text{N}$  (IV) gave 3-acetylmonooacetone-d-xylose, b. 79–80°,  $[\alpha]_D^{20}$   $-1.440^\circ$ ,  $[\alpha]_D^{25}$   $-2.55^\circ$  ( $\text{CHCl}_3$ ). III with  $p\text{-MeC}_6\text{H}_4\text{SO}_3\text{Cl}$  in IV gave the 3-*tosyl* deriv., m. 41–2° ( $\text{C}_6\text{H}_5\text{N}$ ),  $[\alpha]_D^{20}$   $-18.02^\circ$  ( $\text{CHCl}_3$ ), which on hydrolysis with alc. KOH gave III, thus showing that no apparent Walden inversion occurs during the hydrolysis III on hydrolysis with 1%  $\text{H}_2\text{SO}_4$  at 100° for 1 hr gave sirupy *d-xylose* (V),  $[\alpha]_D^{20}$   $-2.16^\circ$  (abs. EtOH),  $[\alpha]_D^{25}$   $-13.28^\circ$  ( $\text{H}_2\text{O}$ ), *p*-bromophenyldiazotone, m. 69–70° (iso-PrOH),  $[\alpha]_D^{20}$   $-26.05^\circ$  (IV); *p*-bromophenylosazone, m. 175–6°,  $[\alpha]_D^{20}$   $-64.7^\circ$  (IV-EtOH), becoming const. after 18 hrs. at 20° 4; phenylosazone, m. 170–80°,  $[\alpha]_D^{20}$   $-66.6^\circ$  (IV-EtOH), becoming const. after 18 hrs. at 0° 5 V and  $\text{Ac}_2\text{O}$  in IV gave the *tri-Ac* deriv., b. 105–6°,  $n_D^{20}$  1.4422,  $[\alpha]_D^{20}$   $-60.87^\circ$  ( $\text{CHCl}_3$ ). R. C. Elderfield

Crystalline *d*-gulonemethose and derivatives P. A. Levene and Jack Compton *J Biol Chem* 111, 335–40 (1935).—By application of the Kiliani cyanohydrin reaction to pentomethyloses, hexomethyloses have been made available. To *d*-xylose (50 g.) in  $\text{H}_2\text{O}$  (110 cc.) was added 80%  $\text{HCN}$  (30 cc.) and  $\text{NH}_4\text{OH}$  (4 cc.) After standing 4 hrs. at 0° and 40 hrs. at room temp. the mixt. was poured into  $\text{N Ba}(\text{OH})_2$  (1 l.) and boiled 2 days. Removal of Ba with  $\text{H}_2\text{SO}_4$ , concd. and crystn. from abs. EtOH gave *d*-gulonemethylosalicylate (I) (22 g.), m. 150–1° (AcOH),  $[\alpha]_D^{20}$   $-63.0^\circ$  ( $\text{H}_2\text{O}$ ), changing after 14 days to  $-63.8^\circ$ . From the mother liquor of I sirupy *d*-gulonemethylosalicylate (25 g.) was obtained. That the new  $\alpha\text{-OH}$  group in I is to the right of the chain is shown by the rotations of Na *d*-gulonemethylosalicylate,  $[\alpha]_D^{20}$   $-11.03^\circ$ , the free acid,  $[\alpha]_D^{20}$   $-5.0^\circ$  and the *p*-bromophenyldiazotone, m. 132–3°,  $[\alpha]_D^{20}$   $-8.8^\circ$ . I on methylation by Purdie's method gave a mixt. of material, b. 125–45°, chiefly trimethyl-*d*-gulonemethylosalicylate (II), and open chain tetra-*Me* *d*-gulonemethylosalicylate, b. 90°. II was oxidized with concd.  $\text{HNO}_3$  at 100° for 7 hrs. and after methylation of the product with 2%  $\text{HCl}$ -MeOH gave *d*-dimethyl dimethoxyacetalate, m. 67–8°. To I (2 g.) in  $\text{H}_2\text{O}$  (15 cc.) and to Congo red at 0° was added 2% Na-Hg (63 g.) over 2 hrs. Neutralization to litmus, concn, pptn. of the Na-SO<sub>3</sub> with abs. EtOH, and removal of impurities by pptn. with  $\text{Et}_2\text{O}$  gave the sirupy *d*-gulonemethose (III) (0.7 g.),  $[\alpha]_D^{20}$   $-35.7^\circ$  ( $\text{H}_2\text{O}$ ); *p*-bromophenyldiazotone (IV), m. 135–6° (EtOH),  $[\alpha]_D^{20}$   $-16.10^\circ$  (abs. EtOH), const. after 2 weeks at 9.80°,  $[\alpha]_D^{20}$   $-49.12^\circ$  ( $\text{CH}_3\text{N}$ ), const. after 2 weeks at 31.70°; *p*-bromophenylosazone, m. 195–6°,  $[\alpha]_D^{20}$  0° ( $\text{C}_6\text{H}_5\text{N}$ -EtOH), const. after 2 hrs. at 15.3°; phenylosazone, m. 182–3°,  $[\alpha]_D^{20}$  0° ( $\text{C}_6\text{H}_5\text{N}$ -EtOH), const. after 1 hr. at 177°. From IV by decompn. with  $\text{BaH}_2$ , III was obtained crystn., and m. 130–1° (AcOH),  $[\alpha]_D^{20}$   $-42.3^\circ$  ( $\text{H}_2\text{O}$ ), const. after 30 min. at  $-38.03^\circ$ . R. C. E.

Crystalline structure of the sugars. II. Methylated sugars and the conformation of the pyranose ring. E. G. Cox, T. H. Goodwin, and A. I. Wagstaff. *J. Chem. Soc.* 1935, 1493–504; cf. *C. A.* 29, 6575.—From the study of the x-ray data recorded in conjunction with others previously published it is concluded that the C atoms in the  $\text{C}_5\text{O}$  ring of simple and methylated aldopyranoses are nearly coplanar, the O ring being displaced out of their planes. Reasons are adduced for supposing that the ring form is not appreciably different in soln. and the conformation deduced from the x-ray data is shown to account satisfactorily for the various chemical reactions (formation of  $\text{MeCO}$  compds.). The x-ray data confirm the configurations assigned to  $\alpha$ - and  $\beta$ -glucose. 2,3,4-Trimethyl- $\alpha$ -lyxose, orthorhombic needles, showing forms  $g\{011\}$ ,  $h\{100\}$  ( $q/g = 81^\circ$ ),  $a = 12.76$ ,  $b = 9.29$ ,  $c = 8.19$  Å,  $n$  (no. of asym. mols in unit cell, 4, whence  $d$ , (calcd.) = 1.31 g./cc. (obs. 1.26); the space group is  $P2_12_12_1$  ( $Q$ ); the min.  $n$  is 1.48 and is perpendicular to the  $a$ -axis. 2,3,6-Trimethyl- $\alpha$ -glucose, acicular orthorhombic combinations of  $a\{100\}$  and  $b\{010\}$ , terminated by  $c\{001\}$ ;  $a = 20.45$ ,  $b = 11.90$ ,  $c = 4.79$  Å,

$n = 4$ ,  $d$ , (calcd.) = 1.28 (obs. 1.28); space group  $P2_12_12_1$  ( $Q$ ),  $a = 1.465$  (parallel to  $c$ ),  $\gamma = 1.51$ . 2,3,4,6-Tetramethyl- $\alpha$ -glucose, elongated orthorhombic combinations of  $a\{100\}$  and  $m\{110\}$ , terminated by  $c\{001\}$  ( $a$   $m = 51^\circ$ ;  $a = 18.29$ ,  $b = 14.85$ ,  $c = 4.74$  Å,  $n = 4$ ,  $d$ , (calcd.) = 1.22, space group  $P2_12_12_1$ ;  $a$  (parallel to  $c$ ) = 1.475,  $\gamma$  (parallel to  $a$ ) = 1.54. 2,3,4,6-Tetramethyl- $\alpha$ -galactose, orthorhombic,  $a = 25.20$ ,  $b = 10.07$ ,  $c = 4.79$  Å,  $n = 4$ ,  $d$ , (calcd.) = 1.28 (obs. 1.24); space group  $P2_12_12_1$ . 2,3,4,6,7-Pentamethyl- $\beta$ - $\alpha$ -glucoheptose, monoclinic crystals with approx. hexagonal outline,  $a = 10.87$ ,  $b = 8.10$ ,  $c = 8.45$  Å,  $\beta = 92^\circ 10'$ ,  $n = 2$ ,  $d$ , (calcd.) = 1.25 (obs. 1.23); space group  $P2_1$  ( $C_2$ ). 2,3,4-Trimethyl- $\beta$ -methylglucoside, orthorhombic needles,  $a = 17.54$ ,  $b = 16.08$ ,  $c = 4.45$  Å,  $n = 4$ ,  $d$ , (calcd.) = 1.25 (obs. 1.23), space group  $P2_12_12_1$  ( $Q$ ),  $\alpha = 1.405$ ,  $\gamma > 1.525$ . 2,3,4,6-Tetramethyl- $\alpha$ -methylmannoside, monoclinic,  $a = 10.22$ ,  $b = 7.89$ ,  $c = 8.41$  Å,  $\beta = 97^\circ 14'$ ,  $n = 2$ ,  $d$ , (calcd.) = 1.235, space group  $P2_1$  ( $C_2$ ). 1,3,4-Trimethylfructose (observed in a simple micro-desiccator), orthorhombic prisms,  $a = 18.49$ ,  $b = 8.60$ ,  $c = 7.14$  Å,  $n = 4$ ,  $d$ , (calcd.) = 1.29, space group  $P2_12_12_1$ ,  $\alpha = 1.49$ ,  $\gamma = 1.525$ . 2,3,6-Trimethyl- $\beta$ -methylglucoside, fine needles,  $c = 4.41$  Å;  $\alpha = 1.47$ ,  $\gamma = 1.555$ . 2,4,6-Isomer, fine needles,  $c = 4.41$ ,  $\alpha = 1.465$ ,  $\gamma > 1.535$ . 2,3,4-Trimethyl- $\beta$ -methyl-d-arabinoside,  $c = 4.87$  Å. 2,3,4-Trimethyl- $\alpha$ -methyl-arabinoside,  $c = 4.85$  Å,  $\alpha = 1.475$ ,  $\gamma > 1.555$ . 2,3,4,6-Tetramethyl- $\beta$ -methylgalactoside, flattened needles,  $c = 4.74$  Å,  $\alpha = 1.47$ . 2,3,4-Trimethyl- $\beta$ -xyloolactone, flattened needles,  $c = 4.60$  Å,  $\alpha = 1.465$ .  $\alpha$ -Ethylglucosufuranoside, small needles,  $c = 5.69$  Å. 2,3,5-Trimethyl- $\gamma$ -lyxonolactone, monoclinic prisms,  $a = 4.42$  Å,  $\alpha < 1.47$ . 2,3,5-Trimethyl- $\gamma$ -arabonolactone, needles (probably orthorhombic),  $c = 4.60$  Å,  $2E = 80^\circ$  approx. Hexamethylxylofuranoside- $\alpha$ -lyxofuranoside, fine needles with  $c = 4.20$  Å. 1,3,4,5-Tetramethyl- $\beta$ -fructose,  $\alpha = 1.51$ ,  $\gamma = 1.53$ . 2,3,4-Trimethyl- $\alpha$ -xylose,  $\alpha = 1.48$ ,  $\gamma = 1.555$ . The original should be consulted for the discussion.

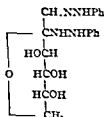
C. J. West  
Detosylation of 4- and 5-tosylacetone-*d*-methylrhannosides. P. A. Levene and Jack Compton *J. Am. Chem. Soc.* 57, 2306–10 (1935).—The usual methods of prep. acetone-*d*-methylrhannofuranoside (I) are attended by the simultaneous formation of acetone-*d*-methylrhannopyranoside (II), at times to the extent of 30–40%. The 5-tosyl deriv. (III) of I, m. 82–3°,  $[\alpha]_D^{20}$   $-13.6^\circ$  (MeOH,  $c$  3.168), II gives a 4-tosyl deriv. (IV), m. 61–2°,  $[\alpha]_D^{20}$   $-21.94^\circ$  (MeOH,  $c$  3.030). Alk. hydrolysis of III leads to the production of an unsatd. compd. in 90–5% yield, together with I; the unsatd. compd.,  $\text{C}_6\text{H}_9\text{O}_5$ , b. 56–60°,  $n_D^{20}$  1.4491; IV yields II quantitatively; these hydrolyses are accomplished without Walden inversion III with m. p. 80° is a mixt. contg. in addn. to pure III, a small proportion of the IV; alk. hydrolysis of this product leads to the formation of the unsatd. deriv. together with a small proportion of II. Reductive alk. hydrolysis of III gives I; IV gives II. The rates of hydrolysis of I and II are given as curves.

C. J. West  
Crystalline *d*- $\alpha$ -galactose and some of its derivatives. Raymond M. Hann, Alice T. Merrill and C. S. Hudson. *J. Am. Chem. Soc.* 57, 2100–3 (1935); cf. *C. A.* 28, 5046.—Details are given of the prep. from *d*-galactose of *d*- $\alpha$ -galacteonic amide, *d*- $\alpha$ -galacteonic acid (I) and its lactone. I (0.3431 g. in 25 cc.  $\text{H}_2\text{O}$  at 20°) shows a mutarotation from 2.5° to  $-23.3^\circ$  in 10 days. The lactone (45 g.) on reduction with Na-Hg gives about 25 g. *d*- $\alpha$ -galactose (II), crystg. as the monohydrate in 4 months, which shows  $[\alpha]_D^{20}$   $-22.5^\circ$  (1.1560 g. in 10 cc.  $\text{H}_2\text{O}$ ), changing to  $-14.1^\circ$  in 72 hrs. (for anhyd. sugar,  $-26^\circ$  and  $-15.3^\circ$ ). MeOH with 1.5%  $\text{HCl}$  gives 65% of  $\beta$ -*d*- $\alpha$ -galactopside, m. 132° (m. p. cor.),  $[\alpha]_D^{20}$   $-70.2^\circ$  (0.2708 g. in 10 cc.  $\text{H}_2\text{O}$ ); the rate of hydrolysis in 0.05 N  $\text{HCl}$  at 100° was 0.0004 (mins and decimal logarithms), its *penta-Ac* deriv., m. 108°,  $[\alpha]_D^{20}$   $-20.4^\circ$  (0.2354 g. in 10 cc.  $\text{CHCl}_3$ ).  $\beta$ -Et *d*- $\alpha$ -galactopside, m. 135°,  $[\alpha]_D^{20}$   $-65.4^\circ$  (0.2310 g. in 10 cc.  $\text{H}_2\text{O}$ ); *penta-Ac* deriv., m. 92°,  $[\alpha]_D^{20}$   $-24.9^\circ$  (0.3570 g.

in 10 cc  $\text{CHCl}_3$ . The benzyl mercaptal of II, m.  $191^\circ$ ,  $[\alpha]_D^{25}$   $30.3^\circ$  (0.2212 g in 10 cc  $\text{CH}_2\text{N}_2$ ); its *hexa-Ac* deriv., m.  $120^\circ$ ,  $[\alpha]_D^{25}$   $-10.6^\circ$  (0.2633 g in 10 cc  $\text{CHCl}_3$ ); the *hexa-Bz* deriv., m.  $129^\circ$ ,  $[\alpha]_D^{25}$   $-48.1^\circ$  (0.2950 g in 10 cc  $\text{CHCl}_3$ ). Attention is called to the parallelism of phys. and chem. properties, as well as rotational behavior, of substances of configurationally related structures in the sugar group, as illustrated from substances of the mannose and  $\alpha$ -galactose series.

C. J. West

Methylation of glucose phenylsulfonate and its formulation as a derivative of fructopyranose. Elizabeth E. Percival and Edmund G. V. Percival. *J. Chem. Soc.* 1935, 1398-402.—Glucosazone and  $\text{Me}_2\text{SO}$ , in  $30^\circ$   $\text{NaOH-Me}_2\text{CO-EtOH}$ , heated at  $70^\circ$  for 10 min., yields the *5-Me* deriv. (I), plates, m.  $116-17^\circ$ ,  $[\alpha]_D^{25}$   $-44^\circ$  ( $\text{CHCl}_3$ ,  $c$  0.7),  $-49^\circ$  ( $\text{EtOH}$ ,  $c$  0.7), changing to  $-12^\circ$  in 36 hrs.; the equal mixt. forms fine needles. Treatment with  $p\text{-O}_2\text{NC}_6\text{H}_4\text{CHO}$  yields an osone, from which I is regenerated in 5 min. at room temp. by  $\text{PhNHNH}_2$ ,  $\text{AcOH}$ . Reduction of the osone with  $\text{Zn}$  and  $\text{AcOH}$  gives *5-methylfructose* (II), with  $[\alpha]_D^{25}$   $-50^\circ$ . In following the course of glycoside formation in the cold, it was found that, whereas fructose gives 100% furanose, II gives 62% of a pyranose in 24 hrs., while the quantity of furanose (14-15%) remains const. during this time. Heating II with 3%  $\text{MeOH-HCl}$  at  $75^\circ$  for 5 hrs. and methylation gives 1,3,4,5-tetramethylfructose (III) (after hydrolysis). Glucosazone, treated 3 times with  $\text{Me}_2\text{SO}$ , gives a tri-Me deriv. as a red syrup; this yields a trimethylfructose by way of the osone;  $\text{MeOH-HCl}$  reacts slowly at room temp. to form almost exclusively a pyranose, indicating substitution in position 5 and a free HO group in position 6. Further methylation yields III, proving that the sugar was essentially 3,4,5-trimethylfructose. Position 6 in the glucosazone is therefore either prevented from undergoing methylation by steric effects, which is improbable, or is concerned with ring formation and it is considered probable that the osazone contains a pyranose ring and has the structure



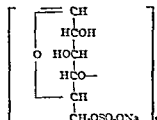
C. J. West

Pseudogalactal. Hermann Lohaus and Otto Widmaier. *Ann.* 520, 301-4 (1935).—Triacetylgalactal (20 g.), boiled 20 min. with 20 parts of  $\text{H}_2\text{O}$ , gives 11 g. diacetyl-galactal,  $\text{C}_{12}\text{H}_{18}\text{O}_6$  (I), b.p.  $163-5^\circ$ ,  $[\alpha]_D^{25}$   $-26.9^\circ$ ; *p*-nitrophenylhydrazine, m.  $178^\circ$ ; *Et* acetal,  $\text{C}_{16}\text{H}_{24}\text{O}_8$  (from  $\text{HC(OEt)}_2$ ), b.p.  $136-8^\circ$ ,  $[\alpha]_D^{25}$   $-39.70^\circ$ . I and  $\text{Ba(OH)}_2$  give isogalactal, whose *p*-nitrophenylhydrazine, m.  $187^\circ$ , repeated distn. gives protogalactal I with  $\text{Pd}$  and  $\text{H}_2$  gives dihydro- $\beta$ -galactal diacetal, b.p.  $148-53^\circ$ ,  $[\alpha]_D^{25}$   $41.2^\circ$  (*p*-nitrophenylhydrazine, m.  $148^\circ$ ); *Et* acetal, b.p.  $107-8^\circ$ ,  $[\alpha]_D^{25}$   $23.78^\circ$ ; dihydrogalactal, m.  $128^\circ$ ,  $[\alpha]_D^{25}$   $48.1^\circ$ .

C. J. West

Structure of sodium sulfonic acid ester of galactan from *Indeae laminarioides* (Rhodophyceae). W. Z. Hassid. *J. Am. Chem. Soc.* 57, 2046-50 (1935); cf. *C. A.* 27, 5782.—The Na sulfonic acid ester of galactan,  $[(\text{C}_6\text{H}_7\text{O}_2)_n\text{OSO}_3\text{Na}]_n$  (I), from the red alga, *Indeae laminarioides*, has a mol wt. of 1560 and therefore  $n$  is 6, the titration curve and the sp. cond. indicate that I is a salt of a strong acid. By electrolysis, the Na was removed and the substance, initially neutral, became acid,  $p_n$  of about 3, this was not obtained pure, because all the Na could not be removed and also because part of the  $\text{SO}_3\text{H}$  group was removed. Acetylation gives a product with 2 Ac groups per 1 mol galactose. Acid or alk. hydrolysis gives a galactan (II) with  $[\alpha]_D^{25}$   $82.2^\circ$  and  $78^\circ$  ( $\text{H}_2\text{O}$ ), resp. The

1 methylated product contains 21.23% MeO and has  $[\alpha]_D^{25}$   $17.2^\circ$  ( $\text{CHCl}_3$ ); hydrolysis gives a dimethylmethylgalactoside. II gives a trimethylgalactan with 44.5% MeO,  $[\alpha]_D^{25}$   $32.4^\circ$  ( $\text{H}_2\text{O}$ ,  $c$  0.8), which yields a trimethylgalactose on hydrolysis,  $[\alpha]_D^{25}$   $129^\circ$  ( $\text{H}_2\text{O}$ ,  $c$  0.8); oxidation with Br and then with  $\text{HNO}_3$ , and subsequent esterification give a dimethylarabodimethoxyglutarate, hydrolyzed to dimethylhydroxyglutaric acid. A tentative structure of I is



This is, apparently, the 1st time that a homogeneous galactan, consisting entirely of galactose units, has been obtained from a plant.

C. J. West

The molecular constitution of benzene. G. B. Banno. *Gazz. chim. ital.* 65, 371-423 (1935).—Following an extensive and crit. review of the present status of the problem of the constitution of aromatic nuclei, certain quantum concepts which have a bearing on this problem are discussed. The methods of Pauling (*C. A.* 27, 2870) and of Huckel (*C. A.* 26, 5826) for explaining the aromatic properties of  $\text{C}_6\text{H}_6$  are then discussed critically, in addition to which certain concepts already advanced by B. (cf. *Intern. Congress Pure Applied Chem.*, Madrid, 1934; cf. *C. A.* 29, 7236) are reviewed. In this connection it is shown that, from the phys. point of view, the assumption of a ring structure for  $\text{C}_6\text{H}_6$ , thiophene, pyrrole and furan is plausible and satisfactory, since it explains the chem. behavior of these compds. and their derivs. One hundred references.

C. C. Davis

Mechanism of polymerization reactions IV.  $\alpha$ -Phenylbutadiene. E. Bergmann. *J. Chem. Soc.* 1935, 1359-60; cf. *C. A.* 26, 2976.—The dimer of  $\text{PhCH}=\text{CHCH}=\text{CH}_2$  is 2-benzyl-1-styryl-3'-cyclopentene,  $\text{PhCH}=\text{CHCH}(\text{CH}_2\text{Ph})\text{CH}=\text{CHCH}_2$ , since oxidation gives

$\text{BrOH}$  and  $\text{HO}_2\text{CCH}(\text{CH}_2\text{CO}_2\text{H})\text{CH}(\text{CH}_2\text{Ph})\text{CO}_2\text{H}$  (I). The mechanism of the dimerization is discussed. Since other possible formulas for the dimer are  $\text{PhCH}=\text{CHCH}=\text{CHCH}(\text{CH}_2\text{Ph})\text{CH}=\text{CHCH}_2$ , which should yield 3-

7 phenylbutane- $\alpha,\beta,\gamma$ -tricarboxylic acid (II) on oxidation and  $\text{PhCH}=\text{CHCH}(\text{CH}_2\text{Ph})\text{CH}=\text{CHCH}_2$ , which should

yield the  $\alpha,\gamma,\delta$ -tricarboxylic acid (III), these acids were studied. III, m.  $163-5^\circ$  (decomp.), was prepd from  $\text{CH}_3(\text{CO}_2\text{Et})\text{CH}(\text{CH}_2\text{CO}_2\text{Et})\text{CH}(\text{CH}_2\text{Ph})\text{CO}_2\text{Et}$  with  $\text{EtONa}$  (*Et* ester, b.p.  $230-40^\circ$ , m.  $42^\circ$ ), followed by hydrolysis and decarboxylation at  $130^\circ$ . II, m.  $168^\circ$  (Duff and Ingold, *C. A.* 28, 1905). Neither II nor III was identical with I. In the synthesis of II by the benzylation of the tetracarboxylic acid from  $\text{CHNa}(\text{CO}_2\text{Et})_2$  and  $\text{Et}$  fumarate, some I is formed.

C. J. West

Stability against interchange of the iodine atoms in diphenylphosphonium iodide. F. Juliusburger, B. Topley and J. Weiss. *J. Chem. Soc.* 1935, 1295-6.—By use of radioactive NaI prepd by neutron bombardment, the interchange of I with the 2 I atoms in diphenylphosphonium iodide (I) was studied. On crystals of I from  $\text{H}_2\text{O}$ -alc. contg. active NaI the resulting solid was strongly radioactive. Diphenylphosphonium hydride derived from it was converted to I with inactive NaI and was inactive. Therefore interchange occurs with the neg. I only. Boiling 20 min. in alc. and  $\text{H}_2\text{O}$ -alc. gave no interchange. I with active neg. I heated at  $103^\circ$  with  $\text{PhI}$  for 5 min. when purified and the neg. I replaced with inactive I gave inactive I. A check portion not so treated was still active.

Foster Dee Snell

Decomposition of *p*-iodoaniline. F. B. Dains, R. G. Brewster and John A. Davis. *J. Am. Chem. Soc.* 57, 2326-7 (1935).—*p*-IC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> (400 g.), on standing 3 years in a glass-stoppered bottle, gave 95 g. 2,4-IC<sub>2</sub>H<sub>3</sub>NH<sub>2</sub> and about 190 g. unchanged *p*-IC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, with some 80 g. black insol. mass.

C. J. West  
Molecular rearrangement of sulfenilides [sulfenilamides]. II. Maurice L. Moore and Treat B. Johnson. *J. Am. Chem. Soc.* 57, 2234-6 (1935); cf. C. A. 29, 7300<sup>1</sup>.—Certain sulfenilamides have been rearranged by digestion in EtOH-NaOH to give the corresponding *o*-mercaptodiphenylamines, whereas, upon heating alone, the same compd. gives *p*-amino sulfides. The free *o*-amino sulfides, so far as exam'd, do not suffer a rearrangement under the same conditions, hence the question arises, whether the sulfenilamides pass through the intermediate stage before forming the *o*-mercaptan deriv. As yet this point has not been clarified. 2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SNHPh gives 87% of 2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHCH<sub>2</sub>CH<sub>2</sub>SNH-2; the *o*- and *p*-toluides give 80 and 90% of the corresponding derivs. These were characterized as the thiomethyl ethers, m. 97-8°, 94° and 84-8°, resp.

C. J. West  
Identification of carboxylic acids as carboxylates of benzylamine and *o*-phenylethylamine. C. A. Buchler, Louise Carson and Rachel Edds. *J. Am. Chem. Soc.* 57, 2181-2 (1935).—The following compds. of PhCH<sub>2</sub>NH<sub>2</sub> with acids are sufficiently stable to permit AcOEt or abs. EtOH to be used for purification. Benzylammonium carboxylates (acid given): HCO<sub>2</sub>H, m. 95-7° (all m. ps. cor.); AcOH, m. 96.3-7.1°; EtCO<sub>2</sub>H, m. 46.7-7.5°; PrCO<sub>2</sub>H, b<sub>10</sub> 109-10°; BuCO<sub>2</sub>H, m. 51.7-2.7°; ClCH<sub>2</sub>CO<sub>2</sub>H, m. 119.9-20.9°; Cl<sub>2</sub>CHCO<sub>2</sub>H, m. 120.3-1.3°; PhCH<sub>2</sub>CO<sub>2</sub>H, m. 122-2.6°; *o*-MeC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 145.4-6.4°; PhCH<sub>2</sub>CHCO<sub>2</sub>H, m. 135.9-6.3°; BrOH, m. 127.2-8.4°; *o*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 112-13.2°; *m*-isomer, m. 134-1.6°; *p*-isomer, m. 197.6-8.6°; *m*-BrC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 160.6-1.2°; *o*-ClCH<sub>2</sub>CO<sub>2</sub>H, m. 150.5-1.5°; *p*-isomer, m. 149.2-9.8°; *p*-isomer, m. 160.3-1.3°; *o*-HOCH<sub>2</sub>CO<sub>2</sub>H, m. 94.3-5.5°; *m*-isomer, m. 188.2-9.6°; *p*-isomer, m. 216.6-17.2°; *o*-MeOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 121.5-2.3°; *m*-isomer, m. 113.1-14.1°; *p*-isomer, m. 144.8-5.6°; *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 143.9-5.7°; *m*-isomer, m. 174.9-5.5°; *p*-isomer, m. 197.3-8.9°. *o*-Phenylethylammonium carboxylates: HCO<sub>2</sub>H, m. 97.8-8.8°; AcOH, m. 92.1-2.5°; EtCO<sub>2</sub>H, b<sub>10</sub> 120-1°; PrCO<sub>2</sub>H, b<sub>10</sub> 125°; ClCH<sub>2</sub>CO<sub>2</sub>H, m. 94.7-5.5°; PhCH<sub>2</sub>CO<sub>2</sub>H, m. 115.6-16.4°; *o*-MeC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 113.3-13.7°; PhCH<sub>2</sub>CHCO<sub>2</sub>H, m. 144.5-5.1°; BrOH, m. 140.6-1.2°; *o*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 111-11.6°; *m*-isomer, m. 168.9-9.4°; *p*-isomer, m. 185.6-6.4°; *m*-BrC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 148.9-9.7°; *o*-ClCH<sub>2</sub>CO<sub>2</sub>H, m. 130.9-1.9°; *m*-isomer, m. 144.7-5.3°; *p*-isomer, m. 152.2-3.2°; *o*-HOCH<sub>2</sub>CO<sub>2</sub>H, m. 102.1-2.9°; *m*-isomer, m. 137.2-8.4°; *p*-isomer, m. 199.3-9.9°; *o*-MeOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 158.7-9.1°; *m*-isomer, m. 131.4-1.8°; *p*-isomer, m. 132.8-3.4°; *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 129.5-30.1°; *m*-isomer, m. 170.7-1.1°; *p*-isomer, m. 195.8-6.8°.

C. J. West  
Amides obtained from benzenesulfonic acid. L. Chas. Rairford and Stewart E. Hazlet. *J. Am. Chem. Soc.* 57, 2172-4 (1935).—The following amides were prep'd. by treatment of 2 mol. proportions of the amine in 3 vols. dry EtO with 1.25 mols. of crude PhSOCl in 2 vols. EtO, with continuous agitation at 0° (the name of the starting amine is given): *o*-ClC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, m. 107-8°, 76% yield; *m*-isomer, tan, m. 100°, very poor yield; *p*-isomer, m. 155.5°, 78%; *o*-BrC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, lavender, m. 110-11°, 20%; *m*-isomer, pink, m. 112-14°, 19%; *p*-isomer, lavender, m. 150.5°, 47%; *o*-MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, pink, m. 124-5°, 44%; *m*-isomer, m. 102-3°, 44%; *p*-isomer, yellow, m. 100-1°, 56%; *o*-MeOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, purple, m. 108-13°, very poor yield; *m*-isomer, m. 133-4°, poor yield; *p*-isomer, purple, m. 133-4°, 89%; *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>Et, m. 114-15°, 29%; 2,5-Me<sub>2</sub>(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>2</sub>, yellow, m. 133°, 26%; 8-CuH<sub>4</sub>NH<sub>2</sub>, tan, m. 143-4°, very poor yield; PhCH<sub>2</sub>NH<sub>2</sub>, m. 100-4°, 26%; 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Ph, m. 165.5°, high yield; 2-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl-4, yellow, m. 206° very poor yield; 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl-4, pink, m. 165-6°, very poor yield. Anilides of benzenesulfonic acid were

prep'd. as follows: *o*-BrC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, m. 130-1°, 72%; *m*-isomer, m. 117.5-18.5°, 69%; *o*-MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, lavender, m. 122-3°, 97%; *m*-MeOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, m. 82.5-3.5°, quant. yield; *p*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>Et, m. 183.5-4°, quant. yield; 2,5-Me<sub>2</sub>(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>2</sub>, m. 175-6°, 88%; 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Ph, m. 147-8°, 67%; 2-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl-4, m. 136-8°, 74%; 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl-4, m. 145-5.5°, quant. yield.

C. J. West  
Some derivatives of *p*-fluorobenzenesulfonic acid. Raymond M. Hann. *J. Am. Chem. Soc.* 57, 2167-8 (1935).—Dry HCl passed through AlCl<sub>3</sub> in a soln. of PhF in CS<sub>2</sub> until sat'd., followed by SO<sub>2</sub> (3 hrs.), gives 75% of Na *p*-fluorobenzenesulfonate dihydrate (I), diamond crystals. I and benzyl *p*-thiourethane (II) in 0.25 N HCl give the II salt of *p*-fluorobenzenesulfonic acid, m. 161° (m. ps. cor.). Oxidation of I with H<sub>2</sub>O<sub>2</sub> and addn. of II give the II salt of *p*-fluorobenzenesulfonic acid, m. 166°. Reducing I and PhCOCH<sub>2</sub>Br in 95% EtOH gives *p*-fluorophenyl phenacyl sulfone, m. 151°. With *p*-ONC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl there results *p*-fluorophenyl *p*-nitrobenzyl sulfone, pale yellow, m. 185°. I and ClCH<sub>2</sub>CO<sub>2</sub>H give *p*-fluorobenzenesulfonylethylacetic acid, m. 110-11° (II salt, m. 144° (decompn.)). *p*-bromophenacyl ester, m. 126°. I and ClCH<sub>2</sub>Ac give a quant. yield of *p*-fluorophenylsulfonylethylacetone, m. 66°; CO(CH<sub>3</sub>)<sub>2</sub>, and I give *α*,*γ*-di-*p*-fluorobenzenesulfonylethylacetone, m. 144°.

C. J. West  
Halogenation of phenolsulfonic acids in nitrobenzene. Ralph C. Huston and Arthur H. Neely. *J. Am. Chem. Soc.* 57, 2176-8 (1935).—PhOH (31.3 g.) and 50 g. concd. H<sub>2</sub>SO<sub>4</sub>, heated 2 hrs. at 100-10°, addn. of 100 g. PhNO<sub>2</sub>, and then 15 g. fuming H<sub>2</sub>SO<sub>4</sub> (temp. not above 10°), followed by 107 g. Br<sub>2</sub> and 50 g. PhNO<sub>2</sub> (dropwise during 2 hrs.), give 10.4% of 2-BrC<sub>6</sub>H<sub>4</sub>OH and 72.7% of 2,6-Br<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH; using 95 g. H<sub>2</sub>SO<sub>4</sub>, gives 9.5% 2,6-Br<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH and 45.5% of 2-BrC<sub>6</sub>H<sub>4</sub>OH. With 68 g. H<sub>2</sub>SO<sub>4</sub>, Cl<sub>2</sub> gives 70.3% of 2,6-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH and 17% 2-ClC<sub>6</sub>H<sub>4</sub>OH; min. yields of 24% of 2,6-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH and 72% 2-ClC<sub>6</sub>H<sub>4</sub>OH were obtained with 100 g. H<sub>2</sub>SO<sub>4</sub>. *o*-MeC<sub>6</sub>H<sub>4</sub>OH (36 g.), 55 g. concd. H<sub>2</sub>SO<sub>4</sub>, 60 g. Br<sub>2</sub> and 50 g. PhNO<sub>2</sub>, give 60% crude 6-Br deriv. and 13% 4,6-Br<sub>2</sub> deriv. Cl<sub>2</sub> gives 30% of the 6-Cl deriv. and 12% of the 4,6-Cl<sub>2</sub> deriv. Bromination or chlorination of the 4- and 6-sulfonic acids of *m*-MeC<sub>6</sub>H<sub>4</sub>OH in PhNO<sub>2</sub> gives the 2- and 4-Br and the 2,6-Br<sub>2</sub> derivs. and the 2-Cl, 2,4- and 2,6-Cl<sub>2</sub> derivs. Sulfonic acid groups on the C<sub>6</sub>H<sub>5</sub> ring of PhOH and derivs. are stable toward halogenation in the presence of acid if carried out in an inert anhyd. solvent.

C. J. West  
Pyrolysis of allyl-*p*-phenetidine. F. L. Carnahan. *J. Am. Chem. Soc.* 57, 2210-11 (1935).—The Na compd. of phenacetin and allyl bromide, followed by hydrolysis, yield 31% of allyl-*p*-phenetidine (I), b<sub>10</sub> 134.5°, b<sub>10</sub> 265° (decompn.), d<sub>20</sub> 1.0090, n<sub>D</sub> 1.5493; *p*-toluenesulfonylethyl, m. 81° (80% yield). Refluxing I at atm pressure gives 0.465 mol. *p*-EtOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 0.40 mol. C<sub>6</sub>H<sub>6</sub> and 0.447 mol. resinous material. The initial step appears to be a splitting into radicals at the C-N bond.

C. J. West  
The condensation power of pyrocatechol derivatives. Paolo Dreyfuss. *Gazz. chim. ital.* 65, 498-501 (1935).—A review and discussion, based on the work of various investigators, and with special attention to the previous work by D. (Dist., Bonn 1933). The power of condensation of derivs. of *o*-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> at the 4- and 5-positions is evident from recent literature. This condensing power should be taken into account in all investigations of pyrocatechol derivs., for on the one hand this power opens new possibilities in syntheses, but on the other hand may give rise to undesired reactions.

C. C. D.  
Alkyl ethers of 2,2-bis(4-hydroxyphenyl)propane. I. Some dialkyl ethers. G. R. Yohe and J. F. Vitche. *J. Am. Chem. Soc.* 57, 2259-60 (1935).—The ethers of HOC<sub>6</sub>H<sub>4</sub>C(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OH were prep'd. with the dialkyl sulfates and alkali (or Am deriv.) from the alc. and SO<sub>2</sub>Cl<sub>2</sub>. Me ether, b<sub>10</sub> 190°, m. 69-61.5°, n<sub>D</sub> 1.5696; Et ether, m. 49-50°, n<sub>D</sub> 1.5556; Pr ether, b<sub>10</sub> 200-2°, n<sub>D</sub> 1.5448, d<sub>15</sub> 1.0156; Bu ether, b<sub>10</sub> 212-13°, m. 20°, n<sub>D</sub> 1.5368, d<sub>15</sub> 0.9961; Am ether, b<sub>10</sub> 225-30°, m. 34.5-5.5°, n<sub>D</sub> 1.5306.

The yield of the first 4 is about 50%, of the Am deriv. 1 28%.

C. J. West  
Addition of chloroform and bromoform to *p*-chloro-benzaldehyde. Joseph W. Howard. *J. Am. Chem. Soc.* 57, 2317-18 (1935); cf. C. A. 29, 1704<sup>1</sup>. — *p*-Cl-C<sub>6</sub>H<sub>4</sub>CHO (46 g) and 60 g CHCl<sub>3</sub> with 4 g KOH, on standing 3 hrs, give 20 g *trichloromethyl-p-chlorophenyl-carbinol* bp 187-8°, d<sub>4</sub><sup>20</sup> 1.523, acetate, m 120-1°; propionate, m 71-2°; benzoate, m 128-9°; butyrate, m 172-3°, d<sub>4</sub><sup>20</sup> 1.331. The *tribromomethyl deriv.* b<sub>p</sub> 198-200°, m 90-1°.

C. J. West  
Rearrangement of thiocyanates into isothiocyanates. Ernst Bergmann. *J. Chem. Soc.* 1935, 1361-2. — *Cinnamyl thiocyanate* (I), m 63°, results in 15 g yield from 12 g KSCN and 20 g PhCH<sub>2</sub>CH=CH<sub>2</sub>Cl in 75% EtOH; on refluxing for 1 hr, I yields *cinnamyl isothiocyanate* (II), yellow, b<sub>p</sub> 162°; thus the classical scheme of rearrangement applies to this reaction. Catalytic reduction of I yields *γ-phenylpropyl thiocyanate*, b<sub>p</sub> 80°, also prepd from PhCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Br (III) and NH<sub>4</sub>SCN; it has an intensely viscous effect. II and PhNH<sub>2</sub> in Et<sub>2</sub>O-EtOH give PhCH<sub>2</sub>CH<sub>2</sub>NHCSNHPh III and K phthalimide yield *phthalido-γ-phenylpropylamide*, m 61°, which also results from catalytic reduction of PhCH<sub>2</sub>CH<sub>2</sub>N(CO)<sub>2</sub>CH<sub>2</sub> (Pogner, Ber. 26, 1890 (1893)). This shows that cinnamylamine does not have the isomeric structure PhCH(NH<sub>2</sub>)CH=CH<sub>2</sub>.

C. J. West  
Emulsion XXIII. Influence of substitution in the benzene nucleus on the fermentative splitting of phenol *β*-*D*-glucosides. II. Burkhardt Heffrich and C. Pauline Burt. *Ann.* 520, 156-63 (1935); cf. C. A. 29, 6817<sup>1</sup>. — *o*-HO-C<sub>6</sub>H<sub>4</sub>OMe (2.9 mol) and 1 mol acetobromoglucose with 1 mol. NaOH in Me<sub>2</sub>CO-H<sub>2</sub>O, after 4 hrs. at room temp, give 55% of the *tetra-Ac deriv.*, m 155-7°, [α]<sub>D</sub><sup>20</sup> -30.2° (CHCl<sub>3</sub>), of *guaiacol β-D-glucoside* (I), m 155.5-7.5°, with 1 mol. H<sub>2</sub>O, [α]<sub>D</sub><sup>20</sup> -68.8° (H<sub>2</sub>O); it is very hygroscopic. *Isoeugenol β-D-glucoside* (III), m 187.5-9.5°, [α]<sub>D</sub><sup>20</sup> -60° (abs. EtOH); *tetra-Ac deriv.*, m 140-3°, [α]<sub>D</sub><sup>20</sup> -24.8° (CHCl<sub>3</sub>) (9.2 g yield from 15 g II). The 3-benzyl ether (IV) of protocatechuic aldehyde *β*-*D*-glucoside, with 1 mol H<sub>2</sub>O, m 165-8°, [α]<sub>D</sub><sup>20</sup> -60.2° (abs. EtOH); the compd is very hygroscopic. *Tetraacetylprotocatechuic aldehyde 4-β-D-glucoside* (11.6 g) and acetobromoglucose with NaOH in Me<sub>2</sub>CO-H<sub>2</sub>O at room temp, for 20 hrs give, after deacetylation, about 8.5 g *protocatechuic aldehyde 4-β-D-glucoside 3-β-D-glucoside* (V), with 1 mol H<sub>2</sub>O, [α]<sub>D</sub><sup>20</sup> -100.5°. With an emulsion with a *β*-glucosidase value of 1.2, the following percentage was decomposed in the time given: I, 10 min 24.8%, III, 10 min, 19.5%, IV, 6 min, 16.3%; V, 33 min 41.2-51.2%; with an emulsion with *β*-glucosidase value of 5.5, V in 6 min is decomposed to the extent of 48.7-59%.

C. J. West  
Condensations of benzoylformanilide with acetone, ethyl phenylacetate and diethyl malonate. John V. Scudi and H. G. Lindwall. *J. Am. Chem. Soc.* 57, 2302-3 (1935); cf. C. A. 29, 7307<sup>1</sup>. — PhNHCOBz and Me<sub>2</sub>CO with a little Et<sub>3</sub>NH at room temp for 6 hrs. give 80% of 2-phenyl-2-hydroxy-4-pentanoneanilide, PhC(OH)(CONHPh)CH<sub>2</sub>Ac, m 137-8°; phenylhydratone, m 174-5° (decompn); further action of PhNHCOBz (2 weeks) gives 25% of 2,6-dihydroxy-2,6-diphenyl-4-heptanone-1,7-dianilide, CO(CH<sub>2</sub>CH(OH)(CONHPh)Ph)<sub>2</sub>, m 171-2°; PhNHCOBz and PhCH<sub>2</sub>CO<sub>2</sub>Et after 3 months at room temp give 90% of *α,β-diphenylmalenil*, yellow, m 178-9°, reduction gives 80% of *α,β-diphenylsuccinil* (I), m 234-5°. With CH<sub>3</sub>(CO)<sub>2</sub>Et, there results 95% of 2-phenyl-2-formanilide-1,1,3,3-tetracarboxypropane, m 107-8°; vacuum distn gives 75% of *α-carboxy-β-phenylmalenil*, yellow, m 111°, reduced to the succinil, m 104-5°; hydrolysis gives I.

C. J. West  
Resolution of *dl*-mandelic acid with (-)-ephedrine. Robert Rogers. *J. Chem. Soc.* 1935, 1544. — Details are given of the resolution; 130 g *dl*-acid yield 45 g *l*- and 30 g *d*-acid; most of the ephedrine is recovered.

C. J. West

Lichen acids. VI. Constituents of *Ramalina scopulorum*. Francis H. Cud and Alexander Robertson. *J. Chem. Soc.* 1935, 1379-81; cf. C. A. 27, 3463<sup>1</sup>. — The lichen *Ramalina scopulorum* (1478 g), extd. with Me<sub>2</sub>CO for 15 hrs, gives about 20 g stictic acid (I) (Zopf's scopulonic acid, C. A. 1, 1877), which seps. from 80% Me<sub>2</sub>CO with 2 mols H<sub>2</sub>O; it is identical with the acid from *Lobaria pulmonaria* (Aubiana, C. A. 27, 4227, 4787); the diacetate m 235-6°, and the tetraacetate, m 226-7°; the dianil, m 234°. With 10% aq. KOH there results stictic acid, which is considered substantial evidence for the presence of a lactone ring in I with MeI and Ag<sub>2</sub>CO<sub>3</sub> give the compd, C<sub>12</sub>H<sub>10</sub>O<sub>5</sub>(OMe)<sub>2</sub>, m 174°; Ag<sub>2</sub>O gives an isomer, m 242-3°. C. J. W.

Attempted preparation of vanilloylformic acid. W. E. Barch. *J. Am. Chem. Soc.* 57, 2330 (1935). — The prep. of vanilloylformic acid by Mottern's method (C. A. 29, 135<sup>1</sup>) could not be repeated; difficulty was also experienced in repeating his prep. of apocyanin. A. B. Lamb reports that 2 other investigators could not repeat the prep.

C. J. West  
Configuration of the *Δ*<sup>1</sup>-tetrahydro- and the hexahydro-terephthalic acids. W. H. Mills and G. H. Keats. *J. Chem. Soc.* 1935, 1373-5. — Slight modifications of Baeyer's method produced from 400 g. terephthalic acid 170 g. *trans-Δ*<sup>1</sup>-tetrahydro deriv. (I) and 40 g. *cis*-(II)-isomer, m 228° and 161°, resp. I was resolved with brucine in H<sub>2</sub>O; the pure *l* form has [α]<sub>D</sub><sup>20</sup> -279° and m 222°; the other isomer could not be purified. Catalytic reduction of I and II gives the corresponding hexahydro acids, m 309° and 167°, resp. In these cases the more sol. form has the *cis*-configuration. For II at 22° K<sub>1</sub> = 0.82 × 10<sup>-4</sup>, K<sub>2</sub> = 0.81 × 10<sup>-4</sup>, I, K<sub>1</sub> = 1.18 × 10<sup>-4</sup>, K<sub>2</sub> = 1.19 × 10<sup>-4</sup>, while I is slightly stronger, the ratio K<sub>1</sub>/K<sub>2</sub> is nearly the same for both acids. From Ingold's formula this gives values of *r* for II and I of 8.14 and 8.26 Å, resp.

C. J. West  
Hydrophthalides VII. Sergio Berlingozzi and Victoria Senatori. *Gazz. chim. ital* 65, 424-40 (1935); cf. C. A. 26, 2728<sup>1</sup>. — In addn to the sapon of benzylidene-*Δ*<sup>1</sup>-tetrahydrophthalide (I) by aq KOH to form *o*-phenylacetyl-tetrahydrobenzoic acid (II), already described (C. A. 26, 2728), expts show that sapon under different conditions leads to a different product. Alc. I (8.5 g. in 100 cc) and aq. KOH (5 g. in 25 cc.), refluxed 5 min. and treated with H<sub>2</sub>SO<sub>4</sub>, yield 6 g. of an isomer (III), C<sub>10</sub>H<sub>8</sub>O<sub>4</sub>, of II, m 115°, sol in cold dil. Na<sub>2</sub>CO<sub>3</sub> (reprecip. by mineral acids). *Ag salt*. With aq. NaOH and NH<sub>4</sub>OH HCl, III forms the oxime, C<sub>10</sub>H<sub>8</sub>O<sub>3</sub>N, m 62° (cf. 154-5° of the oxime of II). III in boiling aq. KOH, acidified with H<sub>2</sub>SO<sub>4</sub> and let stand, forms II, whereas II in boiling aq. alc. KOH on acidification yields unaltered II. This shows that II is the stable and III the unstable form. Evapn of the mother liquor from the crystn of III yields *β*-phenyl-*α,γ*-diketotetrahydroindene,

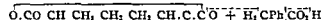
C<sub>14</sub>H<sub>12</sub>CO CHPh CO (IV), canary-yellow, m 104°; its solns. in aq. alkalis are intense violet-blue, and IV is pptd. by acids; it also is formed by evapn of a soln. of I and NaOMe in MeOH or NaOEt in EtOH and treatment of the residue with dil H<sub>2</sub>SO<sub>4</sub>. The yield is higher with MeONa. The mother liquor in this case contains a trace of III, the proportion of which is larger from EtONa than from MeONa. Ice-cold II (4 g.) in 2% aq. KOH (100 cc) and aq. 2% K<sub>2</sub>CO<sub>3</sub> evolve BzH and yield, after steam-distn of the filtered liquid and acidification of the residue with H<sub>2</sub>SO<sub>4</sub>, 0.8 g of BzOH, while the mother liquor extd with Et<sub>2</sub>O yields 0.5 g of BzOH. The wash liquor from the BzOH made alk. with NH<sub>4</sub>OH and treated with CaCl<sub>2</sub> and AcOH gives (CO<sub>2</sub>)<sub>2</sub>Ca, while the filtrate treated with H<sub>2</sub>SO<sub>4</sub> and extd with Et<sub>2</sub>O yields a compd (V), m 50°, with a strong odor of PhCH<sub>2</sub>CO<sub>2</sub>H, and which is probably the latter. With KMnO<sub>4</sub> under the same conditions, III gives twice as high a yield of BzOH, a little (CO<sub>2</sub>)<sub>2</sub>Ca, BzH and V. III (4 g.) in 5% aq. NaOH (80 cc.) and 5% Na-Hg (30 g.), let stand, and acidified with HCl, ppts 3.2 g. of *o*-phenylacetyl-hexahydrobenzoic acid (VI), m 98°, stable on prolonged

heating at 100°; converted into an anhydride by concd.  $\text{H}_2\text{SO}_4$  at 100°, which excludes its being a  $\gamma$ -HO acid. It is also formed by refluxing either II or III in 10% aq. NaOH with powd. Zn, and acidifying with HCl. VI and PhHNH $_2$ , heated cautiously at 100°, and the product purified by soln. in aq.  $\text{Na}_2\text{CO}_3$  and reprecip. with HCl, yield the phenylhydrazide,  $\text{C}_6\text{H}_5\text{NHNH}\cdot\text{C}_6\text{H}_4\cdot\text{CH}(\text{CO}_2\text{H})_2$ , m. 50° (decompn.). VI is oxidized by  $\text{KMnO}_4$ . Na-Hg has practically no action on II. II, III or VI (2 g.) in 5% aq. NaOH (20 cc.) and 4% Na-Hg (100 g.), refluxed, neutralized with HCl, the filtered liquid acidified, the ppt. extd. with dil.  $\text{Na}_2\text{CO}_3$  and water, and purified with  $\text{Et}_2\text{O}$ , yields approx. 1.2 g. of benzylhexahydrophthalide, m. 79°, and approx. 0.5 g. of bis(benzylhexahydrophthalide)pyracene, m. 113°. Neither is sol. in cold dil.  $\text{Na}_2\text{CO}_3$ , while both are sol. in boiling aq. alk. hydroxides (reppd by acids), and these solns. are fairly resistant to  $\text{KMnO}_4$ . When I was first prepd. (loc. cit.), the position of the double bond was not established and therefore that in II was also uncertain. The prepn. and properties of II and III, particularly their behavior with  $\text{NH}_4\text{OH}$  and analogous cases in the literature which are discussed, indicate that they differ only in the position of the double bond. Their reactions with alk.  $\text{KMnO}_4$  lead to the conclusion that III has the

constitution  $\text{HO}_2\text{CCH}(\text{CH}_2)_3\text{CH}(\text{CH}_2)_3\text{CH}(\text{COOCH}_2\text{Ph})_2$ ,

while that of II might be either  $\text{HO}_2\text{CCH}(\text{CH}_2)_3\text{CH}(\text{CH}_2)_3\text{CH}(\text{COOCH}_2\text{Ph})_2$  (VII) or  $\text{HO}_2\text{CCH}(\text{CH}_2)_3\text{CH}(\text{CH}_2)_3\text{CH}(\text{COOCH}_2\text{Ph})_2$ .

Hydrogenation of II and III and analogous reactions cited from the literature lead in turn to the conclusion that II has the constitution VII. The condensation of  $\Delta^1$ -tetrahydrophthalic anhydride with  $\text{PhCH}_2\text{CO}_2\text{H}$ , described in earlier work, probably takes place according to the reaction:



This should be of utility in the synthesis of certain natural substances, e. g., sedanoic acid, sedatonic acid and its internal anhydride.

C. C. Davis  
Migration of allyl groups in the ethylacetoacetate series. Ernst Bergmann and Herbert Corte. *J. Chem. Soc.*, 1935, 1363-5.— $\text{PhCH}(\text{CHCH}_3)_2$  (35 g.) and  $\text{AcCHNaCO}_2\text{Et}$  in dry  $\text{C}_6\text{H}_6$ , boiled 48 hr., give Et  $\beta$ -cinnamylacetoacetate (I), b $_d$  200°, and 2 g. Et  $\beta$ -cinnamylacetoacetate, b $_d$  258°. Hydrolysis of I yields  $\gamma$ -benzylidenecinnamic acid and  $\alpha$ -phenyl- $\Delta^1$ -hexen-4-one.  $\text{PhCH}(\text{CHCH}_3)_2$  and Et  $\beta$ -ethoxycrotonate and  $\text{NH}_4\text{Cl}$ , heated 1 hr. at 160°, give Et  $\beta$ -cinnamylacetoacetate (II), b $_d$  162°, and Et  $\beta$ -cinnamylacetoacetate (III), b $_d$  200-10°. This reaction is primarily an O-cinnamylation, thus affording support for Claisen's view that the 1st stage in the alkylation of Et methoxycrotonate involves the "enolic" O. Alk. hydrolysis of II yields cinnamyl isopropenyl ether, b $_d$  120-2°, and  $\beta$ -phenyl- $\beta$ -vinylpropionic acid, b $_d$  162°; the alkali treatment of II to some extent leads to a rearrangement which is accompanied by a "transposition" of the alkyl group. Heating II at 260° for 6 hrs. gives a small yield of I; this rearrangement is the reason for the formation of III in the above reaction; in the 1st stage is formed II, which is rearranged into I and the enolic form of the latter is again cinnamoylated on the HO group.

C. J. West  
Reversibility of the Friedel-Crafts condensation. Hydrogenation phenomena. L. L. Alexander, A. L. Jacoby and Reynold C. Fuson. *J. Am. Chem. Soc.*, 57, 2209-9 (1935).— $\text{PhCHCH}_2\text{CH}_2\text{Bz}$  is formed from  $(\text{p-Cl-C}_6\text{H}_4)_2\text{C}(\text{CH}_2)_2\text{CHBr}$  or from  $\text{PhCH}_2\text{CHBr}$  with  $\text{AlCl}_3$  in  $\text{C}_6\text{H}_6$  satd. with HCl; likewise, 5 g.  $\text{PhCH}_2\text{CHBr}$  and  $\text{BrCH}_2\text{C}_6\text{H}_4\text{CO}_2\text{Et}$  with Zn in  $\text{C}_6\text{H}_6$  give 79% of the Et ester, m. 79-80.5°, of  $\beta$ -phenyl- $\beta$ -( $p$ -chlorophenyl)- $\beta$ -hydroxypropionic acid, light yellow, m. 188.5-9° (decompn.);  $\text{Ac}_2\text{O}$  and  $\text{AcONa}$  give  $\beta$ -( $p$ -chlorophenyl)cinnamic acid, m. 168°, which with  $\text{AlCl}_3$ , HCl and  $\text{C}_6\text{H}_6$  yields  $\text{PhCHCH}_2\text{CH}_2\text{CO}_2\text{H}$ . ( $\text{PhCH}_2$ ) $_2$  results in 70% yield from  $(\text{p-Cl-C}_6\text{H}_4)_2\text{C}(\text{CH}_2)_2\text{CHBr}$ .

in 20% yield from  $(\text{PhCH}_2)_2$  and in about 50% yield from  $\text{p-Br-C}_6\text{H}_4\text{CH}_2\text{CHBr}$ . C. J. West

Asymmetric syntheses. IV. The action of optically active nitrates on 2-bromofluorene. J. T. Thurston and R. L. Shriner. *J. Am. Chem. Soc.*, 57, 2163-6 (1935); cf. C. A. 28, 1451.—2-Bromofluorene (I) and *dl*-octyl nitrate (II) with  $\text{EtOK}$  in abs.  $\text{Et}_2\text{O}$  at 45° give 62.5% of 9-nitro-2-bromofluorene from the K salt, which gives a green color with  $\text{FeCl}_3$ . I and *d*-II at 35° give a K salt with a 4.48°, *l*-II gives a salt with  $-1.71^\circ$ . Acidification of the salts gives optically inactive *ac*-9-nitro-2-bromofluorene (III), yellow, m. 132°; *i*- $\text{FeCl}_3$  gives a green color; boiling with  $\text{EtOH}$  gives the normal NO $_2$  compd. (IV), m. 170°. Br gives 2,9-dibromo-*p*-nitrofluorene, yellow, m. 139°. The Ag salt of IV and MeI give an unstable nitronic acid, which readily decomposes into 2-bromofluorenone oxime (V) and  $\text{HCHO}$ . IV does not absorb Br, while III absorbs 50% of the theory, V absorbs 50.76% and the *dl*-K salt, 89.2-90.4%. C. J. West

Action of Grignard reagent on certain fuchsones. Percy L. Julian and Wm. J. Gist. *J. Am. Chem. Soc.*, 57, 2030-2 (1935).—Fuchsones or  $\text{p-MeOC}_6\text{H}_4\text{CPh}_2\text{Cl}$  with  $\text{MeMgI}$  gives  $\text{p-MeOC}_6\text{H}_4\text{CPh}_2\text{Me}$ ; this is a case of 1,6-addn. and confirms the results of Bayer and Villiger (*Ber.*, 36, 2703 (1903)). Diphenyl-4-methoxyphenylhydrazine m. 162°; the chloride m. 151°, with  $\text{MeMgI}$  there results 1,4-diphenyl-1,4-methoxyphenylhydrazine, b $_d$  220-30°, m. 144°, naphthofuchsones give the same product, thus indicating a 1,6-addn. None of the 1,6-addn products could be isolated from the reaction of these fuchsones with  $\text{PhMgBr}$ . Anthrafluorene gives a 1,2-addn. product,  $\text{MeMgI}$  yielding 9-methyl-10-benzohydroxyidene-9,10-dihydroanthran-9-ol, m. 223°; oxidation gives  $\text{Ph}_2\text{CO}$  and anthraquinone. 9-Phenyl-10-benzohydroxyidene-9,10-dihydroanthran-9-ol (I) gives a chloride, m. 220° (decompn.); mol. Ag gives in  $\text{C}_6\text{H}_6$ , a deep reddish brown soln., almost completely decolorized by atm. O. The peroxide could not be crystd. The bromide m. 165°. I is stable and does not rearrange into its isomer with the anthracene structure; its halogen derivs. likewise have apparently the dihydroanthracene structure. C. J. W.

Reaction of Grignard reagents with some succinic anhydrides. Chaim Weizmann, Ottlie Blum-Bergmann and Felix Bergmann. *J. Chem. Soc.*, 1935, 1370-1.— $\alpha$ - $\text{C}_6\text{H}_5\text{MgBr}$  and 10 g. succinic anhydride give 2.5 g. of  $\alpha$ - $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{CO}_2\text{H}$ , m. 133°; the  $\beta$ -isomer, m. 184-5°, was similarly prepd. Phenylsuccinic anhydride (8.7 g.) and  $\text{PhMgBr}$  give 2.2 g. of  $\gamma$ -hydroxy- $\alpha,\gamma$ -triphenylbutyric acid lactone, m. 162°;  $\alpha$ - $\text{C}_6\text{H}_5\text{MgBr}$  gives the  $\gamma,\gamma$ -di( $\alpha$ -naphthyl) analog, m. 211°; the acid portion was a brown sirup, whose reduction product b $_d$  265-70°. 3-Phenyl-1,2,3,6-tetrahydrophthalic anhydride and  $\text{PhMgBr}$  give 2-phenylmethyl-6-phenyl-1,2,3,6-tetrahydrobenzoic acid lactone, m. 227°; the acid portion could not be purified. C. J. West

Alcoholic ammonia as a reagent in the nitrosubene series. David E. Worrall. *J. Am. Chem. Soc.*, 57, 2209-301 (1935).—Isoxazoline oxides are prepd. by adding a small vol. of  $\text{EtOH}$  satd. with  $\text{NH}_3$  to a nitrosubene ether alone or mixed with  $\text{PhCH}_2\text{NO}_2$  or  $\text{p-Br-C}_6\text{H}_4\text{CH}_2\text{NO}_2$ ; no reaction was observed with carefully dried  $\text{NH}_3$  and nitrosubene in dry  $\text{Et}_2\text{O}$  after 48 hrs. 4-*p*-Bromophenyl-3,5-diphenyl deriv., m. 172-3°; 3,4-diphenyl-5-*p*-bromophenyl deriv., m. 213-15° (decompn.); 3-phenyl-4,5-bis(*p*-bromophenyl) deriv., m. 160-1°; 3,5-diphenyl-4-anisyl, m. 145-6°; 3-phenyl-4-anisyl-5-*p*-bromophenyl, m. 143-4°; 3,5-diphenyl-4-piperonyl deriv., m. 165-6°; 3-phenyl-4-piperonyl-5-*p*-bromophenyl deriv., m. 192-3°. Isoxazoles were prepd. by the action of  $\text{EtOH-KOH}$  on the oxides: 3,5-diphenyl-4-*p*-bromophenyl, m. 182-3°; 3-phenyl-4,5-bis(*p*-bromophenyl), m. 179-80°; 3-phenyl-4-anisyl-5-bromophenyl, m. 188-9°; 3,5-diphenyl-4-piperonyl, m. 227-8°; 3-phenyl-4-piperonyl-5-bromophenyl, m. 204-5°; 3,5-diphenyl-4- $\alpha$ -chlorophenyl, m. 127-8°.  $\alpha$ -Nitro- $\alpha$ -phenyl- $\beta$ -phenyl- $\beta$ -benzylaminoethane, m. 137-8°; dil. HCl gives  $\text{NH}_3$ ,  $\text{BzH}$  and  $\text{PhCH}_2\text{NO}_2$ ; this compd. probably results from the reaction of  $\text{PhCH}_2\text{NO}_2$  with a condensation of  $\text{NH}_3$  and



BzH (PhCH(OH)N CHPh) The action of EtOH-NH<sub>3</sub> upon *o*-chlorobenzylphenylnitromethane gives  $\alpha$ -nitro-*o*-phenyl- $\beta$ -*o*-chlorophenyl- $\beta$ -*o*-chlorobenzalaminomethane, m 144-5°. The role of NH<sub>3</sub> in these transformations is discussed. C. J. West

Pyrolysis. Elimination of two hydroxyl groups from a glycol Alexander Schonberg and Robert Michaelis. *J. Chem. Soc. 1935, 1403-4*—1,4-Bis(diphenylmethyl)- $\Delta^1$ -cyclohexadiene (Thiele and Balhorn, *Ber. 37, 1469*(1904)) yields with HNO<sub>3</sub> and AcOH Ph<sub>2</sub>C(OH). C<sub>11</sub>H<sub>13</sub>(OH)Ph<sub>2</sub> (Ullmann and Schläpfer, *Ber. 37, 2303* (1904)). Similarly it is believed that the action of HNO<sub>3</sub> in AcOH on chromanorufen (I) yields 5,12-dihydroxy-chromanorufen (II), decomp 260°. (cf. Lieberman and Barrolier, *C. A. 28, 3391*). Heating II at 290-310° gives I, also results on boiling in AcOH. II and Ph-NMe<sub>2</sub> in AcOH (heating 4 hrs) gives 5,12-bis(*p*-dimethylaminophenyl)chromanorufen, pale yellow, decomp 340°. I is unchanged on heating with PhNMe<sub>2</sub>. C. J. West

Grignard reactions with phthalic anhydrides. Chaim Weizmann, Ernst Bergmann and Felix Bergmann. *J. Chem. Soc. 1935, 1367-70*—*o*-Aryl benzoic acids are conveniently prepd from RMgX and C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>O and derivs, the formation of a HO acid or its lactone can usually be prevented by using excess of the anhydride, i. e., by adding the RMgX to the anhydride; yields are 70-80%. Of the 16 compds prepd, the following are new: *o*- $\beta$ -Naphthylbenzoic acid, m 128° (decompn) as hydrate, *di*- $\beta$ -naphthylphthalide, m 168°; *o*-(6-methoxy-2-naphthyl)benzoic acid, m 166°; *o*-phenanthrolylbenzoic acid, m 174-5°; 4-(57)-methoxy-2-benzoylbenzoic acid, from 4-MeOC<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>O and PhMgBr, m 167°; 2-*a*-naphthyl analog, m 198°; tetrachloro-*o*-4-anisoylbenzoic acid, m 186-7°; tetrachloro-*di*- $\beta$ -naphthylphthalide, m 144-5° (decompn); 2-*a*-naphthyl-3-naphthoic acid, m 228-9°;  $\beta$ -isomer, m 240°; 2-(4-methoxybenzoyl)-3-naphthoic acid, m 234°. Cyclization of some of these compds is difficult because of the ease of sulfonation. C. J. West

Some bromine derivatives of indene and indan. H. D. Porter and C. M. Suter. *J. Am. Chem. Soc. 57, 2022-6*(1935)—Indene bromohydrin (I), m. 126-8°, with P<sub>2</sub>O<sub>5</sub> in CCl<sub>4</sub> or on heating at 155-60° for 2 hrs, gives 45-55% of 2-bromindene, b<sub>p</sub> 125-7°, m 38-0° (Jacobi, *C. A. 25, 1517*, considered this the 1-isomer); refluxing with EtONa gives 2-indanone, the Mg deriv. with CO<sub>2</sub> gives indene-2-carboxylic acid, HBr in AcOH gives the dibromide (II), also prepd in 61% yield from I and 47% HBr by heating 3 hrs on the water bath; Br gives apparently 1,2,3-tribromindene, d<sub>4</sub> 212, n<sub>D</sub> 1.6615, oxidation gives *o*-C<sub>6</sub>H<sub>4</sub>(CO<sub>2</sub>H)<sub>2</sub> and hydrolysis yields a dibromosandal, n<sub>D</sub> 1.6350. Heating II at 200-10° for 1 hr. gives the 2-Br deriv. (16% yield); refluxing in dry tetralin gives a 30% yield II and abs. EtOH with C<sub>11</sub>H<sub>12</sub>N<sub>2</sub> refluxed 4 hrs, give 38% of 1-ethoxy-2-bromindene, b<sub>p</sub> 147-8°, n<sub>D</sub> 1.5515, d<sub>4</sub> 1.344; the structure was confirmed by transformation into 1-indanone oxime with NaNH<sub>2</sub> in liquid NH<sub>3</sub>, followed by steam distn. from 10% H<sub>2</sub>SO<sub>4</sub> and addn of NH<sub>4</sub>OH HCl. Br gives a mixt. of 1,2,3-tribromindene and 2,2-dibromo-1-indanone; refluxing with 10% H<sub>2</sub>SO<sub>4</sub> gives 1-indanone (III). The prepn of *trans*-indene glycol (IV) from I is described. III results in 80% yield by refluxing I with 7% H<sub>2</sub>SO<sub>4</sub> for 10 hrs, while IV in 1 hr. gives 71% of the 2 isomer. C. J. West

Synthesis of phenanthrene and hydrophenanthrene derivatives. II. The hydrocarbon synthesis. Louis F. Fieser and Emanuel B. Hersberg. *J. Am. Chem. Soc. 57, 2192-6*(1935); cf. *C. A. 29, 7972*—3,4-Dihydro-naphthalene-1,2-dicarboxylic anhydride and butadiene, heated 85 hrs. at 100°, give 63% of 1,4,9,10,11,12-hexahydrophenanthrene-11,12-dicarboxylic anhydride (I), b<sub>p</sub> 160-70°, m 83-5-4°; 2,3-dimethylbutadiene, heated 20-25 hrs. at 100°, gives 94-7% of the 2,3-di-Me deriv. (II), b<sub>p</sub> 175-85°, m 95-6°. 2,3-Dimethyl-1,4,11,12,13,14-hexahydrophenanthrene-13,14-dicarboxylic anhydride, m 195-65°. 1,2-Dihydrophenanthrene-3,4-dicarboxylic anhy-

dride and butadiene at 100° for 100 hrs. give 76% of 5,6-benzo-1,4,9,10,11,12-hexahydrophenanthrene-1,11,12-dicarboxylic anhydride, m. 161 5-2°; 2,3-di-Me deriv. m 128-8 5° (68% yield). Soln in 6 N KOH and acidification gives the dicarboxylic acid (III) corresponding to II, m 170-7°; II and MeOH with NaOH gives the Me ester of III, m. 157-0°; CH<sub>3</sub>N<sub>3</sub> gives the di-Me ester, m 93 5-4°; catalytic reduction of II gives the 1,2,3,4,9,10,11,12-octahydro deriv. m. 117-18°; the corresponding acid, with 0.5 mol C<sub>6</sub>H<sub>6</sub>, m. 183-4°. Definite products could not be obtained from heating II with KOH at 325-420° but I gives hexahydrophenanthrene, b<sub>p</sub> 179-80° (55% yield); Se gives 86% of phenanthrene. Heating a mixt. of tetra- and hexa hydro derivs. from II and KOH gives 2,3-dimethylphenanthrene, m. 78-8 5°; picric acid, light yellow, m 146-7°; quinine, orange, m. 237 5-8 5° (guinoxaline deriv. m 208-9°). C. J. West

Phenanthrene series IX. Amino alcohols derived from 1,2,3,4-tetrahydrophenanthrene. Frisch Mosettig and Alfred Burger. *J. Am. Chem. Soc. 57, 2189-92* (1935); cf. *C. A. 29, 5106*—1-Keto-1,2,3,4-tetrahydrophenanthrene (1-tetantrone) gives 90% of the 2-Br deriv. in 84-5° (all m. ps. cor.); 2-dimethylamino deriv. (HCl salt) (I), m 218-20° (decompn), 75% yield (picric acid, yellow, m 180° (decompn)); 2-diethylamino deriv. (HCl salt) (II), m. 143-8°, 33%, 2-piperidino deriv. yellow, m 138-40°, 81%; 2-(1,2,3,4-tetrahydroisoquinoline) deriv. (HCl salt), pale yellow, m 226-7° (decompn), 56%; catalytic reduction of I gives 2-dimethylamino-1-hydroxy-1,2,3,4-tetrahydrophenanthrene, m 105-8° (HCl salt, m 223-4° (decompn)); Bz deriv. (HCl), m. 170-81° (decompn); diethylamino analog, m 70-2° (HCl salt, m 221-3° (decompn)); 3,5-dinitrobenzoyl deriv. pale yellow, m 203-11° (decompn); 2-piperidino analog (III), m 121-6° (HCl salt, m 259° (decompn)); 1,2,3,4-tetrahydroisoquinoline analog, m. 155-6° (HCl salt, m 227° (decompn)). 3-Bromo-4-keto-1,2,3,4-tetrahydrophenanthrene, m 104-5°, 87%, 3-dimethylamino deriv. (HCl), m 208-10° (decompn); 3-diethylamino deriv. (HCl), m 184-5° (decompn); 3-piperidino deriv. (HCl), m 248-50° (decompn); 3-(1,2,3,4-tetrahydroisoquinoline) deriv. (HCl), m 164-6° (decompn); 3-dimethylamino-4-hydroxy-1,2,3,4-tetrahydrophenanthrene (HCl), m. 230° (decompn) (Bz deriv. m 80-90°, HCl salt, m 177-8°); 3-diethylamino deriv. (HCl), m 239 5-40° (decompn) (picric acid, yellow, m 206-8° (decompn)); Bz deriv. (as picric acid), yellow, m 173-4° (decompn); 3,5-dinitrobenzoyl deriv. pale yellow, m 216-17 5°; 3-piperidino deriv. (HCl), m 246-7° (decompn) (Bz deriv. (HCl), m 181-2° (decompn)); 3-(1,2,3,4-tetrahydroisoquinoline) deriv. (HCl) (IV), m 230-1° (decompn) 1-Hydroxyphenanthrene, m 153°, results in 50% yield from 2-bromo-1-tetantrone with PhNEt<sub>3</sub> for 4 hrs and in 25% yield by heating 1-tetantrone with Se at 295° for 14 hrs and as a by-product from II. Another by-product of II is the compd C<sub>11</sub>H<sub>12</sub>OH OC<sub>6</sub>H<sub>4</sub>II, m. 119°. 4-Hydroxyphenanthrene (V), m 113°, compd. C<sub>11</sub>H<sub>12</sub>OH OC<sub>6</sub>H<sub>4</sub>II, m 107°; this results from V and 4-tetantrone. The effective analgesic dose of III is 20 mg per kg, of IV, 15 mg per kg, comparable with the effective doses of 10 and 20 mg for codeine and pseudocodeine, resp. C. J. West

Synthesis of substances related to the sterols. IV. Some derivatives of chrysene. H. J. Lewis, G. R. Ramage and Robert Robinson. *J. Chem. Soc. 1935, 1412-14*, cf. *C. A. 29, 7966*—In the reduction of 4-MeOC<sub>6</sub>H<sub>4</sub>CHCO<sub>2</sub>Me (*C. A. 27, 4230*), Me  $\beta$ , $\gamma$ -diisopropylidipate-b may be distd off, b<sub>p</sub> 285°, m 67°; the free acid (I) m 189°. Ring closure of I in C<sub>11</sub>H<sub>12</sub> gives 2,11-diketo-5,14-dimethoxy-1,2,9,10,11,18-hexahydrochrysene-b, m. 220°, this may also be prepd by hydrolysis of the crude mixt from the reduction. Clemmensen reduction gives 5,14-dimethoxy-1,2,9,10,11,18-hexahydrochrysene b, m 140-1° Reduction of 50 g 3-MeOC<sub>6</sub>H<sub>4</sub>CHCO<sub>2</sub>Me with Al-Hg in Et<sub>2</sub>O gives mainly Me  $\beta$ -3-methoxyphenylpropionate, b<sub>p</sub> 148°, with 1 g of Me  $\beta$ , $\gamma$ -di-3-methoxyphenylidipate, m. 139 5°; the free acid (II) m 247-8°, heating II

with 50%  $\text{H}_2\text{SO}_4$  at  $100^\circ$  for 2.5 hrs. gives 2,11-diketo deriv., m. about  $350^\circ$ , of 6,15-dimethoxy-1,2,9,10,11,18-hexahydrochrysene, m.  $109^\circ$ . Piperonal and AcOMe with Na give Me 3,4-methylenedioxy-*cinnamole*, m.  $133-4^\circ$ ; the only reduction product is Me  $\beta$ -3,4-methylenedioxy-phenylpropionate, b.  $165-70^\circ$ , m.  $31-5^\circ$ . 3,4-(MeO) $_2$ - $\text{C}_6\text{H}_3\text{CH}_2\text{CH}_2\text{CO}_2\text{Me}$  on reduction yields Me  $\beta$ -3,4-dimethoxyphenylpropionate, b.  $175-80^\circ$ , m.  $37^\circ$ , and Me  $\beta$ -7-di-3,4-dimethoxyphenylpropionate, m.  $181^\circ$ ; the free acid m.  $255^\circ$ . 80%  $\text{H}_2\text{SO}_4$  gives the 2,11-diketo deriv., m.  $330^\circ$  (decompn.) of 5,6,13,14-tetramethoxy-1,2,9,10,11,18-hexahydrochrysene, m.  $204^\circ$ . Air oxidation of the reaction product of cis-2,11-diketo-1,2,9,10,11,18-hexahydrochrysene and MeMgI yields 2,11-dimethylchrysene, m.  $237^\circ$ , 1,7,5-transbenzene comp., reddish orange, m.  $222^\circ$ , styphnate, orange, m.  $207^\circ$  (decompn.). V. Condensation of phenylsuccinic anhydride with veratrole under the influence of aluminum chloride. Robert Robinson and P. C. Young. *Ibid* 1414-16.—Phenylsuccinic anhydride (4 g.), 0.6 veratrole, 7.5 g.  $\text{AlCl}_3$  and 15 cc.  $\text{PhNO}_2$ , allowed to stand 48 hrs., give 84% of  $\beta$ -veratroyl- $\alpha$ -phenylpropionic acid, m.  $142-3^\circ$ , mono- $\text{NO}_2$  deriv., pale yellow, m.  $170-9^\circ$ , this contains the  $\delta$ -isoveratroyl group. The Me ester, m.  $100-100.5^\circ$ , results by esterification of the acid and also from  $\text{PhCH}(\text{CO}_2\text{Me})\text{CH}_2\text{COCl}$  and veratrole with  $\text{AlCl}_3$  in  $\text{CS}_2$ . Reduction with  $\text{Zn-Hg}$  and coned.  $\text{HCl}$  yields  $\alpha$ -phenyl- $\gamma$ -veratrylbutyric acid, m.  $83^\circ$ , with 80%  $\text{H}_2\text{SO}_4$ , there results 1-keto-6,7-dimethoxy-2-phenyl-1,2,3,4-tetrahydronaphthalene, m.  $140-1^\circ$ ; with  $\text{BrCH}_2\text{CO}_2\text{Et}$  and  $\text{Zn}$  in  $\text{C}_6\text{H}_6$ , there results probably Et 6,7-dimethoxy-2-phenyl-3,4-dihydronaphthylacetate, m.  $159-61^\circ$ , the acid does not give a ppt with  $\text{Br-H}_2\text{O}$  and hence is probably a  $\beta$ , $\gamma$ - and not an  $\alpha$ , $\beta$ -unsat. acid. VI. Robert Robinson and James Walker. *Ibid* 1530-3.—Continuation of the work in part V was abandoned when it was found that the condensation of  $p$ -methoxyphenylsuccinic anhydride with veratrole was not a smooth process. Reduction of Et 3,4-dihydro-1-naphthylacetate with Na in EtOH gives 68% of  $\beta$ -tetrahydro-1-naphthylalkyl alc., b.  $158-60^\circ$ , and not vinyl-dihydronaphthalene. The synthesis of Me 2- $\beta$ -1'-naphthylalkyl-1-methylcyclopentan-2-ol-1-carboxylate, b.  $206-14^\circ$ , n $_D^{20}$  1.5340, is described (the intermediate compds. have been described); its methylamide m.  $100^\circ$ ; the ester could not be dehydrated. Hydroxymethylene-cyclohexanone and  $\text{Me}_2\text{SO}$ , give the  $O$ -Me ether, b.  $75-80^\circ$ , n $_D^{20}$  1.4554; it decomposes rapidly, turning green in a few min. and dark green after 1-2 hrs.; with  $\text{AcCH}_3\text{CO}_2\text{Et}$  and EtONa, followed by condensation and hydrolysis with EtONa, 6-hydroxytetralin-7-carboxylic acid results. The aromatic nucleus of 2-methylchroman could not be reduced catalytically. VII. Wm. Sage Rapson and Robert Robinson. *Ibid* 1533-43.—Et 1-methyl-2'-cyclopentene-1-carboxylate (I), b.  $70-1^\circ$ , results from the HO ester and  $\text{P}_2\text{O}_5$  in  $\text{C}_6\text{H}_6$ ; the acid b.  $110^\circ$ . Reduction of the Et ester gives 70% of 1-methyl-2'-cyclopentyl-1-carbinol (II), b.  $102-5^\circ$  ( $p$ -nitrobenzoate, m.  $67^\circ$ );  $\text{PCl}_5$  in petr. ether gives the chloride, b.  $40-50^\circ$ , which does not condense with  $\text{AcCH}_3\text{NaCO}_2\text{Et}$  in EtOH or  $\text{C}_6\text{H}_6$ . Dehydration of II with  $\text{KHSO}_4$  gives a dihydro-luolen(?) $\beta$ , b.  $112-13^\circ$ . Electrolytic reduction of 35 g. of  $\text{m-MeOC}_6\text{H}_4\text{CHO}$  (4 amps., c. d. 0.025 amp./sq. cm.) for 3 hrs. gives 20 g. of  $\text{m-MeOC}_6\text{H}_4\text{CH}_2\text{OH}$  (III) and 9 g. of 3,3'-dimethoxydihydrobenzoin  $\alpha,\alpha'$ -di-Me ether, m.  $112-13^\circ$ . III was converted through the chloride and nitrile to Et  $m$ -methoxyphenylacetate, which was reduced with Na and EtOH to  $\beta$ - $m$ -methoxyphenylalkyl alc., b.  $135-7^\circ$ ; the chloride, b.  $129-30^\circ$ , reacts sluggishly with  $\text{AcCH}_3\text{NaCO}_2\text{Et}$ ; Et  $\beta$ - $m$ -methoxyphenylacetate b.  $180^\circ$ ; hydrolysis gives  $\gamma$ - $m$ -methoxyphenylpropyl Me ketone, b.  $169^\circ$  (semicarbazone, m.  $100^\circ$ ); condensation with I did not give a pure  $\beta$ -diketone. The K salt of Et trans-cyclopentane-1-carboxylate-2-cyanoacetate and  $\text{PhCH}_2\text{CH}_2\text{Br}$  give Et  $\alpha$ -cyano- $\alpha'$ -(trans-2-carboxycyclopentyl)- $\gamma$ -phenylbutyrate, b.  $195-200^\circ$ ; hydrolysis with coned.  $\text{HCl}$  (boiling 30 hrs.) gives  $\alpha$ -cyano- $\alpha'$ -(trans-2-carboxycyclopentyl)- $\gamma$ -phenylbutyric acid, m.  $210^\circ$  (decompn.); boiling with  $\text{H}_2\text{SO}_4$  and AcOH gives  $\alpha$ -(trans-2-carboxy-

cyclopentyl)- $\gamma$ -phenylbutyric acid, m.  $160-1^\circ$ ; the trans-anhydride, m.  $112^\circ$ , on heating at  $250^\circ$  for 15 min., gives the cis-anhydride, which did not crystallize; the cis-acid m.  $133^\circ$ . The trans-acid with  $\text{H}_2\text{SO}_4$  gives 1-keto-2-(trans-2'-carboxycyclopentyl)-1,2,3,4-tetrahydronaphthalene, m.  $164-5^\circ$ ; heating with  $\text{Ac}_2\text{O}$  and AcONa and decoupling of the anhydride gives the cis-isomer, m.  $155-6^\circ$ . The Me ester of the trans-acid m.  $45^\circ$ ; this did not react with  $\text{BrCH}_2\text{CO}_2\text{Et}$  and  $\text{Zn}$ . Both acids react with  $\text{SOCl}_2$  in  $\text{CHCl}_3$  to yield the trans- and cis-forms of 1-hydroxy-2-(2'-carboxycyclopentyl)-3,4-dihydronaphthalene lactone, m.  $162^\circ$  and  $60^\circ$ , resp. The transform monoclinic needles elongated along  $a$ , with the faces  $\{011\}$ ,  $\{100\}$  developed, the dimensions of the unit cell are  $a = 8.2$ ,  $b = 23.4$ ,  $c = 13.6$ ,  $\beta = 68^\circ$ ; with a density of about 1.315, the no. of mols. in the cell is 8, the cis-form crystallizes in triclinic plates,  $a = 7.27$ ,  $b = 16.75$ ,  $d(001) = 11.0$ ,  $\gamma = 75^\circ$ ,  $d(100)$ , no. of mols. in the unit cell, 4. Catalytic reduction of the trans-lactone yields 2-(2'-trans-carboxycyclopentyl)-1,2,3,4-tetrahydronaphthalene, m.  $107^\circ$ . Et cyclopentanone-1-carboxylate in  $\text{PCl}_5$  in light petroleum gives Et 2-chloro-2-cyclopentene-1-carboxylate, b.  $95-8^\circ$ , and a small quantity of the acid, m.  $115$  to  $116^\circ$ ; this ester did not condense with  $\text{AcCH}_3\text{NaCO}_2\text{Et}$  or  $\text{CH}_3\text{Na}(\text{CO}_2\text{Et})$ ,  $m\text{-HOC}_6\text{H}_4\text{CHO}$  and  $\text{PhCH}_2\text{CH}_2\text{Cl}$  with Na give  $m$ -benzoylbenzaldehyde, b.  $215-18^\circ$ , m.  $54^\circ$ , hippuric acid and  $\text{Ac}_2\text{O}$  give  $m$ -benzoylbenzylidenebenzylisoxazolone, yellow, m.  $129^\circ$ ; hydrolysis gives  $m$ -benzoylbenzylidenebenzylisoxazolone, m.  $145^\circ$  but it decomposes on keeping. alk.  $\text{H}_2\text{O}_2$  gives  $m$ -benzoylphenylacetic acid (IV), m.  $120^\circ$ . Since  $\delta$ -benzoylation could not be avoided in further work methoxylated intermediates were used. The dry Na salt of IV, 6,3-O $_2$ N-(MeO) $_2\text{C}_6\text{H}_3\text{CHO}$  (V) and  $\text{Ac}_2\text{O}$ , heated on the water bath for 24 hrs., give 2-nitro-5-methoxy- $\alpha$ -( $m$ -benzoylphenyl)cinnamic acid, pale yellow, m.  $148^\circ$ , 2-NH $_2$  deriv., m.  $137^\circ$ ,  $\text{m-MeOC}_6\text{H}_3\text{CH}_2\text{CO}_2\text{Na}$ , V and  $\text{Ac}_2\text{O}$  give 2-nitro-5-methoxy- $\alpha$ -( $m$ -methoxyphenyl)cinnamic acid, yellow, m.  $148^\circ$ , 2-NH $_2$  deriv., m.  $185^\circ$ , the diazo reaction gives 63% of a mixt. of 44% 2,7-dimethoxyphenanthrene-9-carboxylic acid, pale yellow, m.  $255^\circ$ , and 56% of the 2,5-isomer, pale yellow, m.  $191^\circ$ . Decarboxylation gives 2,7-dimethoxyphenanthrene and the 2,5-isomer, m.  $117^\circ$  (picrate, orange-red, m.  $151-6^\circ$ ). 2,5-Dihydroxyphenanthrene, m.  $180^\circ$ , di-Ac deriv., m.  $144^\circ$ . In the prepn. of  $\beta$ -2-hydroxy-1-naphthylethyl Me ketone a by-product is 2-methyl-5,6-(1,2-naphtho)- $\gamma$ -pyran, b.  $190^\circ$ , m.  $41-5^\circ$ . The K deriv. of Et cyclopentanonecarboxylate and octyl bromide, boiled 7 hrs. in  $\text{CaH}_2\text{Me}$ , give Et 1-octylcyclopentan-2-one-1-carboxylate, b.  $157-65^\circ$  (85% yield) (semicarbazone, m.  $117^\circ$ ), hydrolysis with Ba(OH) $_2$  gives  $\alpha$ -octyladipic acid, m.  $74^\circ$ , and 2-octylcyclopentanone, b.  $135-8^\circ$  (semicarbazone, m.  $183^\circ$ ); reduction of the ketone yields 2-octylcyclopentanol, b.  $161^\circ$ ,  $140^\circ$ ; bromide, b.  $135-40^\circ$ . Octyl- $\delta$ -cyclopentene and  $\text{MeO}_2\text{CCH}_2\text{CH}_2\text{COCl}$  with  $\text{SnCl}_4$  or  $\text{AlCl}_3$  give 2-( $\beta$ -carbomethoxypropionyl)-1-octyl- $\delta$ -cyclopentene, b.  $173-7^\circ$ , n $_D^{20}$  1.4518. Work of R. Hurt on cyclopentanone-2- $\beta$ -propionic acid is described. Refluxing the acid with  $\text{AcCl}$  gives the enolic lactone, b.  $116-7^\circ$ ; cyclopentanol-2- $\beta$ -propionic acid lactone, b.  $139-9^\circ$ .  $\text{PhCH}_2\text{CH}_2\text{CH}_2\text{Ac}$  and 85%  $\text{H}_2\text{SO}_4$  at  $0^\circ$  give 74% of 1-methyl-3,4-dihydronaphthalene, b.  $107-8^\circ$ . C. J. West

Amino alcohols derived from dibenzofuran. Erich Mossetig and Richard A. Robinson. *J. Am. Chem. Soc.* 57, 2186-9 (1935).—2-Acetyldibenzofuran (I) and Br in abs. Et $_2\text{O}$  at  $0^\circ$  give 55% of the 2- $\omega$ -bromoacetyl deriv., m.  $105-6^\circ$ ; MeNH $_2$  in Et $_2\text{O}$  gives 90% of the 2-(2'-dimethylamino-1-ketocetyl) deriv. (II), m.  $82-3^\circ$  (HCl salt, with 1 mol.  $\text{H}_2\text{O}$ , m.  $212-35^\circ$ ); the HCl salt of the diethylamino deriv. m.  $200-12^\circ$ ; the 2-piperidino deriv. m.  $97-100^\circ$  (HCl salt, m.  $255-65^\circ$ ); the HCl salt of the 2-ethylamino deriv. m.  $254-6^\circ$  (decompn.). Reduction of 2-isotonitrosocetyl-dibenzofuran gives 64% of 2-(2-amino-1-ketocetyl)dibenzofuran, the HCl salt of which decomposes  $251-6^\circ$ . The catalytic reduction of II gives 90% of the 2-(2-dimethylamino-1-hydroxyethyl) deriv., m.  $88-9^\circ$  (HCl salt, m.  $173-4^\circ$ ; benzoic acid ester, m.  $99-100^\circ$ );

diethylamino analog, m. 75-6° (HCl salt, m. 157-9°); 1,2-piperidino analog, m. 116-17.5° (HCl salt, m. 250-1°); benzoic acid ester, m. 119°; 2-ethylamino analog, m. 99-101° (HCl salt, m. 210-19.5°); 2-amino analog, m. 132° (90% yield) (HCl salt, m. 261° (decompn.)). Catalytic reduction of I gives 90% of 2-(1-hydroxyethyl)-dibenzofuran, m. 63-4°; 2-propionylidibenzofuran, m. 101-5-5° (semicarbazone, m. 184-6°); 2-[2-(Methylamino)-1-ketoethyl]dibenzofuran-HCl, m. 235-50°; Methylbis(2-dibenzofurylmethyl)amine-HCl, m. 235-45°.

The NH<sub>2</sub> alics do not give a morphine-like picture in the cat but are generally more analgesic and more toxic than the corresponding NH<sub>2</sub> alics in the phenanthrene series.

C. J. West

Arsenicals containing the furan nucleus II. Action of chlorine. Some substituted furan arsenicals. Wesley G. Lowe and Chiff S. Hamilton. *J. Am. Chem. Soc.* 57, 2314-17 (1935), cf. C. A. 29, 5109°—Chlorination of difuryldichloroarsine, difuryldichloroarsine (I) and trifurylarsine yields 2-chlorofuran tetrachloride, slightly yellow, pungent penetrating odor, d<sub>4</sub><sup>20</sup> 1.620, satd. EtOH-KOH splits off 40% of the Cl; the action of aq. AgNO<sub>3</sub> is complex, varying conditions of temp. and acidity leading to a pptn of 60, 70 and 75% of the total Cl as AgCl. The chlorination product of I gives 8% of difurylarsinic acid, m. 138°, and a very small quantity of trifurylarsinic dichloride, m. 132°. 5-Chloropropionic acid (Na salt) and HgCl<sub>2</sub> give 65% of 5-chloro-2-chloromercaptofuran (II), m. 181°, AsCl<sub>3</sub> in C<sub>6</sub>H<sub>6</sub> gives tri-5-chlorofurylarsine (III), m. 63°, 5-Br analog (IV) of III, m. 106° (40% yield); the action of HgCl<sub>2</sub> upon III in EtOH gives II, IV behaves similarly, these results indicate that the aromaticity of the furan nucleus is less in these compds than in trifurylarsine itself. The chlorination of furan arsenicals is regarded as competitive oxidation of As and the furan nucleus.

C. J. West

Dibenzofuran VII Derivatives of tetrahydrodibenzofuran Henry Gilman, E. W. Smith and L. C. Cheney. *J. Am. Chem. Soc.* 57, 2095-9 (1935), cf. C. A. 29, 5109°—1,2,3,4-Tetrahydrodibenzofuran (I) yields a 7-NO<sub>2</sub> deriv. identical with the 8-NO<sub>2</sub> compd. of Ebel (C. A. 23, 2439) and the 8-nitrohexahydrodibenzofuran of von Braun (C. A. 17, 1958). Br in AcOH gives 3-nitrodibenzofuran. The amine is also the same as the aminohexahydrodibenzofuran of v. Braun, its Ac deriv. m. 146° (not 123°). I with Ac and SnCl<sub>4</sub> in C<sub>6</sub>H<sub>6</sub> yield a 7-Ac deriv. b.p. 210°, m. 66-7°, again identical with v. Braun's product, I<sub>1</sub> in NaOH gives 57% of 1,2,3,4-tetrahydrodibenzofuran-7-carboxylic acid, m. 247-8° (Me ester, m. 72-5-3°). Br in AcOH gives a quant. yield of 7(or 3)-dibenzofurancarboxylic acid (Me ester, m. 138-5°). I and C<sub>6</sub>H<sub>5</sub>OH in CCl<sub>4</sub> give 38% of 3a 1,2,3,4-tetrahydrodibenzofuran-7-sulfonate, the acid chloride with NH<sub>4</sub>OH yields 77% of the sulfonamide, m. 207.5-8.5°, dehydrogenated to dibenzofuran-3-sulfonamide, m. 241-3°, this was also prepd. from 3-aminodibenzofuran, through dibenzofuran-3-sulfonic acid, waters without melting, and its chloride, m. 148-5°. I and PhLi (p-MeC<sub>6</sub>H<sub>4</sub>Li or BuNa), followed by the action of CO<sub>2</sub>, give 4-8% of the 6-carboxylic acid, m. 197°; Br in AcOH gives dibenzofuran-4-carboxylic acid, m. 207-8°, reduction of this acid with Na and EtOH appears to give 1,2,3,4-tetrahydrodibenzofuran-4-carboxylic acid, m. 168°. The 6-MeO deriv. of I with S at 275° gives 4-methoxydibenzofuran. The various reduced dibenzofurans are discussed, there is no doubt of the existence of the tetrahydro and the perhydro derivs (C. A. 27, 2459), a dihydro deriv has not been definitely established and there is a question about the hexahydro deriv, which may be known as a percent type.

C. J. West

Synthesis of rotenone and its derivatives VII Tetrahydrobutyric acid. Alexander Robertson and Geo. L. Rusby. *J. Chem. Soc.* 1935, 1371-3, cf. C. A. 29, 6797°—Tetrahydrobutanol (C. A. 27, 5739) (5 g.) with 7n(CN), HCN and HCl in Et<sub>2</sub>O gives 4 tetrahydrobutyric aldehyde (2,4-dihydroxy-3-isooxymethylbenzaldehyde) (I), m. 112-5°, reddish violet FeCl<sub>3</sub> reaction, semicarbazone, m. 104°, diacetate (II), m. 47.5°. Oxidation of II in

Me<sub>2</sub>CO with KMnO<sub>4</sub> yields the diacetate, m. 140-1°, of tetrahydrobutyric acid (III), m. 203° (Haller and LaForge, C. A. 26, 3253). The 4-Me ether (IV) of I, b.p. 180°, reddish violet FeCl<sub>3</sub> reaction, semicarbazone, m. 193°, the acetate is a viscous oil, oxidation of which yields the Me ether of III, m. 154-5°. IV and 4-methoxyacetoveratone with HCl in AcOEt gives 3,7,3',4'-tetramethoxy-8-isoamylavilyum chloride, deep red prisms, the ferrichloride, red plates with green reflex, m. 131-2°. The azlactone of asarylaldehyde, boiled with 12% aq. NaOH, satd. with SO<sub>2</sub>, filtered and heated with concd. HCl, yields 2,4,5-trimethoxyphenylpyruvic acid, pale yellow, m. 198° (decompn.), through the oxime there was prepd. 2,4,5-trimethoxyphenylacetone, m. 85°, soln. of the crude oxime in boiling water gives mainly the nitrile.

C. J. West

Indole series. VI. Synthesis of ketotryptophan and further studies of 3-alkylation of oxindoles Percy L. Julian, Josef Pkl and Frank E. Wautz. *J. Am. Chem. Soc.* 57, 2020-9 (1935), cf. C. A. 29, 3074°—1,3-Dimethyloxindole (49 g) and bromoacetal (55 g) with EtONa, refluxed 1 hr., give 55% 1,3-dimethyloxindolylacetal, b.p. 182-5-3°; hydrolysis with 5% HCl at room temp. gives a quant. yield of 1,3-dimethyloxindolylacetaldehyde (I), b.p. 177-8° (semicarbazone, m. 206°), 64 g I and 35 g. NaHSO<sub>3</sub> in H<sub>2</sub>O-Et<sub>2</sub>O with 25 g KCN give 50 g of the cyanohydrin, m. 142°; alc. NH<sub>4</sub>, followed by HCl, gives 6-1,3-dimethyloxindolylamine, m. 188° (decompn.), above its m. p. it yields 1,3-dimethyloxindolylethylamine. Condensation of oxindole with bromoacetal presented difficulties and even 1-methyloxindole did not react smoothly. 1-Methyl-5-hydroxyoxindole, m. 187°, 5-EtO deriv. m. 92°. 3-Acyloxindoles were prepd. by adding 1 mol. oxindole and 12 mols of ester to a 10% soln of EtONa contg. 1.3 mols. Na and decomp. the Na salt with dil. HCl. These are catalytically reduced to 3-alkyloxindoles, yields being 50-70%. 1-Methyl-3-acyloxindole, m. 109°. 1-Methyl-3-ethyloxindole, m. 169°. 1,3-Dimethyl-3-acyloxindole, m. 79°. 1-Methyl-3-carbethyloxindolylacetaldehyde (II), m. 67°. 6-1-methyloxindolylpropionic ester (III), b.p. 160°. 1-Methyl-3-dimethylaminoacyloxindole, m. 219°. 1-methyl-3-β-dimethylaminoethyloxindole (IV), b.p. 185° (picrate, m. 103°); the 5-EtO analogs, m. 196° and b.p. 221° (V) (picrate, m. 157°). 1-Methyl-2-methoxy-3-formyloxindole gives 1-methyl-2-methoxyoxindolylcarbinol, m. 62°. II and III are of interest in the synthesis of ketotryptophan, since oxindole may be condensed with phthalimidomalonic ester, reduced and hydrolyzed IV and V are related to bufotenine.

C. J. West

Autoxidation phenomena in the indole series P. Pfeiffer and H. L. de Waal. *Ann.* 520, 185-200 (1935).—1-Phenylindan-3-one (I), m. 78°, yields a 2-benzal deriv., straw-yellow, m. 158°; anisal deriv., bright yellow, m. 174°. 3',4'-dimethoxy-2-benzal deriv., deep yellow, m. 174°. 2-Isotroster deriv., from I and iso-AMNO<sub>3</sub> with EtONa-EtOH, concd. HCl, or simply on standing, decomposes 201-2°, NH<sub>4</sub>OH gives 1-phenylindandione dioxime, pale gray, m. 196-6-5° (decompn.). I and PhNO in 90% EtOH with NaOH give 80% of the monanil (II) of III, C<sub>11</sub>H<sub>9</sub>ON, deep blue-violet, m. 149-50°, and a little of a compd., C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N, yellow, m. 223°. II with concd. H<sub>2</sub>SO<sub>4</sub> (cooling) gives 75% of 1-phenylindandione (III), m. 137-8°, semicarbazone, yellow, m. 252° (decompn.), azine, straw-yellow, m. 173°. Oxidation of II with H<sub>2</sub>O<sub>2</sub> in EtOH gives the compd., C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N, citron-yellow, m. 180°, with 20% KOH in a little EtOH this yields an anilide acid, o-HO,CC<sub>6</sub>H<sub>4</sub>CHPhCONHPh or o-PhNHCO,CC<sub>6</sub>H<sub>4</sub>CHPhCO<sub>2</sub>H, m. 192°; warming this with AcOH, H<sub>2</sub>SO<sub>4</sub> 1 hr. gives the dicarboxylic acid o-HO,CC<sub>6</sub>H<sub>4</sub>CHPhCO<sub>2</sub>H, m. 157-8°. I and p-MeN-C<sub>6</sub>H<sub>4</sub>NO give the dimethylammonianil of III, deep blue-violet, m. 148-50°, autooxidation yields the isooquinole deriv., C<sub>11</sub>H<sub>9</sub>CPH(OH)CO N(C<sub>6</sub>H<sub>4</sub>NMe<sub>2</sub>)CO, orange-

red (C<sub>6</sub>H<sub>5</sub>) or orange (EtOH), m. 214-5°, with KOH this yields the isooindole deriv. (IV), C<sub>11</sub>H<sub>9</sub>CHPh N-

( $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_5$ )  $\text{CO}_2$ , pale yellow, m.  $270^\circ$  (sublimate, m.  $223-4^\circ$  (decomp.)); heating with  $\text{AcOH-H}_2\text{SO}_4$  for 6 hrs. gives phenylphthalide (VI). IV yields in 50% yield from V and  $p\text{-H}_2\text{NCH}_2\text{NMe}_2$  (VII). VI and  $p\text{-H}_2\text{NCH}_2\text{COOH}$  give the compd.,  $p\text{-(Me}_2\text{NCH}_2\text{NHCOO)C}_6\text{H}_4\text{C}_6\text{H}_4\text{NMe}_2$ , yellow, m.  $243^\circ$ . The methanoid of III, blue-white, m.  $118^\circ$ ; amination gives an *isomeric* blue-white,  $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_5$ , m.  $201^\circ$ , prepd. from VI and  $p\text{-MeOCH}_2\text{NMe}_2$  in 80% yield. C. J. West.

Ergot alkaloids. VI. Lysergic acid. Walter A. Jacob and Lyman C. Craig. *J. Biol. Chem.* 111, 455-63 (1935). d. c. d. 29.  $585.5^\circ$ —Dihydrolysergic acid (0.4 g) was ground with KOH (2 g) and fused at  $293-300^\circ$  for 20 min. in a stream of  $\text{H}_2$ . The volatile products were condensed in an air trap and then in  $\text{HCl}$ . The material removed from the  $\text{H}_2\text{O}$  soln. of the residual melt by extraction with  $\text{Et}_2\text{O}$  was combined with the condensate in the air trap and sublimed. The sublimate was dissolved in  $\text{Et}_2\text{O}$  and exhd. with d. l.  $\text{HCl}$ . Fractional distn. of the part remaining in the  $\text{Et}_2\text{O}$  gave a petr. ether-insol. residue, m.  $192^\circ$ , b.p.  $240^\circ$ , of undetd. structure, and a petr. ether-sol. substance (pure, m.  $146-50^\circ$ ), apparently 2-methyl-3-dimethyl- of which a synthetic prepn. has  $145-50^\circ$ . From the  $\text{HCl}$  ext., after making alk., a residue,  $\text{C}_{10}\text{H}_{11}\text{N}_3$ , b.p.  $145-50^\circ$ , m.  $68^\circ$  (petr. ether) (pure, m.  $193-200^\circ$ ), of undetd. structure was obtained from the original alk. ext., after acetication, exhd. with  $\text{Et}_2\text{O}$  and dam. up to  $80^\circ$ ,  $p\text{-MeCH}_2\text{COOH}$ . I was isolated as the  $p$ -bromophenacyl ester. The residue remaining from I apparently was a mixt. of mid-chain bicyclic acids. From the  $\text{HCl}$  trap in the fusion,  $\text{MeNH}_2$  was isolated. Dihydrolysergic acid gave a *metanoid*, m.  $25^\circ$  ( $\text{MeOH}$ ). The bearing of these and earlier observations on the structure of lysergic acid (II) is discussed, and II is assigned the tentative structure of 3-propenyl-4-methyl-3,4-dihydro-4-carboxylic-5-carboxylic acid. R. C. E.

Fieser-Rosenmund rearrangement of  $N$ -acetylcarbazole. Erich Munster. *J. Am. Chem. Soc.* 57, 2127-8 (1935).  $N$ -Acetylcarbazole (21 g.) and 15 g.  $\text{AlCl}_3$  in 125 cc.  $\text{PhNO}_2$  gave, after standing 15-20 hrs., 12-13 g. crude 3-acetylcarbazole (I) and a mixt. of I, the 1-isomer (II) and carbazole, b.p.  $130^\circ$ , the distillate consisting of II and carbazole, sep'd. by sublimation at  $113^\circ$  and  $10^\circ$  mm.; II m.  $128^\circ$ ; *oxime*, m.  $179-80^\circ$ . C. J. West.

Synthesis of certain phenylated benzoxazoles and derivatives. Vladimir J. Mieska and Miriam T. Bogert. *J. Am. Chem. Soc.* 57, 2121-4 (1935).—The solution of  $\text{PhSO}_2\text{Cl}$ ,  $o$ - and  $p$ - $\text{HOC}_6\text{H}_4\text{Ph}$  from the still residues in the prepn. of  $\text{PhOH}$  by fusion of  $\text{PhSO}_2\text{Na}$  with  $\text{NaOH}$  is described, yields being approx. 33, 21 and 1%, resp. 3,4- $\text{O}_2\text{N}(\text{HO})\text{C}_6\text{H}_3\text{Ph}$  (I) in concd.  $\text{H}_2\text{SO}_4$  at  $71^\circ$  for 3 hrs. gives 80% of the *Et* salt of 3-amino-4-hydroxy-phenylsulfonamide. I (3 g.) and 5.2 g.  $p\text{-Me}_2\text{CHSO}_2\text{Cl}$  in 5 g.  $\text{PhNO}_2$ , heated 1 day at  $100^\circ$ , give 68% of 3-methylphenyl-4-*p*-toluenesulfonyl-*Et* salt, bright yellow, m.  $114-5^\circ$  (m. ps. cor.); the benzoxide (II) m.  $159^\circ$  (80% yield). Boiling 3,4- $\text{H}_2\text{N}(\text{HO})\text{C}_6\text{H}_3\text{Ph}$  and 5% excess  $\text{EtCl}$  for 30 min. gives 35% 2,3-dimethylphenyl-*Et* salt, m.  $132.5^\circ$ ;  $p\text{-ONH}_2\text{CH}_2\text{COCl}$  gives 52% of the 2-*p*-hydroxyphenyl analog, m.  $228.5^\circ$  (a by-product is 12% of 3-*p*-hydroxyphenyl-2-hydroxyphenyl, orange-yellow, m.  $249.3^\circ$ ). II and  $\text{SnCl}_4$  in  $\text{AcOH}$  gives 63% of 3-*p*-amino-phenyl-5-*p*-acetylbenzoxazole (III), pale yellow, m.  $174.2^\circ$ ; reduction of the  $\text{NO}_2$  compd. gives 75% of the same compd. III and  $\text{NaNO}_2$  in concd.  $\text{HCl}$  at  $0^\circ$  give 40% of 2-*p*-benzoxazolinyl-5-*p*-phenylbenzoxazole— $\text{HCl}$ , yellow, amorphous, m.  $130.5^\circ$ ; d. l.  $\text{HCl}$  gives 85% of the 2-*p*-hydroxyphenyl-*Et* salt, cream, m.  $270^\circ$ . III in concd.  $\text{H}_2\text{SO}_4$  with fuming  $\text{H}_2\text{SO}_4$  (80% free  $\text{SO}_3$ ) gives 80% of the *isomeric* acid, probably  $\text{Ph}(\text{EO})_2\text{C}_6\text{H}_3\text{C}(\text{O})\text{C}_6\text{H}_4\text{NH}_2\text{SO}_2\text{H}$  m.

3,5-Dimethylphenyl-3-benzoxazole, pale yellow, m.  $119.5^\circ$  (65% yield); 2,3-dimethyl-5-aminobenzoxazole, pale yellow, m.  $221.8^\circ$  (51% yield); the  $\text{EtOH}$  salt shows a greenish fluorescence; the *sulfonic acid*,  $\text{C}_{12}\text{H}_{11}\text{O}_4\text{NS}$  with 1 mol.  $\text{H}_2\text{O}$ , decomposes above  $375^\circ$ . C. J. West.

Triarylypyrylium borates. W. C. Dover and Robert Robinson. *J. Chem. Soc.* 1935, 138-40.— $\text{PhBr}$ , satd. with  $\text{BF}_3$  and heated at  $120^\circ$  for 15 min., gives 25% of 2,4,6-trimethylphenyl borate, yellow, m.  $235-6^\circ$ , a mixt. of 4.5 g.  $\text{PhBr}$  and 5 g.  $\text{PhCOOH}$ ;  $\text{C}_6\text{H}_5\text{Me}$  gives 61% of the same compd.; the pale greenish yellow soln. in concd.  $\text{H}_2\text{SO}_4$  exhibits an intense greenish blue fluorescence; it appears to crystallize with 0.6 mol.  $\text{H}_2\text{O}$  as  $\text{AcONa}$  gives the pseudo base, 2,4,6-trimethylphenylpyrylium borate, orange, m.  $345-7^\circ$ , crystals in 1% yield from  $p\text{-MeOCH}_2\text{HAc}$  and in 52% yield from a mixt. of the ketone with  $p\text{-MeOCH}_2\text{COCH}_3$ — $\text{CM-CH}_2\text{O}_2\text{M}$ — $p$ . These yields are superior to the  $\text{FeCl}_3$  method of Dübner. C. J. West.

Dipole moments and structures of some quinoline derivatives and the orientation of Clais and Hoffmann's  $\alpha$ - $\text{NO}_2$  quinolines. Catherine G. Le Fevre and Raymond J. W. Le Fevre. *J. Chem. Soc.* 1935, 147-55.—The dipole moment,  $\mu \times 10^{-18}$  of quinoline is determined as 2.13, quinoline 1-N, 6-methylquinoline 2.21, 3-methylquinoline 2.00, 2,4-dimethylquinoline 2.30, 3-methylquinoline 2.55, 6-isomer 4.12, 5-isomer 3.57. The direction of the moment has been calculated, the results being tabulated as indications counter-clockwise denoted by  $-$  to the  $+$  —  $+$  —  $+$  (direction 3-Me  $-33.5^\circ$ , 4-Me  $-18.3^\circ$ , 2,4-Me  $-17^\circ$ , 3- $\text{NO}_2$   $+33.5^\circ$ , 6- $\text{NO}_2$   $-17.3^\circ$ , 5- $\text{NO}_2$   $+43.5^\circ$ ) (the  $-$  is considered the more correct for the 5- and the 5- $\text{NO}_2$  d- $\text{NO}_2$ ). It is concluded that in the quinoline d- $\text{NO}_2$  group, except the  $\alpha$ -Me group compounds, the mobilities, typical of  $\text{C}_6\text{H}_5$  and  $\text{C}_6\text{H}_5\text{N}$  of the ring and double bonds are so diminished by ring fusion that the skeleton is best formulated as  $\text{C}-\text{C}-\text{C}-\text{C}-\text{C}$  and that

the component elec. moment, arising from this structure, acts along directions varying from  $15^\circ$  to  $5^\circ$  to the axis of the 3- $\text{C}-\text{C}$  atoms, to meet it at some point outside the mol. on the side of the 5- $\text{C}$  atom. The dipole moment of quinoline is 2.50, of the 1- $\text{Cl}$  d- $\text{NO}_2$  5.23 and the 5- $\text{NO}_2$  d- $\text{NO}_2$  of Clais and Hoffmann (*J. prakt. Chem.* 47, 227 (1903) 3.77; it is suggested that this is the 5- $\text{NO}_2$  d- $\text{NO}_2$ . This is supported by the fact that the component moments of the  $\text{NO}_2$  group and the quinoline nucleus should make an angle of approx.  $71^\circ$ , whereas the 5- $\text{NO}_2$  structure should give a value of the order of  $1.9^\circ$ ; the found value for the 5-isomer is  $115^\circ$ . C. J. West.

Attempts to find new azinulines. IV. Derivatives of 8-methylquinoline. Wm. O. Kermack and Thomas W. Wright. *J. Chem. Soc.* 1935, 147-61; c. d. 29.  $732.5^\circ$ —3,2-MeO-MeOCH<sub>2</sub>CH<sub>2</sub>Ph,  $\text{FeSO}_4$ ,  $\text{AcOH}$  and 9%  $\text{H}_2\text{SO}_4$ , reduced 4 hrs., give 8-methoxy-2-methylquinoline (I), whose  $\text{EtBr}$  salt, light yellow, m.  $208^\circ$ , and *picrate*, yellow, m.  $232-3^\circ$ . I in  $\text{CHCl}_3$  and with  $\text{HBr}$  and treated with  $\text{Br}_2$  gives the 3- $\text{Br}$  d- $\text{NO}_2$  m.  $114-17^\circ$  ( $\text{EtBr}$  salt, light yellow, m.  $207^\circ$ ). 6-Nitro-m- $\text{NO}_2$  and  $\text{Br}_2$  in  $\text{CHCl}_3$  give the 4- $\text{Br}$  d- $\text{NO}_2$  m.  $140^\circ$ .  $\text{MeSO}_3\text{H}$  and  $\text{Et}_2\text{O}$  in  $\text{C}_6\text{H}_6$  give 4- $\text{Br}$ -8-methyl- $\text{NO}_2$   $\text{Me}$  d- $\text{NO}_2$  m.  $111-11^\circ$ ; reduction with  $\text{Fe}$  in concd.  $\text{HCl}$  in  $\text{MeOH}$  gives 4-bromo-8-methoxy-2-methyl- $\text{NO}_2$  (II), light pink, m.  $79-80^\circ$ ; the  $\text{EtBr}$  salt, white,  $\text{Me}$  d- $\text{NO}_2$  m.  $113-13^\circ$  (80% yield), reduction gives 91% of 2-bromo-8-methoxy-2-methyl- $\text{NO}_2$  m.  $71-7^\circ$  ( $\text{Et}$  d- $\text{NO}_2$  m.  $121-2^\circ$ ); the Sandmeyer reaction gives III. I with the Sandmeyer reaction gives the 3- $\text{Br}$  d- $\text{NO}_2$  of I, the 7- $\text{Br}$  d- $\text{NO}_2$  of I m.  $134-3^\circ$ . 6-Nitro-8-methylquinoline yields a 3- $\text{Br}$  d- $\text{NO}_2$  salt, light yellow, m.  $138-9^\circ$ . 3-Bromo-8-methylquinoline, reduced overnight with  $\text{H}_2\text{SO}_4$ , gives 3-phenyl-8-methyl- $\text{NO}_2$  m.  $73-6^\circ$ ; 3- $\text{NO}_2$  d- $\text{NO}_2$  salt, light brown, m.  $143-9^\circ$ . 3-Nitro-8-bromo-2-methylquinoline and propylene in  $\text{Et}_2\text{O}$  give the 3-phenyl-8-methyl- $\text{NO}_2$  d- $\text{NO}_2$ , whose  $\text{EtBr}$  salt m.  $24-9^\circ$ .  $p\text{-OCH}_2\text{CH}_2\text{NMe}_2$ ,  $\text{EtNH}_2$ ,  $\text{CH}_3\text{COCl}$ ,  $\text{EtCl}$  (IV),  $\text{K}_2\text{CO}_3$  and a trace of  $\text{O}_2$  in benzene in  $\text{C}_6\text{H}_6$ , reduced 4 hrs., give 3-( $p$ -methoxyphenyl)phenyl-2-methylquinoline (V), whose *picrate*, light yellow, m.  $176-8^\circ$ ;  $\alpha\text{-NH}_2$  d- $\text{NO}_2$  as the *picrate*, light yellow, m.  $134^\circ$ ; 2-( $p$ -methoxyphenyl)-2-methylquinoline *picrate*, yellow, m.  $150-1^\circ$ . The  $p\text{-NO}_2$  isomer of V, as the *picrate*, yellow, m.  $113-4^\circ$ .  $\text{EtNH}_2$ .

and IV give  $\beta$ -diethylaminoethylpropylamine, whose picrate, yellow, m 133-5°, butylamine analog, b 207-12° (70% yield) (dicaprate, yellow, m 234°); isobutylamine analog, b 191-200° (70% yield) (dicaprate, yellow, m 141°); the following piperidine compds were analogously prep'd; the dicaprates form yellow plates,  $\beta$ -piperidinoethylpropylamine, b 230-30°, 60% yield (dicaprate, m 163°); butylamine, b 230-40°, 70% (dicaprate, m 191-2°); isobutylamine, b 230-40°, 70% (dicaprate, m 167-8°); methylamine, b 190-200°, 45% (dicaprate, m 174°);  $\beta$ -piperidinodimethylamine, b 200-10°, 65% (dicaprate, m 154°).  $C_6H_5Br$  and  $MnEt_2$  in  $EtOH$  give 50% of sym-dimethylthietylenediamine, b 150-60° (picrate, m 160°). 8-Bromomethylpiperidine and  $Et_3NCH_2CH_2NHMe$  with  $K_2CO_3$  in  $CaH_2$  give 8-( $\beta$ -diethylaminoethyl-methylaminoethyl)guanine, the tri-HBr salt of which m 215-16°,  $\beta$ -diethylaminoethylmethylaminoethyl deriv. (tri-HBr salt, m 218-19°, picrate, yellow, m 131-2°),  $\beta$ -diethylaminoethylpropylaminomethyl deriv. (picrate, light yellow, m 113-15°, dicaprate, deep yellow, m 163-4°), diethylaminoethylbutylaminomethyl deriv. (dicaprate, yellow, m 178-80°),  $\beta$ -diethylaminoethylisobutylaminomethyl deriv. (dicaprate, deep yellow, m 160-71°),  $\beta$ -piperidinodimethylaminomethyl deriv. (tri-HBr salt, m 210°), butylamine analog, m 211-12°, isobutylamine analog (dicaprate, yellow, m 210-11°); methylamino analog (dicaprate, yellow, m 205-6°);  $\beta$ -piperidinodimethylaminomethyl deriv. (tri-HBr salt, m 222°), sym-bis(8-quinolylmethyl)dimethylthietylenediamine di-HBr, m 232°. Bis(8-quinolylmethyl)- $\beta$ -diethylaminoethylamine, m 97-8°, picrate, yellow, m 191°, 8-( $\beta$ -diethylaminoethylaminoethyl)guanine tri-HBr, m 223-4°. 1- $\beta$ -Bis(8-quinolylmethyl)aminoethylpiperidine, m 97-8°, picrate, pale yellow, m 228-0°. 1,4-Bis(8-quinolylmethyl)piperazine, with 0.5 mol  $H_2O$ , m 153-4°; HBr salt, m 205-7°. Although inactive in bird malaria, some of these compds possess marked local anesthetic activity.

## C J West

Phenanthridine series IV. Synthesis of plasmochin-like derivatives. Leslie P. Walls. *J Chem Soc* 1935, 1405-10, cf. C. A. 28, 2003°.—3-Nitro-9-methylphenanthridine (15 g) with  $Na_2Cr_2O_7$  in  $AcOH$  gives 14.5 g of 3-nitrophenanthridone, red, m above 350°. 5,9-Br-(AcNH) $C_6H_4Ph$  (30 g), refluxed with  $POCl_3$  for 2 hrs, gives 25 g of 3-bromo-9-methylphenanthridine, m 129-5-30°; oxidation gives 3-bromophenanthridone, yellow, decomp 302°; heating with  $POBr_3$  at 180° for 5 hrs gives 3,9-dibromophenanthridine (I), m 170-1°. 2-Nitrofluorenone with  $Na_2S$  in concd  $H_2SO_4$  gives 2(or 7)-nitrophenanthridone, dark green needles (from  $PhNO_2$ ), sublimes slowly at 250° and 20 mm in yellow needles, decomp 340°. The 2(or 7)-Cl deriv., green, m 340°, 2,7-dinitrophenanthridone, brown-yellow, m above 340°. 2(or 7)-Aminophenanthridone, m 285°, results by reduction of the  $NO_2$  deriv. or from 2-aminofluorenone; the diazo reaction gives a mixt. of the 2- and 7-II-O derivs, which by crystn from  $CH_2Cl_2$  the 2-II-O deriv, m 341-3°, Ac deriv, m 273-4° (decompn). Me ether, m 251° (9-Cl deriv, m 173°). The 7-II-O deriv, m 320-2° (decompn); Ac deriv, m 261-4° (decompn); Me ether, m 271-2° (9-Cl deriv, m 107°).  $\alpha$ -Diethylamino-5-aminopentane dicaprate, yellow, m 134-5°, with 9-chlorophenanthridine this yields 8-diethylamino- $\alpha$ -methylbutylamino-9-phenanthridine, whose dicaprate, yellow, m 196-7°. The sulfate is a hygroscopic glass. I and  $Et_3NCH_2CH_2NH_2$  give 3-bromo-9- $\beta$ -diethylaminoethylamino-9-phenanthridine, analyzed as the di-HCl salt, which contains 3 mols of  $H_2O$ , dicaprate, yellow, decomp 150°. 3-Bromo-9- $\beta$ -diethylamino- $\alpha$ -methylbutylamino-9-phenanthridine forms a dicaprate, yellow, decomp. 217-18°, and a di-HCl salt 2-Methoxy-9- $\beta$ -diethylaminoethylamino-9-phenanthridine gives a dicaprate, yellow, m 207° (decompn), and a hygroscopic di-HCl salt 2-Methoxy-9- $\beta$ -diethylamino- $\alpha$ -methylbutylamino-9-phenanthridine dicaprate, decomp 192-3°, the di-HCl salt is very hygroscopic. The di-HCl salts have a sweet taste and some local anesthetic action, they are very sol in  $H_2O$  to colorless solns of pH about 5, their lack of chemotherapeutic value

appears to be largely explicable on the grounds of their toxicity, in which they compare unfavorably with well-known antimalarials.

C. J. West

Pyrimidines CXLVIII. Action of chlorine on mercaptopyrimidines. James M. Sprague and Treat B. Johnson. *J Am Chem Soc* 57, 2252-5(1935); cf. C. A. 29, 5078°.—The corresponding 4-chloropyrimidines yielded the following 2-mercaptopyrimidines: 4-ethoxy-carbonyl, b 175°, n<sub>D</sub><sup>20</sup> 1.5420, 90% yield, 4-EtO, b<sub>1</sub> 123-4°, n 1.5405, 88%, 4-ethoxy-5-methyl, b<sub>1</sub> 135-8°, n<sub>D</sub><sup>20</sup> 1.5305, 90%, 4-ethoxy-5-bromo, b<sub>1</sub> 140°, n<sub>D</sub><sup>20</sup> 1.5787, 82%; 4-ethylmercapto, b<sub>1</sub> 135-7°, n<sub>D</sub><sup>20</sup> 1.5974, 92%, 4-ethylmercapto-5-methyl, b<sub>1</sub> 158-61°, n<sub>D</sub><sup>20</sup> 1.6100. The action of  $Cl_2$  upon these compds. in  $H_2O$  at 20° gave the following 2-ethylsulfonypyrimidines: 4-ethoxy-5-methyl, m 67-8°, 80%, 4-chloro-5-methyl, m 67.5-8°, 60%, 4-ethoxy-5-bromo, m 89.5-90.5°, 77%; 4-chloro-5-bromo, m 81-2°, 97%, 4-EtO, b<sub>1</sub> 183-5°, n<sub>D</sub><sup>20</sup> 1.6225, 50%; 4-Cl, m 57-8°, 84%, 4-chloro-5-carbonyl, m 72.5-3.5°, 60%; 4-ethylsulfonyl, m 87-8°, 63%, 4-amino-5-carbonyl, m 143.5-4.5°, 93%. 2-Ethylmercapto-4-chloro-5-carbonylpyrimidine and  $Cl_2$  at 5° give 60% of 2-ethylsulfonyl-4-chloro-5-carbonylpyrimidine (I), very unstable; 15-20% of the reaction gives 2,4-dichloro-5-carbonylpyrimidine (II), m 36-7°;  $EtSO_3Cl$  is also formed; at 30-40° there results 60-75% of II, 7% I and 47-75% of  $EtSO_3Cl$ . On acid hydrolysis the pyrimidine sulfones yield the corresponding ketopyrimidines.

C. J. West

The alkaloid of chin-shuh-hu. K. K. Chen and A. Ling Chen. *J Biol Chem* 111, 653-8(1935).—The Szechuan variety of the Chinese drug, chin-shuh-hu, contains an av. of 0.52% of total alkaloids and 48 g of dendrobine,  $C_{19}H_{21}O_3N$ , was obtained from 25 kg. of the powder. Dendrobine  $HCl$ , m 193°, [a]<sub>D</sub><sup>20</sup> -40.3° in  $H_2O$ , hydrobromide, m 250-0.5°, [a]<sub>D</sub><sup>20</sup> -34.3° in  $H_2O$ , hydrosulfide, m 294-4.5°, [a]<sub>D</sub><sup>20</sup> -29° in  $H_2O$ , nitrate, 198.5-9°, [a]<sub>D</sub><sup>20</sup> -30.2° in  $H_2O$ , sulfate, m 178°, [a]<sub>D</sub><sup>20</sup> -30.7° in  $H_2O$ , oxalate, m 137-8°, [a]<sub>D</sub><sup>20</sup> -50.8° in alc.; picrate, m 210°, flavanate, decomposes 212°, methiodide, m 240°, [a]<sub>D</sub><sup>20</sup> -28.8° in alc.; chlorosulfate, m 183-4°, chloroplatinate, m 283°. All m ps are cor. The Kweichow variety yields no dendrobine, indicating that the chin-shuh-hu of commerce consists of several species.

A. P. Lothrop

Structure and configuration of cyarose. Robert C. Elderfield. *J Biol Chem* 111, 527-35(1935); cf. C. A. 29, 4741°.—By the prep'n of 2 different fully methylated lactones from cyarose (I), the allocation of the OMe group on C atom 3 was confirmed. Methyl- $\gamma$ -cyaromaron lactone (II) is identical with dimethyl- $\gamma$ -diploxonic lactone (III), hence I has the same configuration as diploxose (IV) and is 3-methyl-2-deoxy-d-allo-(altro-)hexomethyl-ose. I (2 g) in 50%  $HNO_3$  (40 cc) after standing 3 days at room temp and removal of  $HNO_3$  gave the lactone of  $\alpha$ -hydroxy- $\beta$ -methoxyglutaric acid, m 150-2° (iso-Bu-COOEt), [a]<sub>D</sub><sup>20</sup> -1° ( $H_2O$ ), bi-N-methylamide, m 138° (EtOAc), [a]<sub>D</sub><sup>20</sup> -55° ( $H_2O$ ). Oxidation of I by the method of Hudson and Isbell with  $Br-H_2O$  gave  $\gamma$ -cyaromaron lactone (V), b<sub>1</sub> 110-11°, n<sub>D</sub><sup>20</sup> 1.4635, [a]<sub>D</sub><sup>20</sup> -25° ( $H_2O$ ), after 75 hrs no change, phenylhydrazide, m 154°, [a]<sub>D</sub><sup>20</sup> 2° (MeOH). V methylated with Purdie's reagent, gave II, b<sub>1</sub> 78-80°, n<sub>D</sub><sup>20</sup> 1.4437, [a]<sub>D</sub><sup>20</sup> -30° ( $CHCl_3$ ), phenylhydrazide, m 139°, [a]<sub>D</sub><sup>20</sup> -6°. I (10 g) on boiling with 0.25% abs MeOH-HCl (200 cc) 15 min gave a mixt. of  $\alpha$ - and  $\beta$ -Me cyaromaronoside, b<sub>1</sub> 54-64°, n<sub>D</sub><sup>20</sup> 1.4475, which, methylated by Purdie's method, gave Me methylcyaromaronoside (VI), b<sub>1</sub> 45-8°, n<sub>D</sub><sup>20</sup> 1.4341. VI was hydrolyzed by heating at 100° in 0.1 N HCl for 10 min and the crude product oxidized with  $Br-H_2O$ , giving methyl- $\beta$ -cyaromaron lactone (VII), b<sub>1</sub> 88-90°, n<sub>D</sub><sup>20</sup> 1.4509, [a]<sub>D</sub><sup>20</sup> 30°; phenylhydrazide, m 115-6°, [a]<sub>D</sub><sup>20</sup> 17.5°. VII on oxidation with concd  $HNO_3$  at 90-4° for 8 hrs gave a dimethoxyglutaric acid, isolated as the di-N-methyl amide, m 173-4°, [a]<sub>D</sub><sup>20</sup> -56° ( $H_2O$ ). IV in 0.1% abs MeOH-HCl after standing 1 hr. at room temp gave a mixt. of the Me furanoside and pyranoside of IV, which was directly methylated as

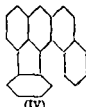
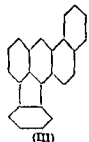
before. The mixt. of di-Me derivs.  $b_2$ , 43-6°. Hydrolysis and oxidation of this as before gave a mixt. of lactones from which III was isolated by distn. at 0.2 mm. All the lactones of this series reduced Fehling's soln. Curves are given for Me glycoside formation of I and IV

R. C. Elderfield

**Partial synthesis of ribose nucleobases II Muscle inosinic acid.** P. A. Levene and R. Stuart Tipson *J. Biol. Chem.* 111, 313-23 (1935), cf. C. A. 29, 174. The structure assigned to muscle inosinic acid, viz. hypoxanthine 5-phosphoriboside (I) has been confirmed by synthesis. Inosine (II) (5 g) was added to a soln. of anhyd.  $ZnCl_2$  (20 g) in  $Me_2CO$  (500 cc). After boiling until all the II was in soln. and standing overnight, concn to 60 cc. and addn. of  $Et_2O$  gave 10.5 g. of acetone-insoluble  $ZnCl_2$  compd. After decompn. of this with  $Ba(OH)_2$ , removal of Zn and excess Ba with  $CO_2$  and extra with hot  $Me_2CO$ , 52% of acetone-insoluble (III), m. 240-5° ( $MeOH$ ),  $[a]_D^{25} - 0.9^{\circ}$  ( $MeOH$ ),  $[a]_D^{25} - 85^{\circ}$  ( $C_6H_5N$ ), was obtained. III with  $p-MeC_6H_4SO_3Cl$  in  $CaH_2N$  (IV) gave 5-isosylmononucleosine, m. 185-6° ( $Me_2CO$ ),  $[a]_D^{25} 35.0^{\circ}$  (IV). The tosyl group of this is in position 5 since it was readily removed by NaI in  $Me_2CO$ . Since it is a ribofuranose, III is the 2,3-isopropylidene deriv. III (3 g) in 100 cc. of IV at -30° was added to a soln. of  $POCl_3$  (1 cc) in 5 cc. of IV at -40°. After standing at -15° for 2 hrs., the mixt. was cooled to -35°, 90% IV (10 cc) was added dropwise and finally ice. Neutralization with  $Ba(OH)_2$  and concn. gave the Ba salt of phosphomonoacetosine (2 g). This was hydrolyzed by standing at room temp. 5 hrs. in HCl (effective acidity 0.05 N HCl), giving Ba inosinephosphate,  $[a]_D^{25} - 36.80$  (0.1 N HCl) identical with natural muscle Ba inosinate. To a soln. of adenosine (10 g) in 500 cc. of  $H_2O$  contg.  $Ba(NO_3)_2 \cdot H_2O$  (21 g) was added  $AcOH$  (15 cc). After several days in the dark, concn. to 65 cc. gave II in quant. yield after working the mother liquors. The *m-Ac* deriv. of II m. 239°,  $[a]_D^{25} - 38.3^{\circ}$  ( $CHCl_3$ )

R. C. Elderfield

**Cholanthrene and related hydrocarbons.** Louis F. Fieser and Arnold M. Schigman *J. Am. Chem. Soc.* 57, 2174-6 (1935); cf. C. A. 29, 4370. The Clemmensen reduction of 4-bromo-1-hydrindone gives 77% of 4-bromohydrindene,  $b_1$ , 118°, the Grignard reaction yields 50% of 4-( $\alpha$ -naphthyl)hydrindene (I), viscous yellow oil,  $\beta$ -isomer (II), m. 68-9° (46% yield). Heating I at 400-5° for 25 min. gives 34% of cholanthrene, m. 173-5° (cor.) (picrate, reddish purple, m. 169-70° (cor.)); II gives 8,9-dimethylene-1,2-benzanthracene, pale yellow, m. 178.5-7° (cor.) (picrate, purple-black, m. 158-9° (cor.)). The prepn. of fluorenone-1-carboxylic acid and fluorene-1-carboxylic acid is described; the acid chloride of the latter and  $\alpha-CuH_4MgBr$  give 50% of 1-( $\alpha$ -naphthyl)fluorene, m. 113-14°; pyrolysis at 415° gives 60% of 15,16-benzodehydrocholanthrene (III), bright yellow, m. 181-1.3° (cor.); picrate, brick-red, m. 174.5-5° (cor.). 1-( $\beta$ -Naphthyl)fluorene, pale yellow, m. 159-62° (84% yield), pyrolysis gives 1',2'-naphtho-1,3-fluoranthene (IV), golden yellow, m. 178-9° (cor.); picrate, crimson, m. 191-2° (cor.).



C. J. West

**Hydrogenation under the action of selenium.** I The action of selenium on cholesterol at 230°. Charles Dörre and Vladimir A. Petrov *J. Chem. Soc.* 1935, 1391-3. Cholesterol with Se at 230° for 25 hrs. gives about 40%

of cholestanone (I); no other single compd. could be isolated; decreasing the time to 10 hrs. gives I with smaller quantities of cholestanol and cholestenone; after 40 hrs. only I was isolated. I and  $PhNHNH_2$  in  $AcOH$  give the tetrahydrocarbazole deriv., m. 180-1°, in 60-70% yield; picrate, bronze-brown, m. 209-10°. The "surface area" of this deriv. is 43 sq. A, thus supporting the angular structure C. J. West

Acids with uneven no. of C atoms in vegetable oils and fats (Manjunath, Siddappa) 27. Gnidusene (Nakamiya) 27. Creatinephosphoric acid (Zeile) 11A.

**Partial oxidation of hydrocarbons.** Stephen P. Burke and Charles I. Fryling (to Doherty Research Co.). U. S. 2,018,994, Oct. 20. For the production of partial oxidation product such as aldehydes, alcs., ketones, etc., a hydrocarbon-contg. gas such as a natural gas or  $C_4H_{10}$  in admixt. with an O-contg. gas is heated (suitably in a metal tube) in the presence of a small amt. of a lower alkyl ether such as methyl ether which serves to facilitate reaction at a somewhat lower temp. Cf. C. A. 29, 1767, 2174.

**Stabilizing organic compounds.** I G. Farbenindustrie A.-G. Brit. 432,480, July 22, 1935. Stabilized aq. solns. of aromatic amino hydroxy, polyhydroxy and polyamino compds., basically substituted compds. of the quinoline and acridine series and alkaloids are produced by adding a small quantity of an org. mercapto compd., which may contain 1 or more mercapto groups and should be sol. in  $H_2O$ . In an example, 1.79 g. of 8-( $\alpha$ -diethylamino-4-pentylamino)-6-methoxyquinoline is dissolved in N HCl, 60 g. quinone-HCl and 90 g. of sacrosine anhydride are added and the mixt. is dild. to 150 cc., 0.292 g. of thioglycolic acid and 11.7 cc. of N NaOH are added and the soln. is made up to 200 cc.

**Organic halogen compounds.** Chemische Fabrik Von Heyden A.-G. Fr. 784,238, July 22, 1935. Stable prepn. of org. compds. contg. active halogen are made by dissolving these compds. in Cl substitution products of aromatic compds., e.g., diaryl ethers. Thus, a 3% soln. of chlorophthalimide in heptachlorodiphenyl ether (I) may be preserved without change for months. Other examples are dichloroamide of toluenesulfonic acid in  $PhCl$ , o-chlorotoluene, o-dichlorobenzene or I.

**Chlorinating aliphatic saturated hydrocarbons.** Edgar C. Britton, Gerald H. Coleman and Bartholdt C. Hadler (to Dow Chemical Co.). U. S. 2,018,345, Oct. 22. Cl is caused to react with a said aliphatic hydrocarbon contg. 3 to 5 C atoms, to form a mixt. contg. said chloro hydrocarbons, HCl and olefinic compds., and the HCl formed during the chlorination is caused to react with the olefinic compds. at a temp. below about 200° to convert the latter into said chloro hydrocarbons.

**Halogenating biphenyl.** Campbell R. McCullough (to Swann Research, Inc.). U. S. 2,019,015, Oct. 29. Biphenyl vapor is mixed with a gaseous halogen such as Cl and heated to above the b. p. of biphenyl but below the temp. at which a flaming reaction would occur. A lower halobiphenyl may be further halogenated similarly. App. is described.

**Separating olefins from other gases.** The Distillers Co. Ltd., Walter P. Joshua and Herbert M. Stanley. Fr. 784,506, July 22, 1935. See Brit. 428,106 (C. A. 29, 6901).

**Alcohols of high molecular weight from esters.** Norman D. Scott and Virgil L. Hansley (to E. I. du Pont de Nemours & Co.). U. S. 2,019,022, Oct. 29. The reaction of alkali metal and hydrolytic alc. on esters of high mol. wt. may be carried out with substantially no side reaction between the hydrolytic alc. and Na to evolve gaseous  $H_2$  if the ratio between the alc. and the ester added to the reaction mixt. is at all times equal to or not more than 2 moles of hydrolytic alc. for each mole of ester group to be reduced. For example, if the ester to be reduced is a simple ester of a monobasic, said. fatty acid, e.g., methyl stearate, 2 moles of alc. will be used to 1 mole of the ester

The corresponding glyceride, e. g., stearin or olein, will require 6 moles of alc. per mole of glyceride, since the glyceride molecule contains 3 ester groups. Finely divided alkali metal is first preferably suspended in a hydrocarbon material such as xylene and this suspension then agitated with a hydrocarbon solvent soln. (for which also xylene may be used) of the ester treated and the hydrolytic alc. Examples are given of the treatment of methyl laurate, bayberry tallow, cottonseed oil, beef tallow, China wood oil, spermaceti and cacao butter, with details of procedure, and mention is made of similar treatment of abietic acid esters, wool grease, menhaden oil, cod-liver oil, linseed oil and of phenylacetic esters.

Esterifying liquid polyhydroxy aliphatic alcohols by reaction with ketenes. Per K. Frolich and Peter J. Wietzich (to Standard Oil Development Co.). U. S. 2,018,753, Oct. 29. In forming an ester, reaction is effected between a polyhydric alc. such as glycerol and a ketene at a temp. below 100°.

Unsaturated esters. I. G. Farbenindustrie A.-G. Brit. 431,398, July 5, 1935. See U. S. 1,993,413 (C. A. 29, 3686').

Acetotricarballylic esters. Frithjof Zwilmeyer (to National Aniline and Chemical Co.). U. S. 2,017,882, Oct. 22. Esters such as monoethyldiamylacetotricarballylate, b<sub>1</sub> (slight decomposition about 230°) and other esters of the general formula R'OOCCH<sub>2</sub>CH(COOR')CH<sub>2</sub>CH<sub>2</sub>COOR are viscous liquids or low-melting solids insol in water, sol in alc., acetone and other org. solvents, and are obtainable by the reaction of an acetooctic ester having the formula, AcCH<sub>2</sub>CO<sub>2</sub>R, with a halosuccinic ester having the formula R'OOCCH<sub>2</sub>CH<sub>2</sub>COOR', R, R' and R'' representing hydrocarbon radicals of which the hydrocarbon radical represented by R is different from the hydrocarbon radicals represented by R' and R'' and X representing Cl or Br can be prep'd by the reaction of an acetooctic ester in the presence of Na alcoholate and of an abs. alc. with a halosuccinic ester having the specified general formula, and can be used as plasticizers or modifying agents with synthetic resins, cellulose esters, etc.

Aldehydes from dicarboxylic acids. Otto Drossbach and Adolf Johannsen (to General Aniline Works). U. S. 2,018,350, Oct. 22. For producing an aldehyde such as benzaldehyde from a dicarboxylic acid such as phthalic anhydride, the acid is treated with a reducing gas such as H<sub>2</sub> at 200–400° in the presence of a hydrogenation catalyst activated by addn of Pb, Be, Ce, U, Cr, Fe, Cu, Mn or Co or oxides of these metals.

Amines. I. G. Farbernd A.-G. (Willy Schmidt and Karl Huttner, inventors). Ger. 618,109, Sept. 2, 1935 (Cl. 12g 1.01). Addn to 599,103 (C. A. 28, 5833'). A carboxylic acid or a functional deriv. thereof is treated with H<sub>2</sub> at a raised temp. and pressure in the presence of a hydrogenation catalyst and (1) a reagent which is converted into an amine under the reaction conditions, e. g., a nitrile, oxime or nitro compd., or (2) NH<sub>3</sub> (or an amine) and another compd. which reacts therewith to form an amine, e. g., cyclohexanol or an aldehyde or ketone. In either case, the amine formed *in situ* reacts with the carboxylic acid or deriv. to yield an amine compd. A hydrocarbon radical derived from the acid. Thus, Me laurate and PhCH<sub>2</sub>CN yield PhCH<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>CH<sub>2</sub>Me, while Me laurate, cyclohexanol and NH<sub>3</sub> yield C<sub>6</sub>H<sub>11</sub>NHCH<sub>2</sub>CH<sub>2</sub>H<sub>2</sub> (other examples are given).

Alkyl amines. I. G. Farbernd A.-G. Fr. 784,599, July 22, 1935. Secondary alkylamines contg. more than 8 C atoms are made by causing the corresponding alkyl halides to react with aq. NH<sub>3</sub> or lower primary aliphatic amines. Examples are given of the prepn of dodecyl (m. 99–1°), didodecyl (m. 58°), dodecyl (m. 47.5°), dodecyltetradecyl, methyltetradecyl (m. 28–9°), butyltetradecyl, methylbutyltetradecyl (m. 18–9°), and methylbutyltetradecyl (m. 4–5°), and methyltetradecylamine, b. 151–5°.

Aromatic amines. I. G. Farbernd A.-G. (Walter Speer, inventor). Ger. 618,922, Aug. 31, 1935 (Cl. 12g 5). Primary or secondary aromatic amines are converted into secondary or tertiary amines, resp., by reaction with aldehydes (or ketones) and esters of HCOOH. The reaction

may be effected at 210–70° under pressure. Examples are given in which (1) *o*-naphthylamine (I), acetone and HCOOME yield *N*-monoisopropyl-I, b. 185–8°, (2) *I*, acetophenone and HCOOME yield *N*-(*o*-methylbenzyl)-I, b. 233–5°, (3) *N*-ethyl-I, cyclohexanone and HCOOME yield *N*-ethyl-*N*-cyclohexyl-I, b. 168°.

Increasing the color stability of primary aromatic amines such as aniline. Robert P. Weiss and John M. Weiss (to Weiss and Downs, Inc.). U. S. 2,019,032, Oct. 29. About 2% or less of maleic anhydride is added as a color stabilizing agent.

Hydroxyamine bases. Soc. pour l'ind. chim. & Bâle Brit. 432,143, July 22, 1935. Such bases, in monomeric form or of low mol. wt., contg. active CH<sub>2</sub> groups are prep'd by condensing hydroxyaryl compds., or others thereof, that contain more than 1 active CH<sub>2</sub> group, with aromatic amines, the condensation being interrupted preferably as soon as the product shows diminution of soly. in alc. and, in any case, as soon as the product shows diminution of soly. in Me<sub>2</sub>CO. The products may be freed from impurities by treatment with inert gases or vapors, e. g., steam, preferably under reduced pressure. Among 9 examples, (1) a soln. of crude cresol in NaOH is treated with CH<sub>2</sub>O and the product is treated with PhNH<sub>2</sub>, monoethylamine or diethylamine in the presence of HCl, (2) polychloromethylcresol (obtained by treating crude cresol with a mixt. of CH<sub>2</sub>O and HCl) is treated with PhNH<sub>2</sub> in presence of NaHCO<sub>3</sub> and (3) *p*-hydroxybiphenyl is dissolved in NaOH, treated with CH<sub>2</sub>O and the product is treated with PhNH<sub>2</sub> in presence of AcOH.

Salts of *N*-nitroamines. Soc. pour l'ind. chim. & Bâle Swiss 175,350, May 1, 1935 (Cl. 36g). Salts of *N*-nitroamines of nuclear halogenated primary aromatic amines are prep'd by treating the corresponding diazo compd. with an oxidizing agent in a weakly alk. soln. Thus, a suspension of dichloroamine-HCl in HCl, water and ice is diazotized and the resulting diazo compd. filtered off and treated with NaClO in a weak NaOH soln. to give the Na salt of 2,5-dichlorophenylnitroamine. Other examples are given.

Amine oxide. Soc. pour l'ind. chim. & Bâle. Swiss 175,351, May 16, 1935 (Cl. 36g). Dodecylmethylamine oxide is prep'd by treating dodecylmethylamine with a peroxidic oxidizing agent such as O<sub>2</sub>, H<sub>2</sub>O<sub>2</sub> or Caro's acid. The compd. is used in the textile industry.

Transformation products from alcohols. Alexander Wacker Ges. fur elektrochemische Industrie G. m. b. H. Fr. 784,656, July 22, 1935. Alcs. are transformed with liberation of H<sub>2</sub> by heating them in the presence of alk. or alk. earth metal alcoholates and hydrogenating or dehydrogenating catalysts such as Co, Ni, Zn, Ag or their compds. Thus, BuOH is transformed mainly to 2-ethyl-1-hexanol and thus to 2-ethyl-1-hexanoic acid, b. 55–6°, and benzyl alc. to BzH, BrOH and (probably) isohydrobenzoin, m. 122°.

Lactones. Firmench & Cie. Successeurs de La Soc. anon. M. Naef & Cie. Swiss 175,340, May 1, 1935 (Cl. 26a). Lactones with at least 11 C atoms in a hetero ring are prep'd by heating carboxylic acids of the general formula X–R–COOH in which R represents an aliphatic residue contg. at least 10 C atoms in the chain and X a reactive substituent, in an indifferent medium, the starting materials being strongly diluted. Thus, a dil. soln. of the anhyd. La salt of 14-bromotetradecanoic-1-carboxylic acid in decalin is heated to boiling in the presence of LiBr as catalyst to give the lactone of 14-hydroxytetradecanoic-1-carboxylic acid. Other examples are given.

Hydrate-sulfonates. Arthur Zitscher and Wilhelm Seidenfaden (to General Aniline Works). U. S. 2,018,103, Oct. 22. Compds. corresponding to the probable general formula acyl–NHCH<sub>2</sub>Y·NH<sub>2</sub>·NH<sub>2</sub>SO<sub>3</sub>–alkali metal, where Y stands for alkyl, alkoxy or halogen, are produced by acting with a reducing agent on the corresponding 2,5-disubstituted 4-acylamino benzene-1-diazosulfonic acid, and may be used for the production of azo dyes. For this purpose, they may be first mixed with combining components contg. a hydroxy or an *O*-alkali metal group, where

the adjacent position is capable of attaching an aryl azo group, provided that neither of the dye components contains a group producing soly. in water. The compus., thus produced, are then brought onto the fibers in the presence of an alkali and the goods, thus treated, are thereafter subjected to a steaming operation. Numerous examples are given.

**Polyuclear phenols and nitration and sulfonation products thereof** E. I. du Pont de Nemours & Co. Brit. 431,945, July 18, 1935. The phenols are prepd. by condensing PhOH or its homologs with an aldehyde or ketone in the presence of an acid condensing medium, e. g.,  $H_2SO_4$ , and an acid-sol B compd. The products are dihydroxy-di- or -tri-aryllkanes. Di(4-hydroxyphenyl)dimethylmethane is best prepd. by using PhOH and  $Me_2CO$  in theoretical proportions, 4-5 mols. of  $H_2SO_4$  per mol. of PhOH, an acid concn. of 65-70% and keeping the temp. in the early stages at 25-35° and later at 45-55°. About 0.5-5%  $H_2BO_3$ , calcd on the PhOH, is sufficient. The products may be used in making synthetic resins, as antioxidants and polymerization inhibitors for motor spirit, paints and varnishes, as insecticides (either as the phenol or its salts) or dye intermediates, or can be nitrated to form explosives or sulfonated to give wetting, dispersing or emulsifying agents.

**4-Alkyl- and 4-alkyl-dihydroresorcinols** Max Klingensuss (to Hoffmann-La Roche Inc.). U. S. 2,018,213, Oct. 22. 4-R-Dihydroresorcinols (R representing an alkyl or aralkyl group) are produced by subjecting solns of 4-R-resorcinols to catalytic hydrogenation in the presence of at least one equiv. of a strongly basic compd. 4-Ethylidihydroresorcinol forms colorless crystals m 109° easily sol. in org. solvents and difficultly sol. in water. Several examples are given.

**Hydrogenating pyridine and its derivatives.** Wilbur A. Lazier (to E. I. du Pont de Nemours & Co.). U. S. 2,019,419, Oct. 29. A compd. such as pyridine or an alkyl pyridine in liquid phase and  $H_2$  under superatm. pressure and temp. conditions, are brought into contact with a catalyst prepd. by heating a multiple chromate of a N base and a hydrogenating metal such as a Cu-NH<sub>3</sub> chromate to its spontaneous decompn temp and then reducing the resulting compn. in  $H_2$ . Numerous examples are given.

**Catalytic hydrogenation of pyridine and its derivatives, etc.** Wilbur A. Lazier (to E. I. du Pont de Nemours & Co.). U. S. 2,018,880, Oct. 29. Hydrogenation is effected under superatm. pressure in the presence of a Ni catalyst which may be at least partly in oxidized form together with an oxidized form of a metal such as Cr forming an oxide more acidic than Ni oxide.

**Polymerizing gaseous olefins to form liquid hydrocarbons.** Vladimir Ipatieff (to Universal Oil Products Co.). U. S. 2,018,066, Oct. 22. An olefinic gas is subjected to polymerizing conditions of temp. and pressure in the presence of a solid phosphoric acid catalyst and with such an added amt. of steam that the vapor pressure of the acid at the operating temp. and pressure conditions is balanced by the steam in the gas so that loss of water from the catalyst is substantially prevented.

**Polymerization of isobutylene.** I. G. Farbenindustrie A.-G. Brit. 432,190, July 15, 1935. Polymerization products of much higher mol. wt. and of greater viscosity are obtained when polymerizing isobutylene (I) by means of  $BF_3$  according to Brit. 401,297 (C. A. 28, 2387) if specially pure I obtained by conversion of iso-Bu or tertiary Bu alcs and subsequent purification by distn is employed. The low temps. of Brit. 401,297 are no longer necessary, as room temp. or temps. up to 100° may be used. In an example, I obtained by leading iso-BuOH over  $Al_2O_3$  at 400° and subsequently distg at 2-3 atm. in a column 5 m high, is cooled to -15° and  $BF_3$  led in. An oil of viscosity 250° Engler at 100° is obtained. If the polymerization is at -80°, a product is obtained that has a mol. wt. of 40,000, undistd. I treated under the same conditions giving a product of mol. wt. 10,000-15,000.

**Polymerizing acrylic esters.** Otto Röhm. Fr. 784,095, July 22, 1935. The polymerization of acrylic esters is

regulated by the addn of relatively small amounts of essential oils or substances which contain unsatd. cyclic bonds of the terpene series, e. g., turpentine oil or colophony.

**Carbohydrate polymers.** Holzhydrolyse A.-G. (Eduard Färber, inventor). Ger. 618,163, Sept. 16, 1935 (Cl 12o. 6). Aldoses are heated to 120-80° for a few min. with substantially less than 0.1% of mineral acid, without the use of reduced pressure, inert gases or liquid diluents. Polymerization products resembling glucose sirup are obtained. Cf Ger. 513,126 (C. A. 25, 1266).

**Condensation products from phloroglucinol.** Leopold Laska and Oskar Haller (to General Aniline Works). U. S. 2,018,137, Oct. 22. See Ger. 603,688 (C. A. 29, 817).

**Solid diazonium salts.** Karl Schnitzspahn (to General Aniline Works). U. S. 2,018,095, Oct. 22. The inorg acid diazonium salts of 4-halo-2-aminodiphenyl ethers, at least one phenyl nucleus of which may be substituted by methyl or a halogen, are caused to sep. in solid form by adding to their aq inorg acid solns an alkali salt of the inorg. acid such as  $Na_2SO_4$  or KBr. Numerous examples are given.

**Solid diazo compounds.** Soc. pour l'ind. chim. à Bâle. Swiss 177,115 to 123, Aug. 1, 1935 (Cl 36g). Addns to 174,081. 4-(4'-Methyl)-phenoxyacetylamin-2,5-diethoxy-1-aminobenzene is diazotized in the presence of  $HCl$  and the product which is a stable cryst. powder useful for the prepn. of azo dyes (177,115), is salted out. Other starting materials can be used, such as 4-phenoxyacetylamin-2,5-diethoxy-1-aminobenzene (177,116), 4-(3'-methyl)phenoxyacetylamin-2,5-diethoxy-1-aminobenzene (177,117), 4-(2'-methyl)-phenoxyacetylamin-2,5-diethoxy-1-aminobenzene (177,118), 1-amino-2-methoxy-5-methyl-4-benzoylaminobenzene (177,119), 1-amino-2-chloro-5-methoxy-4-benzoylaminobenzene (177,120), 1-amino-2-methoxy-5-chloro-4-benzoylaminobenzene (177,121), 1-amino-2,5-diethoxy-4-benzoylaminobenzene (177,122) and 1-amino-2,5-dimethoxy-4-benzoylaminobenzene (177,123).

**N-Vinyl compounds.** I. G. Farbenind. A.-G. (Walter Reppe and Ernst Keyssner, inventors). Ger. 618,120, Sept. 2, 1935 (Cl 12p. 2). N-Vinyl derivs. of compds. contg. a pyrrole ring are prepd. by treating the compds. with  $CaH_2$  in the presence of a basic substance, e. g., an alkali hydroxide. The reaction may be effected in a solvent or diluent at 160-90° under pressure. Examples are given of the prepn. of N-vinylpyrrole, b 122°, N-vinylindole, b 70-5°, N-vinylcarbazole, m 62-4°, b<sub>1</sub> 170-80°, and N-vinyltetrahydrocarbazole, b<sub>1</sub> 125-30°.

**Benzene derivative.** Soc. pour l'ind. chim. à Bâle. Swiss 177,262, Aug. 1, 1935 (Cl. 36g). The compd. 1,2-diaminobenzene is prepd. by treating 1,2-dichlorobenzene with aq.  $NH_3$  at about 150° and 80 atm. in the presence of Cu. The Cu is present in sufficient quantity to cause the formation of a complex Cu salt of the resulting 1,2-diaminobenzene, the Cu being subsequently split off as  $CuS$ . An example is given.

**Diphenylmethane derivatives.** Berthold Biernert and Robert Held (to General Aniline Works). U. S. 2,018,775, Oct. 29. See Ger. 610,319 (C. A. 29, 3690\*).

**Resorcinol derivatives.** William E. Austin. Fr. 784,441, July 22, 1935. Alkyl resorcinols are prepd. by condensing resorcinol with a secondary alc. or an alkyl halide having at least 7 C atoms in the alkyl group, preferably in the presence of a condensing agent. Examples are given of the prepn. of secondary octyl and heptyl resorcinol. Fr. 784,442. Secondary alkyl chlororesorcinols are prepd. by chlorinating secondary alkyl resorcinols or by condensing chlororesorcinol with a secondary alc. or alkyl halide having at least 4 C atoms in the alkyl group.

**Polyhalogen derivatives of pyrene.** Heinrich Vollmann and Hans Becker (to General Aniline Works). U. S. 2,018,935, Oct. 29. See German pat. 600,055 (C. A. 28, 6444\*) and French pat. 761,627 (C. A. 28, 4009\*).

**Compounds of alkali metals and hydrocarbons.** E. I. du Pont de Nemours & Co. Fr. 784,440, July 22, 1935. Addn. compds. are made by causing an alkali metal to



react with a hydrocarbon of the  $C_nH_n$  series in the presence of a polyether or a mixt of such ethers, and, if desired, an inert diluent. Polyethers signify the completely alkylated products of polyhydric alcs., as well as alkyl orthoformates, methylal or other acetals. Thus, a compd which appears to be 1,4-disodium naphthalene may be prepd.

**Aluminum formate** Oskar Jochem and Theodor Hennig (to Zschimmer & Schwarz chemische Fabrik Dolau) U S 2,019,415, Oct 29. A solid formate of the formula  $Al(HCOO)_3 \cdot 3H_2O$  and which can be recrystallized from aq. solns is obtained by crystn. from an Al formate soln. of at least 1.10 sp. gr.

**Tetraalkyl lead** E. I. du Pont de Nemours & Co. Fr 784,222, July 22, 1935. The mud obtained from the reaction product of Pb-Na and an alkyl halide is steam-distd. in the presence of one or more dispersing agents capable of preventing agglomeration of the Pb in the mud. The distn. is interrupted when the greater part of the tetraalkyl Pb has been removed and a wetting agent is added to the residue so that when the distn. is renewed the whole of the tetraalkyl Pb is recovered. The dispersing agent is preferably a mixt. of motor oil and thio-carbamide, but  $Na_2CrO_4$ ,  $Na_2Cr_2O_7$ , Na silicates or  $Na_2SO_4$  may also be used. The wetting agent may be a sulfonated oil. Cf. C. A. 29, 817.

**Tertiary nitriles** I. G. Farbenind. A-G (Max Bockmühl and Gustav Ehrhart, inventors). Ger. 616,876, Aug. 13, 1935 (Cl. 12o 11). Addn. to 473,329 (C. A. 23, 2387). A secondary nitrile of the formula  $RR'CHCN$ , where R and R' are alkyl groups, is treated with a sodium alkyl, e. g., EtNa, to form a compd  $RR'CNACN$ , which is then alkylated (preferably without isolation) as described in Ger. 473,329. An example is given in which Na covered with ether is treated in turn with  $HgEt_2$  and  $Et_3CHCN$  and then, after 1 hr., with  $CH_3CHCH_2Cl$ ,  $Et_3CH$ ,  $CHCH_3$ ,  $CCN$ ,  $b_1$ ,  $71-4^\circ$ , is obtained.

Concentrating dilute acetic acid, etc. Horace F. Oxley and Walter H. Groombridge (to Celanese Corp. of America) U S 2,018,031, Oct. 22. The dil. acid is extd. with an org. liquid such as ether, which is a solvent for the acid, the org. liquid is at least partially sep'd. from the ext., and water is removed from the acid soln. so obtained in the form of an azeotropic mixt. with the vapor of another org. liquid such as  $C_2H_5$ , of such character that the b. p. of the azeotropic mixt. is higher than that of the extg. agent (the hot azeotropic vapor mixt. being passed in heat exchange with the ext.). Water and  $C_2H_5$  are sep'd., and the  $C_2H_5$  is further used in the process.

**Ascorbic acid** Tadeus Reichstein. Swiss 175,263-4, Aug. 1, 1935 (Cl. 116a). Addns. to 169,855 (C. A. 29, 1863). *d*-Ascorbic acid is prep'd by adding HCN to *d*-xylosone and sapon. the product (175,263). The I form is obtained by using *l*-xylosone (175,264). A small amt. of a basic substance may be present as a catalyst.

**Ketogulonic acid** F. Hoffmann-La Roche & Co. A-G, Swiss 174,080, Apr. 16, 1935 (Cl. 36o). The compd. 2-keto-*l*-gulonic acid is prep'd by treating *l*-sorbose with a cyclic ketone and oxidizing the resulting dimethylene ether deriv. in alk. soln. to convert the  $CH_2OH$  group to a  $COOH$  group. The product is dimethylene ether-2-keto-*l*-gulonic acid. The acid is split off by warming with water. Thus, powd. *l*-sorbose, cyclohexanone and a little concd.  $H_2SO_4$  are shaken together. The product is diluted with ether and potash soln. added. After drying, the ether and free cyclohexanone are dist'd. off and the residue dist'd. *in vacuo* to give dicyclohexanone-*l*-sorbose. This is treated with an aq. KOH soln. and HCl and ice to give dicyclohexanone-2-keto-*l*-gulonic acid, m. 132-33°. This is heated to give 2-keto-*l*-gulonic acid.

**2-Methyl-3-hydroxyquinoxaline-4-carboxylic acids** I. G. Farbenind. A-G. Fr 784,265, July 22, 1935. These are prep'd by heating a halogenated acetone with salts of isatic acids in the presence of alk. earth hydrates. Examples are given of the prep'n. of the acid and its 6-phenyl (m. 246° with elimination of  $CO_2$ ) and 6,8-dichloro (m. 259-60°) derivs.

**Polyhydroxy carboxylic acids from carbohydrates** Oskar

Spengler and Adolf Pfannenstiel. Ger. 618,164, Sept. 3, 1935 (Cl. 12o 11). Reducing sugars are treated in aq. alk. soln. with  $O_2$ , as distinct from air, at atm. or slightly raised temp. Carboxylic acids having 1 C atom less than the starting material are obtained in 65-70% yield. Examples are given in which glucose, fructose, invert sugar and mannose yield *d*-arabonic acid, galactose yields *d*-lyxonic acid, arabinose yields *l*-threonic acid and maltose yields a glucosidoarabonic acid.

**Anhydrides** Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. Fr 784,458, July 22, 1935. Anhydrides, e. g., propionic, butyric and caproic anhydride, are made by causing  $Ac_2O$  to react with the fatty acids and if necessary removing by distn. the  $AcOH$  formed during the reaction.

**Acetic anhydride** Consortium für elektrochem. Ind. G. m. b. H. (Martin Muggan and Johann Sux, inventors). Ger. 618,270, Sept. 6, 1935 (Cl. 12o 12). See Fr. 780,889 (C. A. 29, 5864).

**Ethylene oxide** Société française de catalyse generalisee. Brit. 431,966, July 18, 1935. This corresponds to Fr. 771,650 (C. A. 29, 819).

**Pentaerythritol** Louza Elektrizitätswerke und chemische Fabriken A.-G. [Gampel]. Swiss 176,918, Aug. 1, 1935 (Cl. 36o). Technically pure pentaerythritol is obtained from the crude material formed by condensing  $AlCl_3$  and  $CH_3O$  by washing off the dipentaerythritol, pressing the crystals, and washing with a sat'd soln. of pentaerythritol. Examples are given.

***o*-Dihydroxybenzenes** E. I. du Pont de Nemours & Co., Frederick B. Downing and Richard G. Clarkson. Brit. 432,276, July 24, 1935. Addn. to 425,230 (C. A. 29, 5458). In the process of 425,230, the alk. hydrolyzing agent is partly added as the reaction proceeds, the process, if desired, being conducted continuously.

**Hydroxypyrene** Walter Kern (to Soc. pour l'ind. chim. à Bâle). U. S. 2,018,792, Oct. 29. Pyrene monosulfonic acid is subjected to a caustic alkali fusion, and a hydroxypyrene is obtained m. 180-2° and easily sol. in dil. alkalies. Various details and modifications of procedure are given. Cf. C. A. 29, 4776.

**Bromohydroxybiphenyls** Chem. Fab. von Heyder A.-G. (Karl Willy Rüttler and Wolfgang Gundel, inventors). Ger. 616,848, Aug. 6, 1935 (Cl. 12q, 15.01). Addn. to 615,133 (C. A. 29, 6247). Mixts. of 3- and 5-bromo-2-hydroxybiphenyl, such as are obtained by brominating *o*-hydroxybiphenyl, are sep'd. into their components by converting the mixts. into Ca salts, and sep'd. the salts by virtue of their different soly. properties, the Ca salt of the 5-bromo compd. being almost insol. in water. A sp. process is described. The 3-bromo compd. b. 173-8°, the 5-bromo compd. m. 53-4°, b. 180-2°.

**Dibenzoxanthone** Soc. pour l'ind. chim. à Bâle. Swiss 174,643, Apr. 1, 1935 (Cl. 36o). Dibenzoxanthone b. 179-81°, is isolated from the residues of 2,3-dihydroxy-naphthoic acid manuf. by extg. the residues insol. in  $l$ ys with a solvent of the pyridine series. Cf. C. A. 29, 6946.

**Purifying and decolorizing cinchophen** Ernest F. Grether (to Dow Chemical Co.). U. S. 2,018,354, Oct. 22. An  $NH_3$ , alkali metal, alk. earth metal or Mg salt of cinchophen in solid form and which may be assoc'd. with impurities is acidified with more than its chem. equiv. of a concd. liquid water-sol. carboxylic acid such as glacial  $HOAc$ .

**Phenanthrene** Soc. pour l'ind. chim. à Bâle. Swiss 175,343, May 1, 1935 (Cl. 36o). The above is prep'd. by treating  $\alpha,\beta$ -diphenylethane with a dehydrogenating agent such as a metal halide. The reaction takes place preferably in the presence of  $CS_2$ .

**Pregnanolones** Schering-Kahlbaum A.-G. Brit. 431,833, July 16, 1935. These are prep'd. from pregnadiol by (1) esterification to the monoester, oxidation to pregnanediol monoester and sapon., or (2) oxidation to pregnandione, followed by partial reduction, or (3) esterification to the diester, partial sapon. to the monoester and sapon.  $Ac_2O$  is used for esterification,  $CrO_3$  for oxidation and H in presence of Pt catalyst for reduction. Re-

duction may also be accomplished by treatment in the presence of catalysts with compds. that yield H and at the same time form their corresponding oxidation products, e. g., tetrahydronaphthalene, cyclohexanol. An example of each method above is given.

Vanillin. Jemac A.-G. Swiss 175,671, June 17, 1935

## 11—BIOLOGICAL CHEMISTRY

PAUL E. HOWE

### A—GENERAL

ARTHUR W. DOX

Effect of ultraviolet light on enzymic reactions I. Diastase Sobhanlal Banerjee and H. K. Sen *J Indian Chem Soc* 12, 370-83 (1935)—The activity of org catalysts in the decomposition of  $\alpha$ -ketocarboxylic acids is decreased in the presence of ultraviolet light. This retrogression can be retarded by the use of accelerators provided that they serve as filters against the ultraviolet. The diastatic enzyme in malt is known to be rendered completely inactive by ultraviolet light (C. A. 18, 3068) and the protective action of inorg salts, e. g.,  $MgCl_2$ , has been investigated. It has been observed that several amines, amino acids and  $NH_4$  salts, etc., not only increase the activity of the diastase, but also protect the enzyme from complete destruction by the action of the ultraviolet rays. Expts with malt diastase with and without catalyst, both sets being irradiated and nonirradiated, were made. Filtered solns of 0.15-0.2 g of dry diastase in 100 cc of  $H_2O$  were prepd. and the diastatic value was detd. by the Lintner method. Varying amts of the enzyme (0.1, 0.2, 0.3 cc, etc.) were added to 10 cc of 2% starch solns contg 1 cc of 0.1% catalyst soln, previously adjusted to pH 5.6 (neutral to Wisslow's indicator, a mixt of methyl red and blue) by the addn of 0.01 N NaOH or HCl. The expts. were carried out in quartz tubes irradiated by a 3000 candle power Hg vapor lamp with a 12 in arc at a distance of 2 ft. It was found that asparagine,  $PHNH_2$ ,  $CH_3CO_2H$ ,  $NH_4$  citrate, gelatin, tyrosine, etc., cause varying degrees of increment of diastatic activity and exert protective action on malt diastase against ultraviolet light. A distinction must be drawn between an accelerator and a protector though it is possible that some substances have both functions. The protective action probably depends upon the capacity of the substance to absorb partially or wholly the destructive wave lengths of the ultraviolet light. C. R. Addinall

Some analyses of thyroglobulin. Abraham White *Prot Soc. Exptl Biol Med.* 32, 1558-60 (1935), cf C. A. 29, 4777—The av. values of 2 analyses expressed as percentages of the ash-free and moisture-free protein are: N 15.58, S 1.46, I 0.75, glutamic acid 6.50, aspartic acid 1.59, tyrosine 3.17, tryptophan 1.8, cystine 2.05, proline 4.47, histidine 0.62, arginine 8.22 and lysine 1.93.

C. V. Bailey  
The significance of fumaric acid for the respiration of animal tissue I. Annau, I. Banga, B. Gözsy, St. Huszák, K. Laki, B. Straub and A. Szent-Györgyi. *J physiol. Chem.* 236, 1-68 (1935). Introduction, scope, methods A. Szent-Györgyi. *Ibid.* 1-20—A theoretical discussion of the papers that follow. The respiration of pigeon thoracic muscle in a modified Thunberg app indicates a cyclic dehydrogenation, oxidation and reduction of C-dicarboxylic acids: succinic  $\rightarrow$  fumaric  $\rightleftharpoons$  oxalacetic  $\rightarrow$  succinic. Fumarase, one of the most active enzymes known, hydrates fumaric to malic acid but seems to play no part in respiration. Malic acid inhibits the respiration. Influence of C-dicarboxylic acids on tissue respiration I. Banga *Ibid.* 20-31.—The apparent increase in respiration when fumarate is added to the muscle is merely a "conservation" of the system or protection from denaturation. The effect is not observed in the first few min., but only after the control has begun to show a decline. Fumarate does not initiate an artificial respiration system; it merely prolongs the activity of the

normal system. It counteracts also the inhibitory effect of malonate. Oxalacetate behaves precisely like fumarate, and likewise hydroxymalate and hydroxymaleate, but acetylenedicarboxylate and the 3 tartarates are inert. Oxidation and reduction of C-dicarboxylic acids. K. Laki. *Ibid.* 31-42.—The dehydrogenation of succinic acid by comminuted pigeon thoracic muscle suspended in phosphate soln was detd by the decolorization of 1-naphthol-2-sulfonate-4,6-dichlorophenol and also by detn of the fumaric acid formed. The curve shows a sharp break at the succinate concn where the dye is in mol. excess. The max velocity of dehydrogenation occurs as long as an equiv of succinic acid is present. This dehydrogenation is a reversible process in which the fumarate can serve as H-acceptor. Here the reaction may be followed by observing the coloration of leuco dye. It is independent of the concn of fumaric acid as long as the latter is in mol. excess. Both reactions are inhibited by malonate. L-malic acid inhibits aerobic and anaerobic oxidation of succinic acid at the outset, until the fumarase has had time to dehydrate it to fumaric acid or an equil. mixt. Equal inhibition by malate and fumarate indicates equal adsorption on the succinodehydrogenase. Like fumarate, l-malate is a H-acceptor as can be demonstrated by coloration of leuco-methylene blue and leuco-indigocarmine. The reaction is not influenced by co-dehydrogenase. Maleate behaves in the same way, and with its reduction product, succinic acid, forms an oxidation-reduction system. It requires no coenzyme for activation. As H donor fumaric acid is most effective in activating fumarodehydrogenase at the natural fumarate concn. of the tissue, 20 mg %. Here again the presence of coenzyme does not influence the type of curve. The donor effect of l-malic acid is due to a preliminary conversion to fumaric acid. Malic acid is a feeble H-donor but, like malic acid, inhibits tissue respiration even in low concn. It competes with succinic and fumaric acids for adsorption on the enzyme surface and displaces them without performing their function. Acetylenedicarboxylic acid and d-, l- and meso-tartaric acids are inactive. The first is incapable of reducing pos. dyes or oxidizing strongly neg. leuco dyes such as leuco-neutral red or leuco-indigocarmine. For hydrogenation of oxalacetate the 3 tartaric acids require the very neg leuco-neutral red. The hydrogenerating activation of oxalacetate is strongly accelerated by coenzyme, no effect being obtained in the absence of muscle tissue. It is not inhibited by malonate. Hydroxymalate and hydroxymaleate behave precisely like oxalacetate. Microdetermination of fumaric acid and its application F. B. Straub. *Ibid.* 42-53—Two methods were employed for extn. of the fumaric acid: (a) The tissue suspension was treated with an equal vol. of 96% EtOH, allowed to stand 6-10 hrs. in the refrigerator and centrifuged. An aliquot was acidified with  $H_2SO_4$  and then extd. with  $Et_2O$ . (b) The tissue pulp was treated with 20%  $CCl_3CO_2H$ , then filtered or centrifuged and an aliquot extd. with  $Et_2O$ . Both EtOH and  $Et_2O$  must previously be treated with alk.  $KMnO_4$  and distd. to remove reducing substances. The  $Et_2O$  ext. was evapd and the fumaric acid in the residue detd. by titration with 0.01 N  $KMnO_4$ , the latter being added at the rate of 1 drop every 3 seconds until a permanent color was obtained. Added fumaric acid (0.1 mg) may thus be detd. within an error of 6 to -4%. The fumaric acid content of muscle is about 10 mg %. Under aerobic incubation it does not diminish in quantity although it activates respiration, but

under anaerobic conditions a slight gradual decrease occurs. In the presence of malonate a considerable decrease occurs aerobically, but anaerobically the usual slight decrease is inhibited. Added oxalacetate undergoes reduction to fumarate, the recovery of fumarate therefrom amounting to 65%. The micro-method was applied also to the determination of fumarate formed by dehydrogenation of succinate in the presence and absence of malonate. Microdetermination of succinic acid and its application. B. Gossy *Ibid* 54-8.—The method is based on the biol. oxidation of succinic acid by the muscle itself and measurement of the  $O_2$  consumption in a respirometer. The difference in  $O_2$  consumption between parallel detns with and without addn of malonate represents the  $O_2$  consumed by succinic acid. The total error is about -15%. In the presence of malonate the fumarate of muscle is reduced aerobically to succinate, and the succinate oxidation specifically inhibited. Comparative experiments with fumaric acid and malonic acid. E. Annau *Ibid* 58-65.—Respiration by liver and kidney is influenced in the same manner by addn of fumarate and malonate as is that by muscle, only to a smaller extent, especially with kidney. Formation of oxalacetate is also less, and occurs in Ringer soln rather than in phosphate soln. In general the  $O_2$  consumption is greater with tissue sections than with tissue pulp. The formation of acetone bodies, as detd by the  $Na_2Fe(CN)_5NO$  reaction, occurs in the presence but only slightly in the absence of malonate. In this reaction malonate and fumarate are antagonistic. Tissue sections and tissue pulp show no difference in this respect. Experiments in vivo. Istvan Huszák *Ibid* 66-8.—The alterations in respiratory function of animal tissues through the influence of dibasic acids were demonstrated also by intravenous injections in rabbits. Malonate resulted in acetone excretion which reached a max in 30-60 min. Malonate + fumarate gave no evidence of acetone excretion, but the blue nitroprusside reaction characteristic of oxalacetate appeared in 5 min.  $\beta$ -Hydroxybutyrate gave rise to a strong acetone reaction in the urine, and only a faint reaction when fumarate was injected simultaneously. Rabbits injected with oxalacetate gave a faint reaction for  $AcCOH$  in the urine but a much stronger reaction when arsenite was injected simultaneously. Arsenite thus delays the reduction of oxalacetate. This disturbance in tissue respiration may be a possible explanation of the toxicity of As. The relative non-toxicity of malonate shows that the tissues in vivo behave like tissue sections in Ringer rather than in phosphate soln. A. W. Dorr

Liver enzymes III. The aldehydease of the liver. Ludwig Reichel and Herbert Kohle. *Z. physiol. Chem.* 236, 147-57 (1935), cf. C. A. 28, 4079<sup>1</sup>.—Aldehydease preps. obtained by repeated pptn. of liver ext. with  $MeAc$ - $P_2O_5$ ,  $EtOH$ - $Et_2O$  or  $EtOH$ , show  $pH$  optimum at 7.5-7.8 in the dismutation of  $EtCHO$ . The max. dismutation is effected in 120 min. At 69° the enzyme is completely destroyed. Dismutation is inhibited by  $HCN$ , probably because of cyanohydrin formation. Under anaerobic conditions quinone acts as  $H$ -acceptor and the aldehyde is completely oxidized to acid. Methylene blue is less effective as  $H$ -acceptor, but in high concns it favors acid formation. Under aerobic conditions it is effective in lower concn, probably because of re-oxidation of the leuco dye adsorbed on the active surface. For detn. of  $EtCHO$  in the presence of quinone and methylene blue the aldehyde was displaced by a current of  $CO_2$  at a temp. of 80° and absorbed in  $NaHSO_3$ . The  $H$ -acceptor present in the cell is cytochrome, which undergoes reduction and then re-oxidation by indophenoloxidase. The latter enzyme is very sensitive and does not withstand the  $MeAc$  and  $EtOH$  treatment, hence it is not present in the dry preps. By addn. of indophenoloxidase preps. obtained from heart muscle the oxidation of  $EtCHO$  to acid by liver preps. was increased to 100%. The dehydrogenation process by aldehydease thus requires the presence of cytochrome and indophenoloxidase. Among other natural acceptors adrenalinquinone is inactive, but laccin-flavin is very effective. With fresh liver in the absence of

$O_2$ , the proportions of  $PrOH$  and  $EtCO_2H$  formed from  $EtCHO$  correspond approx. to a dismutation reaction IV. The alcohol dehydrogenase of the liver. *Ibid* 158-67.—Alc. dehydrogenase preps. obtained from liver ext. by repeated  $MeAc$  pptn. in the presence of  $N_2$  are much less stable than aldehydease preps. and lose their activity in 6-8 weeks in *vacuo* or in a few days when exposed to air. They do not dehydrogenate  $PrOH$  in the absence of  $O_2$ . Contrary to the statement of Battelli and Stern (C. A. 5, 308) they do not carry the reaction beyond the aldehyde stage. The acid formation by fresh liver must be attributed to a dehydrogenation of aldehyde by the aldehydease system. Optimum activity of alc. dehydrogenase is at  $pH$  6.7-7.3 and a max. conversion occurs in 80 min. At  $pH$  7.8 the destruction temp. is 47.5°. The enzyme is not sensitive to  $HCN$ . In the presence of  $O_2$ , methylene blue serves as intermediate acceptor and the reaction is accelerated. Quinone is less effective, probably because of its greater affinity for the enzyme. Among the natural acceptors ascorbic acid is ineffective while adrenalinquinone is active. The oxidation is increased by addn. of dried spleen preps. which contain no dehydrogenase, but the factors responsible have not been detd. A. W. Dorr

Enzymic amyolysis V. Amylophosphatase from barley. Ernst Waldschmidt-Leitz and Karl Mayer. *Z. physiol. Chem.* 236, 168-80 (1935); cf. C. A. 29, 8012<sup>1</sup>.—Exts. of barley and malt show in addn. to starch saccharification an enzymic splitting off of  $H_2PO_4$  from org. compds. The dephosphorylating enzyme, designated amylophosphatase, can be freed from the saccharifying enzymes,  $\alpha$ - and  $\beta$ -amylase, by selective adsorption of the latter on kaolin at  $pH$  5. The amylase free amylophosphatase is capable of liberating all of the  $H_2PO_4$  organically combined in starch paste. The reaction, which is optimal at  $pH$  5.6, not only splits off  $H_2PO_4$ , but also results in a proportional decrease in viscosity and a measurable formation of reducing groups. Unlike the amylases, this enzyme is not activated by amylokinase. A similar dephosphorylation and liquefaction of starch paste is effected by amylase-free phosphatase from kidney. But the barley enzyme is the more active of the 2 in proportion to its activity toward glycerophosphate. The final effect of either enzyme is the formation of P-free cleavage products, which, after electro-dialysis and  $EtOH$  pptn., give a pure blue color with 1 and reduce  $NaOI$ . The reducing power corresponds to a chain length of 36 glucose mols. Although the products constitute a mixt., the observation is significant in view of the fact that amyloamylose with amylase yields a crystalline cryst. 18 glucose groupings and erythroamylose yields a cryst. hexose. Among the structural units of the starch mol. groupings of 6 glucose mols. or a multiple thereof seem to be of especial significance, and the linkages between them are especially amenable to enzymic cleavage. The P-free dextrin formed by amylophosphatase action is easily split by amylase, more easily in fact than starch paste. It is conceivable that the  $H_2PO_4$  occurs in ester linkage not with one but with several glucoside chains, and thus binds them together. Their cleavage would then decrease the size of the mol. and account for the lowering of viscosity. A. W. Dorr

Iron studies IX. The question of the identity of blood catalase with the easily cleavable blood iron. Georg Barkan and Jutta Oleks. *Z. physiol. Chem.* 236, 197-200 (1935); cf. C. A. 28, 1861<sup>1</sup>.—Blood catalase is not inhibited by a treatment with  $CO$  which diminishes the amt. of Fe cleavable from blood by acid treatment to  $1/2$  the normal value. This is considered further evidence that catalase is not identical with the ionizable Fe of blood. A. W. Dorr

Cytochrome IV. Cytochrome C. Karl Zeile. *Z. physiol. Chem.* 236, 212-15 (1935), cf. C. A. 28, 1851<sup>1</sup>.—In its oxidized state cytochrome C has much greater affinity for adsorbents, such as kaolin and silicic acid, than in its reduced state. This property was utilized in detg. the hemin content of cytochrome C. The difference in wt. of cytochrome adsorbed from reduced and oxidized solns. by a given wt. of adsorbent divided into the difference in cytochrome hemin detd. spectrophotometrically in the 2

ad-xobates gives the hemin content of the erythrocyte. The values thus obtained averaged 3.4%. By the use of electrophoresis app. the isoelec. point of cytochrome was found to be at  $pH$  8.2. A. W. Doy.

**Prostate phosphatase.** Waldner, K. and Hako. *Wochenschr. Z. physiol. Chem.* 236, 237-40 (1935). cf. C. A. 29, 6815. — Phosphatase is of common occurrence in the urine of males but not in that of females or old persons. It is present in high concentration in the ejaculate, and in the prostate gland which is the source of the enzyme. The other ex glands contain considerably less. The phosphatase is active in acid but not in alk. med. in its several times as active toward phenyl phosphate and  $\alpha$ - and  $\beta$ -glycerophosphate as toward hexosediphosphate. It is completely inactivated by heating 5 min. at 60°. The function of this enzyme is probably not carbohydrate metabolism. A. W. Doy.

**Visual purple.** Otto Brummer, Eugen Baroni and Wolrad Kleiman. *Z. physiol. Chem.* 235, 237-42 (1935). — Freshly dissected retinas from the eyes of steers and pigs were ext'd with abs. MeOH while carefully excluding air, the ext. was treated with an equal vol. of benzene and the soln. sep'd into 2 layers by addn. of  $H_2O$ . The benzene layer contg. the pigments was washed with  $H_2O$ , conc'd under  $CO_2$  and exp'd. with alc. KOH at room temp. The benzene layer was again sep'd by addn. of  $H_2O$  and the cholesterol ext'd. out by cooling and further by pptn. of the alc. soln. with diutecum. The benzene soln. of the pigment was then adsorbed on  $AlO_3$  and the pigment identified as  $\beta$ -carotene. The color change during adsorption and the light sensitivity of the adsorbate suggested the desirability of a comparison with visual purple. Photometer readings of the adsorbate in reflected light gave a curve quite similar to that calcd. from the Hsiova and Raveri data on visual purple. In both instances the max. is at 500 m $\mu$ . The results suggest a possible connection between carotene and visual purple. A. W. Doy.

**Creatinephosphoric acid.** Karl Zeile. *Z. physiol. Chem.* 235, 250-52 (1935). — Attempts were made to synthesize creatinephosphoric acid by 2 methods. The 1st method, a phosphorylation of  $SC(NH_2)_2$ ,  $MeSC(NH_2)_2$ , or  $HN(CNH_2)_2$ , and coupling the product with sarcosine, was unsuccessful. The 2nd method, direct phosphorylation of creatine with  $POCl_3$ , yielded a creatinephosphoric acid isomeric with the natural product from muscle. Condensation of  $(PhO)POCl_3$  with thiourea yielded  $diethylenephosphoric acid$ , m. 143°. Treatment of this with  $H_2O$  to remove the S took out  $H_2S$  and gave  $diethylenephosphoric acid$  which ex'd. out as  $Hg$  salt m. 135°. Treatment of  $MeSC(NH_2)_2$  with  $(PhO)_3POCl_3$  yielded merely the salt,  $metathiazuric diethylenephosphoric acid$ , long needles, m. 170°. Phosphorylation of creatine in pyridine with  $POCl_3$  gave a creatinephosphoric acid which could not be satisfactorily purified through the Ca salt. Finally, a  $creatinephosphoric acid$  was obtained by heating creatine in pure  $POCl_3$ . This was purified by repeated recrystn. of the Ca salt after careful removal of excess  $POCl_3$ . It was not identical but was isomeric with the natural product. The 2 substances showed differences in rate of hydrolysis, differences in the accelerating influence of molybdate and differences in dissociation curves. A. W. Doy.

**The actions of coenzyme.** Hans v. Euler and Ragnar Vestin. *Z. physiol. Chem.* 237, 1-7 (1935). — Since unactivated coenzyme no longer activates fermentation but shows diminished activity toward glycolysis, a detn. of the effect of inactivation on the phosphorylation process became desirable. Both active and inactivated coenzyme were found to have practically the same activating effect on the phosphorylation of glycogen by muscle ext. in the presence of hexosediphosphate, and on the formation of lactic acid. Coenzyme thus contains 2 catalytically active groups, the more labile of which influences fermentation and oxidation-reduction, while the more stable group participates in a phosphorylation assoc'd with lactic acid formation. The latter is probably an adenylic acid group. The enzyme system of yeast does not seem to effect a phosphorylation of pyruvic acid or creatine. A. W. Doy.

**Poisoning of fumarase.** Kurt P. Jacobson and Jow Tapadach. *Comp. rend. soc. sci.* 120, 248-50 (1935). cf. C. A. 29, 4585. — Freshly prep'd. fumarase soln. (from liver juice) were less readily inactivated by  $AgNO_3$  than old prepns. L. E. Glaser.

**Problems connected with the principle of humoral transmission of nervous impulses.** O. Loewi. *Proc. Roy. Soc. (London)* B115, 24-31 (1935). — The Ferner lecture. Joseph S. Hephern.

**Recent advances in science biochemistry.** W. O. Kernick. *Science Progress* 30, 245-252 (1935). — A review of recent work on the biochemistry of muscular contraction with especial reference to the products formed from glycogen. Joseph S. Hephern.

**Photosynthesis in intermittent light, in relation to current formulations of the principles of the photosynthetic mechanism.** G. E. Fries. *Biol. Rev. Cambridge Phil. Soc.* 10, 4-10 (1935). — A review. F. H.

**Mathematical physics of metabolism systems with reference to living cells.** N. Richey. *Physiol. Rev.* 15, 343-9 (1935). cf. C. A. 29, 117. — The effects of forces produced by metabolism on the functions and processes of the living cell are treated mathematically. C. E. P. J.

**The freeing and resensitizing of animals.** M. T. Zarwitschew. *Isis and Empiricism* 89, 133-44 (1935). — The blood stream of cold blooded animals is characterized by high  $CO_2$  and anoxic currents. When conditions are made such that the blood of warm blooded animals oxygen these materials in the proper ratio, the body temp. of the animal can be lowered without causing death. The lowering of the body temp. of rabbits, fish and other organisms is discussed. A. H. Johnson.

**Evaluation of ultraviolet radiation for use in medicine.** W. W. Cobble. *Physiol. Rev.* 15, 1-25 (1935). — The procedure proposed is to det. the ultraviolet solar spectral energy curve by means of a photoelectric cell (of Ti or Cd, which responds to only a narrow spectral range) and 1 or 2 filters, combinations with the bath. ter. To obtain the intensity in atm. units the photoelectric cell and auxiliary elec. current meter (microammeter and an plecter) are calibrated against a standard of ultraviolet radiation, a 110 v. quartz  $Hg$  arc lamp which has been standardized by means of the thermopile and filter. So long as the spectral photoelectric response remains unchanged, the evaluation of ultraviolet solar radiation of wave length shorter than and including 3130 Å, by means of a photoelectric cell, appears to be just as reliable as a direct measurement with a thermopile and filter. Differences in transmission through the above-mentioned filters were easily observed with photoelectric cells Ti No. 4 and Ti No. 2, which differed but slightly in spectral photoelectric response. After extensive use the spectral photoelectric response curves of the cells employed in the present research showed no variations comparable to the difference in the spectral response curves of cells No. 4 and No. 2. It is therefore believed that the observed seasonal changes in the transmission of ultraviolet solar radiation through the filters are owing to variations in atmospheric transparency and not to changes in the spectral response curves of the photoelectric cells. Comparative ultraviolet solar intensities in the tropics (at San Juan) and in higher altitudes (radiations, sea-level stations, Washington) are discussed in the light of C. S. Jones. Eighteen references. J. A. Kennedy.

**Noteworthy methemoglobin properties.** R. M. Mayer. *Dist. Z. ges. physiol. Med.* 23, 112-14 (1935). cf. C. A. 29, 5521. — Methemoglobin was distinguished from other hemoglobin compds. by spectral sensitiveness due to variation of H-ion concn. The  $\beta$ -band spectrum of the normal zone was transformed to the spectrum characteristic of hemoglobin and then into that of methemoglobin. Pyridine added produced brown. Excess  $K_2Fe(CN)_6$  caused a sep'n. of methemoglobin. Methemoglobin was obtained from oxhemoglobin as well as hemoglobin only by an oxidation process with evolution of the loosely bound  $N$  (not bound to porphyrin Fe by a principal valence). Francis Kriew.

**Proto-synthetic enzymes. VIII. The proto-synthetic systems of**

papaain. Max Bergmann and Wm F. Ross. *J. Biol. Chem.* 111, 659-66 (1935), cf. *C. A.* 29, 7348<sup>a</sup>.—In natural papaain there are 2 different proteolytic enzymes, a proteinase and a polypeptidase, which differ not only in their substrates but also in their behavior on oxidation. The activity of the former is reversible and of the latter not reversible after oxidation. The polypeptidase is inhibited by PhNHNH<sub>2</sub>, suggesting an aldehyde group, and the effect of I and H<sub>2</sub>O<sub>2</sub> indicates the presence of an easily oxidized group. It is possible to differentiate between 4 stages of activity for papaain: (1) natural enzyme, effective on gelatin, without effect on synthetic substrates (or peptones) but capable of being activated for the latter (by HCN, H<sub>2</sub>S or thioglucose), (2) activated natural enzyme, effective both on gelatin and synthetic substrates, (3) oxidized enzyme (H<sub>2</sub>O<sub>2</sub> or I), no activity upon either type of substrate but with potential activity for gelatin and (4) enzyme activated after oxidation, effective on gelatin but ineffective and incapable of being activated for synthetic substrates. There is no evidence whether the essential groups of the proteinase and polypeptidase are contained in the same mol or whether the 2 enzymes are 2 sep mol individuals. The synthetic substrates used have been recently described. A P. Lothrop

Yeast zymon. I. The effect of some electrolytes upon carbon dioxide production. Homer E. Stavels, L. M. Christensen and Ellis I. Fulmer. *J. Biol. Chem.* 111, 771-83 (1935).—There is an increased amt of inorg phosphate in zymon preps made from yeast which has stood for several days at refrigerator temp and this in part makes these preps more active. The initial max rate of CO<sub>2</sub> production is increased by appropriate concns of NH<sub>4</sub>Cl, MgSO<sub>4</sub>, NaCl and KCl and the steady concn rate is also increased by the 1st 3 salts and by CaCl<sub>2</sub>, NH<sub>4</sub>Cl being the most potent. NH<sub>4</sub>Cl and MgSO<sub>4</sub> increase the rate of CO<sub>2</sub> production in the presence of added inorg phosphate but the effect of the 2 salts is not additive. A  $pH$  of 5.8 to 6.2 which is approx the  $pH$  of the interior of the yeast cell is the optimum  $pH$  for CO<sub>2</sub> production. The induction period which occurs when the concn of zymon is relatively small is shortened by all of the salts which increase the CO<sub>2</sub> production rate. Twenty six references. II. The effect of ethanol upon the production of carbon dioxide. *Ibid.* 785-90.—The production of CO<sub>2</sub> by dried yeast, yeast juice, living yeast and zymon is increasingly decreased by varying concns of EtOH in the order named: A 1.85% alc soln reduces the activity of zymon 50% whereas a concn of 14% is necessary to reduce the activity of dried yeast to the same extent. The sensitivity of zymon is not decreased by the addn of inorg phosphate. Appropriate concns of NH<sub>4</sub>Cl not only decrease the sensitivity of zymon toward EtOH but actually increase the rate over that produced by the salt in the absence of EtOH.

III. The effect of some electrolytes and of ethanol upon the phosphate content during fermentation. *Ibid.* 791-802.—The optimum  $pH$  for disappearance of inorg phosphate from a zymon fermentation must be 6.2 to 6.4, the value for hexosephosphate synthesis by living yeast. The time which elapses before the inorg phosphate content reaches a min. value is markedly decreased by electrolytes, the potencies of the cations being in the order NH<sub>4</sub><sup>+</sup> > 1/2Mg > Na > K > control > 1/2Ca. The time is greatly increased and the min. steady state value of the inorg phosphate content increased by relatively low concns of EtOH. The phosphate content of a dried yeast fermentation must be little affected by EtOH, an effect similar to that on CO<sub>2</sub> production. NH<sub>4</sub>Cl, KCl and NaCl, but not MgSO<sub>4</sub> or CaCl<sub>2</sub>, decrease the min. steady state value of the inorg phosphate content when EtOH is also present. During autofermentation of zymon the inorg phosphate content increases with time. Any concn of EtOH up to 8% causes a slight decrease and NH<sub>4</sub>Cl has a similar effect so that the stimulation of CO<sub>2</sub> production by this salt is not due to increasing the available phosphate. MgSO<sub>4</sub> and Na<sub>2</sub>AsO<sub>4</sub> cause a slight increase in phosphate content. The influence of the radicals in R-NH<sub>2</sub>Cl compds in decreasing the time which elapses before the inorg phosphate content reaches a min. value is in the order CaH<sub>2</sub>O<sub>2</sub> > H >

1 CH<sub>3</sub>, the order of increasing dissociation constants of the corresponding amines. Glucosylamine-HCl is itself not esterified by phosphate. A. P. Lothrop

The quantitative changes in the enzymes present in the liver and in various tissues due to impaired renal functions. Sadamu Murata. *Japan J. Gastroenterol.* 7, 69-87 (1935).—The enzymes asparaginase, amylase, glycylglycinease and lipase were estd in the livers of rabbits at daily intervals after extirpation of one or both kidneys.

2 These liver enzymes with the exception of the third seem to increase slightly as the result of nephrectomy.

Manganese in marine plankton. L. H. N. Cooper. *J. Marine Biol. Assoc. United Kingdom* 20, 201-2 (1935).—The plankton from a cu m. of sea water have 0.5 to 3.7 of Mn. C. M. McCay

Hemolysis in vitro is not reversible. Andrea Manai. *Biochim. terap. sper.* 22, 269-70 (1935).—Hemolysis in hypotonic solns can be stopped by addn of hypertonic liquid to produce isotonic concn. But the process is not reversible. A. E. Meyer

The influence of ascorbic acid on the viscosity of gelatin solutions. Luisa Pozzi. *Biochim. terap. sper.* 22, 356-62 (1935).—The viscosity is decreased by ascorbic acid beyond the effect that can be ascribed to the acidity. The possible relation to scurvy is discussed. A. E. Meyer

Biological action of metals irradiated with the mercury-quartz lamp. V. Effect of irradiated metals on the condition of the capillaries. V. G. Budlyn. *Acta med. scand.* 86, 88-94 (1935); cf. Goldberg, *C. A.* 29, 4374<sup>a</sup>.—Solns of Fe and Cu irradiated with the Hg-quartz lamp produce capillary dilatation in frogs when injected subcutaneously. Similarly, perfusion of the isolated rabbit ear with such a soln gives evidence of a capillary relaxation. VI. Effect of irradiated iron and copper on the water content of the blood. *Ibid.* 95-9.—Intravenous injection of 10 cc. hypotonic soln of Fe or Cu irradiated with the Hg-quartz lamp causes within the 1st hr. a rise in the hemoglobin content of the blood. Similar effects were observed on splenectomized rabbits with an increased no. of erythrocytes. The viscosity of the rabbit serum also increases under this treatment. These results are interpreted as signifying a shifting of water from the blood into the tissues. S. Morgulis

Intracellular proteinases. XVI. Further studies on the activation and inhibition of proteinases. Ernst Maschmann and Fritz Helmert. *Biochem. Z.* 280, 184-203 (1935); cf. *C. A.* 29, 8023<sup>a</sup>.—Full details of the methods for producing activation and inhibition of proteolytic processes are given. The reactions involved, however, are very complicated and no interpretation can be given of them as yet. XVII. Effect of different arsenic compounds on the activity of liver cathepsin. Ernst Maschmann. *Ibid.* 204-12.—Arsphenamine activates while solocalvaran and benzazon inhibit the hydrolysis of gelatin by cathepsin. Arsphenamine does not reactivate inactive cathepsin. Furthermore, it inhibits the hydrolysis of chapein. All cathepsin organ exts give a definite SH-test, except the digestion mixts in which the hydrolysis has been inhibited, where the SH reaction is neg, but no conclusion is drawn as to the relationship between the 2. Neither can any conclusion be drawn as to the effect of As compds because this depends not only on the enzyme prepn but also on the nature of the substrate. S. Morgulis

Effect of ultraviolet light on yeast sucrase. VI. The yeast sucrase-activating wave length region. G. Gorbach and H. Ruess. *Biochem. Z.* 280, 213-16 (1935); cf. *C. A.* 29, 2187<sup>a</sup>.—Brief radiation of yeast autolyzates or dialyzates by wave length 365-366 mμ causes an increased sucrase activity. The blue fluorescence of the enzyme soln is of no consequence since this can be intensified by means of ficulin without increasing the enzyme activity. S. Morgulis

The significance of phosphate esters for the progress and regulation of blood glucolysis. II. Decomposition of hexose monophosphate in hemolyzed blood following phosphorylation in a reaction coupled with the dephos-

phorylation of adenosine triphosphate. Zacharias Dische. *Biochem. Z.* 280, 248-64 (1935); cf. *C. A.* 29, 823<sup>1</sup>.—When the hexose monophosphate undergoes splitting in hemolyzed blood it is phosphorylated at least to the extent of  $\frac{1}{2}$ , at the expense of  $H_2PO_4$  from the hydrolysis of adenosine triphosphate. Since it is possible that a synthesis accompanies this hydrolysis, it is not improbable that the entire  $H_2PO_4$  used in the phosphorylation of the hexose monophosphate comes from this source. The velocity of the esterification process varies with the concn of the hexose ester. Both the Embden and the Neuberg esters become phosphorylated at the same rate, therefore, the ketose and aldose components partake equally in this process. The previous idea of D. that the monophosphate breaks up directly into 1 mol. triose ester and 1 mol. triose is now held untenable. The phosphorylation of the hexose monophosphate by transference of  $H_2PO_4$  from adenosine triphosphate is, therefore, a very essential phase in the combination of the partial processes of glucolysis, and presupposes a rephosphorylation of the adenylic acid. This has actually been shown to be the case in muscle (Parnas) and in muscle ext (Meyershof and Lohman). D. described a mechanism of rephosphorylation of adenylic acid in intact red cells as well as in hemolyzed blood, where the decoupling of hexose diphosphate in the presence of 0.05 M NaF is strongly accelerated by pyruvic acid, whereby an increase in the easily hydrolyzable P and a decrease of the directly determinable P is demonstrated. In this process the pyruvic acid changes to lactic acid, and the P ester formed is approx. equal to the lactic acid so formed. The oxidation-reduction process between pyruvic acid and hexose diphosphate or triose phosphate, which is coupled with the resynthesis of adenosine triphosphate, proceeds much more rapidly than the oxidation-reduction between the triose ester mols. The phenomenon has also been observed in minced rat muscle to which pyruvic acid had been added in the presence of NaF. In the fermentation of yeast press juice there is an equivalence between the amt. of  $CO_2$  set free and the amt. of  $H_2PO_4$  esterified as mono- and diphosphates. Instead of the older view, that this results from a coupling of phosphorylation and breakdown of sugar, the coupling is regarded as taking place between partial fermentation processes and the phosphorylation (with inorganic  $H_2PO_4$ ) of adenylic acid formed in the phosphorylation of sugar by adenosine triphosphate. This explains the equivalence between the  $CO_2$  and esterified  $H_2PO_4$ , although the esterified  $H_2PO_4$  appears partly as mono- and partly as diphosphate of hexose in variable proportions. In blood glucolysis and probably also in muscle glucolysis the rephosphorylation of adenylic acid is coupled with the oxidation-reduction reaction between pyruvic and triose esters, whereas in fermentation the coupling is with the oxidation-reduction reaction between AcII and triose ester. S. Morgulis.

Activation of the proteolytic enzymogen system of the stomach. Rich. Ege and Jens Obel. *Biochem. Z.* 280, 265-75 (1935).—Neutral or slightly acid exts. from gastric mucosa contain relatively little preformed coagulating enzyme, but with a H-ion concn. of  $10^{-4}$  to  $10^{-3}$  the enzyme is immediately activated. Such exts. were made from the stomach of a no. of different animals. Activation of the enzyme begins only when the acidity of the ext. approaches a H-ion concn. of  $10^{-4}$ , and its velocity increases with the H-ion concn., but not in the same manner for the different animals. The zymogen is much more stable in an alk. medium than the active enzyme prepn. S. Morgulis.

The state of the glycogen inside the cell. I. A ternary complex from clupein, nucleic acid and glycogen or dextrin. St. J. v. Przybylski, W. Giedroyc and H. Rafalowski. *Biochem. Z.* 280, 286-92 (1935).—Even within the biol.  $\beta$  range clupein contains groups which react with glycogen. Furthermore, in a mixt. of clupein, glycogen and nucleic acid in definite proportions and at pH 7.0 a complex is actually produced consisting of all 3 components. The amt. of nucleic acid must not exceed the available free arginine. In the fish sperm nuclei no such ternary system can be formed. S. Morgulis.

Studies on the affinity relationship of animal and plant dipeptidases. W. Grassmann, L. Klenk and T. Peters-Mayer. *Biochem. Z.* 280, 307-24 (1935).—According to Bergmann and Zervas (*C. A.* 28, 4075<sup>1</sup>), dipeptidase splits only CO-NH combinations which have free  $\alpha$ -amino and COOH groups. While the nature of C chains is of no significance so far as the qual. process of the dipeptide hydrolysis is concerned, this does affect the hydrolysis quantitatively. Expts. were made with dipeptidases from yeast or kidney. Studies of the affinity show that this varies with different substrates: glycylglycine (G-G) < alanyl glycine (A-G) < leucyl glycine (L-G). The affinity of (A-G) is 4-10 times as great as that of (G-G) and that of (L-G) is at least 10 times greater than that of A-G. Although the affinity const. varies with the enzyme prepns., the activity  $\beta$ -curves are shifted uniformly in all instances, and the affinity between enzyme and dipeptide is influenced by the C chain to which the free NH<sub>2</sub> group is attached, increasing with the length of the chain. The nature of C chain bearing the COOH group is less significant in detg. the affinity. For yeast dipeptidase the max. rate of hydrolysis is obtained with the following substrate concns.: G-G 0.125 M, G-A 0.024 M, and G-L 0.024 M. Since amino acids inhibit the peptide hydrolysis and the substrate affinity decreases in the order L-G > A-G > G-G, the inhibiting effect of the amino acid under similar conditions must vary with the nature of the substrate in the order L-G < A-G < G-G, which has been invariably found with both yeast or kidney dipeptidases. Furthermore, independently of the substrate concn., the inhibiting effect increases with the nature of the amino acid as follows: leucine > valine > alanine > glycine. S. Morgulis.

Transformation of aromatic and of hydroaromatic compounds by microorganisms II. Transformation of quinic acid and inositol. Konrad Bernhauer and Bruno Gölisch. *Biochem. Z.* 280, 394-5 (1935); cf. *C. A.* 26, 4591.—*B. glaucum* and *Pen. griseofulvum* form protocatechuic acid from quinic acid. Inositol is not changed to an aromatic compd. but the mol. is split with the formation of AcOH and probably also of lactic acid. S. M.

Blood iodine problem. A. Sturm, K. Möstner and K. Maass. *Biochem. Z.* 280, 390-412 (1935).—The tech. problem of blood I detn. is discussed, and the need of special training in carrying out these analyses is emphasized. Blood I values of over 20% found on consecutive days in the quiet, fasting condition are regarded as being definitely hyperthyroid. In myxedema or in congenital athyreosis the blood I remains permanently at the low level of 3-5%. S. Morgulis.

The lecithin and polysaccharide symplexes. St. J. v. Przybylski and R. Majmin. *Biochem. Z.* 280, 413-15 (1935).—Studies on lecithin-starch symplexes show that about 50 mols. of lecithin combines with 1 mol. of starch. The lecithin-glycogen symplex contains 60% glycogen and is easily sol. in  $H_2O$  or in NaOH. The lecithin-dextrin symplex is also very sol. and contains about 4 lecithin particles to 1 dextrin. S. Morgulis.

Blood iodine values. Helmut Doernne. *Biochem. Z.* 280, 412-7 (1935).—The blood I values obtained by the open combustion method are considered to be correct. The low values by the Fellenberg method can be reproduced also by the Schwalboid-Reith or by Pfeifer's method, if these are properly carried out. On the other hand, the higher values found by the "closed" combustion procedure are attributed to traces of unused oxidation reagent which in acid medium set free I<sub>2</sub> from the KI. Another source of error in the closed system is the incomplete removal of Cl or Br in the presence of traces of org. impurities. S. M.

Studies on the chemical embryology of *Hymobius*. Makoto Takamatsu. *J. Biochem. (Japan)* 22, 203-11 (1935).—A study of the *Hymobius* egg shows that the relative  $H_2O$  contents decrease in the course of development, both in the embryo and in the jelly, but increase in the perivitelline fluid. The latter has a  $\beta$  of about 6.9-7.0. The total ash content reaches a max. at the middle period of development (3-4 weeks) and its compn. is recorded. The total N content of the embryo decreases gradually while the nonprotein N increases. There is a

marked rise in the creatine and creatinine content, after the second week of incubation, while the urea gradually falls to about  $\frac{1}{2}$ , an increase was found in the purine N, also in the arginine and histidine N fraction but most noticeably in the lysine N. The study of the egg enzymes shows a very close similarity to the situation found in the bird or reptile egg, the gly cogenase and gly cerophosphatase activity being especially strong. S Morgulis

The liberation of arginine in proteolysis. Shigekazu Kamiya. *J. Biochem. (Japan)* 22, 203-77 (1935).—In the digestion of casein or gelatin by pepsin found by erepsin the total arginine is set free, whereas by the enzymic action of pepsin, trypsin, then erepsin only  $\frac{1}{2}$  of this amino acid is thus liberated. The arginine detn was carried out by means of the highly specific arginase-urease procedure or by means of flavimic acid. The results by both methods agreed. S. Morgulis

Studies on the blood  $pH$  estimated by the glass electrode method II. Effects of anticoagulants on the  $pH$  of the blood. Hisato Yochimura. *J. Biochem. (Japan)* 22, 279-95 (1935), cf. C. A. 29, 4432.—Five %  $K_2C_2O_4$ , 5% Na citrate, 1% NaF, 2% NaF + 1%  $K_2C_2O_4$ , 0.5% hirudin or 1% heparin when added to blood in the proportion of 1 to 10 has no effect on the  $pH$  of freshly drawn blood. Addn of the same proportion of 10%  $K_2C_2O_4$  causes a marked shift in the blood to the alk. side, whereas with 2% NaF, or 3% NaF + 1%  $K_2C_2O_4$  or 10% Na citrate the shift is to the acid side.  $K_2C_2O_4$  is the most suitable anticoagulant because it hardly affects the  $pH$  in the concn. used and, by suppressing glycolysis, it prevents the acid change. It is recommended to add 2%  $K_2C_2O_4$  to blood in the ratio of 1 to 9, to sep the plasma, especially from leucocytes, at room temp within an hr, and to estimate the  $pH$  on this plasma. Does the  $pH$  of the blood change during clotting? *Ibid* 297-302 (1935).—In blood clotting at 16° there is no shift to the acid side. On the contrary, the acid change is found in blood in which clotting has been prevented by means of an anticoagulant. S. M.

The mechanism of anaerobic decomposition of hexose-phosphoric acids by animal tissues. III. Simple hydrolysis of hexosephosphoric acids by phosphatase. Takehiko Takaishi. *J. Biochem. (Japan)* 22, 303-21 (1935), cf. Nakamura, C. A. 29, 2589.—Formation of AcCHO is not apparently a const. phenomenon in the production of hexosemonophosphoric acid from hexose diphosphate by autolyzed tissue. The monophosphoric acid and free sugar can be produced directly by the simple hydrolysis of the ester by phosphatase. S. Morgulis

Chemical studies of the proteins of foodstuffs V. The contents of cystine and tryptophan. Tetsuo Tomiyama. *J. Biochem. (Japan)* 22, 341-2 (1935); cf. C. A. 29, 4390.—Silkworm pupa protein contains 0.86% cystine, 3.83% cystine + methionine, 1.50% tryptophan. The proteins of sardine and of soybean contain of these substances 0.76, 3.90 and 1.40%; and 1.05, 3.01 and 1.35%, resp. S. Morgulis

The Bence-Jones' proteins. Hikoya Tatsui. *J. Biochem. (Japan)* 22, 343-50 (1935).—The material was obtained from a patient who, on x-ray exam., revealed general atrophy of the entire skeletal system. A protein substance was found in his urine which differed from ordinary protein only by its peculiar heat behavior. The substance was prep'd by warming the urine acidified with AcOH to 55° and sep'd by centrifuging. The ppt., washed with  $H_2O$  and alc., has 13.50-13.75% N, 0.30-0.41% S and 5.04% ash. Dissolved in 0.1 N NaOH it has a  $pI$  = -95.78. Its isoelec. point is at  $pH$  4.72. A complete analysis of its amino acid compn. shows that the diamino fraction in different samples constitutes 20.3-26.6% of the total N. Digestion with pepsin for 4 hrs results in complete conversion to peptone, subpeptone and proteose. Digestion with trypsin results in the liberation of 41% of the total N as amino N in 48 hrs. S. M.

Oxygen utilization in the vital oxidation of succinic acid in relation to the  $pH$  and oxygen tension. A study of the toxic action of oxygen. Jørgen Lehmann. *Skand. Arch. Physiol.* 72, 79-91 (1935).—At  $pH$  less than 7.4 the max utilization of  $O_2$  by a succinic acid-succinic dehydro-

genase system is attained at 44-55 mm. Hg (6-8%  $O_2$ ), and at greater tensions, up to 713 mm., the  $O_2$  utilization is neither increased nor inhibited. At  $pH$  greater than 7.4 the max.  $O_2$  utilization is still at the same ("critical") tension but in this case the  $O_2$  at higher tensions inhibits the oxidation progressively with increasing  $O_2$  tension. The toxic effect is exerted on the enzyme only in the presence of the succinic acid, and the injurious effect is irreversible. S. Morgulis

The esterase activity of human blood plasma. Bo Vahlquist. *Skand. Arch. Physiol.* 72, 133-60 (1935).—To decide whether human plasma contains a specific choline esterase or the hydrolysis is brought about by the ordinary lipase, a study was made by various methods. Catephoretically both activities moved strictly parallel in the elec. field and independently of the migration of the albumin and globulin. Similarly quinine, atoxyl and physostigmine inhibited the action of the esterase no matter what substrate was employed (acetylcholine, tributyrin or Me butyrate). Parallel detns. of choline and tributyrin esterase activity were made on different individuals under a great variety of conditions. The correlation of all these results was so great that the correlation coeff. was  $0.92 \pm 0.02$ . All 3 modes of attack on this problem indicate, therefore, that there is no specific acetylcholine esterase. The esterase content is not appreciably affected by ingestion of food, muscular exercise, nervous excitement, menstruation or pregnancy. Under conditions of abnormal muscular spasms such as bronchial asthma or ulcus ventriculi the values are relatively low but still within the normal range. Only in tuberculosis is the esterase content abnormally low. The esterase apparently can only act to protect the organism against an accumulation of acetylcholine in the blood. S. M.

The influence of heavy water on the development of amphibian eggs. Hans H. Usung. *Skand. Arch. Physiol.* 72, 192-8 (1935).—A concn. of  $D_2O$  above 5% retards the segmentation of frog eggs, the retardation increasing rapidly with rising concn. At concns. above 30%, normal segmentation no longer occurs, and at 25% development stops at the first or second cleavage division. Eggs from *Bufo varius* developed normally and hatched in 4 days in 10%  $D_2O$ , but in 30 and 40% the development proceeded normally for 24 hrs., then the eggs all died. The  $O_2$  consumption of *Bufo* eggs from the same batch shows great uniformity. The eggs in 30%  $D_2O$  show the same  $O_2$  consumption as those in  $H_2O$  for the first 14 hrs of development, when it gradually decreases to zero during the next 24 hrs. S. Morgulis

Osmosis in living cells and change in weight. G. Fallster. *School Sci. Rev.* 17, 136 (1935). O. R.

Osmosis through living cells. G. Fallster. *School Sci. Rev.* 17, 136 (1935). O. Reimnuth

Some experiments with amylase. C. O. Björling. *Svensk Farm. Tid.* 39, 453-7, 470-4, 485-92, 501-4 (1935); cf. Ohlsson and Rosén, C. A. 29, 1209.—The amylase extd. from ungerminated grain attacks starch and yields maltose and a substance which by all the phys. and chem. tests proved to be starch. A. R. Rose

Phosphate exchange in glycolysis. Hans v. Euler and Ragnar Vestin. *Svensk Kem. Tid.* 47, 231 (1935).—In German. Cozymase loses its function in fermentation if heated but the activation of glycolysis is not altered. In the system Muscle ext. + glycogen + hexose phosphate + cozymase, it makes no difference if the cozymase is heated or not. A. R. Rose

Purification of the active phosphatase found in dog feces. Arthur R. Armstrong. *Biochem. J.* 29, 2020-2 (1935).—A method is described for the prepn. of a very active phosphatase contg. powder from feces. The yield was 0.1-0.5 g./kg. feces of material contg. 130-185  $\times 10^3$  units per g. (estn. by the phenyl phosphate method). The only optimum  $pH$  range for the enzyme was around 9.6. The max. activation by  $Mg^{++}$  was below 0.0003 M. E. W. Scott

Liver glycogen II. Acyl derivatives and "regenerated" glycogens. David J. Bell and Hans Kosterlitz. *Biochem. J.* 29, 2027-30 (1935), cf. C. A. 28, 6735; 29,

823\*—Rabbit and fish liver glycogens did not appear to undergo any degradation when acetylated or benzoated and then regenerated from these acyl derivs. The org. P contents of these glycogens were low and decreased after the above treatment. Glycogen triacetate and tribenzoate prepds. from fish or rabbit glycogen possessed the same properties. III. The molecular units of fish and rabbit glycogen David J. Bell. *Ibid.* 2031-5.—Both fish and rabbit glycogens were shown by means of methylation to have the same structure. Hydrolysis of the completely methylated glycogens gave tetramethylglucopyranose, 2,3,6-trimethylglucose and dimethylglucoses and showed that both glycogens were built up of unbranched chains of 12 glucose units E. W. Scott

The inhibition of respiration by cyanide. Wm E van Heyningen. *Biochem J* 29, 2036-9(1935)—The respiration of liver, kidney and spleen slices in 0.01 M phosphate-saline medium was inhibited to the extent of 75-85% by 0.001 M HCN and 80-90% by 0.01 M HCN. The discrepancy between these results and the much lower results of Dixon and Elliott (*C A* 24, 812) was due to several factors, e. g., the loss of HCN from the media and the high phosphate concn. used by D. and E.

E. W. Scott  
A water-soluble precursor of choline found in the kidney and other tissues Frederick J. Booth. *Biochem. J.* 29, 2071-6(1935); cf. *C A* 29, 6911\*—An extremely water-soluble substance (I) with chem. properties which indicated that it was the choline ester of sphingosinophosphoric acid was isolated from fresh kidney tissue by pptn. as the  $HgCl_2$  double salt. The double salt was sol. in cold water (6°) and was purified by repeated pptn. with  $HgCl_2$ . The water-sol. ester of choline isolated from fresh brain and liver tissue possessed properties similar to I. I had no action on the everted frog rectus. It was decomposed by acetylation, apparently forming acetylcholine.

E. W. Scott  
Total plasma protein in normal and fasting rats. W. C. Cutting and R. D. Cutter. *Am. J. Physiol.* 113, 150-8 (1935).—A perfusion method for detg. total blood vol of rats is described. By the use of this method and an estm. of the concn. of protein in the blood, the total plasma protein of a rat can be measured. During fasting the total plasma protein decrease is proportional to time. The protein of lymph resembles that of plasma in its compn. and its probable entrance into the same bodily activities.

E. D. Walter  
Isolation of phosphocholine from beef liver. Fumito Inukai and Waro Nakahara. *Proc. Imp. Acad. (Tokyo)* 11, 260-1(1935); *Sci. Papers Inst. Phys. and Chem. Research* 28, 10-13(1935).—A yield of 0.3 g. of phosphocholine as the cryst. picrate was obtained from 200 kg. of fresh beef liver. The crystals softened at 225° and m. 228°. Elementary analysis agreed well with the theoretical. Expts. indicated that the crystals were an addn. compd. of 1 mol. of picric acid and 2 mols. of phosphoric choline ester. The picrate of the synthesized compd. showed no depression of m. p. when mixed with the picrate of the naturally isolated substance. R. P. W.

Ricinusp, its nature and specificity. Herbert E. Longenecker and D. E. Haley. *J. Am. Chem. Soc.* 57, 2019-21(1935).—A dry, stable and highly active prep. of Ricinus lipase (I) has been obtained from hulled castor beans by extr. the fat with low-boiling petr. ether, pulverizing and sifting the residue of the bean through a 60-mesh sieve. With 0.1-g. sample, a sample freshly prepd. showed 86% hydrolysis of olive oil, while a sample 10 years old showed 17-49% hydrolysis. A  $H_2O$ -in-oil emulsion appears to be desirable for I action; the formation of this emulsion is aided by shaking and by lower temps. of incubation. Rates of hydrolysis of a variety of oils of vegetable and animal origin are reported for study on the nature of action and specificity of I. Under the exptl. conditions, I catalyzed the hydrolysis of the following oils, listed in order of decreasing percentage hydrolysis after a given time: peanut, castor, corn, cottonseed, soybean, rape, olive, linseed, neat's-foot, peach kernel, coconut, whale, fish and sperm. An analysis of the data on the

basis of the no. of moles hydrolyzed reveals that I showed no specificity in its attack on glyceride mols. contg. C chains of different lengths. C. J. West

Occurrence of decomposition products of chlorophyll III. Isolation of pyroporphyrin from beef bile. Paul Rothmund. *J. Am. Chem. Soc.* 57, 2179-80(1935). cf. *C A* 29, 839\*—Pyroporphyrin (I) was found in beef bile under physiol. conditions, its quantity changed only very slightly with the season of the year, traces of coproporphyrin (II) were also always present. The sepn. from phylloerythrin and from other pigments in bile was performed by fractionation of an  $Et_2O$  ext. of the pigment with  $HCl$ . It is assumed that the occurrence of I in beef bile is due to decompn. of chlorophyll, while the presence of II is accounted for by the decompn. of hemoglobin. No evidence for the presence in bile of other chlorophyll porphyrins could be obtained, neither was it possible to demonstrate the occurrence of other porphyrins from blood pigment C. J. West

Action of light rays (ultraviolet) on the skin and accumulation of cholesterol Angel H. Roffo. *Compt. rend.* 201, 566-8(1935); cf. *C A* 29, 5077\*—White rats which had their backs or ears exposed to the sun or ultraviolet showed a marked increase in cholesterol as compared with those areas not exposed. Julius White  
Flavinphosphoric acid from the liver H. Theorell, P. Karrer, K. Schöpp and P. Frei. *Helv. Chim. Acta* 18, 1022-6(1935); cf. *C A* 29, 2538\* Julius White

Sterols as starting material for hormones, vitamins and other physiologically important compounds A. Windaus. *Nachr. Ges. Wiss. Göttingen, Math.-physik. Klasse* III, [N. S.] 1, No. 7, 50-83(1935) Julius White

Guraly Pereira, D. Jose. La ciclopoyesis en el organismo animal. Madrid. Victoriano Suarez. 101 pp. Ptas 6

## B—METHODS AND APPARATUS

STANLEY R. BENEDICT

A simple arrangement for the study of the absorption of electric waves in passing through the body G. Petrucci. *Nuovo cimento* 12, 361-7(1935).—This arrangement permits the detn. of the absorption by tissue of the human body of waves several cm. in length. The absorption of NaCl solns. shows a min. at 6%. This behavior is related to the dielec. const. of these solns. J. B. Austin

Determination of blood urea by enzymic action and direct nesslerization. Adolfo E. Raices. *Rev. med. quim. patol. femenina* 5, 531-40(1935).—To 5 cc. of tungstic acid blood filtrate in a test tube, add 2 drops of acetate buffer (15 g. Na acetate in 75 cc.  $H_2O$  + 1 cc. glacial acetic acid, made up to 100 cc. with  $H_2O$ ) and 1 cc. of urease ext. dild. 1:10. Keep in the water bath at 50° for 15 min. (or for 25 min. at room temp.), shaking 2 or 3 times. Transfer, after cooling, to a 25-cc. graduated test tube. Add 1 cc. ghatti gum soln. (2%) and 2.5 cc. Nessler reagent; make up to 25 cc.; compare with the usual standard made up with  $(NH_4)_2SO_4$ . The urease ext. is prepd. as follows: to 15 g. of  $NH_4$ -free permittite add 100 cc. of 0.001 N  $H_2SO_4$ , 30 g. urease and 150 cc. glycerol. Shake for  $\frac{1}{2}$  hr. and filter with suction. This filtrate, slightly turbid, is very active and can be kept at room temp. without alteration for several months.

E. S. G. Barron  
A simple apparatus for demonstrating the action of the heart and circulation of the blood. Ralph J. Shaw. *Science Education* 19, 103-4(1935).—An app. is described and illustrated which indicates the action of the circulatory system. W. H. Boynton

Economy of urine investigations. II. Microscopic examination of urinary sediments. Paul Schugt. *Pharm. Ztg.* 80, 909, 922-4(1935); cf. *C A* 25, 5184\*—Microscopical methods have been critically examd. and improvements suggested when apparently needed for eventual use in pharmacopoeial revision. W. O. E.

Determination of glucemia. I. Deproteinization by cadmium hydroxide. Christian Dumazert. *Bull. soc. chim. biol.* 17, 1163-70(1935)—See *C A* 27, 4827. II.



Iodometric microdetermination of glucemia *Ibid* 1171-17—See C. A. 29, 7366<sup>1</sup> L. E. Gilson

Acid base equilibrium [of the blood] and Henderson's equation D. D. Van Slyke *Bull. soc. chim. biol.* 17, 1184-6(1935)—Reply to Reding, *et al.*, C. A. 29, 2989<sup>4</sup>. L. E. Gilson

The Donaggio reaction G. Sollazzo<sup>1</sup> and V. Cortese. *Boll. chim. farm.* 74, 625-37(1935).—Expts confirmed the Donaggio phenomenon as one of pure colloidal protection, caused by a thymine-molybdate lake. Pos unies showed no diminution in reaction after putrefaction for 15-30 days but became neg when subjected to the action of pepsin and AcOH. A substance giving a pos reaction was isolated from pos unies by pptn with abs alc. This substance was water sol and was pptd with NaCl, FeCl<sub>3</sub>, tannic and picric acids but gave no xanthoproteic or biuret reactions.

Gravimetric method for the determination of fat and cholesterol in blood G. Gorbach and R. Kadner. *Mikrochemie* 18, 266-71(1935).—Bang's methods (C. A. 12, 2333, 13, 2216) for detg. fats and lipids in blood have been much used and have undergone various modifications (cf. Rappaport, C. A. 29, 1477<sup>4</sup>). In this paper a new microextn app is described which permits further improvement. Details concerning the procedure are given.

Sugar determination by the ferricyanide electrode Philip A. Shaffer and Ray D. Williams. *J. Biol. Chem.* 111, 707-23(1935).—A method is described for the detn. of reducing sugars by the ferricyanide electrode by which satisfactory results are easily and conveniently obtained over a wide range of sugar concns., if interfering substances are absent. Blood sugar may be detd on as small a quantity as 0.02 to 0.05 cc. of blood if the filtrates are prep'd by Zn(OH)<sub>2</sub> pptn and the method gives approx. correct values for "true sugar". The method is applicable to the analysis of similar quantities of other solids. It is not advocated as a substitute for satisfactory titrimetric or colorimetric procedures unless the no. of analyses to be made, the vol. of blood available or other circumstances justify its choice. For only occasional analyses or in the hands of workers not somewhat familiar with the technic of potential measurements, it would be less satisfactory than the standard methods now generally used.

A new method of extraction of nucleoproteins V. Ducceschi and A. Cardin. *Biochim. lerap. sper.* 2, 265-8 (1935).—Pancreas tissue was kept with 10 parts of a 1% formaldehyde soln at 37° for 24 hrs. The following extn. gave a purer product than extn. of untreated pancreas.

Gastric analysis by fractionated testing Marie Permentier, R. Letulle and C. Bergès. *Presse méd.* 43, 581-3 (1935).—The technic is described and the advantages are pointed out.

Colorimetric methods for the determination of urinary protein, plasma protein, urinary and plasma albumin and for the serial salting out of these proteins Hilding Berglund and Walter de M. Scriven. *Acta Med. Scand.* 86, 82-7(1935).—Measure 1-10 cc. urine into a 15-cc. centrifuge tube calibrated at 10 cc. Dil with H<sub>2</sub>O, if necessary, to 10 cc. vol. Add 1 cc. 50% trichloroacetic acid, leave 10 min at room temp and 10 min at 50°, centrifuge 10-30 min to pack down the coagulum, decant, wash 4 times with 4% trichloroacetic acid, mixing each time and leaving in a water bath at 50° before centrifuging. Dissolve the ppt in 1-2 drops 10% NaOH and dil to the 10-cc. mark with H<sub>2</sub>O. Det. N by the Folin-Wu method in an aliquot. To calc. the mg. % N in urine divide 400,000 (if the standard was set at 20) by the product of the unknown colorimeter reading, vol. of urine analyzed and vol. of the aliquot. To det. the albumin fraction in the urine, weigh out 11.02 g. anhyd. neutral Na<sub>2</sub>SO<sub>4</sub> into a 50-cc. vol. flask, add 5 cc. buffer of pH 7.0 and 10-40 cc. urine, depending upon the protein concn. Dil to the mark, keep at 45° and shake frequently until the salt has all dissolved. Leave at room temp and adjust the vol., then after 1 hr. filter through double thickness until a clear filtrate is obtained. Det.

the N in a 5-10-cc. aliquot of the filtrate. For plasma the procedure is as follows. Dil 1 cc. plasma to 100 cc. in a vol. flask, det. the total N in 2 cc. and correct for the non-protein N. Measure another sample, usually 1 cc., into a 50-cc. flask, add 2 cc. pH = 7.0 buffer and sufficient Na<sub>2</sub>SO<sub>4</sub> soln to give a final concn. of 22.05%. Mix well, let stand 1 hr. at room temp. and filter until clear. Det. the N in the filtrate. The stock Na<sub>2</sub>SO<sub>4</sub> soln is prep'd by dissolving 650 g. of the cryst. salt in 1 l. Det. by titration of 20 cc. the amt. of 0.1 N NaOH required for neutralization. Add the calcd. amt. to 980 cc. and make up to vol. Det., by igniting the residue from 2 cc., the exact concn. and calc. the no. of cc. to prep. 50 cc. of 22.05%. The stock soln. should be kept at about 40°. For the fractionation of the urinary protein, measure into each of five 50-cc. vol. flasks 5 cc. of the buffer and 5.35, 7.1, 8.9, 11.02 and 12.1 g. Na<sub>2</sub>SO<sub>4</sub>, add to each 10-40 cc. urine (depending upon protein concn.), keep 1 hr. at 37°, filter clear and analyze 5-10 cc. of the filtrate for N. Total N - N fraction 1 = fibrinogen N; N fraction 1 - N fraction 2 = euglobulin N; N fraction 2 - N fraction 3 = pseudoglobulin-I N; N fraction 3 - N fraction 4 = pseudoglobulin-II N; N fraction 4 - N fraction 5 = albumin-I N and N fraction 5 = albumin-II N. S. M.

An approximately quantitative method for the determination of stereoporphyrins I. Boas. *Biochem. Z.* 280, 227-31(1935).—The acetone and alc. employed in the purification of feces dissolve out appreciable quantities of porphyrins which should not be neglected. The various porphyrins (copro-, deuter- and proto-porphyrin) are extd. from the ether soln. by shaking with 0.1, 0.4 or 2 to 5% HCl, resp. The no. of times this must be repeated with a specified amt. of the acid until the soln. no longer shows the absorption band is utilized as a measure of the particular porphyrin.

Volumetric determination of hemoglobin Istvan Rusznayk and E. B. Hatz. *Biochem. Z.* 280, 242-7(1935).—Sat. a measured quantity of defibrinated blood with CO, drive off excess CO with N<sub>2</sub>, set the bound CO free with K<sub>4</sub>Fe(CN)<sub>6</sub>, and the CO is carried off in a stream of N<sub>2</sub> into a soln. of KBr in PdCl<sub>2</sub> (reaction: PdCl<sub>2</sub> + CO = Pd + COCl<sub>2</sub>) which causes the sepn. of an equiv. quantity of metallic Pd. The finely divided Pd is taken up in a measured amt. of standard 0.02 N KBrO<sub>3</sub> and acidified. An equiv. amt. of Br<sub>2</sub> is set free which reacts forming Pd-Br<sub>2</sub>. The excess Br<sub>2</sub> is detd. titrimetrically with 0.02 N Na<sub>2</sub>AsO<sub>4</sub>. One cc. 0.02 N KBrO<sub>3</sub> = 0.224 cc. CO and 1.34 cc. CO = 1 g. hemoglobin. The calcn. is, therefore, (a - b) × 22.4/1.34 = g. hemoglobin, where a and b = cc. Na<sub>2</sub>AsO<sub>4</sub> soln. used in the blank and the detn., resp.

Method for the determination of carbon in biological fluids F. Lauersen and K. Voigt. *Biochem. Z.* 280, 276-85(1935).—A combustion train is described for oxidizing org. material in a slow stream of dry, CO<sub>2</sub> free air, and passing the CO<sub>2</sub> formed through Ba(OH)<sub>2</sub>. The amt. of Ba(OH)<sub>2</sub> used up is measured by titration with 0.01 N HCl with Tashiro's mixed indicator. The acid is added until an intense red color is produced and the excess is titrated back with 0.01 N NaOH until the color changes to a grass green. The prepn. and method for keeping of the Ba(OH)<sub>2</sub> soln. are discussed.

A systematic study of the value of different hypophysis stains with comments on the number of the anterior lobe epithelial cells and their reciprocal relations A. L. Burgdorf. *Endokrinologie* 16, 148-60(1935).—Three types of staining on formalin fixed material, namely, hematoxylin-eosin, methylene blue-eosin and staining of the colloids (Kraus), are sufficient for all purposes of detn. In normal human hypophyses the relation between the acidophile, chief and basophile cells is 30:8:45:8:23:4. The granules of both chromophile cell types (acidophile and basophile) are specifically secretory in nature.

Barbiturates. XI. Further contributions to methods of barbiturate research Charles R. Linegar, James M. Dille and Theodore Koppanvi. *J. Am. Pharm. Assoc.* 24, 847-52(1935); cf. C. A. 29, 886<sup>4</sup>.—Two practical methods

of clearing highly colored urines for the purpose of detg. barbiturates are described: (1) Add 2 g. Na molybdate per 25 cc. of urine and sufficient 20-30%  $H_2SO_4$  to produce a heavy green ppt., filter and ext. the filtrate with  $CHCl_3$  as usual, the method does not destroy barbitol and is effective in removing pigments from certain concd. urine specimens, but is inferior to the following one. (2) Add 5 cc. of 10% Cu sulfate soln. to 25 cc. of urine, make alk. with 10 cc. of 10% Na tungstate soln., mix, filter, add 4 cc. of 5%  $H_2SO_4$  to 30 cc. of filtrate, let stand about 20 min., filter and ext. 25 cc. of filtrate with  $CHCl_3$  as usual. Urine has a limited buffering capacity manifested in the conversion of Na barbitol into the acid form even in alk. urines; the amt. of barbitol so converted is inversely proportional to the amt. of Na barbitol originally added to the urine. Large vols. of blood (after Folin-Wu pptn.) can be extd. with  $CHCl_3$  without obtaining interfering materials in the ext. even after concn. The liquid-air method of direct extn. of barbiturates can now be applied to the central nervous system after removing the phospholipides from the  $CHCl_3$  ext. with  $Me_2CO$ . A. P. -C.

The estimation of ascorbic acid by titration. Earle W. McHenry and Murray Graham. *Biochem. J.* 29, 2013-19 (1935) cf. *C. A.* 29, 5472<sup>2</sup>.—Important modifications in the titration procedure of Harris and Ray (*C. A.* 27, 4279) were described, i. e., soln. of the indophenol (I) in a phosphate buffer  $pH$  7.2, its standardization against  $FeSO_4(NH_4)_2SO_4$  soln. of this soln. 15-20 times when small amts. of ascorbic acid ( $CO_2H$ ) were estd., triple extn. of the sample with 3%  $CCl_4CO_2H$  and addn. of KCN in small amts. during the extn. Interfering plant pigments were removed (1) by titration of the ext. over  $CHCl_3$  which reddened as soon as excess I was present or (2) by extn. of the ext. with  $BuOH$  or  $AmOH$  before titration. Vegetable tissues contained appreciable amts. of reversibly oxidized I. Several vegetables gave an increased titration value after short heating or after acid hydrolysis, presumably because of the liberation of II from a water-sol. compd. insol. in the extg. soln. E. W. Scott

A microchemical test for choline and its esters in tissue extracts. Frederick J. Booth. *Biochem. J.* 29, 2064-6 (1935).—Choline (I) in 1:50,000 concn. in tissue exts. could be identified as the periodide. Florence's reagent (1.65 g. KI, 2.54 g. I, water, 30 g.) 3 drops was added to 1 drop of the unknown. Acetylcholine gave the test after 2 min. hydrolysis with 1% NaOH. The water-sol. precursor of I gave no ppt. with Florence's reagent. The sensitivity of this test for choline could be increased 100-fold by concn. of the tissue ext. E. W. Scott

The new color reaction of vitamin A. Jeno Rosenthal and János Erdelyi. *Biochem. J.* 29, 2112-13 (1935); cf. *C. A.* 29, 5470<sup>2</sup>, 6265<sup>7</sup>.—In reply to Anderson and Levine (*C. A.* 29, 4798<sup>7</sup>), R. and E. point out that guaicol (I) must be added to the new color test for vitamin A (II) when a detn. of II is carried out, since carotene does not give a color when I is present and the color obtained is quite stable and easily measured. E. W. Scott

Urobilin. A practical modification of the technic of the Schlesinger reaction in urine analysis. O. M. Mighlaccio. *Dis. med.* 6, 224 (1933); *Anal. Assoc. quim. Argentina* 23, 10B (1935).—Place the sample in a centrifuge tube, add an equal quantity of 10% alc. soln. of  $Zn(OAc)_2$  and centrifuge. In the clear portion of the liquid there will be a fluorescence when urobilin is present. E. M. Symmes

Detn. of moisture in small quantities of substance (Erdős). 7. Methods of observing electrophoresis of bacteria, blood corpuscles, etc. (Waelisch) 2.

\* Apparatus for preparing extracts from living organic materials. Henri D. Sauvage. *Fr.* 784,430, July 22, 1935.

## C-BACTERIOLOGY

LAWRENCE H JAMES

Relative productivities of certain culture media. R. E. Noble. *J. Am. Water Works Assoc.* 27, 1143-60 (1935).—Mass inoculation is not sufficiently sensitive, nor are 4 or 5 tests of value. The quant. measure of productivity

should be based on 30 or more tests. Buffered lactose broth, generally, is just as productive as standard lactose broth. Brilliant green lactose bile broth, brilliant green lactose bile agar and ferrocyanide-citrate agar were among the media tested. D. K. French

Selective media for coli aerogenes isolation. C. C. Ruchholt and John F. Norton. *J. Am. Water Works Assoc.* 27, 1134-42 (1935).—Of all the trial media studied standard lactose broth showed the biggest productivity. Brilliant green bile medium has been studied as a confirmatory medium. Of the other media only the fuchsine broth and possibly the methylene blue-bromocresol purple broth are considered worthy of further study. D. K. French

Sugar decomposition of dysentery bacilli. A. Uchida. *Japan Z. Mikrobiol. Path.* 29, 031-41 (1935).—U. found that the  $pH$  increased from 7.9 to 8.2 15 days after the cultivation of various dysentery types in peptone water without sugar. I. S. Yun

Bacterial decompos. of the rubber in the latex of *Hevea* (Spence) 30. Analysis of small quantities of gas (Bruce) 7.

## D-BOTANY

THOMAS G PHILLIPS

Plant-growth substances XVI. The constitution-specificity of heteroauxin. Fritz Kögl and D. G. F. R. Kostermans. *Z. physiol. Chem.* 235, 201-16 (1935). cf. *C. A.* 29, 7997<sup>2</sup>.—Heteroauxin (indole-3-acetic acid) has the same physiol. action as the more complex auxins a and b in deflecting the growth of oat coleoptiles. The only grouping it has in common with the other 2 auxins is the  $CO_2H$ . However, auxins a and b are rendered inactive by esterification with  $MeOH$ , while the  $Me$  ester of heteroauxin retains more than  $1/2$  the activity of the free acid. With increasing size of alkyl groups in the  $CO_2H$  the activity decreases at about the same rate, and with a secondary alkyl ( $Me_2CH$ ) the decrease is much greater than with primary alkyl ( $Pr$ ). Possibly the activity of the esters is due to a partial sapon whereby the active acid is liberated. On the other hand, a comparison of auxin a with heteroauxin shows that both have disocn. const. of the same order of magnitude. A no. of derivs. were prepd. for the purpose of detg. the effect of various substituents, or other modifications, on the activity of heteroauxin. By esterification of indole-3-acetic acid with  $CH_3N$ ,  $MeCHN$ , and  $EtCHN$ , the  $Me$ ,  $Et$  and  $Pr$  esters were prepd. as oils and purified as picrates, m. 125°, 84° and 105°, resp. The  $iso-Pr$  ester was obtained from the Ag salt and  $Me_2CHI$  (picrate, m. 100-1°).  $Me$  2,3-dihydroindole-3-acetate (picrate, m. 178°) was obtained by hydrogenation of  $Me$  indole-3-acetate in  $EtOH$  with  $HCl$  and  $PtO_2$  catalyst, and sapon to the free acid (picrate, m. 168°).  $Me$  2-methylindole-3-acetate, m. 65°, was obtained by  $CH_3N$  methylation of the free acid. 2-Ethylindole-3-acetic acid, m. 100-1°, was prepd. by condensing homolevulinic acid with  $PhN_2H$  and refluxing the phenylhydrazine in  $EtOH$  with  $H_2SO_4$ , and 5-methylindole-3-acetic acid, m. 151°, by the same treatment of the hydrazine from  $Et$  formylpropionate and p-tolylhydrazine. The latter gave a  $Me$  ester which formed a picrate m. 122-3°. Other derivs. tested for biol. action were 2,5-dimethylindole-3-acetic acid, m. 172-3° obtained from levulinic acid and  $p-MeC_6H_4N_2H$ , and reduction with  $SnCl_4$ ,  $\alpha$ -(8'-indolyl)-propionic acid, m. 102° (picrate, m. 146-7°),  $\beta$ -indolylcarboxylic acid,  $\beta$ -indolylpropionic acid,  $\alpha$ -indolylcarboxylic acid,  $\beta$ -indolyl-lactic acid and  $\beta$ -indolylpyruvic acid. In general, where the acids had biol. activity, this was diminished or destroyed by esterification, and hydrogenation of the indole ring destroyed activity. Where the  $\alpha-Me$  deriv. was active the  $\alpha-Et$  deriv. was inactive. Since tryptophan is undoubtedly the parent substance from which heteroauxin is derived, some of the intermediate products might be expected to show activity. The derived pyruvic acid was highly active, while the lactic acid was inert. Methylation in the benzene ring is less deleterious than methylation in the pyrrole ring. It appears that there are "sensi-

tive regions" in the mol in which the slightest alteration diminishes activity, while other regions are comparatively indifferent

A. W. Dox

Recent advances in science plant physiology. Walter Stiles *Science Progress* 30, 313-17 (1935) —A review of recent work on growth hormones in plants J S H

Carrying out the Went auxin test in daylight H Söding *Ber deut botan Ges* 53, 331-4 (1935) —A procedure obviating the use of a dark room is described L P M.

Callus and root formation by  $\beta$  indolylacetic acid F. Laibach *Ber deut botan Ges* 53, 359-64 (1935) — $\beta$ -Indolylacetic acid in lanolin applied as a paste (cf. C. A. 28, 3443) is active in inducing callus formation and in the initiation of roots Lawrence P. Miller

A test method for the determination of the callus forming action of growth-substance pastes F. Laibach and O. Iyechuch *Ber deut botan Ges* 53, 464-77 (1935) —The increase in thickness of a decapitated *Vicia faba* epicotyl (under strictly specified conditions) is taken as a measure of the callus forming potency. The effect of various concns of  $\beta$  indolylacetic acid (I) is shown by a curve. An increase in thickness of 10% (a 1/10 unit) under the conditions of the method is produced by a concn of 73  $\gamma$  (I) in 1 g paste Lawrence P. Miller

Comparative investigations on growth stimulants in native plants Friedrich Boas *Ber deut botan Ges* 53, 495-511 (1935). —Exts. of various plants were active in inducing growth of yeast and in increasing the yield of *Aspergillus niger*. Among the most effective were *Viscum album* and *Maltharia chamomilla* L. P. Miller

The annulling of the growth-substance effect by living plant parts P. Kornmann *Ber deut botan Ges* 53, 523-7 (1935) —Agar blocks which induce bending in the oat coleoptile because of having taken up growth substance from the oat or corn plant become inactive or have greatly reduced activity if parts of plants of a different species from that which originally furnished the growth substance are placed in contact with the blocks for a no. of hrs before the test is made Lawrence P. Miller

Chlorosis of *Hydrangea hortensis* J. D. Wilson and H. A. Runnels *Ohio Agr Expt Sta, Bimonthly Bull* 175, 143-6 (1935) — $\text{CuSO}_4$  in combination with  $\text{Ca}(\text{OH})_2$  and water (Bordeaux mixt) was not alone effective in increasing transpiration rates in plants, since the sulfates of Na, Fe and Mn caused nearly as great an increase when applied to *Coleus* as did Bordeaux mixt Mixts contr.  $\text{ZnSO}_4$ ,  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{K}_2\text{SO}_4$  were considerably less effective The ppts of these various mixts, settled in the reverse order of their effectiveness in increasing transpiration rate, e g, the mixt contr.  $\text{CuSO}_4$  increased the rate of water loss most, but its ppt settled most slowly; whereas that of the one contr.  $\text{K}_2\text{SO}_4$  settled most rapidly, and the mixt caused the smallest transpirational increase. C. R. Fellers

Preliminary study of the effect of a series of temperature changes upon respiratory activity of apples during the post-climacteric in senescent decline. Charles A. Eaves *Sci Agr* 16, 28-39 (1935). —The weighed tube method of estg  $\text{CO}_2$  in a moving air stream was entirely satisfactory for respiratory studies upon relatively large samples of apples. Respiratory activity of the control lots stored at 3°, 10° and 18° was characterized by a rise in  $\text{CO}_2$  output to the climacteric followed by a decline. The length of life of the apple and the onset of climacteric in terms of  $\text{CO}_2$  output are in inverse ratio to the rise in temp. The onset of fungal invasion caused a rise in the curve of respiration. Sharp initial upward temp. gradients 3-15° resulted in a heavy depletion of effective substrate as shown by excessive lowering of respiratory activity. C. R. Fellers

The osmotic and suction pressures of some species of the mangrove vegetation R. E. Cooper and S. A. Pasha *J Indian Bot Soc* 14, 109-20 (1935) —Expts were carried out on *Acanthus sifonolus* L., *Sonneratia apetala* Ham and *Avicennia officinalis* L. The osmotic and suction pressures were highest in the leaves, medium in the stems and lowest in the roots. There was a marked increase in the pressures of each species from Aug to Oct.

during the dry season; the pressures ranged from approx 40 to 90 atm and were very high as compared to other plants. The osmotic pressure of the sea water surrounding the plants was approx. const. (25 atm.) throughout the season. The osmotic pressure of the soil soln was 20-485 atm K. D. Jacob

Respiration of the roots and leaves of the rice plant (*Oryza sativa* L.). F. Baptista *J. Indian Bot Soc* 14, 159-65 (1935) —The roots evolved 82-200 mg and the leaves 163-400 mg  $\text{CO}_2$ /hr/100 g of dry wt. There was a marked fall in the rate of respiration soon after transplantation; the max was reached about the middle of Sept and the rate soon decreased sharply. K. D. J.

Carbohydrate nitrogen ratio of the shoots of some tropical trees R. H. Dastar and M. R. Raut *J. Indian Bot Soc* 14, 269-89 (1935) —Analyses were made of the shoots of *Bombax malabaricum* DC., *Cassia remigera* Wall., *C. fistula* Linn and *Pernandrea regia* Boyer. During the vegetative and reproductive phases the carbohydrate content was higher than at any other period during the year. The N content showed a continual increase from the beginning of the vegetative phase to the end of the flowering period and then decreased sharply. The N content of *B. malabaricum* decreased at the end of the vegetative period. In the 2 species of *Cassia* the C:N ratio was highest during the vegetative phase and lowest in the reproductive phase. The vegetative and reproductive phases of *B. malabaricum* and *P. regia* were not characterized by different C:N ratios. In all species the ratio was very low during the termination of vegetative and reproductive phases. The high carbohydrate content during the vegetative phase is due to photosynthetic activity and during the reproductive phase to the upward flow of carbohydrates from places of storage. K. D. J.

The effect of wounding on respiration in the starring leaves of *Aralia gulfuyensis* A. B. Saran. *J. Indian Bot Soc* 14, 294-304 (1935) —The max. output of  $\text{CO}_2$  was obtained from leaves wounded just after 2.5 hrs of starvation. The effect of wounding gradually decreased as the period of starvation was increased from 2.5 to 72 hrs; it then increased at 96 hrs of starvation. Injection of a 1.5% glucose soln into the leaves increased the  $\text{CO}_2$  output from the wounded leaves when the leaves had an initial respiration value of 4.7 mg  $\text{CO}_2$ /hr, but had no consistent effect when the respiration value was 6.4 mg/hr. K. D. Jacob

Cyanoglucosides in white clover. B. W. Doak. *NZ New Zealand J. Agr.* 51, 180-62 (1935). cf. C. A. 27, 5574; 29, 1851 —The percentages of HCN found in various types of white clover during the 1933-34 and 1934-35 seasons were somewhat lower than those previously reported for the same types grown under N. Zealand conditions, only a few samples contained more than 0.01% HCN. During the 1934-35 season the lower HCN limit of those lines certified as mother-seed lines was in the neighborhood of 0.005%. There was no evidence that white clover contr. 0.013% potential HCN is toxic to livestock. K. D. Jacob

Leaf carotenes G. MacLennan *J. Biol. Chem* 111, 75-84 (1935). —In C. A. 29, 7391<sup>1</sup>  $\alpha$ -carotene in line 2 should read  $\beta$ -carotene and  $\beta$ -carotene in line 4  $\alpha$ -carotene A. P. Lohthorp

Carotene. IX Carotenes from different sources and some properties of  $\alpha$ - and  $\beta$ -carotene Harold H. Strain *J. Biol. Chem.* 111, 85-93 (1935). —In C. A. 29, 7391<sup>1</sup>  $\alpha$ -carotene in line 6 should read  $\beta$ -carotene and  $\beta$ -carotene in the same line  $\alpha$ -carotene. A. P. Lohthorp

Iodine contents of ten species of Chinese marine algae Pei-Sung Tang and Pao-Chun Whang. *Chinese J. Physiol* 9, 285-90 (1935). L. A. Maynard

Method for determining germinative capacity without making a germination test. Aleksander Gurevich *Ber deut botan Ges* 53, 303-18 (1935). —The method depends on the ability of living cells through respiratory activity to reduce dimtobenzene to nitrophenylhydroxylamine which gives a purple color with  $\text{NH}_3$ . Details of the appearance of the tissues which have been treated with these reagents in viable and nonviable seeds of wheat and rye are given

Results obtained by the use of this method were in good agreement with actual germination tests. L. P. M.

Secretion of oil by orchid blossoms. Leopoldine Müller. *Ber. deut. botan. Ges.* 53, 319-64 (1935).—Blossoms of *Dendrobium nobile*, *D. pierardii* and *D. crumenatum* secrete a fatty oil. Droplets obtained from the inner side of the lobellum of *D. nobile* also contain a volatile oil. Lawrence P. Miller

The nutritive economy of the beech (*Fagus silvatica* L.). Ernst Glimmner. *Ber. deut. botan. Ges.* 53, 368-77 (1935).—Thirty-three 110-year old trees about 35 m. high and having a vol. of wood of about  $2\frac{1}{2}$  cu. m. were analyzed at intervals throughout a nonbearing year. During the winter months each tree (exclusive of the buds) contained an av. of 51.8 kg. carbohydrate (including hemicelluloses), 6.1 kg. fat and 2.2 kg. crude protein. From 60 to 80% of these reserves were in the wood, half or more of this in the mature wood. At the beginning of March the trunk begins to thaw out and changes take place in the relative moisture contents of the tissues. The fats and fatty acids begin to be utilized and after 2 months (and before the leaves come out) 90% of these reserves have disappeared. From May 14 to June 1 an av. of 1.1 kg. of adnl. dry wt. is laid down in the leaves and twigs daily. Of the 37 kg. of carbohydrate used during the appearance of the leaves and growth in height about  $\frac{1}{2}$  comes from reserves and  $\frac{1}{2}$  is produced by photosynthesis. The newly formed leaves contain 4.3 kg. protein,  $\frac{1}{2}$  of which is obtained from reserves. The mineral constituents are furnished largely by the branches and twigs. During the period of yellowing of the leaves (Oct. 12-20) 6.1 kg. carbohydrate and 2.6 kg. protein are translocated into the tree and lost due to leaf fall amounts only to 1.7 kg. carbohydrate, 0.4 kg. fat and 1.8 kg. protein. Figures are also given for the changes in wt., vol. and food reserves of the buds during the year. For a study of the effect of bearing (90-100) yr. old trees were analyzed. Of a reserve of 114 kg. carbohydrate 27.25 kg. was used during the active growth period. During flowering and fruiting an addnl. 40-45 kg. obtained from all parts of the tree including the roots was withdrawn; the branch wood lost 86.7% of its reserve, the mature wood of the trunk 48.0%. The carbohydrate/protein ratio of the beech is usually about 5:1; in the process of bearing the tree loses 40-50 times as much carbohydrate as protein. Lawrence P. Miller

The formation of precipitates in plant cells. A. Weller. *Ber. deut. botan. Ges.* 53, 377-414 (1935).—The small globular or granular ppts. which are formed when living cells contg. tannin are treated with basic substances are true ppts. which are formed in the same way as inorg. globular ppts. If plants take up Cu from soln. many of them will form spherules in the epidermal cells of the leaves. These contain Cu as a constituent. Similarly to inorg. spherules (cf. C. A. 29, 2121) these Cu spherules arise from a drop around which a membrane forms. The pptn. takes place within the membrane from the membrane surface toward the center. It shows a web-like structure. The ppts. which form when plant parts are treated with KOH are spherules which result either from the reaction between substances which are sepd. in the living cell, as e. g. Ca phosphate spherules, or they are formed by the withdrawal of water from drops by the KOH, resulting in a membrane around the drops within which pptn. takes place while water diffuses out, as e. g. inulin spherules. Starch grains do not grow by apposition and are not spherocrystals but spherules. They have a membrane which can be brought out clearly by treatment with dil. KOH and have the structure characteristic of spherules. Lawrence P. Miller

Gold in Zea mays. N. Nemec. *Ber. deut. botan. Ges.* 53, 440-2 (1935).—Corn grown in Oslany (West Slovakia) on andesitic soil contained about 1 mg. Au per kg. No Au was found in *Vicia faba major* from the same locality. Lawrence P. Miller

Optimum conditions for the accumulation of citric acid with some considerations of a theory of citric acid formation. T. Chrzastecz and E. Peyron. *Biochem. Z.* 280, 325-30 (1935).—The selection of the proper strain of mold

is very essential for obtaining a large yield of citric acid, and also of its purity. The largest amt. of citric acid is formed by maintaining a fairly const. concn. of 20% sucrose, which must contain 0.3%  $\text{NH}_4\text{NO}_3$ , 0.1%  $\text{KH}_2\text{PO}_4$  and 0.1%  $\text{MgSO}_4$ , also a few drops of  $\text{FeCl}_3$  per l. Zn salts are injurious in any concn. The best procedure is to culture the spores directly in the sugar soln. By changing the sugar soln. the max. production of citric acid is finally attained. It is very desirable to neutralize partially the citric acid with  $\text{CaCO}_3$ , but the fermentation mixt. must remain acid at all times. The fermentation should be carried out at 24-30° for 7-11 days. The hypothesis that quinic acid is an intermediate product in the formation of citric acid is regarded as untenable. S. Morgulis

Independence of the carbon dioxide assimilation of green plants from the presence of small amounts of oxygen and a reversible inhibition of the assimilation by carbon monoxide. Hans Gaffron. *Biochem. Z.* 280, 317-69 (1935).—Mol.  $\text{O}_2$ , either in the free or in the bound state, is not required for the initiation of the  $\text{CO}_2$  assimilation by green plants. Expts. with algae, *Chlorella* and *Scenedesmus*, in the dark, both in the presence of sugar or of  $\text{O}_2$ -absorbing reagents, demonstrate this. Only when the sojourn in the dark is protracted is there evidence of an inhibition of assimilation. The expts. with these algae further demonstrate that  $\text{CO}$  strongly influences their assimilatory process, which may vary from a completely reversible to an irreversibly injurious effect. The reversible, light-sensitive action of the  $\text{CO}$  is compared to its effect on the catalase in the absence of  $\text{O}_2$ , and the observed action of the  $\text{CO}$  is regarded as a specific reaction with the enzyme which in the Blackmann reaction produces likewise a liberation of  $\text{O}_2$ . This occurs only after an anaerobic period in the dark, either because the concn. of the photochem. intermediate "peroxide" product is too small for the  $\text{CO}$  to affect its displacement from combination with catalase or because the compd. reacting with  $\text{CO}$  is generally combined with  $\text{O}_2$ . The results are still not easily accounted for, and it is possible that the inhibition of catalase by  $\text{CO}$  may also involve an inhibition of the production of a primary photochem. substance. S. M.

Mold sterols. I. Sterol formation by *Aspergillus niger*. Konrad Bernhauer and Georg Patzelt. *Biochem. Z.* 280, 348-57 (1935).—Synthesis of sterols runs parallel with the development of the mycelium, which indicates that they play a physiol. function in the molds. Preliminary expts. show that the addn. of crotonaldehyde to fermenting yeast results in a definite increase in the  $\text{SHCl}$  reaction. On molds these expts. could not be repeated because crotonaldehyde is very toxic for them. The possibility of formation of sterol by polymerization of crotonaldehyde or  $\beta$ -methyl-crotonaldehyde is discussed. S. Morgulis

The estimation of starch in plants with special reference to woody plants. J. T. Sullivan. *J. Assoc. Official Agr. Chem.* 18, 621-36 (1935).—A procedure is described for the extn. of starch in plants; it is based on the extn. of the starch with slightly alk.  $\text{CaCl}_2$  soln. and its pptn. by  $\text{I}_2$ . The difficulties encountered in pptg. the starch iodide in presence of  $\text{CaCl}_2$  are avoided by removing the  $\text{CaCl}_2$  and substituting  $(\text{NH}_4)_2\text{SO}_4$  as the flocculating agent; the double pptn. necessary tends to purify the starch ppt. The conditions that must be observed to obtain the max. yield of starch by  $\text{I}_2$  extn. involve the concn. of the  $\text{CaCl}_2$ , the duration of the boiling and the degree of fineness to which the sample is ground. When the proper conditions are used the yield of starch obtained by one extraction agrees with that obtained by successive extns. under less drastic conditions, which indicates that all the starch has been brought into soln. Recovery of potato starch, alone or when added to plant material, indicates that no starch is lost during the process. The quantities of starch obtained by this method represent the true starch values more closely than those obtained by enzymic methods. The application and importance of a more accurate method to the study of the physiology of woody plants are indicated. A. Papineau-Conture

The lignin of algae. Peter Kluson. *Monatsh. Chem.* 14d,

47, 210-20(1935), cf. C. A. 29, 2578<sup>4</sup>.—Ash-free, bark-free algae contain 27% lignin. The analysis indicates a glycerol group where the terrestrial lignins have an aldehyde. This explains the granular gray-white  $\beta$ -naphthylamine ppt instead of the usual fine yellow ppt. The precursors xylose and hexose are 1-1 as in the Coniferae (C. A. 28, 5500<sup>4</sup>). K. speculates on the relation of lignin chemistry to evolution.

A. R. R.  
Comments on Holmberg's article "Thioglycolic acid as a lignin reagent." Peter Klason *Svensk Kem. Tid.* 47, 220-3(1935), cf. C. A. 24, 6007. —Thioglycolic acid reacts with lignin aldehyde groups but some lignins do not have these. Cf. preceding abstr.

A. R. R.  
Influence of weather conditions on the nitrogen content of wheat. II J. W. Hopkins. *Can. J. Research* 13, C, 127-33(1935), cf. C. A. 29, 3000<sup>4</sup>.—Supplementing a previous statistical study, coeffs designed to weight observed temps in proportion to their assumed effect on respiration were computed from the daily observations for three 3-week periods extending from July 1 to Sept. 1. After allowing for the effect of May and June rainfall there was a moderate but significant partial correlation ( $r = +0.23$ ) between N content and the sum of the temp coeffs for the last 2 periods. There was a pos correlation ( $r = +0.74$ ) between height of crop and yield of grain, and a neg correlation ( $r = -0.50$ ) between height and N content. The partial correlation between N content and yield, after eliminating variations in both assocd. with height, was negligible ( $r = -0.07$ ), suggesting that reductions in yield due to restriction of the later stages of translocation did not result in significant modification of the N content of the grain. Results of the investigation as a whole are briefly discussed.

J. W. Shipley.  
A study of moisture changes in standing grain. R. K. Larmour, W. F. Geddes, J. G. Malloch and A. G. McCalla. *Can. J. Research* 13, C, 134-39(1935).—A study of the moisture changes in standing grain during and after the ripening period was conducted at Winnipeg, Saskatoon and Edmonton in 1932 and 1933 with a view to obtaining information on the problem of combine harvesting. Grain was found to be fit for binding 4 to 17 days earlier than for straight combining. There was no evidence that fully ripened grain at moisture contents of 11-13% can absorb sufficient moisture at night, owing to the higher relative humidity, to exceed 14% and become tough. The rate of moisture loss in wet mature grain is much greater than the moisture loss, through the same range, in immature grain.

J. W. Shipley.  
Superiority of silver nitrate over mercuric chloride for surface sterilization in the isolation of *Ophiobolus graminis* Sacc. F. R. Davies. *Can. J. Research* 13, C, 163-73(1935).—It has been difficult to isolate *Ophiobolus graminis* from plant parts previously surface sterilized by the commonly used  $Hg_2Cl_2$  method, although *Helminthosporium sativum* and *Fusarium spp.* are readily isolated following its use. When a  $AgNO_3$  method of surface sterilization was substituted, *O. graminis* was isolated with considerably more success than had previously been obtained. These results can be explained by the difference in reaction of the above-mentioned fungi to these chemicals. When similar concns of these chemicals were added to nutrient agar,  $AgNO_3$  proved less toxic to *O. graminis* than  $Hg_2Cl_2$ , whereas the reverse was true for *H. sativum*.

J. W. Shipley.  
Biochemical studies on "Sotetsu," the Japanese ago plant II. The chemical constituents, especially the sex differences of "Sotetsu" stems. Kotaro Nishida and Antomo Yamada. *Bull. Agr. Chem. Soc. Japan* 10, 193-6(1934).—The cortex has greater sugar, fat, fiber and ash contents and peroxidase activity than the pith, but the cortex has more starch only in the case of male shoots. The more recently flowering female shoots are lower in polystarches and higher in sugar, protein, ash and peroxidase activity than other shoots.

J. T. S.  
The visible structure of the secondary wall and its significance in physical and chemical investigations of tracheary cells and fibers. I. W. Bailey and Thomas Kerr. *J. Arnold Arboretum* 16, 273-300(1935).—The struc-

tural pattern of the secondary wall is clearly visible in some fiber-tracheids and libriform fibers of certain dicotyledons and in them may be observed the effects of specific chem. and mech. treatments. The cellulose matrix of the secondary wall is continuous but interspersed with noncellulosic material, as lignin, and 2 continuous interpenetrating systems may result. Either system may be dissolved without modifying the pattern of the remaining system. The patterns and orientations are discussed and illustrated by photomicrographs.

J. T. Sullivan.  
Chemical composition of certain pasture species at flowering and maturity. R. E. Shapter. *J. Council Sci. Ind. Research (Australia)* 8, 187-94(1935).—Data are given for the protein, fiber, ether ext., N-free ext., sol. and insol. ash and  $P_2O_5$  contents and the dry matter produced in 11 grasses, 4 legumes and 5 miscellaneous species.

J. T. Sullivan.  
Chemical studies on *Rhizopus japonicus*. Hoshik Lun. *J. Faculty Agr., Hokkaido Imp. Univ.* 37, 165-209(1935).—The fungus *Rhizopus japonicus* Vuillemin was cultivated in Raulin's soln. contg. sucrose and tartaric acid as the only org. substances. The compn of the dried fungus is: protein 35.8, crude fat 9.7, crude fiber 7.7, N-free ext. 42.2, ash 5.5, total N 5.8 and nonprotein N 3.6%. The mineral compn of the ash was also detd.

From the ether ext. of 1 kg. of dried fungus there were isolated ergosterol 1.08, fungistrol 2.55, palmitic acid 7.6, stearic acid 1.16 and phosphatides 1.19 g. The unsatd acids were mainly oleic and a small quantity of linoleic. The ergosterol, after irradiation, had an antirachitic value on rats. After removal of the ether-sol substances, the fungus was extd. with 95% alc. and from this soln. there were isolated mannitol 16.3, sucrose 8.6, trehalose 0.26,  $NH_4Cl$  0.15, adenine (as picrate) 0.13, hypoxanthine 0.076, histidine (as picrate) 0.027, betaine (as hydrochloride) 0.14, and stachydrin (as Au chloride) 0.012 g. The fungus contained 14.03% mannose, 1.54% fructose and some glucose. From the residue after the ether and alc. extn. there was isolated 29.7 g. of protein which is nearly insol. in  $H_2O$ , NaCl soln. and alc., easily sol. in NaOH and as pptd. by acid. It contains N 15.25, H 8.752, C 53.233, S 0.211, P 1.288 and O 21.256%. It is a phosphoprotein and L. suggests the name *Rhizopenin*. It is rich in basic amino acids, tyrosine and tryptophan. The ratio of cystine S to total S is 1.18, the I value is 16.167% and the isoelec. point is  $pH$  2.95-3.02. The digestive coeffs. of the fungus by Bergmeyer's method are 72.1 for nitrogenous substances, 78.6 for carbohydrates and 74.2 for ash. Nutrition expts. were pos. for vitamins B<sub>1</sub> and B<sub>2</sub> and neg. for A and C. A growth-promoting substance toward yeast was present.

J. T. Sullivan.  
Raan or B deficiency in swedes (O'Brien, Dennis) 15. Maturity of fruit (Hinton) 12.

## E—NUTRITION

PHILIP B. HAWK

Identity of natural vitamin D from different species of animals. Ottar Rygh. *Nature* 136, 552-3(1935).—The liver and body fat of 18 species of fish, whale, a cow and a human female were examd. for vitamin D. No difference was found between them in antirachitic effect on chicks, 70-80 international (rat) units per chick per day being required. The results confirm those of Dols (*Dusserl. Wageningen* 1935, cf. C. A. 28, 6178<sup>9</sup>) but not those of Bulls, *et al.* (C. A. 29, 1465<sup>9</sup>). None of the preps showed max. absorption in the ultraviolet at 260-70 m $\mu$ , no rotation in EtOH soln. and the degree of esterification with phthalic anhydride and pyridine after 10 days was 100%. Maleic anhydride had no effect upon the present vitamin D. There is no evidence that the vitamin D in all the fats investigated is not the same.

Philip D. Adams.  
Effect of a low-calorie, low-protein diet on blood proteins. Windsor C. Cutting and Richard D. Cutter. *Proc. Soc. Exptl. Biol. Med.* 32, 1032-6(1935).—Twenty-six normal subjects subsisted for 2 days on a 500-calorie protein-free diet with fluids and activity unrestricted.

The plasma values before and after the expl. period were: protein (per 100 cc.) 7.4 g., 7.74 g.; vol. 236 cc., 205S cc.; total protein 170 g., 156.5 g., resp.; the protein fractions were unchanged. The subjects were hungry; they drank little water and lost 2 lb. in wt. C. V. B.

Effect of diet upon blood phosphorus partition of rats with and without insulin. Natalie Van Cleve and Agnes Fay Morgan. *Proc. Soc. Exptl. Biol. Med.* 32, 1636-41 (1935).—Rats aged 28 days were kept for 28 days on a standard diet or on a diet rich in fat or in carbohydrate, in the fasting state the animals were injected with 3U/kg. of insulin and 1/2 hr. later the pooled whole blood of each group showed a decrease in inorg. P only in the high-fat diet, the org. P tended to increase; total P, acid-sol. P and lipid P were practically unchanged. C. V. B.

The specific effect of ascorbic acid on the anemia of scurvy. D. Melville Dunlop and Harold Scarborough. *Edinburgh Med. J.* 42, 476-82 (1935).—The daily oral administration of 60 mg. of ascorbic acid for 17 and 19 days in 2 patients suffering from scurvy caused a marked increase in the red blood count and hemoglobin content. Rachel Browne

Supplementing soil with iron and copper for the prevention of anemia in young pigs. L. H. Moe, W. A. Craft and C. P. Thompson. *J. Am. Vet. Med. Assoc.* 40, 302-11 (1935).—A litter of pigs having access to 50 lb. of soil to which 9 g. of  $\text{FeSO}_4$  and 1.5 g.  $\text{CuSO}_4$  were added maintained a higher hemoglobin level and were significantly heavier 3-4 weeks after birth than pigs having access to no soil or soil without the added Fe and Cu. Rachel Browne

The antirachitic effect of ultraviolet radiation transmitted by a smoky atmosphere. Hugh M. Barrett. *J. Ind. Hyg.* 17, 199-216 (1935).—The irradiation from a quartz Hg arc lamp, after passage through a concn of smoke similar to that encountered in the urban atm., fails to prevent the development of rickets in rats fed on a rachitic diet. A. L. Elder

Spectrum analysis of hen eggs and chick tissues. W. F. Drea. *J. Nutrition* 10, 351-5 (1935).—Spectrographic analysis of material evaporated to dryness in  $\text{SiO}_2$  and Pt dishes showed that a no. of elements pass from the feed or water into the hen's blood, from there to the egg, and finally into all of the chick's tissues and chick's blood. These are Al, Ba, Ca, Cu, Fe, Mg, P, K, Si, Na, Sr, Ti, V and possibly Rb. Of the 'trace' elements, Al, Ba, Ca, Cu, Sr, Ti and Zn were quite uniformly distributed among the tissues. The 'trace' elements more concd. in the hen's blood or egg, and of probable physiol. significance, are Ba, Fe, Sr and V. The presence of considerable V in the blood, femur, heart, kidney and lung of both hen and chick suggests that it may have a hematopoietic function. Whereas Cr, Pb and Mo were found in the hen's blood, only traces were present in the eggs. Mn was present in greater quantity in the yolks as compared with the hen's blood and was absent from the egg white and egg shell. It was concd. in the liver and appeared in slightly lesser amts. in the kidney and gizzard. Mn is of probable nutritional importance. C. R. Fellers

Effects of increasing calcium content of a diet in which calcium is one of the limiting factors. H. C. Sherman and H. L. Campbell. *J. Nutrition* 10, 363-71 (1935).—The Ca content of the dry rat diet was increased from 0.2% to 0.35% by the addn. of  $\text{CaCO}_3$ , and the effect on successive generations of rats noted. High Ca intake was followed by a more efficient utilization of the food, better growth, earlier maturity, several indications of higher adult vitality, a longer period between the attainment of maturity and the onset of senility, and an increase in life expectation of the adult. Infant mortality was decreased as a result of the increased Ca intake. The effect on females was less marked than with males. Conclusion: The level of Ca intake most conducive to optimal well-being is significantly higher than that required for normal growth and maintenance. C. R. Fellers

Calcium and phosphorus needs of preschool children. Amy L. Daniels, Mary K. Hutton, Elizabeth M. Knott, Olive E. Wright and Mary Forman. *J. Nutrition* 10,

373-88 (1935).—The Ca needs of normal children of preschool age can be met by foods contg. 45-50 mg. of Ca per kg., or between 7 and 9 mg. per cal., providing sufficient vitamin D is allowed. P needs, on the other hand, can be met by food contg. between 60 and 70 mg. per kg., or between 9 and 11 mg. per cm. C. R. Fellers

Effect of deficient diets on the total ash, calcium and phosphorus content of bones. Ruth Yeager and Jet C. Winters. *J. Nutrition* 10, 389-97 (1935).—The bones of rats stunted by means of cal., protein and lysine deficiency showed a much larger percentage of total ash, Ca and P than was present in the bones of normal animals of the same wt. but a smaller percentage in each case than is found in the bones of normal animals of the same age. Stunting by means of a low-salt diet resulted in loss of total ash, Ca and P so that the percentages in the bones of the animals so stunted are considerably smaller than those characteristic of normal animals of the same wt. and greatly reduced in comparison with those of normal animals of the same age. There is some indication that Ca/P ratio in bones depends to some extent on the amts. of Ca and P present in the diet. C. R. Fellers

Augmentation of the toxicity of fluorosis in the chick by feeding desiccated thyroid. Paul H. Phillips, Honora E. English, and E. B. Hart. *J. Nutrition* 10, 399-407 (1935).—The chick possesses a high tolerance for F administration. Approx. 70 mg. of F per kg. of body wt. is necessary to inhibit growth in baby chicks. This level of F intake inhibits growth through restricted feed consumption. Intraperitoneal injections of F also restricted feed consumption, indicating that the reaction to F is systemic in nature and independent of any reaction via the digestive tract. The tolerance of intraperitoneal injections of NaF soln. were distinctly lower, 64 mg. of F per kg. body wt. were lethal while 35-40 mg. per kg. of body wt. retarded growth. The feeding of desiccated thyroid in amts. greater than 350 mg. per kg. of body wt. definitely retarded growth, while levels of 200-225 mg. were without effect. Feed consumption was not restricted by desiccated thyroid feeding. Nontoxic levels of desiccated thyroid were made toxic by chronic fluorosis induced by NaF feeding in the growing chick. Growth was greatly retarded and the survival period shortened. F ingestion definitely enhances the toxicity of desiccated thyroid and vice versa. C. R. Fellers

Tankage as a source of protein for dairy cows. J. G. Archibald. *Mass. Agr. Expt. Sta., Bull.* 321, 8 pp. (1935).—High-grade tankage can be safely used as a protein concentrate for dairy cows. C. R. Fellers

Minimum vitamin A requirements with particular reference to cattle. H. R. Guilbert and G. H. Hart. *J. Nutrition* 10, 409-27 (1935).—The total storage of vitamin A and carotene in the liver and body fat of cows 2-18 years old, which had access to green feed in abundance throughout life, was estd. at 0.6-0.7 g. in the younger animals and up to 3.6 g. in the aged cows. From 67 to 93% of the storage was in the liver. The amt. in the other organs was negligible. In the liver the form was vitamin A, whereas in the fat, carotene predominated. The daily withdrawal of vitamin A from the body store when cows were placed on a vitamin A-deficient ration was approx. 9-11 mg. The percentage of carotene recovery by a cow from alfalfa was about 2.7%. Reserves above a certain level are but slowly accumulated. Subcutaneous injections of carotene into a vitamin A-deficient calf caused the disappearance of corneal lesions and improvement in phys. condition. However, wt. gains did not occur. The injected carotene was poorly utilized by the animal's body. Night blindness was the first detectable clinical symptom of vitamin A deficiency and constituted a delicate index upon which min. requirements could be based. The daily carotene requirement of the bovine is 26-33 mg. per kg. live wt. Carotene intake at the level of 29 mg. per kg. daily prevented or cured clinical symptoms and promoted normal wt. increases, yet it resulted in no storage. When the intake fell below this level, night blindness resulted, and wt. gains decreased. Vitamin A deficiency that had progressed to the point of night blind-

ness and convulsions did not inhibit the occurrence of estrus or reproduction (in 1 case). Vitamin A requirement is related to body wt. rather than to energy requirement and the min amt required for mammals is 20-30 mg per kg per day. The requirement is considerably higher for both turkeys and chickens. C. R. Fellers

Vitamin C VII. Germination of seeds and vitamin C 6 Effect of light on the production of vitamin C during germination. Tomuji Matsuoka. *Mem. Coll. Agr. Kyoto Imp. Univ.* No 35, 1-10(1935).—See C. A. 27, 2180 VIII. Chemical nature. 1 Separation of vitamin C from cabbage juice. *Ibid.* 11-23.—See C. A. 27, 2988 IX. Chemical nature. 2 Vitamin C and ether. *Ibid.* 25-30.—See C. A. 27, 3505 X. Chemical nature. 3 Narcotine and vitamin C. *Ibid.* 31-8.—See C. A. 27, 4282 XI. Physiological studies. 1. Antiscorbutic value of vitamin C and method of administering. *Ibid.* 39-47.—See C. A. 28, 2750<sup>2</sup>. XII. Chemical nature. 4 Vitamin C in the liver and adrenal of cattle. *Ibid.* 49-56.—See C. A. 28, 2750<sup>1</sup>. XIII. Chemical nature. 5 Extraction of vitamin C and the solvents. *Ibid.* 57-64.—See C. A. 28, 2750<sup>1</sup>. XIV. Chemical nature. 6 Extraction of vitamin C and pH value of the solution. *Ibid.* 65-70.—See C. A. 28, 2750<sup>1</sup>. G. G.

Vitamin C XV. Physiological studies on vitamin C 2 Antiscorbutic value of vitamin C and its administration. Tomuji Matsuoka. *Mem. Coll. Agr. Kyoto Imp. Univ.* No 35, 71-80(1935); cf preceding abstr.—With guinea pigs the best time to begin the scurvy-cure test is on about the 22nd day of the feeding of the basal ration, when the animals are in the middle degree of scurvy. After 30 days of feeding, when the animals were heavily afflicted they required a large amt of vitamin C and the cure was not effective. Subcutaneous injection of vitamin C ext. prep'd from radish juice did not cure scurvy. The same prep'n given by mouth cured scurvy. Intravenous injection was more effective than subcutaneous injection but not as effective as oral administration. The amt. of vitamin C necessary to prevent scurvy apparently is related to the amt. the pigs have been taking before the test. With guinea pigs previously fed green grass alone, the administration of 30 g. of green grass did not prevent scurvy. Those fed 20 g. of grass per day with the basal diet for 3 generations needed only 15 g. of grass per day to prevent scurvy. XVI. Enzymes and vitamin C. 1. *Ibid.* 81-92, cf. C. A. 28, 8767<sup>1</sup>.—Vitamin C ext. from Japanese orange juice activated diastase to a slight extent. The orange juice itself had a greater diastase-activating power which seems to be due to impurities containing a coenzyme and inorg. salts. The digestion of casein with pepsin was inhibited by orange juice and by vitamin C. The digestion of olive oil with pancreatic lipase was not influenced by orange juice or by vitamin C. XVII. Chemical nature of vitamin C. 6 Change of content of vitamin C in barley. *Ibid.* 93-108.—As judged by scurvy-prevention expts. with guinea pigs, vitamin C in the seeds of barley increases markedly on about the 3rd day of germination, and gradually increases in the plant until the blooming period. From the blooming period to ripening there is a decrease in content of vitamin C, caused by lack of production of vitamin C, and by its transformation into other substances. Juice obtained from barley plants in the blooming period and preserved in an atm. of CO<sub>2</sub>, after removal of impurities, retained its vitamin C content. Vitamin C is produced in barley grown in the dark but the content is greatly increased when it is grown in the light. By following Kogl's method (C. A. 27, 2404) which oats for the study of growth production it was found that vitamin C has no auxin-like properties. E. D. Walter

Presence of vitamin C in Bioplastin. Serrano. E. Ferrara. *Rass. clin. terap. sci.* Vol 34, 1482-9(1935).—Bioplastin Serrano, even in small doses, protects mice from vitaminosis A. The protective action has been shown to be due to vitamin A and carotenoids. H. Lee Gruehl. Studies of relationships between nutritional deficiencies and (a) facial and dental arch deformities and (b) loss of immunity to dental caries among Sea Islanders

and Florida Indians. Weston A. Price. *Dental Cosmos* 77, 1033-45(1935).—The subjects were Melanesians, Polynesians and Florida Indians. The dietary of the mother during gestation and lactation and of the child during the growth period det'd. the degree of reproduction of the ancestral phys. pattern. Nutritional deficiency could change the racial pattern in even a single generation, and tended to reduce the immunity to certain diseases. Joseph S. Heppburn

The influence of calcium carbonate in the feed of laying hens, upon digestion. G. Davis Buckner and Amanda H. Harms. *Poultry Sci.* 14, 380, 375, 380(1935).—The results indicated less digestion of N-free ext. and more digestion of protein in the presence of an abundance of CaCO<sub>3</sub> in the ration. K. D. Jacob

Variations of egg-yolk color and characteristics produced by feeding and environment. W. P. Albright and R. B. Thompson. *Poultry Sci.* 14, 373-5(1935).—When hens that normally produced eggs with light yellow yolks were fed on pigmented feeds, such as green alfalfa, alfalfa hay and yellow corn meal, the yolk color abruptly changed to a dark orange which persisted as long as the pigmented materials were fed. The yolk color was influenced by the administration of fat sol. dyes but other dyes had no effect. K. D. Jacob

A preliminary report on the vitamin G requirement of turkeys. G. F. Heuser. *Poultry Sci.* 14, 376-8(1935).—Turkey rations for the 1st 4 weeks should contain a min vitamin G equiv. of approx 16% of dried skim milk. During the 2nd 4-week period the vitamin G requirement drops to the equiv. of about 10% of dried skim milk. After 8 weeks of age the requirement is not greater than the equiv. of 7% of dried skim milk. The quant. requirements for the 1st 2 periods of 4 weeks each are in direct proportion to the relative growth rate, indicating that the need for the growth-promoting phase of the vitamin G complex is directly related to the rate of growth. K. D. Jacob

Vitamin A content of West Indian shark (*Carcharias* sp.)-liver oil. Conrado F. Asenjo, Luz M. Dalmazo and Joseph H. Axtmayer. *Puerto Rico J. Pub. Health and Trop. Med.* 11, 158-62(1935).—The vitamin A value of the oil from these sharks caught during the month of July was found to be slightly greater than 13,300 Sherman units per g. Thirteen references. J. A. Kennedy

Dietary factors in the production of dental disease in experimental animals, with special reference to the rat I. Dental caries. J. D. King. *Brit. Dental J.* 59, 123-44, 305-16(1935).—Diets of corn starch, rice starch, cane sugar or finely ground yellow maize with deficiency of vitamins and mineral salts did not produce abnormalities in the molar teeth of rats after long exptl. periods. There was a high incidence of Gram + lesions in decalcified sections of the dentine of the lower molars of rats fed on diets composed mainly of coarse yellow maize or whole brown rice while teeth of the upper jaw were relatively free from such defects. The lesions occurred in the neighborhood of the summits, on the anterior aspect of those cusps which had a pronounced forward inclination, and at the base of deep fissures. Frances Krasnow

Egg yolk and bran as sources of iron in the human dietary. F. Ila McCollum Vahlteich, Esther H. Funnell, Grace MacLeod and Mary Swartz Rose. *J. Am. Dietetic Assoc.* 11, 331-4(1935).—Expts for 2 nine day periods on 2 young women showed that the Fe of egg yolk and of bran are of equal efficiency as sources of Fe. A. Le V.

The preparation and nutritional value of hepatoflavin. T. J. Stare. *J. Biol. Chem.* 111, 567-75(1935).—With both rats and chicks liver flavin added to a vitamin-B<sub>12</sub> deficient diet does not prevent the appearance of dermatitis but another substance is present in liver which does prevent this condition. Both the flavin and the unknown substance (vitamin B<sub>12</sub> complex) are necessary for growth. The procedures of Kuhn, Stern, Karrer and others for the prep'n of flavin have been considerably shortened. The principal modifications are the pptn. in alc. soln. by hot Ba(OH)<sub>2</sub>, the *pn* fractionation of the Ag pptn., and

the extn. of the Ag ppt. with hot dil. AcOH and H-S rather than with AcOH alone.

A. P. Lohrop  
The substitution of diethoxyethylamine (cystine amine) for cystine in the diet of the white rat H. H. Mitchell. *J. Biol. Chem.* 111, 699-703 (1935).—No evidence was obtained that cystine amine can perform the functions of cystine in promoting growth in paired feeding expts. with white rats. On the contrary its addn. to a cystine-deficient diet definitely depresses its growth-promoting value and definitely impairs the appetite of the animal consuming it. A severe restriction of food intake in a paired feeding expt. does not invalidate the comparison of rations, it merely delays the appearance of the effects of differences in nutritive value. Cf. Sullivan, Hess and Schrell, *C. A.* 25, 3696 and Jackson and Block, *C. A.* 27, 524.

A. P. Lohrop  
The biological value of mixed cereal proteins Wm H. Adolph and Fa-Wu Cheng. *Chinese J. Physiol.* 9, 245-52 (1935).—The various protein sources were compared at a 10% protein level with growing rats. By use of the N balance method mixts of kaoliang, soybean and maize (1), wheat and cowpea (2), maize and soybean (3) and maize, soybean and millet (4) showed a higher biol. value than millet alone. By use of growth per g. of protein as a measure, (1), (2) and (4) proved superior to millet, kaoliang, soybean or cowpea alone, while (3) proved equal to millet and soybean and superior to kaoliang and cowpea.

L. A. Maynard  
Effect of steaming compared to baking on the nutritive value of wheat bread. Wm H. Adolph and Yung-Tu Tsun. *Chinese J. Physiol.* 9, 275-84 (1935).—The 2 kinds of bread were of equal value for growth in rats and did not differ as regards rate of passage from the stomach or speed of absorption. The digestibility coeffs. obtained in expts with men were not different for the 2 breads. Expts *in vitro* showed a more rapid digestibility of the carbohydrate and protein of the baked bread than of the steamed bread, particularly in the crust portion.

L. A. Maynard  
The interrelationship of vitamins and other dietary constituents I. Vitamins A and D and other dietary constituents in relation to the formation of urinary calculi I. C. Hou. *Chinese J. Physiol.* 9, 299-306 (1935).—In a diet deficient in vitamin A but very rich in vitamin D (3) rats showed urinary calculi. The frequency was much lower on a diet free from D. Diets low in A, when high in protein or low in Ca and P, favored calculi formation. On a high cereal diet supplemented with cod-liver oil no calculi developed.

L. A. Maynard  
Behavior of ascorbic acid of inanition tissues. Emilio Martini and Arturo Bonisore. *Arch. sci. biol. (Italy)* 11, 167-74 (1935); cf. *C. A.* 29, 2580.<sup>3</sup>—In fasting guinea pigs the tissue contents of ascorbic acid are diminished, the ratio of dehydroascorbic/ascorbic acid is not increased as it is in exptl. scurvy. Glutathione, and in general, those substances that reduce I<sub>2</sub> in acid media except ascorbic acid do not vary appreciably. The administration of ascorbic acid can probably retard death from starvation, but death is probably not due to ascorbic acid deficiency in the tissues.

P. F. Mettler  
Effect of small additions of cystine to the diet on the metabolism under quantitative or qualitative and qualitative protein undernutrition. Luis Lorente. *Biochem. Z.* 279, 76-81 (1935).—In all expts. on rats on a protein-poor diet, whether the protein was lentil or casein protein, there was a neg. N balance and the urinary C/N and Vakot-O/N quotients were high. On the addn. of 0.0075 g. pure L-cystine per animal and per day the neg. balance became somewhat less but the effect on the urinary quotients was not entirely clear. In general the high quotients were slightly reduced or remained unaltered by the addn. of the small amt. of cystine.

S. Morgulis  
Detoxication processes in the rabbit during B-avitaminosis. Teki Kumon. *J. Biochem. (Japan)* 22, 213-24 (1935).—The synthesis of glycine with benzoic, phenylacetic or phenylpropionic acid is not materially different in normal or in vitamin B-lacking rabbits. On the administration of hippuric acid, by subcutaneous injection, the excretion of benzoic acid in the urine is very

much diminished during B avitaminosis, although the liver and kidney of both normal and avitaminotic animals possess the hippuric acid-splitting enzyme to the same extent. The formation of conjugated ethereal sulfates following the administration of phenol or indole, is always less in the avitaminotic rabbits and there is also less urinary indican than in the normal animal. Both the normal and the B-avitaminotic rabbit have the same ability of forming glucuronates, but the methylation of pyridine is somewhat decreased during the avitaminosis.

S. Morgulis  
Enzyme action in the avian organism during B-avitaminosis S. Tsunoo, M. Takamatsu, T. Kamachi and M. Inazumi. *J. Biochem. (Japan)* 22, 225-31 (1935).—The pancreatic esterase and the liver amylase are more active in the B-avitaminosis animals than in the normal ones, but the kidney phosphatase action is somewhat weaker in the former.

S. Morgulis  
The relation of vitamin C deficiency to the strength of skin capillaries Gunnar Palk, Karl-Ove Gedde and Gustav I. Gøthlin. *Uppsala Lakareforen. Forh.* 38, No 1, 1-24 (1932-1934).—Vitamin C deficiency was measured on 89 children in the district of Norrbotten, just north of the Arctic circle. Marked deficiency in vitamin C intake was observed in about 20% of these school children. Children of school age require a larger vitamin C intake per kg. of body wt. than do adults.

James C. Munch  
International vitamin standards E. M. Nelson. *J. Assoc. Official Agr. Chem.* 18, 610-11 (1935).—A brief explanation of the present status of international standards for vitamins A, B<sub>1</sub>, C and D. A. Papineau-Couture.

The chemistry of the vitamins II Katharine H. Coward. *School Sci. Rev.* 17, 94-100 (1935). Cf. *C. A.* 29, 6252.<sup>1</sup>

O. Renmuth  
The use of grape juice for weight reduction Report of a series of 31 cases F. Damrau. *Clin. Med. Surg.* 42, 477-80 (1935).—Grape juice before meals leads to weight reduction, apparently because of its content of grape sugar. The latter gives rapid relief of hunger and consequently lowers the total consumption of food.

T. H. Rider  
Dietary protein in relation to sterility I. J. Cunningham and C. S. M. Hopkirk. *New Zealand J. Sci. Tech.* 17, No 1, 420-32 (1935).—The fertility of male and female rats receiving 15-92% protein was tested. The growth rate on all diets was good. All the diets were adequate to promote fertility of the females. Diets contg. 67-82% protein or 15-18% protein derived largely from corn and gelatin induces sterility in males, causing damage to the germinal epithelium of the testicle closely resembling the changes produced by inanition, deficiencies of vitamins A or E or by complete deprivation of fats.

W. Gordon Rose  
A fat-soluble growth factor required by blowfly larvae II. Identity of the growth factor with cholesterol Ralph P. Hobson. *Biochem. J.* 29, 2023-26 (1935). Cf. *C. A.* 29, 5893.<sup>1</sup>—Purified cholesterol (I) supplied an essential growth factor required by blowfly larvae. From the exptl. results, I itself was the fat-sol. factor necessary. The larvae did not require fatty acids or fat-sol. vitamins present in the nonsterol fraction of the unsaponifiable residue.

E. W. Scott  
The effect of feeding cacao shell to cows on the vitamin D content of butter (milk). Stanislaw K. Kon and Kathleen M. Henry. *Biochem. J.* 29, 2051-56 (1935).—Cacao shell, a by-product of chocolate manu., contained 35 I. U. of vitamin D per g. The shell fat contained 300 I. U. per g. and possessed 40% of the total activity. The feeding of cows on 2 lb. of cacao shells daily for 1 month increased the vitamin D content of the butter from the winter to the summer level.

E. W. Scott  
The nature of the "sporogenes vitamin," an essential growth factor for *Cl. sporogenes* and related organisms. Alwin M. Pappenheimer. *Biochem. J.* 29, 2057-63 (1935).—A high active growth prepn. (I) was obtained by extg. the saponifiable plus nonsaponifiable fraction of mare's urine in pyridine-ether soln. (1:1) with concd. HCl. I was purified by prepn. of a Me ester which could be distd. at low pressure. The ester upon sapon. gave an



unsatd. hydroxy acid (mol wt. about 200) which promoted visible growth on a medium of known compn. in concns of 0.04% per ml. The differences between this growth factor and those essential to plants and other microorganisms were pointed out. E. W. Scott

The effect of different percentages of protein in the diet on bachelors and virgin rats. James R. Smonaker. *Am. J. Physiol.* 113, 154-63 (1935). cf. C. A. 25, 5033—The order of efficiency of diets containing different percentages of protein as determined by the total distance run (in revolving cages) was 14.2%, 5104 > 18.2%, 5041 > 22.2%, 4691 > 26.3%, 5802 > 10.3%, 3461 miles. The duration of sexual life was greatest in the 14.2% group and least in the 10.2 and 26.3% groups. The life span of both sexes was shortest in the 26.3% group. It was longest in the males of the 14.2% group and in the females of the 14.2% group. E. D. Walter

Goat-milk anemia. G. O. Kohler, C. A. Elvehjem and E. B. Hart. *Am. J. Physiol.* 113, 279-84 (1935). cf. C. A. 25, 5018—The anemia produced in young rats by restricting them to a diet of whole goat milk was cured by the addition of Fe and Cu salts. The growth of rats fed goat milk mineralized with Fe, Cu and Mn was much inferior to that obtained with cow milk mineralized in the same manner. The deficiency in goat milk for growth was not corrected by the addition of cod liver oil, yeast, liver or creatine vitamin B<sub>1</sub>. Normal growth was obtained by the addition of brain tissue. The addition of a better grade of alfalfa hay to the diet of the goats caused a definite improvement in the growth-promoting properties of the milk. E. D. Walter

The effect of viosterol upon oxygen consumption of frog muscle. Samuel Gelfan. *Am. J. Physiol.* 113, 464-6 (1935). cf. C. A. 27, 5785—Isolated muscles from frogs receiving daily injections of viosterol have a greater O<sub>2</sub> consumption than the controls. E. D. Walter

Feeding experiments with decomposition products of proteins. IV. Silvio Maveda. *Proc. Imp. Acad. (Tokyo)* 11, 238-9 (1935). cf. C. A. 25, 3444—By synthesis from crotonic acid diester according to Abderhalden and Hervas (C. A. 28, 4382),  $\alpha$ -amino- $\beta$ -hydroxybutyric acid (I) was prepd and its growth-promoting ability affirmed. Feeding expts. showed that rats lost weight on a synthetic diet in which proteins were entirely replaced by the mixt. of purified amino acids, but that the same diet supplied with 1% of I caused consistent increase in body weight. Im. 235\* with decarbox. and gives a typical violet coloration with ninhydrin. R. P. Walton

## F—PHYSIOLOGY

HOMER W. SMITH

Extraction of estrin from female urine after acidification with various acids. W. Kenneth Cuyler. *Proc. Soc. Exp. Biol. Med.* 32, 1382-5 (1935).—When 750 cc. of female stock urine was acidified with 50 cc. of either H<sub>2</sub>SO<sub>4</sub> or HCl or AcOH or 15 g. of CCl<sub>3</sub>COOH or tartaric acid, the yield in rat units of estrin per l. was 0, 4, 6, 9.3 and 18.6, resp. C. V. Bailey

Amino acids in human skin. H. C. Eckstein. *Proc. Soc. Exp. Biol. Med.* 32, 1573-4 (1935).—The outer layers of human skin were successively extd. with cold 90% alc., Et<sub>2</sub>O and CHCl<sub>3</sub>; the material was then digested with conc. pepsin for 72 hrs. and for a similar period with conc. trypsin; the residual dry product contained 6.1% of ash and 14.2% of total N. With the Van Slyke partition method, the residual product contained arginine 5.01, lysine 4.68, histidine 0.64, cystine 3.82, tyrosine 3.42% and tryptophan 1.8%. Basic amino acids of human skin. Richard J. Block. *Ibid.* 1574-5. Cornified epithelium from the sole of the foot and scales from patients with exfoliative dermatoses were washed and extd. with hot alc., Et<sub>2</sub>O and digested with pepsin-HCl; 14 g. of the dry residual product yielded arginine 6.0, histidine 0.82, lysine 4.3 and cystine 3.4%. The mol. ratios of histidine:lysine:arginine in true keratins are 1:4:12 (cf. C. A. 25, 5133), the ratios in this material and in that of Eckstein

are 1:6:7; evidently the process of keratinization was not completed. C. V. Bailey

Relation of potency of anterior pituitary-like hormone to hydrogen-ion concentration. C. A. Elden and Marvel-Dare Fellows. *Proc. Soc. Exp. Biol. Med.* 32, 1567-69 (1935).—The urine of pregnant women was most potent in anterior pituitary-like action at pH 6.1-7.3; in this range 0.75 cc. of fresh urine injected intravenously in the rabbit caused rupture of Graafian follicles in approx. 15 hrs. in 3 out of 5 test animals. Above and below this range the potency decreased greatly; no ruptured follicles were found after the injection of as much as 5 cc. of urine at pH 12.0. The hormone may be protein-like in nature or carried down with the foreign protein at the isoelec. point of 6.1. The potency of normally acid or alk. urine was restored by adjusting the pH; potency was permanently decreased in artificially alkalinized urine. The findings do not detract from the value of the Friedman modification of the Aschheim-Zondek test (C. A. 24, 2170) in which 5-10 cc. of urine is always used. C. V. Bailey

Absence of follicle-stimulating hormone in pigeons of young pigeons. Oscar Riddle and James P. Schooley. *Proc. Soc. Exp. Biol. Med.* 32, 1610-14 (1935). Effects of complete and incomplete hypophysectomy on basal metabolism of pigeons. Oscar Riddle, Gunderberg C. Smith and Clarence S. Moran. *Ibid.* 1614-16—Within 10-20 days after complete hypophysectomy the basal metabolism of the adult pigeon was decreased 35% when measured at the animal's crit. temp. (30°), at 20° the decrease was only 17%. When 10-25% of the gland remained in the body the basal metabolism at 30° was maintained, when less than 4% was present the effect was similar to that found in complete absence of the gland. Such tests must be conducted at the animal's crit. temp. C. V. Bailey

Further purification of galactin, the lactogenic hormone. W. H. McShan and C. W. Turner. *Proc. Soc. Exp. Biol. Med.* 32, 1655-6 (1935). cf. C. A. 27, 4289—Obtain the isoelec. ppt. at pH 6.5-5.5 from the alk. digest of fresh or desiccated anterior pituitary glands of sheep, cattle or hogs, wash repeatedly with acetone, dry in a desiccator and grind to a fine powder. Ext. 4 g. of the powder with 2 vol. of glacial AcOH, centrifuge and decant into 12 times its vol. of Et<sub>2</sub>O, repeat until the extn. is colorless, mix the white flocculent ppt. with acetone, let stand 45-60 min., centrifuge, decant, desiccate and grind to a fine powder. The product has approx. 10 fold the concn. of the active principle. C. V. Bailey

Relationship of the parathyroid gland to calcium metabolism. DeForest P. Willard. *Ann. Surg.* 102, 351-6 (1935). Rachel Brown

Chemical constitution of sex hormones. J. H. Blackwood. *Glasgow Med. J.* 6, 113-23 (1935).—A review with 38 references. Rachel Brown

The Zondek-Zondek pregnancy test. With special reference to reaction I. Mabel F. Potter. *J. Obstet. Gynaecol. Brit. Empire* 42, 644-50 (1935).—No relationship exists between the amt. of prolactin A excreted by a pregnant woman and the continuance of the pregnancy to term. Rachel Brown

Basal heat production of the Rhesus monkey (*Macaca mulatta*). Nathan Rabenstein. *J. Nutrition* 10, 357-62 (1935).—The av. basal heat production of 11 adolescent Rhesus monkeys was 608 Cal per sq m per 24 hrs. A similar heat production was found for males and females. Two expts. on 1 animal which received 35 mg. of Na amyltal per kg. gave results almost identical with those obtained on it under normal basal conditions. C. R. Fellers

Is the work of the kidney, due to the excretion of urea, a factor in specific dynamic action? A. G. Eaton, Shirley C. Cordill, J. L. Gonaux and Vera Clav. *J. Nutrition* 10, 429-37 (1935).—Metabolism expts. on 4 human subjects show that the work done by the kidney in the excretion of the urine during rapid excretion of urea is not a material factor in the sp. dynamic action of proteins. The data do not rule out the kidney as a possible seat for other

reactions dealing with the increased heat production following ingestion of proteins or amino acids. C. R. F.

Effect of male hormone on the protein and energy metabolism of castrate dogs. Charles D. Kociakian and John D. Murlin. *J. Nutrition* 10, 437-59 (1935).—Male hormone prepd. from urine gave a marked drop in urinary N with a subsequent increase both of which are due to changes in urea N. Other urine constituents show no significant changes. Creatine is not excreted by castrate dogs fed on a creatine-free diet. The decrease in urinary N does not begin until the 1st day after the injection. After reaching a max., further injections only maintain the decreased protein catabolism at this level it is more efficient to use small repeated doses than single large doses. The heat production of thin castrate dogs is unaffected while that of a fat castrate dog is slightly raised by 10% after repeated injections. The fat metabolism is slightly increased, the protein metabolism decreased, and the carbohydrate metabolism is unaffected in the thin dog, but is decreased in the fat dog. A slight but significant wt. gain occurred in dogs during injection. This added wt. was lost as soon as the injection ceased.

C. R. Fellers

Perfusion of the hind quarters of the dog. Noël Fressinger, Henri Bénard and Félix Pierre Merklen. *Compt. rend. soc. biol.* 119, 1373-5 (1935).—The hind quarters, in muscular repose, were perfused with de-aerated blood. The O consumption was 50-100 cc/lg./hr. There was no increase in urea, even when glycine was added to the blood, and little or no change in polypeptides and uric acid. Blood glucose slowly decreased and lactic acid remained stationary or sometimes decreased slightly. Galactose added to the blood was apparently utilized to some extent.

L. E. Gilson

Endocrinology of the pregnant cat. R. Courrier and Gaston Gros. *Compt. rend. soc. biol.* 120, 5-7 (1935).—In the cat ovulation occurs 26-27 hrs. after the first copulation. Nidation occurs 13-14 days later. The gestation period is 64 days. If the corpus luteum or the ovaries are removed before nidation has occurred nidation is prevented. If removed after nidation but before about the 46th day of gestation interruption of pregnancy by expulsion or resorption of the fetuses results. (In some cases the fetuses were resorbed while the placentas continued to grow.) After the 46th day of gestation the operation has no effect on the pregnancy. Action of folliculin in the pregnant cat. *Ibid.* 8-9.—The injection of 500-1000 rat units of folliculin before nidation causes abortion; after nidation it has no such effect. L. E. G.

Synthesis of aspartic acid from fumaric acid in the liver. Kurt P. Jacobsohn, Joan Tapadinhas and F. B. Pereira. *Compt. rend. soc. biol.* 120, 33-6 (1935).—A dog liver was perfused with de-aerated blood contg. NH<sub>4</sub> fumarate and acetate. About 10% of the fumaric acid was converted to aspartic acid. The latter was isolated from the deproteinized perfusate as its insol. Cu salt. L. E. G.

Remote effects of denervation of the adrenal on the secretion of adrenaline. J. A. Scroggio. *Rev. soc. argentina biol.* 11, 139-46 (1935); *Compt. rend. soc. biol.* 120, 270-2 (1935).—One adrenal was denervated, then studied 1-3 months later by connecting the vein of the operated gland to the jugular of another dog. Nicotine, candicine and cocaine produced adrenaline discharge, but less than normal. Yohimbine did not suppress the effect of candicine. Centripetal elec. stimulation of the vagus, brachial and sciatic nerves had no effect on the gland. L. E. G.

Progress and problems of endocrinology. R. G. Hoskins. *J. Am. Med. Assoc.* 105, 948-51 (1935).—Discussion.

F. P. Griffiths

Effect of thymus extract (Hanson) on the early eruption and growth of the teeth of white rats. M. T. Barrett. *Dental Cosmos* 77, 1088-93 (1935).—Thymus ext. caused very early eruption of the teeth and a marked acceleration in their growth.

Joseph S. Hepburn

Studies of the pH of normal resting saliva. II. Diurnal variation. Robert E. Brawley. *J. Dental Research* 15, 79-80 (1935); cf. C. A. 29, 7428<sup>a</sup>.—The normal resting

saliva of 3403 normal healthy individuals (both sexes, ages 3 weeks-101 yrs) was tested colorimetrically for pH at intervals throughout the day. The av. normal value was 6.75, slightly higher values occurred 1 hr. before breakfast (6.8) and 1 hr. before lunch (6.83), and slightly lower values 1 hr. after breakfast (6.73) and 1 hr. after lunch (6.68). No significant difference existed between the av. value in the morning (6.76) and in the afternoon (6.74). The diurnal variations were independent of age and sex. III. Effects of vitamins A and D in school children. *Ibid.* 87-8.—The subjects were school children, aged 11-17, av. 13, yrs; 164 served as controls, while a daily dose of 6 tablets, each contg. 1500 units vitamin A and 2450 units vitamin D, was administered to each of 113 subjects for a period of 1 yr. At the end of 6 months the av. pH of the normal resting saliva was 6.76 in the expt. proper and 6.75 in the controls. At the end of 1 yr. the av. pH was 6.79 in the expt. proper and 6.78 in the controls. No significant difference existed between the 2 groups.

Joseph S. Hepburn

Iron content of teeth of normal and anemic rats. Sarah Ratner. *J. Dental Research* 15, 89-92 (1935).—The av. Fe content of the upper incisors was 0.0114% in 8 rats fed an anemia-producing ration for 121 days, and 0.0298% in 6 control litter mates fed a complete ration. When the incisors were first treated with 0.1 N NaOH soln. to remove hemoglobin from the dental pulp, the Fe content of the teeth proper was 0.0126% in the anemic rats, and 0.0262% in the controls. A relationship apparently existed between the Fe content of the teeth and their color. J. S. H.

Chemical regulation and the hormone concept. Julian S. Huxley. *Biol. Rev. Cambridge Phil. Soc.* 10, 427-41 (1935).—A review.

E. H.

Preparation of feeds for cattle as it affects digestibility and absorption. E. A. Silver. *Agr. Eng.* 16, 257-9, 270 (1935).—Crude fiber and crude protein deaths were made on samples of the rumen contents of 4 steers and on the alfalfa hays which were fed. The 1st samples, taken before feeding and representing the residue from previous feedings, always contained a relatively high percentage of crude fiber and a low percentage of crude protein when compared with the analyses of the hays fed. The 2nd samples, representing a mixt. of the hay eaten and the residue in the rumen, contained less crude fiber and more crude protein than the previous sample. The subsequent samples, taken every 2 hrs., showed, in general, a rise in crude fiber content and a reduction in crude protein content of the material the longer it remained in the rumen.

K. D. Jacob

Plasma lipides of normal men at different ages. Irvine H. Page, Lshen Kirk, Wm. H. Lewis, Jr., Wm. R. Thompson and Donald D. Van Slyke. *J. Biol. Chem.* 111, 613-39 (1935).—The plasma lipides were detd. by the gasometric methods of Kirk, Page and Van Slyke (C. A. 28, 6755). Variations in age from 20 to 90 yrs. have no determinable influence on either the amt. or the compn. of the plasma lipides. Part of the amino and monoglyceride N obtained in the petr. ether exts. appears to be derived from substances other than phosphatides. The following results for lipides in normal plasma from 66 subjects in mg. per 100 cc. of plasma were obtained: total cholesterol 232 ± 62, free cholesterol 82 ± 17, phosphatides 181 ± 71, neutral fats 225 ± 137, total lipides 735 ± 216. The cholesterol values range much higher than those of Boyd (C. A. 27, 5092, 29, 1810<sup>a</sup>) and of Gardner and Gainsborough (C. A. 21, 2706) but no explanation is apparent.

A. P. Lathrop

The effect of age on the plasma calcium content of men. Esben Kirk, Wm. H. Lewis, Jr., and Wm. R. Thompson. *J. Biol. Chem.* 111, 611-2 (1935).—The Ca detns. were made by the gasometric method of Van Slyke and Sendroy (C. A. 24, 392) and the subjects were all normal males in so far as could be ascertained by clinical examn. They were part of the group who provided the plasma for the accompanying study of plasma lipides (cf. preceding abstr.). No effect of age changes up to age 85 was observed and the variations between age classes were no greater than would be expected by the variations within

age classes. The mean for age class varied from 9.8 to 10.6 mg per 100 cc. A. P. Lothrop

The lipid content of the jelly of Wharton. Eldon M. Boyd *J Biol Chem.* 111, 667-9(1935).—The total lipid content of the jelly of Wharton from the umbilical cord, one of the least active of all the body tissues, varied between 116 and 330 mg % with a mean value of 209, percentages much lower than those of any other tissue of the body. The total lipid was composed on the av of 57% phospholipide, 26% neutral fat, 15% free cholesterol and 2% cholesterol esters. The results are addnl evidence in favor of Bloor's hypothesis regarding the relationship between lipid compn and physiol activity. The almost uniform absence of cholesterol esters suggests that large percentages of this lipid characterize degenerating rather than inactive tissue. A. P. Lothrop

Blood chemistry of swine II. Further studies of blood changes following the ingestion of glucose. Donald F. Eveleth and Margaret W. Eveleth *J Biol Chem.* 111, 753-6(1935); cf. *C. A.* 28, 3779\*.—The feeding of glucose to fasting swine increases the serum Mg and oxalic acid and decreases the inorg phosphate and Ca. It seems likely that oxalic acid may be a decompo product of disintegrating sugar and the marked increase in serum Mg definitely points to some relationship of Mg to glycogen formation. A. P. Lothrop

The copper content of urine of normal children. Allan Ross and I. M. Rabinowitch *J Biol Chem.* 111, 803-5(1935); cf. *C. A.* 27, 3514.—Cu appears to be a const. constituent of the urine of normal children (50 subjects) and is present in amts varying between 0.04 and 0.52 mg per l (av 0.3) and between 0.026 and 0.02 mg per day (av. 0.16). The concns agree very closely with those found in adults but the av amt. per day is appreciably smaller. A. P. Lothrop

The existence in the blood and the urine of substances promoting liver function II. The urine. Nobuo Mizuta and Tatsuo Matsura *Japan J Gastroenterol.* 7, 67-68 (1935); cf. *C. A.* 29, 6635\*.—In normal human and rabbit urine are phenol-like substances which promote the excretion of azofuchsins-G from the liver of rabbits poisoned with U nitrate or cantharidin. These urinary substances are thermostable in acid or neutral medium but readily destroyed by heating with alkali. C. M. McCay

The significance of the liver for the metabolism of lactic acid. Inkei Ohashi *Japan J Gastroenterol.* 7, 88-103(1935).—Normal livers perfused with lactic acid split the d form more effectively than the dl-one. Livers impaired by the usual agents as CHCl<sub>3</sub> decrease in their utilization of lactic acid. C. M. McCay

The functional innervation of the bone marrow. Piero Foà *Arch sci biol (Italy)* 21, 133-66(1935).—Bone marrow is provided with unmyelinated vasoconstrictor nerve fibers. It is possible to produce vasoconstriction and vasodilation which vary the vol of the organ; such variation in vol det. the outpouring of blood cells into the circulation and especially the immature cells. The spinal nerves act through the sympathetic fibers. In the marrow there are pain sensory fibers that could be the center of a neurogenic or neuro-hormonic regulation of hemopoiesis. Cutting the nerve fibers stimulates activity of hemopoiesis. P. F. Metuldi

Glucolysis in maternal and fetal blood. G. Tesaro *Arch sci biol (Italy)* 21, 197-200(1935).—Fetal blood has a more active glucolysis than maternal blood. P. F. Metuldi

The chemistry of muscular contraction. R. Margaria *Arch sci biol (Italy)* 21, 213-31(1935).—A review of the literature. P. F. Metuldi

The calcium content of the red cells in human blood. Paolo Larizza. *Biochim terap. sper.* 22, 289-5(1935).—The erythrocytes contain less Ca than serum and consequently than the whole blood. The av value is 3 mg in 100 g. A. E. Meyer

Blood lactic acid in the dextral abdominal syndrome. F. Rabbioni *Biochim terap. sper.* 22, 307-17(1935).—The lactic acid was increased in cases with cholecystitis. A. E. Meyer

1 The relationship between body weight, body surface and respiratory exchange. Alfio Falaschini *Biochim terap. sper.* 22, 320-50(1935).—Expts. on rats showed that an individual factor influences the metabolism besides surface and wt. The metabolism in younger animals is always more active than in older ones. A. E. Meyer

The biological reaction of lability in human serums. Giovanni Scuderi *Biochim terap. sper.* 22, 351-5 (1935).—Normal human serum, stable in respect to colloids, causes a decrease of the surface tension of the serum in rabbits after intravenous injection. Labile serums produce an increase of the surface tension. Antitoxins which may be present in the serum have no influence on the reaction. A. E. Meyer

Functional relationship between epiphysis and anterior hypophysis II. Action on magnesium, phosphate and calcium contents of the blood. Salvatore Fiandaca. *Biochim terap. sper.* 22, 363-8(1935).—Though an aq ext of the pineal gland has no direct action on the Mg and P in the blood, it prevents the increase caused otherwise by pituitary exts. The Ca is not affected by either ext. It is concluded there is a certain antagonism between both glands. A. E. Meyer

Hypophyseal-endocrine interrelations: Hypophysis and parathyroids. R. Rivoire *Presse méd.* 43, 528-30 (1935).—A review. A. E. Meyer

Recent progress in the knowledge of hormones. Bernardo A. Houssay. *Rev. farm. (Buenos Aires)* 77, 233-46 (1935).—A review. A. E. Meyer

Colloid-osmotic pressure measurements in arterial and venous blood during parturition. R. Kessler and Friedrich Paulsen *Acta Med Scand* 86, 100-26(1935).—The oncotic pressure of serum is about 33 mm H<sub>2</sub>O higher than that of plasma, this difference being practically const. under physiol. and pathol. conditions. The oncotic pressure undergoes regular changes during birth. During the period of dilation the blood of the capillaries becomes somewhat thinned out but as the parturition progresses this diln becomes less. In neuropathia gravidarum the oncotic pressure shows the usual variations of pregnancy but the difference between arterial and venous pressure is very great, being 48 mm H<sub>2</sub>O. S. Morgulis

The organ of histidine in the urine from pregnant women. Regine Kapeller-Adler and Fritz Haas *Biochem Z* 280, 232-41(1935).—No evidence was found of the participation of the placenta in the liberation of histidine. Expts with ground kidney showed that this had no effect on added histidine but liver from males and females, as well as from patients with severe liver damage, or from operatively castrated women destroys it quite actively. Up to 60% of 50 mg added histidine may be thus destroyed by 1 g liver. However, the liver from pregnant women exerts practically no such action. Evidently, the histidinase activity is inhibited and the unchanged histidine appears in the urine. In pregnant animals, on the other hand, the histidinase retains its activity so that no histidine appears in their urine. S. Morgulis

Occurrence of bromine in the normal organism. Theodor Leipert. *Biochem Z* 280, 416-33(1935).—The Br content of normal blood ranges from 0.16 to 0.40 mg %, that of the plasma being somewhat higher (0.18-0.45 mg %). The Br is retained largely in the erythrocytes but its distribution, like that of the Cl, is also affected by the CO<sub>2</sub> tension of the blood. The amt. of Br in the blood is too small to affect the Cl content. The Br is all in the form of its ions and no evidence could be found of a compd with protein or other org substance. Although the excretion of Br through the urine, like that of the Cl, depends upon its concn. in the blood, the Cl/Br ratios of the blood and urine are not similar. The Br always accompanies Cl in the intermediate metabolism, gastric juice secretion, deposition in the tissues, etc., and tissue diuretics lead to a washing out of Br. Milk feeding brings about a shift in the Cl/Br ratio of the organism which manifests itself in a lowering of this ratio in the urine, while the newborn shows in the urine much higher

ratios which are in good agreement with the Cl/Br ratio of the blood (umbilical).

S. Morgulis  
Calcium and phosphorus metabolism of the chick embryo. Tatsumi Kamachi. *J. Biochem. (Japan)* 22, 189-197 (1935).—Embryos were injected with  $\text{CaCl}_2$ ,  $\text{Na}_2\text{HPO}_4$  or  $\text{Yatokonin}$  (a com. Ca fructose-phosphate compd.) and the allantoin fluid as well as the embryo were analyzed for Ca and P at intervals. Such treatment resulted in a diminished Ca content and, since there has been no change in the Ca content of the allantoin fluid, it is assumed that the mobilization of Ca from the shell had been inhibited. Treatment with the  $\text{Yatokonin}$  resulted in a greater P content, not observed with the  $\text{Na}_2\text{HPO}_4$ .

S. Morgulis  
Histidine formation from arginine in the incubated chick egg. Tatsumi Kamachi. *J. Biochem. (Japan)* 22, 199-202 (1935).—Evidence is presented to show that arginine injected into developing chick embryos is partly converted to histidine.

S. Morgulis  
Experimental study of the ligation of the rat sperm duct. Stephan Leiter. *Endokrinologie* 16, 160-74 (1935).—No changes in the testes of the rat follow the ligation of the vas deferens.

S. Morgulis  
Hormone studies on corpus luteum cysts. Arthur v. Prolastner. *Endokrinologie* 16, 174-9 (1935).—Prolan B and folliculin, but no lutin, were found in the corpus luteum cyst fluids.

S. Morgulis  
Physiology of the colon. II. The iron elimination in the intestinal canal of dogs. Ragnar Nicolaysen. *Skand. Arch. Physiol.* 72, 126-32 (1935); cf. C. A. 29, 837-8. During Fe hunger the isolated colon of the dog excretes 0.03-0.06 mg. per day and the rest of the intestinal canal about 2 mg., but no Fe is eliminated through the urine. After an injection of Fe, a small part appears in the urine, whereas the excretion through the intestine is unaffected.

S. Morgulis  
The relative choline-esterase activities of serum and corpuscles from the blood of certain species. Edgar Itedman and Ellen Steedman. *Biochem. J.* 29, 2107-11 (1935).—A method is described for the estn. of choline-esterase (I) applicable to whole blood, corpuscles or tissue. A Barcroft differential app. was used. The blood sera of the ox, sheep and goat were deficient in I, although it was present in appreciable amts. in the corpuscles. Corpuscles from the cat, fowl and duck contained no I. I was absent in the cerebrospinal fluid of the cat and man but occurred in considerable amts in the brain.

E. W. Scott  
The linkage of chemical changes in muscle extract Dorothy M. Needham and Wm. E. van Heyningen. *Biochem. J.* 29, 2040-50 (1935); C. A. 29, 409-54. By use of dialyzed frog and rabbit muscle exts. it was shown that in the presence of Mg and adenylic acid (I), added creatine (II) was converted to phosphocreatine (III) during the breakdown of phosphoglyceric acid (IV). The synthesis involved the reaction of IV (or phosphopyruvic acid) and I to form adenylyphosphate (V) and pyruvic acid, followed by the action of V on II giving III and I. Evidence was cited to show that the coenzyme function of I and V was due to their ability to act resp. as phosphate acceptor and donor.

E. W. Scott  
A water-soluble precursor of choline in the human placenta. David H. Smyth. *Biochem. J.* 29, 2067-70 (1935).—Aq. exts. of human placenta contain a water-sol substance contg. choline (1 mol.) bound to  $\text{H}_2\text{PO}_4$  (1 mol.) and an amino group (1 mol.). The presence of free choline also in the placenta was demonstrated by Florence's test. The choline compd. was apparently the same as that isolated from the kidney by Booth and Milroy (C. A. 29, 63114).

E. W. Scott  
The metabolism of amino acids. V. The conversion of proline into glutamic acid in the kidney. Hans Weis-Malherbe and Hans A. Krebs. *Biochem. J.* 29, 2077-81 (1935); cf. C. A. 29, 8030-1. Proline (I) and hydroxyproline (II) with  $\text{NH}_3$  gave an acid amide similar to glutamine (III) in the presence of rabbit kidney slices. When  $\text{As}_2\text{O}_3$  was added to the medium contg. kidney tissue, I gave  $\alpha$ -ketoglutaric acid (IV) and  $\text{NH}_3$ . The oxidation of I in the kidney was therefore formulated as follows. I

+  $\text{O}_2 \rightarrow$  glutamic acid (1) +  $\text{NH}_3$  III or (2) +  $\text{O} \rightarrow$  IV +  $\text{NH}_3$ . Pyrroloindolecarboxylic acid was not the intermediate in the first step since it was not metabolized by the kidney. II was not an intermediate since it reacted more slowly than I in respect to amino-N and to amide-N formation.

E. W. Scott  
The role of glutathione in muscle glycolysis. Robert Gaddie and Corbet P. Stewart. *Biochem. J.* 29, 2101-6 (1935).—Dialyzed muscle exts. produced lactic acid (I) from glycogen (II), glucose, hexosediphosphoric acid or an equimol. mixt. of pyruvic acid (III) and glycerophosphoric acid in larger amts. when reduced glutathione (IV) was also present than when  $\text{Mg ions}$  (V) and adenylyphosphate (VI) were the only coenzymes present. In the absence of IV,  $\text{MeCOCHO}$  (VII) accumulated, the addn. of IV diminished the amt. of VII or prevented its accumulation. When VI was omitted IV and V allowed the production of I from II by muscle exts., but in this case the omission of IV caused no increase in the amt. of VII. During the formation of I, VII accumulated and then disappeared in part. The amt. lost was not fully accounted for by conversion to I.

E. W. Scott  
Estimation of muscle hemoglobin. Rodger H. Watson. *Biochem. J.* 29, 2114-21 (1935).—The hemoglobins of finely minced muscle (5-6 g.) mixed with sand were quantitatively removed by 2 extns. with  $\text{M}/15$  phosphate buffer,  $\text{pH } 6.5$ , (12-14 ml.). The exts. were sepd. by centrifuging and the hemoglobins were obtained by adsorption on kieselguhr, elution with 0.1%  $\text{NH}_4\text{OH}$ , adsorption on  $\text{Al(OH)}_3$ , and final elution with  $\text{NH}_4\text{OH}$ . The relative concns. of blood and muscle hemoglobins in a soln. could be detd. by the Hartridge reversion spectroscopy and consequently the muscle hemoglobin could be estd. in muscle contg. blood. The detn. of hemoglobin by conversion to acid hematin was found suitable for muscle ext. The method of quant. extn. gave results about 30% lower than those of Whipple (C. A. 20, 2599). A small no. of muscle hemoglobin detns. were given.

E. W. Scott  
The relation of the parathyroid hormone to the state of calcium in the blood. Franklin C. McLean, B. O. Barnes and A. Baird Hastings. *Am. J. Physiol.* 113, 141-9 (1935); cf. C. A. 29, 2217-8. A study was made of the state of Ca in the serum in conditions approximating hypo- and hyperfunction of the parathyroid glands. Hypofunction was simulated by thyroparathyroidectomy, hyperfunction by subcutaneous injection of the parathyroid hormone. Most of the expts. were done on cats, with a few observations upon chronic latent tetany in the dog. Conclusion: Removal of the parathyroid glands leads to a reduction in Ca concn. of the plasma, injection of parathyroid hormone to an increase. Acute postoperative parathyroid tetany, in the cat, was observed only when the  $\text{Ca}^{++}$  concn. in the serum was 0.65 mM. per kg.  $\text{H}_2\text{O}$  or below, and then only irregularly. The symptoms of hyperthyroidism may appear, in the cat, when the Ca concn. in the serum rises above 1.7 mM. per kg.  $\text{H}_2\text{O}$ , and death may occur at concns. between 1.7 and 2.0 mM. per kg.  $\text{H}_2\text{O}$ . Chronic latent tetany, in the dog, is accompanied by a chronic lowering of the Ca concn. in the plasma. The use of Na polyanetholsulfonate as an anticoagulant does not affect the ionization of Ca, and plasma so obtained from dog blood is not toxic to the frog heart.

E. D. Walter  
The relative significance of electrolyte concentration and tissue reaction in water metabolism. H. A. Davis and Lester R. Dragstedt. *Am. J. Physiol.* 113, 193-9 (1935); cf. C. A. 29, 4817-8. A study was made of the ability of normal and dehydrated dogs to retain 0.9% NaCl and 5% glucose solns. when injected intravenously. Three types of dehydration were produced: (1) by simple  $\text{H}_2\text{O}$  deprivation, (2) dehydration plus severe electrolyte loss and marked alkalosis resulting from loss of gastric juice from a special fistula (C. A. 24, 4554) and (3) a similar  $\text{H}_2\text{O}$  and electrolyte loss plus an acidosis resulting from loss of pancreatic juice through a special fistula (C. A. 25, 5453). Normal animals excreted the injected fluids most readily and completely. Group I retained both glucose

and NaCl solns. better than any of the others. Groups 2 and 3 did not retain injected H<sub>2</sub>O much better than normal controls. Group 2 retained H<sub>2</sub>O somewhat better than group 3. Group 3 animals were depleted of base, chiefly Na ion, still they did not retain injected 0.6% NaCl as well as those in group 2. E. D. Walter

The specific gravity of the blood of normal rabbits and cats and splenectomized rabbits before, during and after emotional excitement. L. B. Nice and H. L. Katz. *Am. J. Physiol.* 113, 245-8 (1935); cf. C. A. 28, 2768. The sp. gr. of the blood of normal rabbits and cats showed an immediate and marked elevation during emotional excitement. Peripheral blood showed a greater sp. gr. increase than central blood. Splenectomy raised the basal level and diminished the sp. gr. increase of excited blood. Recovery of the sp. gr. to the normal level is gradual and was delayed in the splenectomized group. Complete recovery was obtained within 30-40 min. after the excitatory period. The factors accounting for these changes are discussed. E. D. Walter

Correlated studies of calcium, inorganic phosphorus, and serum phosphatase in normal animals and in animals influenced by irradiated ergosterol. Smith Freeman and Chester J. Farmer. *Am. J. Physiol.* 113, 279-29 (1935); cf. C. A. 26, 4362. Toxic doses of irradiated ergosterol increase serum Ca and (or) inorg. P and decrease the serum phosphatase activity in dogs and rabbits. Bleeding causes a decrease in phosphatase activity. In dogs, after the initial lowering, the phosphatase remains rather constant. A beef heart (high protein) diet induces a low phosphatase activity, while a bread and meal (high carbohydrate) diet causes a high phosphatase activity. The serum phosphatase of dogs fed irradiated ergosterol shows an inverse relationship to the serum Ca and (or) inorg. P. The results suggest that an inverse relationship exists between the activity of serum phosphatase and the acid sol. org. P of whole blood. E. D. Walter

Basal metabolism and iodine excretion during pregnancy. Lena Enright, Vera V. Cole and F. A. Hitchcock. *Am. J. Physiol.* 113, 221-8 (1935).—Growth of the fetus and supplementary tissues plays an important part in producing the increased metabolism which accompanies pregnancy. With adolescent girls, however, there appears to be another factor which stimulates the metabolism and results in a greater rise than that which occurs with more mature women. The use of iodized salts furnishes an adequate supply of I during pregnancy. There is a marked increase in I excretion in the 3rd week ante partum which suggests a possible change in the thyroid function. In 6 out of 7 cases the vital capacity increased during pregnancy, and in all 7 cases there was a progressive increase in tidal air during the latter part of pregnancy which was followed by a decrease after parturition. E. D. Walter

Plasma protein determinations in lactating women. Silber C. Peacock and Winifred F. Hamman. *Am. J. Physiol.* 113, 235-7 (1935).—Considerable losses of protein in the milk have no apparent effect on the blood proteins, which are in all cases normal quantities. E. D. Walter

Influence of the ovarian hormones, estrin and progesterin, upon the menstrual cycle of the monkey. Geo. W. Corner. *Am. J. Physiol.* 113, 334-50 (1935).—Progesterin has the definite property of inhibiting the menstrual flow in the monkey. Simply maintaining the estrin level by injections of Progynon-B or crude follicular estrin is not sufficient to prevent the onset of natural menstruation. E. D. Walter

An assay of three hormones present in anterior pituitaries of seven types of cattle classified for age, sex and stage of reproduction. Robert W. Bates, Oscar Riddle and Ernest L. Lahr. *Am. J. Physiol.* 113, 253-64 (1935).—Seven classes of cattle hypophyses (embryos, veal, steers, bull, nonpregnant cow, early pregnancy cow and late pregnancy cow) were assayed for their content of prolactin, follicle-stimulating (F. S. H.) and thyrotropic hormones. Thyrotropic hormone was found in fairly equal quantity in the 7 types except that low values were obtained from the glands of embryos (5-7 months) and adult steers. F. S. H., in assays uncomplicated by

the "augmentation" phenomenon, was found in greatest amt. in glands from cows in early pregnancy and least in adult steers. Nearly equal amts. of prolactin (25-50 units) were found in the anterior lobes of veal calves, adult steers, adult bulls and in nonpregnant cows. Large amts. were present in cows in early pregnancy (58 units) and late pregnancy (44 units). A much larger amt. (784 units) was found in the whole glands of 5-7 month embryos. E. D. Walter

Basal metabolism and urinary nitrogen excretion of Oriental women. Abby H. Turner and Francis G. Benedict. *Am. J. Physiol.* 113, 291-5 (1935); cf. C. A. 26, 3613. The basal metabolism of 10 well-nourished foreign-born Oriental women students who had been living for 1-3 years in the United States, in an American college environment and partaking of an American college diet, averaged 12% below the prediction standard and was lower than that of 6 American college mates. The low basal metabolism cannot be ascribed to a low-protein metabolism. E. D. Walter

Relation of the suprarenal cortical hormone to nitrogen metabolism in experimental hyperthyroidism. Giles A. Koeleche and Edward C. Kendall. *Am. J. Physiol.* 113, 335-49 (1935); cf. C. A. 16, 2719. Administration of thyroxine to a suprarenalctomized dog receiving a low maintenance dose of cortical hormone produces a neg. N balance. The hormone of the suprarenal cortex exerts a sparing action against the effect of thyroxine on N metabolism. The neg. N balance can be lessened and a pos. balance sometimes can be maintained by giving, with the thyroxine, sufficiently large amts. of cortical hormone. Expts. show that the amt. of cortical hormone administered is one of the factors which dict. the amt. of loss of N produced by a given dose of thyroxine. The results suggest that the effect of thyroxine on N metabolism may be indicative of the amt. of cortical hormone available in the body of the dog. E. D. Walter

Excretion of inulin, creatinine, xylose and urea in the normal rabbit. Bernard L. Kaplan and Homer W. Smith. *Am. J. Physiol.* 113, 354-60 (1935); cf. Shannon, C. A. 29, 7441. The inulin, creatinine, xylose and urea clearances in the normal rabbit are reported. All of these clearances increase with increasing urine flow, and fail to reach a const. value at urine flows that are maximal under the conditions of these expts. Simultaneous clearances of inulin and creatinine are equal, regardless of urine flow or plasma level of creatinine. The change in the inulin and creatinine clearances in relation to changing urine flow is interpreted as indicating a corresponding change in glomerular activity. The xylose and urea clearances are considerably less than the creatinine-inulin clearance. Administration of H<sub>2</sub>O by mouth or by injection in excessive amts. leads to oliguria, convulsions and death. This, coupled with the physical relationship between glomerular activity and H<sub>2</sub>O excretion, should be considered in all renal function studies in the rabbit. E. D. Walter

Fetal carbohydrate metabolism following adrenalectomy, insulin and glucose experiments on the mother. E. L. Corev. *Am. J. Physiol.* 113, 450-4 (1935); cf. C. A. 20, 7439. Adrenalectomy of the mother rat produced significant depletions in the glycogen content of the maternal and fetal livers. In 19 cases there was an av. reduction of the hepatic glycogen of 70% in the mother and 43% in the fetus, compared to 12-hr. fasted controls. Injections of cortico-adrenal ext. in pregnant rats failed to cause changes in the carbohydrate values studied. Administration of insulin to the mother was followed by decreases in maternal and fetal liver glycogen similar to those after adrenalectomy. Injections of glucose into the mother increased the glycogen concn. in the fetal liver, as well as in that of the mother. The placental glycogen was relatively unaffected by the exptl. procedures used. The fetal muscle glycogen showed no significant change except a slight rise after glucose injection in the mother. E. D. Walter

A histamine-like substance in the gastric juice. Charles L. Brown and Ralph G. Smith. *Am. J. Physiol.* 113

455-63(1935).—The method of extn. of histamine from tissues used by Best and McHenry (C. A. 25, 2450) was applied to human gastric juice, obtained by continuous aspiration. The presence of a histamine-like substance in the gastric juice was demonstrated by biol. methods.

E. D. Walter

Liberation of ammonia by the brain following the natural state of excitation. Joseph Kahn and Mme. Lubov Chekoun. *Compt. rend.* 201, 505-6(1935).—The  $\text{NH}_3$  absorbed by the brain of the fish at  $15-16^\circ$  during normal respiration was 1.14 mg. per hr., during dyspnea, 1.63 mg. At  $19-20^\circ$  these values increased to 1.68 and 2.51, resp. The fish *Carassius* placed in a stream of running  $\text{H}_2\text{O}$  for 1-2 hrs. at  $3-4^\circ$  gave an  $\text{NH}_3$  value of 0.58 mg. Hence a lowering of temp. caused a 50% decrease in  $\text{NH}_3$ .

Julius White

Precursors of coprosterol and the bile acids in the animal organism. O. Rosenheim and T. A. Webster. *Nature* 136, 474(1935).—Cholestone (I) added to a rat diet poor in cholesterol (II) gave rise to a large excretion of fecal coprosterol (III). This is in accord with the idea of R and W. I and coprostanone and not II are the intermediate precursors of III formed from it in the intestine by bacterial reduction.

Julius White

Hormones and their use in cosmetics (Brugmans) 17.

## G—PATHOLOGY

H. GIDEON WELLS

Caisson disease and its relation to tissue saturation with nitrogen. Charles W. Shilling, James A. Hawkins, B. Polak and Raymond A. Hansen. *U. S. Naval Med. Bull.* 33, 434-44(1935).—The N satn. of the tissues is definitely related to the incidence of caisson disease. Forty-six cases are presented.

Philip D. Adams

Placental immunity. I. A method of determining dosage of placental globulin in measles prophylaxis. Samuel Karelitz, Charles K. Greenwald and A. J. Klein. *Proc. Soc. Exptl. Biol. Med.* 32, 1350-62(1935). Cf. C. A. 28, 11891.—Globulin extd. from the placenta contains the measles antibody and diphtheria antitoxin. Globulin extd. from immune human blood serum is equally effective in measles prophylaxis as the equiv. amt. of the blood serum, which is given in doses of 8-40 cc. By comparing the titer of diphtheria antitoxin of pooled 1-cc. specimens of maternal blood serum with that of the globulin extd. from pooled placentas, one obtains equivalents which det. the dose of globulin in measles prophylaxis. II. Comparison of maternal circulating blood immunity with that of placental fluid. Samuel Karelitz and Charles K. Greenwald. *Ibid.* 1362-5.—The fluid expressed from the human placenta has the same diphtheria antitoxin titer as that of the maternal blood serum. This fluid from pooled placentas may be used in place of the pooled maternal blood serum in detg. the dose of placental globulin in measles prophylaxis.

C. V. Bailey

Positive flocculation tests in rabbits inoculated with flocculate from human syphilitic serum. F. Rytz. *Proc. Soc. Exptl. Biol. Med.* 32, 1501-4(1935).—Rabbit serum gave pos. flocculation tests for at least 2 months after the last injection of flocculate from human syphilitic serum; the flocculate thus obtained caused the formation of "flocculins" in another rabbit when injected intravenously. The serum of a rabbit injected with 360 flocculate units developed 1260 units in excess of the no. injected.

C. V. Bailey

Flocculation of some serums by orthophosphoric acid solutions. Pio Gori. *Arch. sci. biocim. ital.* 7, 61-8 (1935).—When serums of different species of animals, dilid. with  $\text{H}_2\text{O}$  (1:10), are mixed with a sufficient quantity of  $\text{H}_3\text{PO}_4$  (0.315 per 1) and warmed to  $80^\circ$  for 5 min., a flocculation is produced. The concn. of  $\text{H}_3\text{PO}_4$  necessary to produce optimum flocculation is, between certain limits, const. and characteristic for serum of each species (pig, horse, cattle, sheep). Results are more const. when old serum is used.

E. S. G. B.

Diagnostic value of phosphatase determinations in the study of bone tumors. Channing C. Simmons and Ch.

ford C. Franseen. *Ann. Surg.* 102, 555-62(1935).—Plasma phosphatase was consistently increased in metastatic carcinoma and osteogenic sarcoma.

R. B.

A serological study emphasizing the hydrogen-ion concentration of the blood—in conjunction with the red-cell sedimentation test, leucocytic index and complement-fixation test. K. T. Sasano. *Am. Rev. Tuberculosis* 32, 458-74(1935).—The  $\text{pH}$  accepted as normal for health appears too broad. To obtain basic and comparable  $\text{pH}$  values it is suggested that they be detd. on fasting blood early in the morning. This is important since exercise affects the  $\text{pH}$  values. The  $\text{pH}$  values of the blood of ambulatory tuberculosis patients do not vary significantly from the values obtained in nontuberculous cases and in normal controls. Strict bed-cases have, as a rule, more alk. blood than ambulatory cases. No definite correlation was found among the  $\text{pH}$  of the blood, sedimentation rate of erythrocytes, the leucocytic reaction and the complement-fixation test for tuberculosis.

H. J. Corper

Liver-function tests in productive and exudative pulmonary tuberculosis. F. Meythaler and C. Pelz. *Beitr. klin. Tuberk.* 85, 696-700(1934).—Comparative tests of the liver function using the levulose tolerance and the primary paradoxical insulin hyperglucemia according to Bürger were performed on 40 cases of pulmonary tuberculosis. Both methods revealed in nearly all the cases consistent deviations from normal in the carbohydrate metabolism of the liver, which was attributed to liver injury in the form of fatty degeneration. It is suggested that carbohydrate and insulin be used to combat the disturbed liver function.

H. J. Corper

The Meuncke serum reaction in tuberculosis. F. Böhm and G. Grüner. *Beitr. klin. Tuberk.* 86, 37-44 (1935).—Three types of Meuncke reactions were tried in tuberculosis, and the centrifugation method was found unsatisfactory because of too many sources of error. Coincident microreactions proved valuable especially in questionable cases. Pleural, ascitic and lumbar fluids proved unsatisfactory for test. Of 293 patients with undoubted tuberculosis 255 (87%) gave a pos. test. Two explanations are given for neg. findings in definite tuberculosis, either the body has lost its power to form antibodies or has not reached the stage of antibody production, or else overproduction of antibodies has resulted in flooding the body so that a detn. of free antibodies is not possible. Au-treated patients showed a weak reaction, while tuberculin treatment resulted in strong reactions. The serums of 80 nontuberculous patients gave neg. reactions; only in typhus fever were pos. reactions obtained. Two cases of asthma were also pos. The Meuncke reaction was found to be as sensitive as the Betredka reaction.

H. J. C.

The value of the so-called phytotoxic index in pulmonary tuberculosis. Julian L. Garcia and Garcia-Minon. *Beitr. klin. Tuberk.* 86, 45-8(1935).—The phytotoxic index is indicated by the toxic action of certain drugs on plant protoplasm. In place of drugs the authors use the serum from tuberculous cases and add this to salt solns., detg. the inhibitory or enhancing effect of the serum on the growth of lupines. The phytotoxic index is the fraction, av. growth of the test group = 100 divided by the av. growth of the control group. The normal is 76. The serums from over 100 cases of tuberculosis (about 220 tests being made) in various stages of the disease were tested and correlated with the clinical, röntgenologic and lab. data. The phytotoxic index possessed no sp.-diagnostic value but is valuable in prognosis combined with röntgenologic and lab. data. In benign forms of tuberculosis figures between 42 and 76 were found. In evolutive caseo-fibrous cases, the phytotoxic index was increased (80 to 100). It is therefore, believed that the phytotoxic index shows a direct relation to the severity of the tuberculous disease. In advanced stages of the disease the serum contains substances, of unknown nature, stimulating the growth of lupine roots. Conc'd. tuberculin added to the salt mixt. retards the growth of the roots in definite relation to its concn.

H. J. Corper

Iron deposition in tuberculous tissues. Bernhard Steinmann. *Beitr. klin. Tuberk.* 86, 84-98(1935).—

5 repeated Menkin's work (C. A. 28, 73687) on the deposition of Fe in caseating tubercles which in rabbits was found to retard the disease process. In human tuberculous tissues he found Fe present only where deposited prior to tuberculous changes. Tuberculous guinea pigs fed Fe preps showed no special depositions of the Fe in the tuberculous tissues. The liver, spleen and lungs contained large amts of Fe, but the tubercles in these organs remained free from Fe. Only where Ca impregnation of tuberculous caseation occurred, was Fe found. A favorable influence of the administration of Fe on the course of the tuberculosis was not noted. Intravenous injection of FeCl<sub>3</sub> into tuberculous rabbits also revealed no effect on the disease. These findings do not agree with those of Menkin.

H. J. Corper

Carbohydrate assimilation in various forms of tuberculosis in childhood, especially with pulmonary infiltrations. E. Mahr. *Z. Tuberk.* 72, 1-11 (1935).—The alimentary blood-sugar curve in children was studied in relation to pulmonary tuberculous infiltrations. In the majority of cases there was a prolonged blood-sugar curve with marked retardation to starvation levels; paralleling the resorption of the infiltration the blood-sugar curves returned to normal. The same deviations were found in cases of erythema nodosum. The carbohydrate tolerance in inactive tuberculosis and in extrapulmonary cases of tuberculosis did not deviate from normal. In hilum gland tuberculosis high blood-sugar values attained. The levulose-tolerance tests showed a marked retardation in the blood-sugar fall in all forms of active tuberculosis, as an expression of the injury of liver cells by toxins, tissue protein disintegration products and also partly as an expression of amyloid changes.

H. J. Corper

The vital capacity and alveolar carbon dioxide tension in pulmonary tuberculosis. Experimental studies. Antonino Rusi. *Z. Tuberk.* 72, 161-75 (1935).—The vital capacity and alveolar CO<sub>2</sub> tension were studied in 50 cases with pulmonary tuberculosis, part of them with pneumothorax and part taking the regular sanatorium cure, and some treated medicinally. The well-known facts were verified that in pulmonary tuberculosis the vital capacity was diminished, the CO<sub>2</sub> tension reduced and arbitrary apnea diminished.

H. J. Corper

Sulfur metabolism in cystinuria. James C. Andrews and Alexander Randall. *J. Clin. Investigation* 14, 517-24 (1935).—The metabolism of various compounds in the cystinuric organism has been studied with the following results. The cystine output is unchanged by administration of NaHCO<sub>3</sub> or Na citrate but daily administration of alkali does prevent deposition of cystine calculi. Administration of glycine and glutamic acid in equiv. amts. is without effect on the rate of cystine excretion. Administration of L-cystine by mouth results in practically complete oxidation of the S from the cystine but administration of D-cystine is followed by slightly less efficient oxidation. Cystic acid is neither oxidized by the normal nor by the cystinuric organism. After administration of D-methionine, there was observed: (1) no significant increase in cystine excretion, (2) no excretion of homocystine and (3) definite but very slight excretion of methionine. The increase reported by Brand and co-workers in the apparent cystine content of these urines is confirmed. In C. A. 29, 4823 instead of "Cystine is excreted unoxidized by cystinuric and by normal subjects. The cystine content of cystinuric urines, etc." read "Cystic acid is excreted unoxidized by cystinuric and by normal subjects. The cystine content of cystinuric urines, etc." E. J. C.

Porphyria in clinical porphyria. W. Hoerburger and H. Fink. *Z. physiol. Chem.* 236, 139-49 (1935).—The porphyria isolated from the urine of patients suffering from porphyria due to Pb poisoning and to arsenamine poisoning was identified by its fluorescence curve as coproporphyrin III, whereas that from a clinical case of acute gastric pain, flatulence and diarrhea was found to be coproporphyrin I.

A. W. Dox

The immuno-chemical system sheep blood-antiserum blood serum. Edvard Brønnum. *Arkiv Kemi, Mineral. Geol.* 12B, No. 10, 3 pp (1935); cf. C. A. 28, 6475.

1 The highly purified Forssman antigen, unobtainable in clear water soln., is completely dissolved after the addition of bile acids. It is resistant to proteolytic enzymes (papain, trypsin), HNO<sub>3</sub> and Ph isocyanate. Diazomethane reacts with the antigen in an ethereal medium, the antigen losing much of its immunological capacity, possibly because of the carbonyl groups in the antigen mol. After the influence of proteolytic enzymes (trypsin, papain) the Forssman antibody loses its immunological activity. The antibody is pptd. at pH 5.5, pptn. occurring only in the absence of electrolytes. The purified antibody is completely stable at 37° within the pH range 5-10. Complete inactivation of the antibody is obtained by HNO<sub>3</sub>, indicating the presence of free amino groups in the antibody mol. Quant. loss of activity is also caused by Ph isocyanate.

Edward Eagle

Serum gelification reactions in human leprosy. G. Fabiani. *Compt. rend. soc. biol.* 120, 13-14 (1935).—In leprosy, tuberculosis, syphilis and kala-azar the addition of 2 drops 40% HCHO or 0.1 cc. lactic acid to 1 cc. serum usually causes gelification. In leprosy the gelification time ranges from a few min. to several hrs.

L. E. G

Variations observed in Weltmann's reaction for cancer. Diminution of the band of coagulation. G. Carrière, P. Martin and J. Driessens. *Compt. rend. soc. biol.* 120, 31-2 (1935); cf. C. A. 29, 3333.

L. E. Gilson

Value of the formal gelification and formal-substance reactions in the diagnosis of canine leishmaniasis. P. Cartana. *Compt. rend. soc. biol.* 120, 63-5 (1935).—Neither reaction is specific.

L. E. Gilson

Blood amino acids in surgery. Adrián J. Bengolea, Carlos Velasco Suarez and Remo S. Ferracani. *Rev. med.-quir. patol. Jemerina* 6, 245-60 (1935).—Lesions of the liver are accompanied by an increase in aminoacidemia. Major surgical operations, regardless of the anesthetic used, usually cause an increase in aminoacidemia for a few days and the increase is greater if there are post-operative complications. Tissue destruction is probably responsible for the increase. A summary of 45 case reports is given. Twenty-five references. The amino acid contents of the blood cells and plasma and their relation to surgical operations. Remo S. Ferracani. *Ibid.* 261-5. In 9 of 25 diversified surgical cases the operation was followed by a decrease in the cell amino acids/plasma amino acids ratio; in the other 16 cases the ratio increased. Presumably the permeability of the cells to amino acids was changed but the mechanism is uncertain. The nature of the ailment and the anesthetic used do not seem to be factors.

L. E. Gilson

The treatment of milk allergy and its basic principles. Bret Ratner. *J. Am. Med. Assoc.* 105, 934-8 (1935).—The proteins of the whey fractions, lactalbumin and lactoglobulin, are usually responsible for allergy. Heating or evap. milk causes coagulation of these proteins and greatly reduces the ability of the milk to produce sensitivity or induce allergy. In cases where allergy is reported as due to casein, error is commonly caused because of the use of impure casein preps in testing for sensitivity. Twenty-five references.

F. P. Griffiths

The so-called mosaic fungus as an intercellular deposit of cholesterol crystals. A. M. Davidson and P. H. Gregory. *J. Am. Med. Assoc.* 105, 1262-4 (1935).—The mosaic sometimes associated with skin infections and often confused with true hyphae is shown to consist of aggregations of cholesterol crystals. The relationship of the deposits to ringworm infection is not known.

F. P. Griffiths

Levulosemia in hepatic disturbances. P. De Lucia and E. Claar. *Minerva med.* 1935, II, 345-59. A transitory levulosemia of 70-140 mg/100 was produced 30-60 min. after the ingestion of 40 g. levulose in 300 cc. H<sub>2</sub>O by normal persons or patients with other than hepatic diseases. Sixty-three % of the persons with altered hepatic function showed a levulosemia greater than 140 mg. which persisted 4-5 hrs.

Helen Lee Gruell

Method of increasing the sensitiveness of animal tumors to x-rays. S. Russ and G. M. Scott. *Proc. Roy.*

*See* (London) B115, 316-20 (1935) —Expts. on over 200 tumors on rats showed that a marked increase in sensitivity to the effects of x-rays without apparent toxic effect was produced by either injection of thorotrast into the tumor or injection of well-established growing tumors of rat sarcoma vitally stained with trypan blue into either the tumor or another part of the body. Vital new red and assume blue did not have this action on the tumors.

Joseph S. Hephner  
Local factors influencing dental canes: A study of organic matter associated with enamel. Paul Pines. *Bull. Dent. J.* 59, 572-91 (1935) —Examin after staining of grooves in occlusal surfaces of unerupted teeth did not prove conclusively the existence of a surface layer. Biochem. tests on the org. material removed from the enamel indicated its keratin nature in some respects. Attempts at decapsulation of the keratinous material were unsuccessful. *Actinomyces* was not found in materials from carious lesions or in saliva from mouths known to contain carious teeth. Decalcification of enamel was shown to proceed at different rates depending upon whether the destruction was proceeding across or along the enamel rods. The effect on tooth-sections, synthetic saliva and lactic acid must was negligible. An acid-resistant matrix has been demonstrated in enamel.

Frances Krasnow  
A microchemical study of human biliary calculi. Thomas W. Ray. *J. Biol. Chem.* 111, 689-97 (1935) —"Analysis of a large no. of human biliary calculi shows there is no essential quantitative difference between the types referred to as cholesterol-pigment-Ca stones and cholesterol-pigment stones. Hence there is no quantitative basis for the present classification of these 2 varieties. Some of the former stones contained even more cholesterol and less mineral matter than some of the latter. Furthermore some stones of supposedly copropoaninities proved to be remarkably alike. There is a surprising regularity in the way the constituents are distributed in some stones but as a rule these substances are laid down without order. Stones from the same gall bladder were found to be very similar from a chemical standpoint." *Mg*, *Ca* and *Pb* were found in about half of the stones; *Fe* was always found, frequently in both the ferrous and ferric form, and *Ca* and *Pb* are constant constituents. *Barium* appears in both types.

A. P. Lethbrun  
The metabolism of inorganic salts and water in hepatic disturbances. III. 1. The metabolism of inorganic salts. (3) Perfusion experiment of the extirpated liver. Lavin Stenger. *Jap. J. Gastroenterol.* 7, 174-177 (1935); cf. C. A. 29, 6848<sup>3</sup> —Rabbit livers were infused with  $\text{CCl}_4$  and then perfused with  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{KCl}$  and  $\text{NaCl}$  salts. The amt. of cations fixed by the tissue was found to be subnormal for each salt. IV 2. The metabolism of water. *Ibid.* 111-14 (1935) —Very limited data indicate that ligation of the common bile duct in rabbits is injury to the liver with  $\text{CCl}_4$  increases the  $\text{H}_2\text{O}$  of the liver, kidneys, intestines and brain.

C. M. McCave  
Sero-hematological investigations on malignant neoplasms. XII. New research on the first reaction. G. D'Alessandro and R. Indovina. *Biochim. Lymph. Soc.* 2, 298-306 (1935); cf. *Fabrics*, C. A. 29, 5914<sup>3</sup> —The *thermo-lability* test can be made more evident by previous lysis of the serum. The lability can be subnormal by bile, but this does not effect an increased stability. Neonatal sera have a higher content of unsat. valences. The ability of the serum-bilirubin complex in serum of neoplasms of the bile tract is attributed to a change in the lipid metabolism.

A. E. Mavor  
Calcium, phosphorus and alkali reserve in the rachitic osteopathy caused by starvation. Giuseppe Nazzari. *Biochim. Lymph. Soc.* 2, 385-9 (1935) —Rabbits receiving 5% lactate by mouth develop a bone disease resembling rickets. The *Ca* in the blood increases, the *P* decreases and the alkali reserve is diminished.

A. E. Mavor  
Hyperlipemia and cholesterolemia. Jans Sing and Chris Stamp. *Acta Med. Scand.* 85, 12-21 (1935) —It is shown that the hyperlipemia in nephrotic subjects bears no relation either to the hypoproteinaemia or to the anemic condition. Crystallinuria, or the urea and creatinine

clearance, and cholesterolemia proceed in a parallel manner, but the urinary cholesterol does not depend upon the blood cholesterol content, or upon diuresis. There is a relationship between the cholesterol and protein excretion, but this is not marked by any degree of constancy.

S. Margolis  
Lactic acid formation in unexcitinated and irradiated tumor tissues. Walter Hartmann. *Biochim. Z.* 280, 175-83 (1935) —The lactic acid formation of tumor tissue depends upon the content of the carbohydrate (blood sugar, glycogen) and upon the disappearance of lactic acid and even under anaerobic conditions. Tumor tissue is more sensitive to the effect of radiation than is normal tissue, the inhibition of lactic acid-forming ability not being a measure of the cell necrosis because the formation of lactic acid from some sources may remain unchanged.

S. Margolis  
Breath in the organism of the mouse. Th. Lorenz and O. Wachtel. *Biochim. Z.* 280, 614-41 (1935) —The *Br* in the organism of the mouse, like that of the normal, is entirely in the form of  $\text{Br}_2$  ions. The wide range of variation in the blood *Br* values points to an alimentary origin, while the generally low values are obviously associated with the low *Cl* concentration due to deficient diet. In the mouse as well as in the normal individuals the blood *Br* content decreases and the *Br* content of the gastric juice increases after a meal. This excludes the necessity to postulate the presence of an endocrine regulatory mechanism in the hypophysis for *Br*. Incidentally the *Br* content of the hypophysis from mouse persons shows as large a degree of variation as that from normal individuals.

S. Margolis  
The effect of parathyroid hormone and of tuberculous on the serum and tissue calcium of guinea pigs. Geoffrey C. Linder. *Biochim. Z.* 280, 215-21 (1935) —The effect of parathyroid hormone on the *Ca* of the serum in the presence of infection and on the *Ca* distribution of the bone, brain and liver of normal and tuberculous guinea pigs was studied. The injection of large doses of *I* caused no significant increase in the serum *Ca* of normal animals, but gave a noticeable increase when tuberculous animals were used. No difference in the degree of bone decalcification was observed in either case. *I* caused no *Ca* increase in the brain of normals, but some increase in those of tuberculous animals. Liver *Ca* was increased by injections of *I* and by tuberculous infection and in the latter case it was further increased by *I*. E. W. Scott

The protein fractions of serum as different anions. I. Purvis. *Folia Biol.* 1935, 142; *Abstr. exp. chim. Argent.* 23, 68 (1935) —Two globulins of different antigenic nature, as shown by ampholytic shock, were obtained. To obtain each buffer, *pr.* with 13.5%  $\text{Na}_2\text{SO}_4$  at 57°, wash with 14%  $\text{Na}_2\text{SO}_4$ , except 4 times at 0° and electrolysis to  $\lambda = 1.4$ . For globulin *pr.* with 21.5%  $\text{Na}_2\text{SO}_4$  at 57°, wash the *pr.* with 21%  $\text{Na}_2\text{SO}_4$ , *pr.* 4 times at the same time washing the cream as before, once with 13.5%  $\text{Na}_2\text{SO}_4$ , dissolve the final *pr.* in distilled  $\text{H}_2\text{O}$ , dialyze and electrolysis to  $\lambda = 1.4$ . The albumin *pr.* obtained by the method of Paul and Semmow (C. A. 23, 247) and that of Erbe (C. A. 15, 17), supplemented by electrolysis to  $\lambda = 1.4$ , are *sp.* analogous to  $\alpha$ - and pseudoglobulin.

E. M. S.  
Blood regeneration in severe anemia. Fractions of kidney, spleen and heart compared with standard liver fractions. F. S. Robscheit-Robbins, G. S. Waller and G. H. Wrigley. *Am. J. Physiol.* 113, 47-70 (1935); cf. C. A. 24, 5345<sup>3</sup> —Expt. evidence indicates that there are several factors, rather than a single factor, responsible for the potent influence of these fractions upon the regeneration of red cells and hemoglobin in eryth. anemia due to blood withdrawal in dogs.

E. D. Waters  
Quantitative theory of the precipitation reaction. II. The reaction between crystalline egg albumin and its homologous antibodies. Michael Heidelberger and Forster E. Kendall. *J. Exp. Med.* 62, 477-72 (1935); cf. C. A. 29, 5129<sup>3</sup> —A quantitative theory of the precipitation reaction based on the laws of classical chemistry has now been found applicable to the crystalline egg albumin-antibody system. Equations derived from the theory permit the calculation of



the behavior of an anti-egg albumin serum over most of the reaction range after a few quant. analyses have been made for the N pptd. The empirical relation, shown to have advantages in the dye-antidye system, may also be used for the Ea-A reaction. Serum from the same animal after successive courses exhibits progressive changes which have been described graphically and quantitatively. These changes are believed to consist in the formation of more and more antibody capable of reacting with a large no. of chemically different groupings in the antigen mol. Evidence is presented that anti-egg albumin is not homogeneous, and that even after prolonged immunization the antiserum contains much low-grade antibody, incapable of forming ppts. unless more reactive precipitin is present. Factors affecting the equivalence point ratio are discussed.

C. J. West

Further observations on the blood cholesterol of rabbits in relation to atherosclerosis. Kenneth B. Turner and Emily H. Bidwell. *J. Exp. Med.* 62, 721-32 (1935); cf. C. A. 27, 4310.—The action of KI in preventing a significant rise in the blood cholesterol (C) of rabbits fed C was temporary. After about 4 months it lost its effectiveness and the blood C rose. In rabbits with hypercholesterolemia resulting from long-continued C feeding, the administration of KI caused a marked rise in the blood C. On the other hand, dried whole thyroid given to such animals produced a sharp fall in the blood C. This fall was temporary and was followed by a rise to new high levels. In thyroidectomized rabbits fed C and KI, both thyroid and thyroxine delayed but did not prevent a rise in blood C. Even with the hypercholesterolemia in these animals, however, the incidence of atherosclerosis was low. Age apparently played a part in detg. the response of the blood C to C feeding. In a group of old rabbits when compared with a younger group the rise in the C of the blood was greater and the subsequent return toward normal was slower when the feeding was stopped.

C. J. West

Physiological reversal of effects of adrenaline hypertension. Raymond-Hamet. *Compt. rend.* 201, 570-2 (1935) Julius White

## H—PHARMACOLOGY

A. N. RICHARDS

Comparative chemotherapeutic studies of arsenoxide (3-amino-4-hydroxyphenylarsenoxide) and neosphenamine. Geo. W. Raiziss and Marie Severac. *Am. J. Syphilis and Neurol.* 19, 473-80 (1935).—The max. tolerated dose for arsenoxide (I) intravenously to rats is 0.018 g. and for neosphenamine (II) is 0.400 g. per kg. body wt. The min. trypanocidal dose of I is 0.0025 g. and of II is 0.020 g. per kg. body wt. Hence, the therapeutic indexes are 7.2 and 20, resp. The max. tolerated dose of I and II in rabbits is 0.011 g. and 0.200 g. per kg. body wt., resp. The min. curative dose of I and II in exptl. rabbit syphilis is 0.012 g. and 0.040 g., resp. The therapeutic indexes are therefore 0.92 and 5. Philip D. Adams

Differential biological reaction of cobaltous compounds and certain cobaltic complexes (cobalthalmines). Jean-Marie Le Goff. *Compt. rend.* 201, 531-2 (1935).—Injection of a cobaltic complex or cobalthamine produces different effects than those previously reported for CoCl<sub>2</sub> (C. A. 22, 1403, 3459). The organism possesses not only the power of differentiating cobaltous compds. and cobaltic complexes but is able to distinguish N and Co in cobalthalmines. Philip D. Adams

The influence of thyroid in the healing of wounds. Howard L. Puckett. *U. S. Naval Med. Bull.* 33, 510-16 (1935).—Desiccated thyroid (I) *per os* seems to increase the rate of healing of superficial wounds in young rats. Daily administration for 3.5 months produced no harmful effects. When I is used over a long period of time its effects on healing become less pronounced, and repeated wounds on the same animal apparently decrease the rate of cicatrization. Philip D. Adams

Physiological activity of some pyrocatechol derivatives. Michael G. Mulinos and Raymond L. Osborne. *Proc. Soc. Exptl. Biol. Med.* 32, 1344-6 (1935).—Pyrocatechol, ethylpyrocatechol and especially chloroacetylpyrocatechol

possess sympathomimetic powers in which the inhibitory effects predominate. In agreement with Dakin the catechol nucleus is, in great part, responsible for the typical sympathomimetic activity of adrenaline. The 3,4-position of the aryl hydroxyls, a 2-C side chain, a hydroxylated  $\beta$ -C, and substituent of some indifferent mol., preferably an amine, are contributory factors in the formation of a typically sympathomimetic drug. C. V. Bailey

Physiology of pyrimidines. VIII. Metabolism of isobarbituric acid in the rabbit. Wm. J. Conway and Leopold R. Cerecedo. *Proc. Soc. Exptl. Biol. Med.* 32, 1600-1 (1935); cf. C. A. 27, 3720, 3752.—In the rabbit as in the adult dog and in man pyrimidines are partly broken down into urea and partly conjugated with H<sub>2</sub>SO<sub>4</sub>. C. V. Bailey

Effects of phenacetin and aspirin respectively upon action of phenobarbital. Alfred Gilman and Henry G. Barbour. *Proc. Soc. Exptl. Biol. Med.* 32, 1634-6 (1935).—In the rat the administration of 50 mg./kg. of Mg phenobarbital produced symptoms of excitement and then depression for several hrs., the addn. of an equal dose of acetylsalicylic acid nullified these effects, the addn. of 5 times the dose of phenacetin in no way diminished the activity of phenobarbital, it diminished the toxic effects when the latter was given in large doses. C. V. B.

Lecithin in experimental arteriosclerosis. Preliminary study. Wm. G. Downs, Jr. *Am. Med.* 41, 460 (1935).—In expts with rabbits the addn. of relatively small amts. of lecithin to the diet prevented the occurrence of exptl. arteriosclerosis of high cholesterol diet origin. R. B.

The biological effects of beryllium. Russell N. Loomis and Emil Bogen. *Am. Rev. Tuberculosis* 32, 475-80 (1935).—Although among many salts tried a definitely favorable influence on the course of exptl. tuberculous guinea pigs, Be in the form of basic tartrate or the chloride in doses of 1 and 5 mg. (or 10 mg. of the basic salt) accelerated the development of tuberculosis in the treated guinea pigs. The same effect was noted whether the animals were infected with large or small doses of virulent human tubercle bacilli and whether infected subcutaneously or by inhalation. Oral administration of the Be did not appear to be effective. Repeated injections and larger doses gave the more const. results. Admixt. in media with bacilli did not affect the growth of the bacilli. The basic chloride, the nitrate, the tartrate, the phosphate and the lactate all gave similar results. The citrate and a suspension of the insol. carbonate gave no perceptible effect. The Be rickets reported in rats (Guyatt, Kay and Branton, C. A. 27, 5785) were not observed in these guinea pigs. H. J. Corper

The effects of the inhalation of hydrogen fluoride. II. The response following exposure to low concentration. Willard Machle and Karl Kitzmiller. *J. Ind. Hyg.* 17, 223-9 (1935); cf. C. A. 28, 2788.—The lower limit of toxic concns. of HF in air lies between 0.03 and 0.0025 mg. per l. Lungs and liver are most severely and consistently affected. III. Fluorine storage following exposure to sub-lethal concentrations. Willard Machle and E. W. Scott. *Ibid.* 230-40.—Distribution of fluorides in ashed tissue was detd. by treating with perchloric acid, and titrating the F with 0.025 N cerous nitrate or 0.005 N Th nitrate. In animals exposed to fluorides, F was found in high concns. in the bones and in instances of prolonged exposure in the teeth. A. L. Elder

Pharmacological properties of ergobasine, a new alkaloid from ergot. E. Rothlin. *Compt. rend. soc. Biol.* 119, 1302-4 (1935).—Ergobasine, C<sub>20</sub>H<sub>25</sub>O<sub>5</sub>N<sub>3</sub>, [α]<sub>D</sub><sup>20</sup> = +90°, acts on the uterus like ergotamine. It is hypertensive but not sympatholytic. It accelerates respiration. It has little or no effect on glucemia and does not produce gangrene at the site of injection. The lethal doses are: for white mice, intravenously, 0.145; rat subcutaneously, 0.5; rabbit, intravenously, 7.5; cock, intramuscularly, more than 10 mg./kg. These doses are about 4 times those of ergotamine. Cf. Stoll and Burkhardt, C. A. 29, 5219, where the formula for ergobasine, by mistake, is given C<sub>20</sub>H<sub>25</sub>O<sub>5</sub>N. L. E. Gilson

Pharmacological reactions of the melanocytes of detached scales of *Carassius vulgaris*. J Verne and V Viter. *Compt. rend. soc. biol.* 119, 1312-13 (1935).—Scales from live carp were placed in hanging drops of serum of various vertebrates. Posterior hypophysis ext (II) caused expansion of the melanocytes, epinephrine (II) and adrenaline (III) caused contraction. Upon standing [several days] sensitivity to II disappeared before sensitivity to III. Nicotine suppressed the sensitivity to III and ergotamine inverted the response to III. I acts directly on the melanocytes and they react even if detached from the scale. II and III act through the nerve fibers and their effect is greatly decreased if the scales are cut into small pieces. They have no effect on isolated melanocytes. L E Gilson

Treatment of arterial hypertension by intravenous injection of octyl alcohol. L Pezzangora. *Compt. rend. soc. biol.* 119, 1331-32 (1935).—The patients were given 15 injections, on alternate days, of 10-15 cc of a 1:100,000 soln. of octyl alc in distd water. A decrease in blood pressure was produced in essential hypertension cases but not in cases of stable hypertension with cardio-renal complications. Diuresis was increased. L E Gilson

Opposed action of the hydrogen and hydroxyl ions on pharmacodynamic reactions of the autonomic system. Action on uterine tonus. M Tiffeneau and D Brown. *Compt. rend. soc. biol.* 119, 1380-2 (1935).—At pH 8.4 the tonic action of adrenaline, histamine or posterior hypophysis ext. on the isolated uterus was considerably greater than at pH 7.4. At pH 8.8 the action was greatly weakened or even inverted. L E Gilson

Effects of hydrogen and hydroxyl ions on the anesthetic action of propyl bromide on fish. M Tiffeneau and D Brown. *Compt. rend. soc. biol.* 119, 1382-4 (1935).—Immersion for 5 min in a 1/750 soln. of lactic acid decreased the time required to produce anesthesia when the fish were transferred to a 1/1500 soln. of PrBr, while immersion for 5 min. in a 1/3000 soln. of Na<sub>2</sub>CO<sub>3</sub> prolonged the time required. L E Gilson

Action of pericloside. T. Solacolu, G. Baltaceanu, V. Vasiliu and G. Hertmann. *Compt. rend. soc. biol.* 120, 3-52 (1935); cf. C. A. 29, 2198<sup>1</sup>.—In dogs weighing 15-16 kg. the intravenous injection of 2-7 mg. pericloside caused a general and recurrent increase in arterial pressure, suppression of urinary secretion, dilation of the kidneys, decrease in spleen vol. and slowing of respiration. L E Gilson

Chlorine of the livers of hyperthyroidized animals. I. Parhon and M. Cahane. *Compt. rend. soc. biol.* 120, 52-3 (1935).—In 6 out of 8 animals given repeated injections of thyroid ext. the Cl content of the liver was lightly increased. L E Gilson

Blocking of the reticuloendothelial system and adrenal-chloroform syncope. Constantin C. Velluda and Ion Gabriel Russu. *Compt. rend. soc. biol.* 120, 57-8 (1935).—Blocking with curarine or Chinese ink prevented the syncope (in dogs) and decreased the hypertensive action of the adrenaline. L E Gilson

Action of sodium bicarbonate on the production of hyperglycemia by pilocarpine and by ligation of the portal vein. Victor Papilian and Victor Preda. *Compt. rend. soc. biol.* 120, 58-60 (1935); cf. C. A. 29, 3725<sup>1</sup>.—Intravenous injections of NaHCO<sub>3</sub> inhibit the production of hyperglycemia by adrenaline but do not affect the hyperglycemic action of pilocarpine; hence the mechanism of the 2 actions must be different. NaHCO<sub>3</sub> prevents the increase in glucose which usually follows ligation of the portal vein. L E Gilson

Histopicrographic study of the fixation of manganese during experimental chronic intoxication by manganese boride. A. Polkard. *Rev. sci. argentina biol.* 11, 155-7 (1935); *Compt. rend. soc. biol.* 120, 564-6 (1935).—Rats were given MnO<sub>2</sub> in their food over long periods. There was some accumulation of Mn in the testes, brain, lungs, liver and renal cortex but not in the renal medulla. After the feeding of MnO<sub>2</sub> was discontinued Mn disappeared from the liver and kidneys first. L E Gilson

Pharmacological difference between the tinctures of

*Strophanthus hispidus* and *S. kombé*. Nicoletta Sabatucci. *Scienza farm.* [2], 3, 49-63 (1935).—Tests made by the isolated tooth heart method of Galatá (*Boll. sci. biol. sper. I*, No 4 (1926)), the pigeon emesis method of Ilanzlik (C. A. 24, 1428) and the frog method of Focke showed the tincture *S. kombé* to be much more active in each case. L E Gilson

Parathyroid extract and viosterol treatment of radium poisoning. L P Craver and H Schlundt. *J. Am. Med. Assoc.* 105, 959-60 (1935).—Alternate periods of administration of parathyroid ext with low-Ca diets or viosterol with high-Ca diets did not cause marked increases in the amts of Ra eliminated by 3 patients suffering from Ra poisoning. F P Griffiths

The relationship of drug therapy to agranulocytosis. Roy R. Kracke and Francis P. Parker. *J. Am. Med. Assoc.* 105, 960-6 (1935).—Amidopyrine, dinitrophenol and closely related drugs cause agranulocytosis. Indiscriminate use of amidopyrine or proprietary drugs which contain it should be avoided. A list of preps containing amidopyrine is given. Seventy-five references. F P Griffiths

How drugs act. H Raymond Ing. *Science Progress* 30, 262-7 (1935).—A review (26 references), with considerable attention to the relationship of drug action to the humoral transmission of nerve impulses. J. S. H

Sedatives in dentistry. H T Roper-Hall. *Brit. Dental J.* 59, 177-86 (1935). I. Rances Krasnow

The action of prostigmine on the cardio-vascular apparatus. Leonardo Donatelli. *Arch. sci. biol.* (Italy) 21, 210-12 (1935).—This substance acts peripherally on the intracardiac vagal endings and centrally on the bulbar pressor centers. Atropine counteracts the functional disturbances. P F Mettli

The treatment of Addison's disease with common salt. G. Miralton, J. A. Collazo and J. Jimena. *Presse med.* 43, 505-7 (1935).—Most cases of Addison's disease improve upon medication with NaCl and the use of cortical hormone can be restricted. Immediate effects are alleviation of the Na-Cl disequil., the dehydration and the acidosis. A. E. Meyer

Vagotonia in hypertension. P. Bernal. *Presse med.* 43, 507-10 (1935).—The action is gradual and without danger. A. E. Meyer

Pharmacognosy of *Troxis divaricata* Spreng. var. *Discolor* Griseb. Luis Florian. *Rev. farm.* (Buenos Aires) 77, 223-6 (1935).—The botanical description is given. The drug contains resins, xanones and an alkaloid. It causes sensory and motor paralysis in animals. It is used in chronic arthritis as a diaphoretic and diuretic. A. E. Meyer

Poisonous gases. Carlos L. Carbonacci. *Semana med.* (Buenos Aires) 1935, II, 998-1018.—A toxicological review. A. E. Meyer

The sugar-excretion threshold of rabbits treated with tonsillar extract. Chikara Tateishi. *J. Biochem.* (Japan) 22, 251-61 (1935).—It is stated that tonsillar ext stimulates the sugar assimilation since the sugar-excretion threshold of rabbits is lowered by a small dose and raised by a large dose of this ext. Administration of cholic acid still further promotes the lowering effect of the ext. S. Morgulis

Arrow poisons. XII. C. G. Santesson. *Skand. Arch. Physiol.* 72, 92-102 (1935); cf. C. A. 29, 1510<sup>1</sup>.—Arrow poisons from the Belgian Congo region apparently contain the alkaloid erythropleme. On the frog, *R. temporaria*, it produces symptoms of nausea, weakness and typical cardiac effects. The m. l. d. for the frog is 21.6 mg of the crude poison per kg. The arrow poison from Borneo, which is the juice from bark of *Anthus lincaria*, produces typical cardiac effects and the m. l. d. is 10 mg. (of different samples) ranged from 3 to 10 mg. per kg. The upas poison, also from Borneo, was investigated and it is thought to contain the *Anthus* poison, and a dose of 0.036 mg. of this kills a 31-g frog in 1 1/4 hrs. The m. l. d. of this poison (Punan poison) is about 2.81 mg. per kg., corresponding to 4% antiarr. S. Morgulis

Effect of cyanic acid on the respiratory metabolism of the organism. Knud O. Møller. *Skand. Arch. Physiol.* 72, 103-14 (1935).—Rabbits under urethan anesthesia

receiving subcutaneous injections of NaCN show a quick, but transient rise in the  $O_2$  consumption. In rabbits receiving continuous injections of NaCN this initial rise occurs only occasionally, while in those inhaling the gas there is no increase at all. From this it is concluded that a sudden increase in HCN concn is the condition responsible for the initial rise in  $O_2$  consumption. S Morgulis

The effect of methylene blue and of the combination hydrocyanic acid-methylene blue on the respiratory metabolism of the rabbit. Knud O Møller. *Skand Arch Physiol* 72, 115-25(1935).—Intravenous injection of methylene blue in amts of 3-30 mg. per kg. causes a strong increase in the  $O_2$  consumption of the rabbit which varies with the injection dose. The larger doses also cause a rise in the body temp. Intravenous injection of 40-50 mg per kg causes a relatively smaller increase in the  $O_2$  consumption than the smaller doses, but a greater rise in the body temp., and lead more frequently to death. The intravenous injection of 20 mg. per kg. either 45 min. before or 15 min. after an injection of 7 mg. NaCN per kg., which is strongly lethal, entirely removes the inhibition of oxidation and the rabbits remain alive. Injected 20-25 min. after the NaCN administration methylene blue restores the  $O_2$  consumption to the normal value, but does not save the animals from death. The methylene blue begins to exert its effect invariably within less than a min. after the injection. S Morgulis

Dehydrogenation processes in the tissues of animals treated with thyroxine. M. Reiss, L. Schwarz and F. Fleischmann. *Endokrinologie* 16, 145-8(1935).—Expts by the methylene blue procedure show that the organs (kidney, liver and muscle) from animals treated with thyroxine have a stronger anaerobic dehydrogenation activity than those from untreated animals. A similar increase was noted in the liver from fasting or fatigued animals, and this increased activity of the liver tissue is attributed to an accumulation of donor substances resulting from glycogen mobilization. S Morgulis

Cardiac output in man after the administration of  $\alpha$ -dinitrophenol. E. Nyman and A. Palmlov. *Skand Arch Physiol* 72, 166-74(1935).—Single or repeated doses of 4 mg per kg in adult human subjects caused a pronounced increase in the  $O_2$  consumption without affecting the minute vol of the heart. The increased  $O_2$  consumption must, therefore, be attributed to the opening up of capillaries. The cardiac output per beat actually decreases indicating a weakening of the heart action. S Morgulis

The injection treatment of varicose veins. F. E. Greenbaum. *Clin Med Surg* 42, 480-3(1935).—A combined soln of quinine and Na morrhuate in which the alkali-insol alkaloid is peptized by the soap is reported to be of value in the obliterative injection treatment of varicose veins. T H Rider

The trypanocidal action of styryl selenazole compounds. C H Browning, R. Gulbransen and V. McCartney. *J. Pharmacol* 54, 36-70(1935).—Mol proportions of 1-methylbenzoseleazole methiodide (I) and  $p$ -acetylaminobenzaldehyde were boiled in abs alc 2 hrs. A deep violet color and a violet-brown ppt developed. The ppt, 1-( $p$ -acetylaminostyryl)benzoseleazole methiodide (II), m 285°. Similarly I condenses with  $p$ -dimethylaminobenzaldehyde to give a greenish black product 1-( $p$ -dimethylaminostyryl)benzoseleazole methiodide (III), m 250° (decolor). Both products were sulfonated with chlorosulfonic acid in HOAc, the sulfates being yellow and cryst., recryst. from HOAc, easily sol in water and not pptd by alkali. No analyses or controls are given for the sulfonates, but they were halogen free. II had a temporary therapeutic effect at the rate of 0.001 g. for an uninfected mouse weighing 20 g. In the same dose III showed no effect in max tolerated dose while the sulfonate of II produced only variable results. T H Rider

Alcohol injected intravenously. Rate of disappearance from the blood stream in man. Henry W. Newman and Windsor C Cutting. *J. Pharmacol*, 54, 371-7(1935).—Max alc concn after intravenous injection is linear and

the amt. required to maintain blood alc at a const level is the same regardless of the level. Conclusion: The metabolism of alc in man proceeds at a const rate, regardless of blood concn. T. H. Rider

The evaluation of gonadotropic hormone preparations on the basis of the rat mouse ratio assay. Warren O Nelson and Milton D Overholser. *J Pharmacol* 54, 378-92(1935).—The relative activities as detd on mice and rats for gonad stimulating preps including exts of pituitary glands, pregnancy urine and pregnant mare serum varied considerably between a ratio of 5:1 and 1:3 for mouse and rat, resp. Conclusion: Variations are due to varying proportions of follicle-stimulating and luteinizing factors. The rat-mouse assay ratio offers a method of differentiating between anterior pituitary and anterior pituitary-like gonadotropic hormone complexes. T H Rider

The pharmacologic action of ergotocin, a new ergot principle. M. Edward Davis, Fred L. Adair, K. K. Chen, and Edward E. Swanson. *J Pharmacol* 54, 398-407(1935).—Ergotocin may be assayed by the U S P cock's comb method, by isolated uterine response (but not the Broom-Clark method). It has little inhibitory action on adrenaline. Ergotocin maleate has a m l d of 250 mg per kg in mice, and 80 mg per kg in guinea pigs by intravenous injection. Other pharmacol actions are described. T H Rider

Cumulative poisoning by lanadign, ouabain and digitoxin in dogs. R. C. Li and H. B. Van Dyke. *J. Pharmacol*, 54, 415-25(1935).—Relative potencies were ouabain 10, lanadign and digitoxin 1 in the frog and 4:1:1, resp., in the dog. Cumulative activity was greatest for lanadign, least for digitoxin, the effect of repeated doses of digitoxin in dogs being clearly different in dogs and cats. T. H. Rider

Ketogenesis-antiketogenesis. I. The influence of ammonium chloride on ketone body formation in liver. Norman L. Edson. *Biochem. J* 29, 2082-94(1935).—The effect of  $NH_4Cl$  on ketogenesis in the liver was studied with the tissue slice technic. Micromethods for the detn of  $\beta$  ketonic acids, both manometrically and volumetrically, and  $\beta$ -hydroxybutyric acid were described. Anan's (C A 28, 4078) statement that  $NH_4Cl$  increased the production of acetoacetic acid (I) from pyruvic acid in the liver was confirmed. In the absence of substrate  $NH_4Cl$  increased the formation of I in the well-nourished liver but not in the starved liver where I was already being formed rapidly. The fatty acids, regardless of the no. of C atoms, caused increased production of I and this was further accelerated by  $NH_4Cl$ . Fatty acids contg an even no. of C atoms formed 3 times as much I as those having an odd no. The effect of  $NH_4Cl$  was inhibited by glycerol. E. W. Scott

A pharmacodynamic study of berberine sulfate. Mario Soto and N. Pedro Sivori. *Rev. asoc med Arg* 47, 109-14(1933); *Anal. asoc quim Argentina* 23, 11B(1935).—An exptl study on frogs and guinea pigs. The nervous system and the respiration are depressed. The action of berberine sulfate on the circulatory organs and respiration. *Ibid.* 11A-21.—A exptl study on dogs, with cardio- and pneumograms. E. M. Symmes

Poisoning by magnesium hyposulfite. Leonidas L. Silva. *Rev. asoc. med Arg* 47, 200-6(1933); *Anal. asoc quim Argentina* 23, 12B(1935).—The min. intravenous lethal dose of Mg hyposulfite in rats is 0.25 g/kg. in dogs 0.30 g/kg. There is no narcosis. F. M. S.

Poisoning by carbon monoxide and psychosis. Nereo Rojas and Jose Belbey. *Rev. asoc. med Arg* 47, 386-92(1933); *Anal. asoc quim Argentina* 23, 12B(1935).—A study of cases. E. M. Symmes

Poisoning by barbital. Treatment by coramine. Alfredo Buzo and Guido Costa Bertani. *Rev. asoc med Arg* 47, 388-401(1933); *Anal. asoc quim Argentina* 23, 11B(1935).—A description of cases treated, with recovery. E. M. Symmes

Is apiole actually toxic? Frappi. *Rev. med Lat-Am* 1933, 825-9; *Anal. asoc quim. Argentina* 23, 12B(1935).—Apiole causes poisoning and lesions in the liver

and kidney, sometimes fatal. The drug is difficult to obtain pure.

J. M. Symmes  
Acid-base changes in the serum of the dog associated with the hyperthermia of dinitrophenol administration. Edward Muntzler, Victor C. Myers, Wayne H. Danielson and Carla Form. *Am. J. Physiol.* 113, 186-92 (1935); cf. C. A. 26, 5051.—Oral administration of 1,2,4-dinitrophenol in nonfatal doses was followed by a decrease in bicarbonate concn. and an increase in chloride concn. in the serum. The serum total base changes were variable and appeared to depend on the dose given. The serum pH remained within normal limits but tended to be lowered from the control level when larger doses were given. After subcutaneous injection of either a nonfatal or fatal dose of the drug both the serum bicarbonate and chloride decreased. The serum total base concn. tended to decrease after a nonfatal injection, while it tended to increase after the fatal dose injection. In both cases there was a definite increase in the undetected acid concn. The serum pH remained within a narrow range. Despite this fact it is believed that hyperventilation probably played a prominent part in the changes observed in the acid-base balance.

F. D. Walter

A comparison of the effects of sympathin and adrenaline on the iris. W. B. Cannon and A. Rosenblueth. *Am. J. Physiol.* 113, 251-8 (1935); cf. C. A. 27, 4288; 29, 7440.—The results of expts. on the iris of the cat are discussed. Tentative conclusion: Sympathin F, unlike adrenaline, may affect not only the dilator but also the constrictor muscle of the iris. The differences between cardio-pulmonary and hepatic sympathin may be attributed to the presence of sympathin I in the former and its absence from the latter.

E. D. Walter

Differential depression of vasomotor mechanisms by adrenaline. Leland C. Wyman and Caroline Tum Soden. *Am. J. Physiol.* 113, 271-8 (1935); cf. C. A. 26, 5345.

E. D. Walter

The influence of adrenaline on the blood sugar, lactic acid and inorganic phosphorus of completely hypophysectomized dogs. I. L. Chaikoff, F. L. Reichert, L. S. Read and M. E. Mathes. *Am. J. Physiol.* 113, 306-11 (1935); cf. Chaikoff, et al., C. A. 29, 7502.—As judged by the 3 blood constituents (blood sugar, lactic acid and inorg. P), completely hypophysectomized dogs are capable of responding to adrenaline. The responses, however, were diminished as compared to those of normal and control animals. The significance of the abnormal adrenaline reaction of the completely hypophysectomized dog is discussed.

E. D. Walter

The influence of sodium fluoride upon the basal metabolism of the rat under several experimental conditions. Paul H. Phillips, H. E. English and E. B. Hart. *Am. J.*

*Physiol.* 113, 441-9 (1935); cf. C. A. 28, 1079.—F in the form of NaF does not lower the basal metabolic rate of the normal rat. NaF enhances the toxicity of hyperthyroidism induced by feeding desiccated thyroid. Insofar as the hyperthyroidism of toxic goiter and that induced by the administration of desiccated thyroid are identical, to that extent NaF therapy is contraindicated. A ration containing a combination of 0.15% NaF and 0.25% desiccated thyroid rapidly produces an exhaustion of body wt. and a fatal collapse, while either substance alone has no marked effect and is not fatal.

F. D. Walter

Pharmacological studies of "Senso," a Chinese drug from the dried skin secretion of toads. IV. The action of  $\psi$ -bufotalin, the active component in Senso, and  $\psi$ -bufotalin bromide. Yoshito Kobayashi. *Proc. Imp. Acad. (Tokyo)* 11, 295-300 (1935); cf. C. A. 29, 1515.—The min effective dose in rabbits and with perfused guinea-pig hearts is about equal for  $\psi$ -bufotalin bromide (I) and  $\psi$ -strophanthin (II). The m. l. d. of (I) in cats according to the Hatcher method is 240 times greater than the m. l. d. for (II). This unusually favorable ratio for the bromide does not hold for the  $\psi$ -bufotalin. The cumulative effect of (I) is slight. A possible structural formula for the compd is proposed.

R. P. Walton

Biochemistry of amygdalin (Vichoever, Mack) 17. Decomposition of Bengal silk cocoons (Dutt) 25. Emetine camphorsulfonate (Grau) 17. Phenanthridine series. IV. Synthesis of plasmochin-like derivs (Walls) 10. Chemistry of the acetylenes. II. Pharmacol properties of the acetylenic linkage (Bachman) 10. Antimalarials. XIV. Derivs of 8-methylquinoline (Kermack, Wight) 10.

## I—ZOOLOGY

R. A. GORTNER

Action of mammalian anterior hypophysis extract on the secondary sexual characteristics of the male *DiscoGLOSSUS* R. Kehl. *Compt. rend. soc. Biol.* 120, 10-12 (1935).—In normal but not in castrated males the ext. causes the deposition of black pigment in the callosities of the thumbs such as naturally occurs during the breeding season.

L. E. Gilson

Studies on annelid muscle. I. Taurine in *Audouinia spirabranchus*, Moore. Alton C. Kurtz and James M. Lick. *J. Biol. Chem.* 111, 577-84 (1935).—Up to 3% of taurine has been isolated from the muscles of the annelid, *Audouinia spirabranchus*, Moore, an annelid, exceeding the values for any nitrogenous extractive hitherto reported. Little or no taurine was found in the muscles of several other annelids and related organisms. Twenty-seven references.

A. P. Lothrop

## 12—FOODS

F. C. BLANCE AND H. A. LEPPER

Chemical composition of some foodstuffs of Northern Argentina. P. Mazzocco. *Rev. soc. argentina biol.* 11, 314-17 (1935).

L. E. Gilson

Vitamin contents of some common food materials in China. Chao-Yü Chen. *Science (China)* 19, 1211-51 (1935).

C. L. Tseng

Some differences in the values obtained by the chemical and biological assays of vitamin C in certain foods. H. C. Hou. *Chinese J. Physiol.* 9, 291-8 (1935).—Good agreement between the 2 methods was obtained with oranges but with vegetables a lower value was obtained by the biol. method than by the chem. method.

L. A. M.

Certified colors for foods are standardized materials. J. K. Hall. *Food Ind.* 7, 545-6 (1935).—The av. amt. of pure dye in the 15 permitted food colors is 90%. The other 10% is largely water of crystal. and salt. Only actual experience and trial with a food product will insure securing the proper shade of color. In the dry state, these dyes will keep indefinitely, but the presence of moisture,

light and high temps is detrimental. Glycerol makes a good preservative for food colors. The use of 1-2 oz. of citric or tartaric acid per gal. of color soln is suggested, but the addn. of 0.25 oz. of  $\text{BzONa}$  is required for good keeping quality. The presence of reducing substances such as  $\text{SO}_2$ , bleaching agents and Fe is very destructive to most food colors.

C. R. Fellers

Chemical reactions involved in food spoilage. J. Gangl. *Oesterr. Chem.-Zig.* 38, 146-51 (1935).—A discussion of the anaerobic fermentation of carbohydrates, the development of rancidity in fats and the decompn. of proteins, and of the chem. tests for detg. the extent of these processes.

W. Gordon Rose

Report on (the analysis of) cereal foods. J. A. LeClerc. *J. Assoc. Official Agr. Chem.* 18, 560-2 (1935).—A brief discussion of the importance of suitable methods for the analysis of cereal products.

A. Papineau-Couture

Determination of moisture in cereal products by distillation with tetrachloroethane. J. M. Tucker and T. E.

Burke *Analyst* 60, 663-7(1935).—The purpose of this investigation was to find a suitable method for detg. the low  $H_2O$  content of baked cereal products, the results of which could be correlated with baking losses. The results of the expts. described show that distn. with  $CaH_2Cl_2$  (b. 140°) gives values which are in some cases the same but usually higher than those obtained by drying at 98.5°. The essential conditions are fairly rapid distn. and sufficient solvent to leave a mobile residue when all  $H_2O$  has been removed. The method is not reliable when appreciable quantities of invert sugar are present. W. T. H.

A modification [in the form of the apparatus] of the Göttinger method for testing the quality of wheat, Georg Ghemeroth *J. Landw.* 83, 227-33(1935), cf. Engelle, *C. A.* 29, 6860<sup>4</sup>.—A dome-shaped flask is attached above the original sample flask to permit the immediate passage of  $CO_2$ . The principal function of the gas is unchanged. The results with the new type of flask were more concordant than those obtained with the former flask in which trapping of gas under the cover often occurred.

John O. Hardesty  
Kernel texture as an indicator of quality in hard red spring wheats. O. S. Aamodt and J. H. Torrie. *Can. J. Research* 13, C, 70-88(1935).—The gray wooded soils found at Fallis, Alberta, provided a satisfactory means of obtaining a differentiation in kernel texture in hard red spring wheats. Correlation studies showed that the varieties behaved more or less similarly from year to year in kernel texture, protein content and loaf vol., but not in partial baking score. Kernel texture was indicated as being a better measure of partial baking score than protein content, while the latter was the better index of loaf vol. A close relation was found between the kernel texture of the varieties grown at Fallis and both the partial baking score and loaf vol. of the same varieties grown at Edmonton. In the case of protein content detd. on the Fallis material no such relation was obtained. The wheat-milk fermentation test was found to be of little value in differentiating between the baking quality of hard red spring wheat varieties. J. W. Shipley

Mass poisoning by wheat infected with *Lohium temulentum*. Julius Orient *Pharm. Monatsh.* 16, 191-3(1935).—Workers reporting cases of poisoning by wheat so infected are mentioned. It is found that uninfected wheat (sp. gr. 0.70-0.80) can be seed from infected wheat (sp. gr. 0.67) by flotation in  $H_2O$ . Of wheat and oat samples collected in 7 localities 8.3-28 and 22-56%, resp., were found to be infected. Chemistry, reactions, symptoms and treatment of poison cases are discussed.

H. M. Burlage  
The value and uses of bran. R. A. Lehmann *Mühle* 72, 1301-3(1935).—Analyses and digestibility values are given for various types of wheat and rye bran used in feeding swine and ruminants. Clinton L. Brooke

Nitrogen distribution and carbohydrate partition in Philippine rice bran. Joaquin Marañon and Luz Cosme. *Philippine J. Sci.* 57, 289-94(1935).—High grade Philippine rice bran that contained no hulls was analyzed and the compn. ascertained. The bran contained a considerable amt. of carbohydrates (44.50%). The N in the bran (2.204%) corresponded to 14.15% protein by calcn. Investigation of the N distribution showed that the major portion of the nitrogenous substances in rice bran consisted mostly of protein, which is composed largely of nonbasic N. The bran had a rather high content of starch (24.16%) and, in addn., contained small amts. of other carbohydrates such as pentosans, nonreducing sugars, gums and also crude fiber. Fourteen references.

F. L. Dunlap  
Grain conditioning. Heinz Gehle *Mühle* 72, 1027-30(1935).—The radiator conditioner is claimed superior to the warm-air conditioner, especially for soft wheats, which require higher temp. and shorter time than hard wheats. Expts. on German wheat using a lab conditioner in which the wheat was slowly heated to 40° and then rapidly raised to 50° effected a marked improvement in baking quality. An electrical experimental conditioner. Otto Haltmeier. *Ibid.* 1029-32.—A lab. conditioner is

described which is heated by passing an elec. current through grain between 2 parallel plates. In one form of the app. the voltage can be varied from 100 to 2000 v., facilitating the study of conditioning at high temps. and short time. The present status of grain conditioning. Hermann Annen. *Ibid.* 72, 1031-4.—The milling advantages derived from conditioning are uniformity in milling quality of different wheats, making it possible to grind soft and hard wheats together, toughening of the bran, better mellowing of endosperm resulting in cleaner sepn. from the bran, decreased power consumption, improved sifting and purifying. The improvement in baking quality resulting from conditioning of soft wheats is due to hardening of the gluten and not, as often claimed, to increased enzymic activity. The gluten of hard wheat cannot be softened by conditioning. Diastatic activity of sprouted wheat can be reduced by suitable treatment. Expts. with the elec. expt. conditioner (cf. above) at temps. between 40° and 60° show a linear increase in gluten swelling and shortening of the gluten with increasing temp., optimum gluten quality being obtained at 55°. Hard and soft wheat conditioning and its control. Ernst Berchner. *Ibid.* 1033-6.—Hard wheats with strong gluten should be conditioned at low temp., since their gluten needs no further strengthening. Hard wheat gluten cannot be made softer or more extensible even by long tempering at high moisture content, and the swelling rate of the gluten is not influenced thereby. Wheats with long dough development time are especially suited for strengthening soft wheat mixes. Soft wheat conditioning is to be considered from the standpoint of gluten hardening, and the extent of hardening is shown by the decrease in extensibility of the gluten. The farinograph and fermentograph do not show the most important changes in gluten quality resulting from conditioning. Dough softening should not be interpreted as gluten softening. Gassing power and diastatic activity are not appreciably influenced by normal heat treatment or tempering; evidence to the contrary is based on tests of the finished flours, whose phys. differences due to conditioning lead to false conclusions. Proteolysis is of minor importance even in wheat mixes contg. sprouted kernels. Dough softening is seldom or never the result of proteolytic degradation of the gluten, but is due to excessive starch degradation and hardening of the gluten. The wheat kernel contains no highly active proteolytic enzymes. Influence of conditioning on the extensibility of dough. Edwin Zeigler. *Ibid.* 1035-8.—Curves obtained with the Chopin extensometer and the farinograph are used to show that low extensibility can be increased and excessively high extensibility decreased by conditioning with warm air. Determination of the conditioning effect. Otto Haltmeier. *Ibid.* 1037-40.—Tests for changes in milling quality of grain effected by conditioning include milling tests on a simple attrition mill and stock analyses with detns. of both wt. and vol. of the sieve fractions and tests of the individual fractions. Baking tests should be combined with detns. of extensibility. Conditioning yesterday and today. Leo Hopf. *Ibid.* 1039-44.—Hard wheat conditioning has nothing to do with drying; its function is to transfer moisture from the exterior to the interior of the kernel and bring about the desired changes in gluten quality and kernel structure by the aid of heat. Soft wheat conditioning is simply an improved form of drying; moisture is transferred from the interior to the exterior of the kernel, and only enough is restored to the interior to alter the gluten quality and structure in the manner desired. Detailed procedures are given for conditioning of hard and soft wheats, and the effect of conditioning on different types of gluten is described. A combined grain peeling and conditioning process. Otto Kettner. *Ibid.* 1043-6(1935).—The app. is illustrated and described. Yield, keeping quality and color of flour, and aroma and flavor of bread are improved by peeling and conditioning of the grain (wheat or rye). Power consumption and cost of milling are lowered, and the grain mills more rapidly. Sound flour can be produced from musty grain, since the outer layers of the kernel are entirely removed. The preparation of grain (for milling)

in small and medium-sized mills. F. Alroth. *Ibid.* 1047-8 Conditioning. Leo Hopf. *Ibid.* 1129-32 — Polemical with Annen and Berliner (cf. above). The disputed softening of hard wheat gluten by conditioning is actually an increase in extensibility resulting from heightened water absorption and hyper-swelling of the gluten. Berliner's claim that the softening of dough attributed to proteolysis is due to starch degradation is inconsistent with the fact that starch-free gluten washed from sprouted wheat flour becomes soft and runny upon standing, in the same manner as dough. The decrease in elasticity of gluten during dough fermentation demonstrates gluten degradation. Zeigler's farinograms (cf. above) show at what temp. the gluten begins to break down under the influence of heat; this is not shown by the swelling no

Clinton L. Brooke

Quality and keeping properties of flour from wheat grown on the black and gray soils of Alberta. O. S. Aamodt and A. G. McCalla. *Can. J. Research* 13, C, 160-7 (1935).—Weight per bushel and milling yields of hard red spring wheats grown on the black soil at Edmonton were the same as for the same varieties grown on the gray soil at Fallis. Grade, protein content and baking quality of Edmonton-grown samples were superior. The flour from most of the Edmonton-grown samples retained its quality for at least 2 years after milling, but flour from most of the Fallis-grown samples had deteriorated so much during storage for 10 months that it was unfit for bread-making. Flour from Reward showed less deterioration than that from any other standard variety grown at Fallis. It also had the best original baking quality, and is the only one of the recommended varieties considered satisfactory for the gray soil area.

J. W. Shipley

Nitrogenous matter of wheats and flours. A. Leyte. *Ann. agron.* [N. S.] 5, 72-88 (1935).—The classification of wheats by the relationship gliadin/glutenin gives different and much less precise results than those by the extensometer. The mech. properties of a dough are similar to those of its gluten. Chemicals which can modify  $W$  (the least energy necessary to tear apart 1 g. of dough) produce great variations in the gliadin/glutenin relationship. The values for  $W$  and gliadin/glutenin relationships are changed in opposite directions. Mech. properties of gluten are a function of the  $pH$  of its medium and are partly dependent on its nonnitrogenous content. They are not changed by a preliminary ether extn. Flours, doughs, gluten and their solns. are sensitive to the action of certain oxidizers, reducing agents, antioxidants and protide precipitants. The transformations seem due to reactions similar to autooxidation and antioxygenation. An unknown substance (nonsensitive to chloroform but which reacts with traces of  $Na_2SO_4$ ,  $NaOH$  and  $KCN$ ) gives gluten its mech. properties. Cystine and cysteine may be important.

F. W. Marsh

Report on viscosity of flour. Tentative method for viscosity determination of acidulated flour-suspension. C. G. Harrel. *J. Assoc. Official Agr. Chem.* 18, 577-92 (1935).—The study was undertaken to verify some of the results of Bayfield (C. A. 28, 2211; 29, 2219) and of Reiman (C. A. 28, 4706). The procedure proposed by Bayfield was slightly modified by specifying: (1) const. wt. of protein instead of const. flour wt., (2) making up the suspension by means of a standard-size mortar and pestle with a stirring time of 2.5 min. instead of 45-60 sec., (3) the introduction of a 60-min. digestion period, and (4) the measuring of the viscosity in a MacMichael viscometer standardized at different temps. by means of a 60% sucrose soln. Judging by the close agreement in collaborative work the proposed method for making the suspensions seems to be satisfactory. The results of the collaborative work between different labs. were not encouraging. The discrepancies were of about the same magnitude whether the results were recorded in a MacMichael viscometer or in centipoises; this does not mean that the standardization of the instrument is unnecessary, because there is a definite tendency for some collaborators to be high or low on all tests. The results submitted by collaborators on the standardization with the same sugar-glycerol solns. sent

out showed no better agreement between different labs. It is possible that the lactic acid used in different labs. was of different character even though the normality was the same.

A. Papineau-Couture

Report on (the determination of) ergot in flour. C. L. Brooke. *J. Assoc. Official Agr. Chem.* 18, 592-3 (1935).—For the detn. of ergot *per se* Okoloff's modification (C. A. 23, 3991) of Hoffmann's reaction was found the most satisfactory method. For detg. the amt. of alkaloids present, the Allport-Cocking modification (C. A. 27, 562) of the colorimetric reaction of *p*-dimethylaminobenzaldehyde and  $H_2SO_4$  with ergot alkaloids was found satisfactory. Quant. extn. of the alkaloids or other constituents is the major problem involved in any detn. of ergot in flour, in the extn. of the ergot alkaloids, good results were obtained by a modification of Musset's method (*Pharm. Zentralhalle* 40, 353 (1899)) in which the flour, after exposure to  $NH_3$  vapor for several hrs., is percolated with  $H_2O$  from which the alkaloids are extd. and eventually transferred to 1% tartaric acid. A. Papineau-Couture

Report on (the determination of) color in flour. H. K. Parker. *J. Assoc. Official Agr. Chem.* 18, 593-8 (1935).—From a study and discussion of the various methods of detg. the color value of flour, it is concluded that extn. methods are valuable for carotenoid pigment content, while reflectance methods are better suited for the study of flour color as a whole and they include the brilliancy factor, which is important in the judgment of flour quality.

A. Papineau-Couture

Report on (the determination of) ash in flour, macaroni products and baked products, chlorides in baked products, and moisture in baked products containing fruit. L. H. Bailey. *J. Assoc. Official Agr. Chem.* 18, 562-3 (1935). *cf. C. A.* 23, 6189.—To det. salt-free ash in alimentary pastes, deduct from the total Cl the Cl normally present in semolina or flour, calc. the balance of the Cl to NaCl and subtract the result from the total ash. For the detn. of  $H_2O$  in baked products contg. fruit (fruit cake, fig newtons), good checks were found by drying for 1 hr. at  $130^\circ$  in an air oven, and also by drying for 5 hrs. in a vacuum oven at  $98-99^\circ$ , but the latter procedure gave appreciably lower results than the former, drying in vacuum at  $70^\circ$  for 5, 9, 11 and 15 hrs., resp., also gave closely agreeing results, but const. wt. was not obtained, and the results were much lower than those obtained by drying at  $98-99^\circ$ . Dried fruits high in sugar content cannot be dried to a const. wt., and since fig newtons and fruit cake contain large proportions of fruit, the method cannot be expected to give satisfactory drying results with such products.

A. Papineau-Couture

Report on (the determination of) the hydrogen-ion concentration (of flour). Rowland J. Clark. *J. Assoc. Official Agr. Chem.* 18, 563-6 (1935). *cf. C. A.* 28, 6487.—The previously described method was studied collaboratively on samples of flour, bread, cake and macaroni. The agreement of all the collaborators on flour and macaroni was very close, there being only 0.2  $pH$  difference between the max. and min. results. The agreement among collaborators on bread and cake was not very close, there being 0.5  $pH$  difference between max. and min. results, indicating that the bread and cake samples were not properly prepd. for distribution, possibly too coarsely ground, not sufficiently mixed, or contained too much moisture, thereby permitting deterioration. All collaborators used the same method of prep. the exts., and although they seemed to experience no difficulty with its actual operation, turbid solns. resulted and caused errors; it would therefore be well to centrifuge and filter the ext. solns. instead of allowing them to settle and decanting, to reduce the turbidity as much as possible. A. P.-C.

Report on (the determination of) diastatic value of flour. M. J. Blish. *J. Assoc. Official Agr. Chem.* 18, 566-9 (1935). *cf. C. A.* 28, 6487.—The previously described method was studied collaboratively. The results of 14 of the 17 collaborators showed satisfactory concordance, those of the other 3 being decidedly low; this agrees with the collaborative study conducted by Sandstedt (C. A. 29, 2754). From a discussion of possible sources of error and

variability it is concluded that (1) for best results the titration should be carried out as soon as possible after the sample is removed from the boiling water bath; (2) the acidity of the  $H_2SO_4$  should be  $3.58 \pm 0.05 N$ ; (3) in cases where a 5-cc aliquot of the clarified flour ext contains more reducing sugars than the specified quantity of  $K_2Fe(CN)_6$  will take care of, a smaller aliquot must be taken, but it is inadvisable to use less than a 3-cc. aliquot and in any case the ratio of flour to buffer soln must not be altered; (4) flours undergo no reduction in diastatic activity over long periods when stored under refrigeration (approx  $0^\circ$ ), but changes have been found to occur in a few weeks at room temp. A. Papineau-Couture

A study of the Kjeldahl method. IV. Metallic catalysts and metallic interferences. R. A. Osborn and J. B. Wilkie. *J. Assoc. Official Agr. Chem.* 18, 604-9 (1935), cf. *C. A.* 28, 5002<sup>1</sup>.—A study of the effect of each of 39 metals upon the accuracy of the detn of the N content of a gluten flour showed that Hg is the most satisfactory catalyst, its only disadvantage being the necessity of pptg the Hg as sulfide following digestion and preceding distn of the  $NH_3$ ; no other catalyst appears to require this treatment. The possibility of poisoning from volatile Hg vapors during digestion was not considered. Of the 39 metals studied in concns of 0.003 M, 10 or 12 catalyzed the Kjeldahl digestion of a gluten flour. From a practical standpoint the safest and best catalysts appear to be Hg, Te, Ti, Fe and Cu. In ordinary amts, or under less violent conditions of digestion, Se, Mo, V, W and Ag may be suitable as catalyst. When present in larger amts, Se, V and  $KMnO_4$  clearly interfere with the accuracy of the detn. Results confirm the interference of Pt as well as the need of pptg Hg as sulfide before distn. Mixed catalysts are not recommended with digestion of flour samples. A. Papineau-Couture

Report on (the determination of) flour bleaching chemicals. Dorothy B. Scott. *J. Assoc. Official Agr. Chem.* 18, 570-2 (1935), cf. *C. A.* 28, 6487<sup>1</sup>.—The tentative official A. O. C. method for the detn of Cl in bleached flour was modified by extg with 1.1 H<sub>2</sub>SO<sub>4</sub> rather than 1.3, 3 changes were made in the Kent-Jones and Herd method (*C. A.* 24, 3435). 50 cc of 4% alc NaOH was used instead of 20 cc to hydrolyze the fat and as a fixative for Cl, the acid exts were not coned, and no NaCl was added for coagulation. The 2 methods as thus modified gave closely agreeing results on amts of 2-250 mg. Cl. A collaborative study of S's test for the detection of  $Bz_2O$  (*C. A.* 28, 5139<sup>1</sup>) showed that the method offered no difficulties and gave reliable results. From a crit. study of S's method, Nicholls' method (*C. A.* 27, 1411) and Munsey's method (*C. A.* 29, 7508<sup>1</sup>), it is concluded that a more satisfactory method than any of the 3 can be worked out. A. Papineau-Couture

Report on (the determination of) crude fiber in baked products. R. G. Capen. *J. Assoc. Official Agr. Chem.* 18, 573 (1935); cf. *C. A.* 28, 6489<sup>1</sup>.—A further study of the official A. O. C. method for crude fiber in grain and stock feeds as applied to baked products not contg fruit (whole wheat crackers, ginger cookies and cakes) gave concordant results in all cases. A. Papineau-Couture

Report on (the determination of) milk solids in milk bread. V. F. Munsey. *J. Assoc. Official Agr. Chem.* 18, 573-7 (1935).—Collaborative study of the Hartmann and Hüllig citric acid method (*C. A.* 27, 5431) gave highly unsatisfactory results (no details of results given), which are believed to be attributable to the small quantity of material used in the detn and to the incomplete extn of the citric acid in the alc solvent in the short time prescribed. A method is described for estg the butter-fat content of bread, it is based on total extn of the fat after acid hydrolysis and treatment of the fat similar to the Reichert-Meissl no detn (use a 1-g sample and titrate with 0.02 N alkali), the value thus obtained being designated as the "fat no."  $C = A(B - 1.0)/31.5$  and  $D = 7.7 (B - 1.0)/2.3 \times 31.5$ , in which A = percentage of total fat on dry basis, B = "fat no.," C = percentage of butter fat on dry basis, D = percentage of milk solids on dry basis, 71.5 = "fat no." of butter fat, 1.0 = "fat no."

on fat from water bread, 7.7% = milk solids on dry basis in milk bread, 2.3% = milk fat on dry basis in milk bread. A collaborative study of the method yielded fair results, the spread of the "fat no." was greater than desirable, which is attributed to failure to conform to the details of the procedure, as the method is essentially empirical. A. Papineau-Couture

(Detection of) soybean flour in smoked meat products. Charles H. LaWall and J. W. E. Harrison. *J. Assoc. Official Agr. Chem.* 18, 644 (1935); cf. *C. A.* 28, 5140<sup>1</sup>.—The previously described tests were applied to frankfurters contg 1, 3 and 10% soybean flour and manufd. commercially under known conditions. Neither cooking of the frankfurter in the casing, as usually practiced, nor smoking affected the qual test based upon liberation of  $NH_3$  from urea by the urease naturally present in soy; if, however, the soy flour is first made into a stiff paste and heated above  $100^\circ$  or under pressure, all of the urease is destroyed, so that a neg. test does not necessarily indicate absence of soy flour; pos. tests should always be confirmed by identifying the characteristic cell structures. A. Papineau-Couture

Milk-fat globules. Henry J. Apple. *J. Am. Vet. Med. Assoc.* 40, 331-2 (1935).—Photomicrographs are given of milk-fat globules from 3 breeds of goats, 4 breeds of cows, human and homogenized cow's milk. R. Brown

Detection of formaldehyde in milk. T. McLachlan. *Analyst* 60, 752 (1935).—In detecting HCHO in milk by the Hekner method, it is sometimes hard to get accurate results if the  $H_2SO_4$  contains an impurity such as  $SO_2$ . It is well, therefore, to add a trace of  $Fe^{+++}$  salt and to test each shipment of acid to see if it is satisfactory. The addn. of a little  $KMnO_4$  sometimes serves to make the acid more sensitive but the effect is only temporary. W. T. H.

Approximate determination of milk solids, including lactic acid, in condensed buttermilk and related products. Joseph W. E. Harrison. *J. Assoc. Official Agr. Chem.* 18, 645-6 (1935).—Condensed buttermilk and similar products are sold on a basis of the total solids contents, and custom has established that the lactic acid content be viewed as a milk solid. Use of the usual drying method for detg total solids is impossible because part of the lactic acid is volatilized and also because charring occurs at  $100^\circ$ . Distn methods give variable results. Loss by volatilization and charring due to lactic acid can be prevented by addn. of ZnO, the following technic being suggested. weigh 2-5 g of sample in a tared flat-bottomed dish contg approx. 2 g of recently ignited ZnO, add 5 cc  $H_2O$ , mix well and dry at  $100^\circ$  for 3 hrs. Sand may also be incorporated if desired. The result for solids thus obtained can be corrected for loss of  $H_2O$  liberated by neutralization of the lactic acid by the ZnO by adding  $1/10$  of the lactic acid detd by titration. A. Papineau-Couture

The methylene blue reduction test, its efficiency and interpretation under Philippine conditions. Jose B. Uchanco. *Philippine J. Sci.* 57, 293-319 (1935).—Statistical examn of the results on 142 samples of milk indicate a high degree of efficiency and dependability in the methylene blue reduction test. Philip D. Adams

Mineral constituents in fresh and canned milk. A. J. Hermans and S. Sagrario Claravall. *Philippine J. Sci.* 57, 323-8 (1935).—Forty-five samples of natural sterilized milk (canned), evapd and powd. milk, sweetened condensed milk, fresh goat, cow and carabao (water buffalo) milk were analyzed for Ca, P, Fe and water, fat, ash, protein and lactose. Fresh cow milk gave a higher percentage of ash and Ca than natural sterilized milk. Carabao milk had the lowest ash content (0.64%) but the ash had the highest amt of Ca (27.99% CaO), and also more fat and protein than other natural fresh milks. Toggenberg goat milk had a higher Ca content than any other breed of goats. Klum and Molico (powd. whole milk) had the highest percentage of fat. Philip D. Adams

Effect of different methods of mixing milk. H. Barkworth. *J. South-Eastern Agr. Coll. B. Ye, Kent No.* 36, 129-30 (1935).—In sampling milk, either dipper-stirring or bucket-mixing gave, in general, a fat content lower than that obtained with an unrevved sample. K. D. Jacob

**The freezing of milk and cream.** F. Bruce Baldwin and J. J. Doan. *J. Dairy Sci.* 18, 629-33(1935).—When whole milk is partially frozen in an undisturbed condition, the fat content in the frozen and unfrozen portions over the entire range of freezing is dependent upon the creaming phenomenon and its speed relative to the speed of freezing, and to some extent upon the size and shape of the container. When the creaming ability of milk is destroyed (as by heating or homogenization) the fat content of the unfrozen portion increases progressively with the degree of freezing, while that of the unfrozen portion decreases at first, but approaches the fat percentage of the original milk as the degree of freezing approaches 100%. Increasing fat contents retard diffusion of milk constituents into the unfrozen portion, and at 25% fat such diffusion is practically nil.

**Frozen milk.** Angel Mantovani. *Rev. facultad quim. ind. agr. (Univ. nac. litoral, Argentina)* 3, 54-53(1934).—Milk which is pasteurized for an hr at 63° and then poured into Sn molds in contact with a CaCl<sub>2</sub> bath at -35° to -30° is frozen in 12-15 min. The product is homogeneous in appearance. Chem. analysis of sections of this product compared with that of cakes frozen over an 8-hr. period are given to show that only the former have uniform compns. The bacterial count shows no change in a month. Practical advantages of dealing with frozen milk are given.

**Relative economies of different forms of milk as sources of protein, calcium and phosphorus.** M. M. Kramer and Bernice L. Kumerth. *J. Am. Dietetic Assoc.* 11, 318-21(1935).—Graphs are presented to show the cost of 1 g. protein, Ca and P from different forms of cow milk, at a range of market values.

**The composition of commercial dried whey.** W. L. Davies. *J. Soc. Chem. Ind.* 1935, 338-41T.—The proximate analyses, N distribution, and partial analyses of the ash of 19 samples are reported. The av. results show: fat content 1.18, ash 7.54, chloride as NaCl 2.28, crude protein 12.5, titratable acidity 2.38%, calcd as lactic acid.

**Vitamin-A assay of ghee.** B. N. Banerjee and S. D. Banawala. *Agr. Live-stock India* 5, 382-8(1935).—The characteristics of pure samples of Indian ghee, prepd from buffalo, goat and cow butter, were: sapon. value 220.4-236.2, I value 26.5-37.9, acid value 0.38-1.0, unsaponifiable matter 1.64-3.86%, n<sub>D</sub> 1.4531-1.4549 and  $\eta_{sp}$  0.9172-0.9206. Goat ghee and buffalo ghee were pure white but gave a trace of yellow color in the Lovibond untometer and were rich in vitamin A. Pure ghee from all sources gave a pos. reaction with the Carr and Price SbCl<sub>5</sub> test, the blue value amounting to 16-22 units per g. of fat; the blue color was completely masked by the presence of as little as 10% of adulterant (hydrogenated vegetable fat). Ghee substitutes had lower sapon values (187-200) and higher I values (75-60) and n than the pure materials. Of the pure materials, goat ghee had the lowest acid value, I value and n and the highest sapon value; it had the lowest carotene value but the highest blue value.

**Kaufmann's thiocyanogen value of Indian butter fat (ghee).** U. D. Budhalakoti and K. C. Mukherji. *J. Indian Chem. Soc.* 12, 455-8(1935).—The inolic acid content of butter fats as detd. by thiocyanogen and I values is fairly const. (cf. Arup, C. A. 26, 3853) and can be relied upon for estg. the purity of butter-fat samples. Authentic samples of butter or butter fat (ghee) from various Indian sources were examd. for the percentage of inolic acid content which was found by multiplying the difference between the I value (W<sub>1</sub>'s method) and the thiocyanogen value (Wizoff's modification of Kaufmann's procedure) by 1.104. The range of I values varied from 30 to 50. The inolic acid content was fairly const. (3.5-5.4). One sample submitted contained no inolic acid and was probably a hydrogenated oil or fat sold as vegetable ghee.

**Effect of beet tops on the flavor and odor of milk.** G. M. Trout and G. E. Taylor. *Mich. Agr. Expt. Sta. Quarterly Bull.* 18, 37-41(1935).—Approx. 1700 samples of

milk from Guernsey, Jersey and Holstein cows were examd. for beet flavor. The beet-top flavor became noticeable in the milk of cows when the animals were fed at least 25 lb. daily. The flavor was most objectionable in night milk and was worst when only beet tops were fed. The supplemental feeding of grain or hay decreased the beet flavor. The flavor was most pronounced when the tops were fed shortly before milking. The presence of frozen or decompd. beet tops in the stable had a deleterious effect on the flavor and odor of the milk. Beet-top flavor did not develop or become pronounced when the milk was held in storage 24-48 hrs. Aeration made the odor and flavor less objectionable but did not eliminate it entirely. Pasteurization of the milk caused a change in flavor but did not produce good quality milk from the off-flavored product. Under ordinary conditions of feeding beet tops, the quality of the milk is insufficiently affected to merit rejection.

**Fishy flavor in butter.** B. Segal. *J. S. African Chem. Inst.* 18, 42-3(1935).—The development of fishiness is due to chem. production of Me<sub>2</sub>N from lecithin, present in the butter in varying quantities depending on the method of manuf.

**Differences in the lactic acid percentages in butters.** E. O. Whittier and C. S. Trimble. *Ind. Eng. Chem., Anal. Ed.* 7, 389-90(1935).—Sweet-cream butter can be distinguished from sour-cream butter by a lactic acid detn. Storage of butter has no effect on the lactic acid content.

**The estimation of salt in butter.** F. H. McDowall and C. L. MacDonald. *New Zealand J. Sci. Tech.* 17, 417-19(1935).—The following method is proposed for the accurate checking of butter samples const. approx. the (British) legal limit (1.5-2.0%) NaCl. 50 ml. H<sub>2</sub>O is added to 5 g. butter dissolved in 15 ml. Me<sub>2</sub>CO. A small amt. of powd. CaCO<sub>3</sub> is added, and the soln. is titrated with 0.12 N AgNO<sub>3</sub>, with K<sub>2</sub>CrO<sub>4</sub> as indicator.

**Determination of butacetyl and methylcarbinol.** C. R. Barnucoat. *Analyst* 60, 653-62(1935).—Various workers have detd. butacetyl in butter but the values obtained range from 2-4 p. p. m. (in highly flavored butters) to 0.05-0.5 p. p. m. All published methods for detg. (AcO)<sub>2</sub> and its precursor (hereafter called carbinol) are based on the sepn. of (AcO)<sub>2</sub> by distn. and pptn. as Ni salt of dimethylglyoxime. First, the conditions most favorable for a complete pptn. of the Ni compd. were studied. A satisfactory reagent is a mixt. of 4 ml. 20% NH<sub>4</sub>OH HCl, 4 ml. 20% NaOAc soln. and 2 ml. of 5% NiCl<sub>2</sub> soln. free from Co and Fe. For carrying out the conversion of (AcO)<sub>2</sub> to the monoxime, the monoxime to the dioxime and finally the pptn. as Ni compd. it is best to heat for several hrs. at 80-90° and then let stand overnight at 70-80°. The second reaction is slow. The p<sub>H</sub> should at the last be 7 or higher. Three days should be allowed for the ppt. to form completely. To sep. carbinol + (AcO)<sub>2</sub> from butter, distil 400 g. of the sample with steam in a flask contg. 500 ml. of 0.1 N H<sub>2</sub>SO<sub>4</sub> which is satd. with NaCl and also contains 4% of FeCl<sub>3</sub> to oxidize the carbinol to (AcO)<sub>2</sub>. To obtain this last compd. alone, distil in an atm. of CO<sub>2</sub>. In both cases, the distillate is passed into a receiver contg. the Ni reagent. The precautions necessary in weighing the Ni ppt. and the possibility of making the ppt. serve for the colorimetric detn. is shown when the ppt. weighs less than 1 mg. The procedure recommended gives consistent results which are a little low but the final conclusion drawn is that in the present state of our knowledge it is difficult to det. whether (AcO)<sub>2</sub> has been intentionally added to butter.

**The effect of soybeans in the rations of dairy cows upon the vitamin A value of butter.** J. W. Wilbur, J. H. Hilton and S. M. Hauge. *J. Dairy Sci.* 18, 661-5(1935).—Soybeans apparently suppress the transference of vitamin A from the ration to the butter. Roasting the soybeans does not prevent the action. It is possible to produce butter of fairly high vitamin A value even when soybeans are used in the rations of the cows provided roughage of high vitamin A potency is fed.

**Retarding rancidity.** Colored transparent cellulose



wrappers Willard L. Morgan. *Ind Eng Chem*, 27, 1257-90(1935) —Blue and invisible ultraviolet light materially accelerates the development of rancidity in such materials as potato chips, crackers, cakes, butter, candies, nuts and soaps, whereas other visible light such as red and yellow have little effect. Consequently rancidity-retarding wrappers may be of any visible color except blue. Highly protective yellow transparent cellulose films have been developed and utilized for food packaging. A large no. of specially prep'd cellulose films of a wide variety and shade of colors were used in detg. which rays in sunlight are accelerators of rancidity. The time at which rancidity first became evident in taste and odor compared with the time required for rancidity to develop in some of the same lot of potato chips simultaneously exposed in uncolored transparent cellulose was used to establish a ratio indicating the rancidity-retarding value of the films. Under the same exposure conditions various lots of foodstuffs which gave a neg. Kreis test developed rancidity at widely different rates. It is believed that this and other espil. difficulties and the failure to appreciate the autocatalytic effect of small amts. of ultraviolet light transmitted by the filters used by various other investigators have been the source of the confusion and conflicting claims which have been published. F. L. Dunlap

Firm white of fresh and storage eggs H. J. Almqvist and F. W. Lorenz. *Poultry Sci*, 14, 340-1(1935) —The percentage of firm white in storage eggs had a pos. high correlation with that of the fresh eggs from the same hens. Eggs with higher percentages of firm white showed a lower percentage of liquefaction of the firm white during storage. K. D. Jacob

Refractometric estimation of the total solids content of whole eggs and of yolks M. Irene Bailey. *Ind Eng Chem*, Anal Ed 7, 385-6(1935) —The percentage of total solids of whole eggs and yolks when plotted against the indices of refraction shows straight-line relationships. Equations are derived to be used in routine work.

Yolk-fat C. H. Lea. *Dept. Sci. Ind. Res. Rept. Food Investigation Board* 1934, 58-9(1935) —Detn. of the free acidity of the  $\text{Et}_2\text{O}$  ext. of the yolks of eggs stored for 3-6 months at 0° indicated that the method is not sufficiently sensitive to be of much service as a measure of incipient decomposition of eggs in shell. The peroxide values obtained were without significance, since the deep yellow color of the extd. oil renders a small zero error in the  $\text{Na}_2\text{S}_2\text{O}_8$  titration practically inevitable. Storage at 10° and 20° yielded slightly higher figures for the free acidity of the fat, but only in those cases where decomposition was already obvious. A. Papineau-Couture

The  $p_n$  of the white of egg E. C. Smith. *Dept. Sci. Ind. Res. Rept. Food Investigation Board* 1934, 50-2(1935) —On storage the  $p_n$  of egg white increases from an original value of 7.47 to 9.2-9.5 because of loss of  $\text{CO}_2$ . The loss of  $\text{CO}_2$  can be restricted or prevented by maintaining a suitable partial pressure of  $\text{CO}_2$  (e. g., 10% at 20-5° or 3% at 0°) in the surrounding atm., and in this respect the egg white behaves very much like a 0.1 N  $\text{NaHCO}_3$  soln. It would appear that the quality of the white is best preserved by maintaining the  $p_n$  between 7.5 and 8.0, but other aspects of storage may require a compromise at a higher or lower  $\text{CO}_2$  concn. than is indicated by this consideration alone. A. Papineau-Couture

Bound water in thick and thin white T. Moran. *Dept. Sci. Ind. Res. Rept. Food Investigation Board* 1934, 52-3(1935) —The amts. of water unfrozen in thick and thin white at different temps. were detd. by the colloid-sac method (C. A. 29, 1921). Conclusion: The high viscosity of thick white is due to its structure, which, in a new-laid egg, is readily observable to the naked eye.

Acid cure for meat Lloyd A. Hall. *Food Ind.* 7, 533(1935) —A typical formula for an acid meat-pickling brine consists of  $\text{NaNO}_2$  0.9,  $\text{NaNO}_3$  0.6,  $\text{NaCl}$  97.65 and anhyd. citric acid, 0.85%. The acid speeds the conversion of  $\text{NaNO}_2$  to  $\text{HIN}(\text{N})$ , the latter being the element which combines with the hemoglobin to form the red

coloring which gives cured meat its appetizing and attractive appearance. In curing processes where nitrate is used, bacterial action reduces the nitrate to nitrite. A liquid pickle is preferred to the use of the solid salts. The use of an acid provides for a regulatory influence on the bacterial actions which take place in the pickling soln.

C. R. Fellars  
Detection of meat reddening chemicals. A. Beythum. *Pharm. Zentralhalle* 76, 545-7(1935) —The addn. of  $\text{NaH}_2\text{PO}_4$  and  $\text{NaOAc}$  to meat is discussed from the standpoint of their possible detection. The German food law now proscribes the use of the acids and salts in connection with meats, but fails to include  $\text{NaOAc}$  as an addn. product, the presence of added  $\text{NaOAc}$  may be possibly detected through an increase in alkyl of the ash. W. O. E.

Chemical changes in the fat of bacon. C. H. Lea. *Dept. Sci. Ind. Res. Rept. Food Investigation Board* 1934, 76-7(1935), cf. C. A. 28, 5146 —The yellowing of bacon fat appears to be a secondary reaction involving the peroxide, but the mechanism of the change as it occurs in the tissue remains as yet undecoded. A. P. C.

Analysis of fish I. Tests for the condition of the oil of fish flesh Maurice E. Stanby. *J. Assoc. Official Agr. Chem.* 18, 116-21(1935) —The following simple, rapid method for the extn. of oil from mackerel (*Scomber scombrus*), without risk of decomposition, is presented to 20 g. of finely ground fish flesh (free from skin and bones) in a 150-cc. shaking bottle add 25 g. anhyd.  $\text{Na}_2\text{SO}_4$  and 100 cc.  $\text{Li}_2\text{O}$  (neutral to phenolphthalein), shake mechanically for 30 min.; det. fat by filtering a 20-cc. aliquot and evap. on the water bath in a tared beaker; titrate free acidity in another 20-cc. aliquot, using the official A. O. C. method without removing  $\text{Et}_2\text{O}$ , det. the peroxide iodometrically in a 3rd 20-cc. aliquot by Wheeler's method (C. A. 26, 3124) as follows: add 50 cc. of a freshly prep'd mix. of 60% c. p. glacial  $\text{AcOH}$  and 40% U. S. P.  $\text{CHCl}_3$  followed immediately by 1 cc. of sat'd  $\text{KI}$  from a pipet, shake with a rotary motion for exactly 1 min., quickly add 100 cc. of 0.03% starch soln. and immediately titrate with 0.01 N  $\text{Na}_2\text{S}_2\text{O}_8$ ; moles of peroxide ( $\text{M}$ ) per 1000 g. of oil = 0.5 (cc.  $\text{Na}_2\text{S}_2\text{O}_8$ ) (normality)/(wt. of oil). Application of the method was found to be valuable in appraising the condition of fresh and frozen mackerel. In a series of tests it was found that 1000  $\text{M}$  varied as follows: fresh 0-0.6, slightly rancid 0-21.4, rancid 18.4-36.4, extremely rancid 33-201. A. Papineau-Couture

Tin and lead in canned fish H. Amphlett Williams. *Analyst* 60, 683-5(1935) —About 150 samples of canned fish, selected at random, were exam'd. for Sn and Pb and 20 samples were reported adversely. The suggestion is made that 0.1 grain per lb. can be adopted as a limit for the quantity of Pb which canned fish shall be allowed to contain. Twelve samples contg. more than 2 grains of Sn per lb. were found. W. T. H.

Studies on maturity of fruit IV. The catalase and oxidase activity of apples in storage as affected by conditions during growth J. C. Hinton. *Univ. Bristol Agr. Hort. Research Sta., Ann. Rept.* 1934, 29-62, cf. C. A. 27, 4271 —Catalase activity of stored apples increased during storage, but in 3 cases it subsequently decreased during the later stages of storage. The rate of increase in catalase activity was highest during the early part of storage and it fell more or less rapidly during the storage period. The fall was proportionate to the value of the max. early rate, being more rapid in those cases where the early rate was higher. The later the date of picking the apples, the higher was the early rate of increase in catalase activity and the steeper was the subsequent fall in rate. With the Newton Wonder variety, differences in the rate of change of catalase during storage were such as to suggest that fruits on ringed trees matured more slowly than fruits from similar not ringed trees. In fruit of the Allington Pippin variety (1) grown under grass cover, (2) grown under clean cultivation and (3) grown on trees on which fruit thinning was carried out, changes in catalase activity during storage were not sufficiently uniform to suggest differences in the rates of maturation of the fruits. There were indications that unusually low temps. prevailing for

some days prior to picking cause a decrease in the catalase activity of the fruit. Oxidase activity of apples decreased markedly but slowly during storage and also during ripening on the tree. Differences in oxidase activity during storage could not be definitely correlated with differences in the cultural treatment of the trees. V. The effect of conditions during growth on the process of softening and on the loss of total weight in apples during storage. *Ibid.* 53-53.—The rate of softening of apples during storage was correlated with the degree of maturity of the fruit at the time of picking. The rate was lower in fruit from trees (1) growing under grass cover, (2) which had been bark-ringed or (3) upon which fruit-thinning had been carried out, than in fruit from the control trees or from trees grown under clean cultivation. The rate of loss of total wt. during storage of the fruit diminished as storage proceeded, over the greater part of the storage period, and it the final stage of storage increased again in the majority of the samples. The behavior of the sample was only approx. defined by the initial rate of loss of wt. The variance with respect to loss of wt. increased as the samples were picked at later dates. The rate of loss of wt., to the extent to which this rate diminished in storage and the variance of the samples were all related to the stage at which the samples had been picked. There seemed to be very little relation between the loss in wt. during storage and the H<sub>2</sub>O content of the fruit. VI. The effect of conditions during growth on some chemical constituents of apples in storage. *Ibid.* 54-105.—The total N content decreased during storage. The rate of decrease was greatest during the early part of storage and was influenced by the stage of maturity at which the fruit was picked. With increasing stage of maturity the rate of loss of N in storage increased up to a certain point, after which the rate of loss again decreased. The acid hydrolyzable fraction decreased markedly during storage but in many cases underwent an increase toward the end of storage life. The rate of loss of sucrose decreased between succeeding picks in the Allington Pippin series but tended to increase between picks in the Newton Wonder series. There was evidence that the loss of sucrose ceased at a time when a residual amt. was still present, this residual amt. increased with succeeding picks. The amt. of increase of reducing sugars in storage and the length of time during which increase in value took place were less with succeeding pickings in each case. The rate of increase in reducing sugars in storage decreased with succeeding pickings in all cases. The ratio of reducing sugars to sucrose increased during storage in all cases. K. D. Jacob.

Report on (the determination of) pectic acid and electro-metric titration and by W. L. Roberts. *J. Assoc. Official Agr. Chem.* 18, 599-602 (1935).—A collaborative study was made of 4 methods: (1) electrometric titration with H<sup>+</sup> electrode, (2) electrometric titration with quinhydrone electrode, (3) titration with phenolphthalein as inside indicator and (4) titration with azoform as outside indicator, for the detn. of the following colored fruit solids: (1) strawberry preserves, (2) raspberry preserves, (3) orange conc. and (4) powder consisting of acid, sugar and amarant. There was good agreement between the H<sup>+</sup> electrode and quinhydrone electrode titrations in all instances, the phenolphthalein titration was consistently higher and the azoform titration consistently lower than the 2 electrometric methods, as was to be expected as the azoform end point is at pH 7, the phenolphthalein end point at pH 8.3 and the electrometric end point at about pH 7.9. While the agreement on the basis of a 25-cc. aliquot was good in most cases, it was rather poor when calcd. on the basis of a 100-g. sample, this could be corrected somewhat by the titration of aliquots containing larger portions of sample. In spite of the fact that hydroquinone is oxidized in alk. soln. in presence of air, the quinhydrone electrode gives good results if the titration is carried out rapidly (within 5 min.). The excellent agreement of the 2 electrometric methods up to pH 8 proves the accuracy of both methods, when the titration is carried out to the equivalence pt. of pH 7.9. A. Papineau-Couture.

Changes in the pectin of fruits during storage. T. N.

Morris. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 200-3 (1935); cf. C. A. 29, 2411.—Previous tests were repeated and more precise data obtained with the help of a jelly-strength tester and the results (which must not be taken as absolute) indicated that: (1) there was a slow loss of setting power with raspberries and gooseberries frozen raw at -10°, and even at -20° a slight loss was indicated; with apples there was no marked change. (2) the setting power was completely lost when raw raspberries and gooseberries were treated with SO<sub>2</sub>, but ext. from apples appeared definitely to have gained in setting power, (3) the setting power was unchanged during cold storage after previous treatment by heat, and showed an increase in each case during storage at ordinary temp. with SO<sub>2</sub> after previous heating and cooling. It is suggested that the increase noted in setting power in presence of SO<sub>2</sub> may represent a stage in the slow conversion of pectin to pectic acid under the influence of the mineral acid. A. Papineau-Couture.

Preservation of fruit with sulfur dioxide. Effect of hot and cold methods on residual sulfur dioxide content in jam. Vernon L. S. Charley. *Univ. Bristol Agr. Hort. Research Sta. Ann. Rept.* 1934, 255-63.—Plums processed in the cold with 100 and 200 p. m. of SO<sub>2</sub> gave jams which contained 20 and 70 p. m. of SO<sub>2</sub>, resp. With this process the use of Ca(HSO<sub>3</sub>)<sub>2</sub> resulted in a lower residual figure, but the data with K<sub>2</sub>SO<sub>3</sub> and calcd. no significant deviation from the ordinary H<sub>2</sub>SO<sub>4</sub> method. A considerable increase in residual SO<sub>2</sub> (84-120 p. m.) was observed in jams made from hot-processed pulp. The skins of the whole fruits preserved in the cold by the stronger soln. became toughened and this condition persisted in the jam, the weaker soln. of SO<sub>2</sub> did not greatly affect the texture of the fruit. The hot-processed pulp yielded darker jams in which the fruit was completely disintegrated. The skins of cold-processed plums contained much more SO<sub>2</sub> (320-983 p. m.) than did the skins and flesh. K. D. Jacob.

Some principles of apple storage. Franklin Kidd. *Horticultural Education Apple Yearbook* 2, 39-42 (1933).—A difference of 1°F. in the av. storage temp. may result in 10% difference in storage life. Safe temps. range from 34° to 40°F. depending on the variety of apples. Control of the compn. of storage atm. is in the direction of reducing the concn. of O<sub>2</sub> and allowing a limited accumulation of CO<sub>2</sub>. Safe atm. range from natural air to 5 to 10% CO<sub>2</sub> and 2.5 to 13% O<sub>2</sub>. Humidity is largely self-controlling. Oiled wrappers control superficial mold or skin browning. Irvin C. Feestel.

Fruit products. IV. Experiments on the clarification of unfermented and fermented apple juice. Vernon L. S. Charley. *Univ. Bristol Agr. Hort. Research Sta. Ann. Rept.* 1934, 246-54. cf. C. A. 23, 223.—Pectin-decomposing enzymes (Pectinase and Filtragol) had a considerable effect in increasing the ease of filtration of apple juice which had been exposed to the action of the enzyme for 24 hrs. The effect on filtration was more important than differences in clarity in the stored juices or ciders caused by the enzymes. A proteolytic enzyme (Maltolysin) greatly increased the ease of filtration of mixed cull apple juice but had comparatively little effect on juice of the Amine Elizabeth variety. Excellent clarification of the juices was obtained by the addn. of gelatin 80-100 and tannin 40-50 g./100 gal. No outstanding differences due to the treatments were observed in the samples which had been bottled for 15 months. When apple juice, later fermented into cider, was treated with enzymes considerable removal of pectin occurred but this was not always followed by any appreciable clarification of the juice; filtration after 24 hrs. was carried out with much greater speed and economy when compared with juices which had not been treated with such enzymes. No significant differences were observed in the filtered samples. K. D. Jacob.

Experiments on the improvement of the juice from culinary and dessert apples by maceration with pressed bitter-sweet pomace. Progress report. II. P. T. H. Peckford. *Univ. Bristol Agr. Hort. Research Sta. Ann. Rept.* 1934, 209-12; cf. C. A. 23, 6885.—The bitter-sweet

character of Bramley's Seedling juice was considerably enhanced by contact with Dabinett pomace. No significant differences were obtained by pressing the juice immediately with the Dabinett pomace or by macerating the juice with the pomace for 14-26 hrs. The rate of fermentation of the macerated juices was approx. the same and was considerably slower than that of the untreated juice and of the juice which was pressed immediately with the Dabinett pomace. The treated juices contained less malic acid than did the untreated juice, but the treatments had no pronounced effect on the sp. gr. of juices or on the ciders made therefrom. K. D. Jacob

Vitamin C content of apples. S. S. Zilva, F. Kidd, C. West and E. O. V. Perry. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 164-5(1935)*; cf. C. A. 29, 242. —The vitamin C potency of the red peel of Bramley's Seedling apples is much greater than that of the green peel, the higher potency being located near the skin. Tests carried out on the "rosy" variety of Bramley's Seedling showed that the potency of the skin was not different from that of the blushed side of the "green" variety. No difference between the 2 varieties could be found with the pulp. Apples gathered in mid-Sept. were stored in pure O<sub>2</sub> and in pure N<sub>2</sub> (contg. less than 0.5% O<sub>2</sub>) in containers continuously ventilated with these gases at 1 in. up to the middle of Dec. Biol. tests carried out (after peeling) by the prophylactic method in doses of 3 g. failed to reveal any difference between the stored apples, their potency remaining that usually possessed by freshly gathered fruit. A. Papineau-Couture

Preservation of vitamin C in canning fruits and vegetables. S. S. Zilva, T. N. Morris and E. O. V. Perry. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 203-5(1935)*. —Ascorbic acid was added to runner beans (which do not contain much vitamin C), to spinach (a fairly good source of vitamin C), to apple and to apple jelly, with the aim of producing products which would have an antiscorbutic potency of the same order as a good natural source of vitamin C, such as the citrus fruits. After canning the products were examd. prophylactically by the biol. method, with the following results: *Runner beans*. —About 25% of the added ascorbic acid was destroyed, most of it had been taken up by the solids, the liquid being practically inactive. *Spinach*. —The solids alone showed definitely less activity than when the liquor was present; the loss of ascorbic acid was not estd. but could not have been great. *Apple*. —As with the beans, the loss was of the order of 25%. *Apple jelly*. —The loss was estd. at 10-20%. A. Papineau-Couture

Identification of ethylene among the volatile products of ripe apples. R. Gane. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 122-3(1935)*; cf. C. A. 29, 2322. —*Mimosa pudica* and *Ruscus communis*, both of which are sensitive to C<sub>2</sub>H<sub>4</sub> at concns. of 8-0 l p. m., showed the effects characteristic of C<sub>2</sub>H<sub>4</sub> when placed in a confined space with apples. Similar results were obtained with tomato plants and sunflower seedlings, less response was obtained from *Salvia splendens*. Apples at 15° were ventilated with a current of pure N<sub>2</sub> after 20 days O<sub>2</sub> was mixed with the issuing gas stream till it had the same N:O ratio as fresh air, and pea seedlings were grown in the resulting atm.; growth was normal in that the curvature and swelling which are characteristic effects of C<sub>2</sub>H<sub>4</sub> were absent. In another expt., in which bananas were used as the biol. indicator, acceleration of ripening occurred. It is as yet uncertain whether production of C<sub>2</sub>H<sub>4</sub> is completely stopped in the absence of O<sub>2</sub> or only greatly reduced. C<sub>2</sub>H<sub>4</sub> is not produced by ripe apples that have been killed by freezing, there is, however, enough C<sub>2</sub>H<sub>4</sub> present in the tissues of an apple frozen in a closed container to affect the growth of pea seedlings, provided that the thawed fruit and seedlings are again kept in a closed space. A. Papineau-Couture

Containers for apple juice and cider. I. Wooden vessels. A. The suitability of various coniferous woods. P. T. H. Pakford. *Univ. Bristol Agr. Hort. Research Sta., Ann. Rept. 1934, 213-16*. —Small samples of wood (prime clear British Columbian pine, Siberian red fir,

Archangel white fir, New Zealand kauri pine and Louisiana red cypress) were immersed for 6 months in bottles of blended, sharp, sweet and bittersweet ciders, resp., stored in a cool place. All the woods adversely affected the flavor of the ciders. Although none appeared to be of outstanding promise, it is possible that Archangel white fir and Louisiana red cypress may prove serviceable if some initial treatment can remove the woody flavor. K. D. Jacob

Cider. D. W. Stewart. *Chemistry & Industry 1935, 879-81*; cf. C. A. 28, 6212. —The chem. changes taking place during cider making are discussed. The presence of potash salts of org. acids and phosphoric acid contributes to the prophylactic and therapeutic value of cider. J. S. Hicks

Enzymic hydrolysis of starch in pectic extractions from apple pomace. Geo. L. Baker. *Del. Agr. Expt. Sta., Bull. 192 (Ann. Rept. 1934), 27(1935)*; cf. C. A. 28, 6371. —This report deals with mixts. of known amts. of pectin and sol. starch in dil. solns. Pectin has a natural inhibiting effect on diastatic enzymes. More diastase is required when the concn. of pectin in the soln. is increased. The optimum temp. for starch hydrolysis in pectin solns. is 30°. The optimum pH for the reaction is 3.0-3.5 when speed of hydrolysis of the starch without destruction of the jelling properties of the pectin is considered. The time required for complete hydrolysis is increased as the pH decreases. Hydrolysis of pectin is greatest at pH 4.0-5.0. The increased hydrolysis of pectin is due partly to the increased activity of pectic enzymes in the diastase prepn., and partly to the natural effect of the pH on pectin. Conditions favorable for the hydrolysis of starch are far from optimum when used in a pectic environment. C. R. F.

Vitamin G content of commercially canned tomato juices. Charles F. Poe and Esther L. Gambill. *J. Am. Dietetic Assoc. 11, 343-5(1935)*; cf. C. A. 29, 3417. —A detn. of the vitamin G content of 18 brands of tomato juice showed little variation from 100 units per lb.

Acidity and resistance to rotting in oranges. J. Barker, C. R. Furlong and N. E. Holmes. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 158(1935)*. —Acidity, total solids and rotting by *Penicillium* were detd. in navel oranges from Berri exptl. orchard (South Australia). In these samples, grown under the same climatic conditions and presumably having the same handling, high acidity was assocd. with low wastage, and with each decrease in acidity there was an increase in wastage. A. Papineau-Couture

The use of aluminum cans for fruits. T. N. Morris and J. M. Bryan. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 195-7(1935)*. —Canning tests were carried out on gooseberry, strawberry, raspberry, red currant, black currant, white cherry and black cherry with plain drawn Al cans and also similar cans that were given 1 coat and 2 coats, resp., of lacquer. All the plain cans became H-swells in a comparatively short period (many had gone in 1 month). In single-lacquered cans only strawberries have so far (4 months) shown any rot. The tests in double lacquered cans have not proceeded long enough for the results to have any value. Success with Al cans for fruits which give trouble is likely to be even more dependent on the perfection of the lacquer coating than in the case of tinplate. Comparison of ordinary rolled sheet Al and of "drawn" Al showed that the latter was corroded slightly more than the former in 0.5% citric acid, but the difference was not striking. A. Papineau-Couture

Corrosion of steel and tinplate by actual extracts of fruits. T. N. Morris. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 180-3(1935)*; cf. C. A. 29, 859. —Steel strips 3 × 1 in. were immersed for 3 days at 25° in sterile exts. of various fruits, the pH values of the exts. were measured, the loss in wt. of the strips and of H<sub>2</sub> produced were detd., and the theoretical H<sub>2</sub> was calcd. from the loss in wt. The most noteworthy results were: (1) although of relatively low acidity, cherry ext. of pH 3.1 formed a bronze-colored deposit which stopped further

corrosion, and when buffered with Na citrate to pH 4.07 it formed a loose yellow deposit which was nonprotective, (3) there appears to be a fairly close correlation between corrosion and the rate of formation of H<sub>2</sub>-swells and perforations by the various fruits tested. Addition of citric acid to cherry ext. reduces corrosion; addn. of 25% sucrose to the ext. slightly reduces corrosion over the whole pH range, but the reduction through the addn. of acid was of about the same order, whether or not sugar was present. Strips of template 3 × 1 in. were rubbed with emery to remove the Sn longitudinally from half the surface, and were immersed in fruit exts like the plain steel strips. The figures for dissolved Fe indicate the fruits which may be expected to give trouble through the formation of H<sub>2</sub>-swells, and those for dissolved Sn those fruits which either suffer severely from discoloration or cause heavy detinning of the cans, on the whole, fruits producing high corrosion of Sn produce low corrosion of Fe, and vice versa. A. P. C.

Experiments on the determination of the quality of potatoes. K. Rathack. *Ernahr. Pflanze* 31, 321-7 (1935).—New methods are given for the detn. of the cooking quality factor, the resistance to cutting, and the increase in firmness accompanying increases in the time of cooking. A loaded were allowed to cut through a cube of cooked potato of given diam. gives a measure of the resistance to cutting. A pos. relation exists between the resistance to cutting and the starch content. The value was neither influenced by the starch:protein ratio nor the protein or fiber contents. The value for the cooking quality factor decreases with the loss in wt. during storage but is independent of the starch content and the starch:protein ratio. Mech. changes in the starch grain during cooking are discussed. The mineral content of the tubers was the chief factor influencing the flavor. John O. Hardesty.

The vitamin C content of two varieties of Chinese amaranth, Hsien Ts'ai Hsiang-Chuan Hou. *Chinese J. Physiol.* 9, 253-6 (1935).—By chem. titration the av. value obtained for the red amaranth was 0.25 mg. of ascorbic acid per g. of the fresh vegetable and that for the green amaranth was 1.09 mg. By biol. assay the ascorbic activity of the red amaranth was about 1/4 of that of Sunkist orange juice, the potency of 2 g. of red amaranth being about the same as that of 1 cc. orange juice. One g. of the green amaranth exerted a somewhat greater antiscorbutic effect than 1 cc. of orange juice. L. A. M.

Laboratory control improves quality of carbonated beverages. Laurence P. Geer. *Food Ind.* 7, 541-2 (1935).—Bacterial and chem. control of containers, ingredients and finished products insure high-quality, sterile beverages. C. R. Fellers.

When standard analysis (in coffee) does not help. A. B. Kennedy. *Food Ind.* 7, 540, 572 (1935).—Pitfalls in the chem. analysis of coffees are outlined. C. R. F.

Microscopic methods for the detection of karaya gum, gum tragacanth and agar-agar. J. D. Wildman. *J. Assoc. Official Agr. Chem.* 18, 637-8 (1935).—Methods which have been used by W. for the microscopical detection of karaya gum and gum tragacanth in catsup, and agar-agar in presence of gelatin in mayonnaise and salad dressing, and in canned chicken are outlined. It is essential that the analyst first become thoroughly familiar with the properties of the various gums and with the foods in question, because no set procedure can be given which will apply to all products. It should be noted that the microscopic method does not apply to most sol. gums. A supplementary test for karaya gum consists in treating 1 drop of the product with 1-2 drops of chlorozinc iodide reagent, which stains the gum masses somewhat greenish, the U. S. P. I. test can be used for distinguishing agar-agar from the other gums generally used in foods. A. P. C.

Volatile oils in mace and nutmegs. Joseph F. Clivenberger. *J. Assoc. Official Agr. Chem.* 18, 161-16 (1935).—West Indian mace and nutmegs give a volatile oil which may be recognized by its low d and n and high α. Volatile oil distd. from nutmegs has a greater pos. α than that distd. from the corresponding mace, this may be due to a loss from the mace of the more volatile fractions of oil, which would give a greater α. Shriveled East India nut-

megs give a larger percentage of volatile oil than do the mature sound ones. The loss of volatile oil from ground mace and nutmegs is relatively rapid, amounting to approx. 80% in 2 months. The volatile oils obtained from ground mace and nutmegs that have been exposed in the lab. show a definite increase in d., n., acid and ester nos. and a distinct decrease in α, these results should be valuable in detg. the conditions under which these products are handled. The characteristics of the volatile oil should serve in the identification of unknown samples of mace and nutmegs. A. Papineau-Couture.

Production and marketing of groundnut in the Madras Presidency. K. Raghavachari. *Madras Agr. J.* 23, 39-62 (1935).—When they were stored in cans for 6 weeks, undried groundnut kernels became highly rancid (acid value 101-175) and were subject to attack by fungi and insects. Deterioration in storage was entirely prevented by drying the kernels at steam-oven temps. K. D. J.

The microscopic examination of cattle foods. Some oil-containing seeds. S. T. Parkinson. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 9-20 (1935), cf. C. A. 28, 68761.—The characteristics are given of the various portions of the seeds of castor-oil bean (*Ricinus communis* L.), croton (*Croton tiglium* L.), curcas (*Jatropha curcas* L.), candle nut (*Aleurites moluccana* Willd.), hemp (*Cannabis sativa* L.), kapok (*Eriodendron anfractuosum* D. C.), sesame (*Sesamum indicum* L.), cacao (*Theobroma cacao* L.), sunflower (*Helianthus annuus* L.), niger (*Guzotia oleifera* D. C.) and madia (*Madia sativa* Mol.). K. D. Jacob.

The composition, nutritional value and digestibility of pasture grasses, experimental investigation of grazing mares on the pastures. Hans Nietsch. *Landw. Jahrb.* 81, 525-75 (1935).—The fluctuation in the compn. of pasture grasses over a period of years was largely due to the weather conditions, the greatest fluctuations occurring in the wettest years. The digestibility of the grasses was decreased at this time, especially in relation to the crude fiber and N-free ext. content. Analyses made in May, July and September showed the highest crude protein in September, but the percentage of digestible protein did not vary with the season. K. C. Beeson.

The conservation of grass—hay, silage or dried grass. Arthur Crichton. *Trans. Highland & Agr. Soc. Scotland* 47, 50-63 (1935).—Samples of well-dried, good-quality, mixed pasture grass, cut in the summer and autumn, resp. contained dry matter 88.8, 90.3; protein 13.1, 17.9; oil 4.1, 5.9; sol. carbohydrates 44.5, 37.2; fiber 20.9, 20.8, and ash 6.2, 8.2%. K. D. Jacob.

Grass drying in hop oasts, 1933-4. A. H. Burgess and N. L. Tinley. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 119-21 (1935).—Pasture grass was cut on May 24th to June 5th (at approx. the flowering stage) and dried in 12-layers in hop oasts for 8 hrs. at 165°F. The dry material contained crude protein 13.66-14.56, EtO ext. 2.93-3.40, fiber 20.55-27.51, ash 6.46-7.45, carbohydrate 43.23-47.17 and H<sub>2</sub>O 5.20-7.86%. On storage of the material the H<sub>2</sub>O content increased to 16%. In an 8-week expt. with sheep the dried grass compared favorably with a mixt. of concentrates of equal analysis. K. D. Jacob.

Harvesting soybeans for hay. C. J. Willard, L. F. Thatcher and J. B. Park. *Ohio Agr. Expt. Sta., Bi-monthly Bull.* 175, 148-54 (1935).—Soybean hay should be cured to a moisture content of below 30% to insure freedom from storage deterioration. The protein content of the hay is lowest at the stage when the pods are just forming. After this stage, the protein slowly increases. C. R. Fellers.

High- vs. low-protein grain with pasture. Russell D. Horwood, G. W. Putnam and J. G. Wells, Jr. *Mich. Agr. Expt. Sta., Quarterly Bull.* 18, 22-4 (1935).—In a feeding trial cows fed a grain mixt. contg. 16.7% digestible crude protein produced 496 lb. more 4% milk than those receiving a grain mixt. contg. 9.3% digestible crude protein. The 3.6% difference is not significant. C. R. F.

Sweet clover silage as a feed for dairy cows. I. T. Atkeson and G. C. Anderson. *Idaho Agr. Expt. Sta., Bull.* 214, 3-11 (1935).—Feeding trials showed that sweet clover was practically equal to corn silage for body wt.,

milk and fat production in dairy cows The silage did not taint the milk or butter from the cows fed sweet clover.

C R. Fellers

**Vitamin A content of pasture plants** IV. White blossom sweet clover (*Melilotus alba* Desv.), orchard grass (*Dactylis glomerata* L.) and meadow fescue (*Festuca elatior* L.) under pasturage conditions and fed green Ella Woods, F W Atkeson, I W Slater, C. D. Arndt and R F Johnson *J Dairy Sci* 18, 639-45 (1935); cf. C. A. 29, 7520\*—Second year and first-year white blossom sweet clover contained, resp., 242 ± 10 and 500 ± 30 rat units of vitamin A activity per g when sampled under pasturage conditions and fed green. Under similar conditions orchard grass and meadow fescue contained, resp., 275 ± 13 and 250 ± 17 rat units Philip D Adams

**The preservation of silage** W. D Reid. *New Zealand J Agr* 51, 139-43 (1935)—Expts were carried out on the preservation of grass silage in drums and pits with addn of fresh whey, HCl, molasses and cultures of *Lactobacillus bulgaricus*, *Streptococcus thermophilus*, *S. lactis* and a cheese starter The quality of the silage was materially improved by addn of either *Lactobacillus bulgaricus*, whey, HCl or molasses The most economical treatments were whey (0.5-4.0%) and molasses (0.5-1.0%), either alone or combined Molasses treated silage usually had a pleasant odor, retained a freshly cut appearance, and when combined with *Lactobacillus bulgaricus*, HCl or whey reduced the objectionable acids and odors to a min

K D Jacob

**Comparative feeding tests on dairy cows, of ensilage prepared from sugar-beet tops and leaves, and grass silage prepared according to the Dutch method** J C de Ruyter de Wildt *Verlag landb Onderzoek Rijkslandbouw proefsta No 41C*, 211-61 (1935)—Sugar-beet tops and leaves were buried in a pit 4 X 10 X 0.5 m Thirty-three % of the org matter was destroyed in 3 months (Oct in Jan) Practically all of the carbohydrates were destroyed Feeding expts on dairy cows indicate that this material was equiv to grass silage, based on the total nutrients present in each feed W Gordon Rose

**Apple pomace silage** A I Perkins and C F Monroe Ohio Agr Expt Sta, *Bumonthly Bull* 175, 154 8 (1935)—Ohio apple pomace contains approx 21 % dry matter, 1.6% protein and 18.6% digestible nutrients It is readily ensiled either alone or along with corn silage Greater use of apple pomace is advocated as a cattle feed

C R Fellers

**Notes on locust meal as a poultry feed** F M Fronda Philippine Agr 24, 425-7 (1935)—The locust meal contained moisture 7.23, fat 17.27, ash 9.87, protein 30.72, fiber 16.32 and carbohydrates 18.59% A L Mehring

**Chemical research and the [packing] industry** (McDowell) 13 Paint in the food industry (Barry) 26 Ultraviolet light as an aid on the farm—feeding stuffs (Grant) 15 Anethole (residue as silage) (Vuihsenkel) 17 Date growing in Arizona (Albert, Hukeman) 15 Detn of Hg in leafy vegetables (Winkler) 7 N economy in agriculture (Virtanen) 15 Effect of temp changes on respiratory activity of apples (Iaves) 11D Bacteriol examn of oysters (Fisher, Acker) 14 Lecithin and oil mxts from soybean sludge (U. S. pat. 2,018,781) 27. App for irradiating milk, etc., with ultraviolet rays (U. S. pat. 2,018,332) 1. Inhibiting the interior corrosion of sealed metallic containers such as cans contg foods (U. S. pat. 2,018,682) 9 App for drying or wiping fruits to remove spray residues (U. S. pat. 2,019,087) 15 Emulsions [used as salad oil] (Brit. pat. 431,642) 13 App for condensing milk, fruit juices, vegetables or beef (U. S. pat. 2,018,049) 1. App for filling (tins contg foods with inert gas heavier than air (U. S. pat. 2,019,422) 1. Dust [flour, feed, starch] disasters (Tricey) 24

**Food preparations** Chemische Fabrik Hl Sander & Co A-G Ger 618,492, Sept 9, 1935 (Cl. 53: 4). Materials contg vitamins, e. g., bran or yeast, are extd. for several hrs with water at 55-60°, and the exts are steri-

lized by heating them several times to 60°. Oils or fats e. g., cod liver oil or oleomargarine, are then mixed with the exts to produce liquid or pasty food preps

**Food product** Léon Badosa-Guardiola. Fr. 784,551 July 22, 1935 Almonds are peeled, ground to a very fine powder, sugar and water are added and the whole is emulsified

**Caramel mixture for use in preparing foods** Teresa T Speed U S 2,019,305, Oct. 29. Dry, finely sub divided caramel of the highly hygroscopic, water-sol type is used with a more quickly sol., less hygroscopic material including sugar sufficiently dry not to cake when the mix is kept in a dry air tight container under com. conditions

**Containers such as fiber cans for foods, etc.** II Chester Aument. U S 2,019,242, Oct. 29. Fibrous material such as paper is used having its fibers said with a non-adhesive acacia gum App. is described

**Apparatus for manufacturing flakes, etc., from plastr materials containing vegetable matter, e. g., cereals** Axel U Sirmark. Brit. 431,504, July 2, 1935.

**Wheat flour** Edward J. Miller, Harry L. Goodwin and Edgar M. Miller U. S. 2,018,060, Oct. 29. Wheat berries are cracked to release the germ, the impure material and the germ are moved in a relatively thin layer through which a slight draft of air is uniformly forced so that impurities lighter than the germ and flour forming stock are carried away by the air, the aeration of the germ is maintained until the germ is set, and the germ and stock for the formation of flour are then crushed An arrangement of app is described

**Bread** Conrad Pietrkowski. Ir. 784,093, July 22, 1935 To the flour or dough is added a nourishing medium for the yeast in a form such that the yeast can consume it gradually and so produce a sufficient and durable amt of CO<sub>2</sub> during the fermentation and baking Thus, 100-500 g of a mixt contg glucose 20, sucrose 30 and raffinose 60% is added to 100 kg. of flour.

**Bread, milk products** Alexander Axelrod. Brit 431,087, July 1, 1935 Addn to 342,083 (C. A. 25, 4635) See Fr. 770,464 (C. A. 29, 517)\*

**Shortening agents for dough, etc.** Herbert Schou Brit. 431,065, July 1, 1935 Dough for use in making cakes, pastry, bread, etc., is mixed with a shortening agent consisting of an emulsion having a fatty continuous phase and an aq dispersed phase Suitable emulsions are prep'd by mixing an oil or fat, e. g., a mixt. of soybean oil and lard, with an oil-sol emulsifying agent, e. g., Palgaard's emulsion oil, then adding an amt. of H<sub>2</sub>O less than 0.2 the amt. of oil or fat and emulsifying the mixt. The H<sub>2</sub>O may contain sugar, salt or albumin and may be replaced by milk

**Apparatus for determining the fermenting power of yeast in dough** Brabender G m b H. Ir. 784,585, July 22, 1935

**Milk product suitable for use in making baked foods, etc.** Alexander Axelrod U S 2,018,391, Oct. 22 Skim milk is subjected to peptonization by use of *Micrococcus casei liquefaciens* or *Streptococcus liquefaciens* and to lactic acid fermentation, the lactic acid fermentation being prevented from detrimentally influencing the pepton formation by limiting the milk-sugar content of the milk and subsequently adding organisms producing lactic acid, and the product may be dried Various details of procedure are given

**Apparatus for irradiating milk, etc.** Henning A Trebbk (to Hanovia Chemical and Manufacturing Co.) Brit 432,011, July 18, 1935.

**Heat-exchange apparatus suitable for freezing or cooling milk, etc.** Clifford Morrow (to H. H. Miller Industries Co.) U S 2,018,462, Oct. 22 Various structural and operative details

**Evaporating milk** John B M Knutsen. Brit 432-075, July 19, 1935 Pre-evap'd milk from a vacuum concg unit is sprayed through a nozzle together with warm dry H from a generating system supplied to an annular ring of nozzles into a drying chamber wherein dried powd milk is deposited, the temp. of the milk in its

successive treatments being that of normal fresh milk. App. is described.

**Device for neutralizing sour cream.** Charley Jorgensen. U. S. 2,018,173, Oct. 22. A neutralizing agent is atomized with air which is then injected into a body of cream near the bottom of the latter. Various structural details are described.

**Packaging cheese.** Charles F. Doane. U. S. 2,018,337, Oct. 22. A large cheese, to prevent growth of mold in it, is cut into comparatively small pieces which are covered with material such as paraffin paper so as to leave a space around the pieces retaining preservative gases generated from the cheese, and the pieces are loosely contained and sealed in a container.

**Fish extracts.** Hans Schmalfuss and Hans Werner Brit 431,143, June 27, 1935. An ext. for human nutrition is prep'd from the flesh of whales, fish or other sea animals, preferably comminuted, by adding  $H_2O$ , and heating the mixt slowly to 80–96°. The liquid ext. is sep'd so as to obtain it free from fat and albumin by filtration through a moistened filter or by centrifuging and is conc'd. under reduced pressure.

**Apparatus for mixing air or inert gases with lard or the like.** Oscar C. Schmidt (to Cincinnati Butchers' Supply Co.). U. S. 2,018,618, Oct. 22. Various structural, mech., and operative details.

**Drying malt sirups, other sirups, vegetable juices, molasses residues, etc.** for use in feeds or fertilizers, etc. Lawrence W. Lewis. U. S. 2,018,797, Oct. 29. A non-hygroscopic product is obtained from a sirup or the like to which an alkali such as lime or NaOH is preliminarily

added to reduce the stickiness of the material, which may then be spray dried.

**Maraschino cherries.** Win. K. Tucker (to Calif. Packing Corp.). U. S. 2,019,030, Oct. 29. Cherries are subjected to the action of  $SO_2$  to reduce natural coloring matter, washed free from  $SO_2$  and then treated with a hypochlorite soln. to bleach out remaining coloring material.

**Invert sugar for making confections.** The Nulomoline Co. Ger. 618,324, Sept. 6, 1935 (Cl. 53/3). See Brit. 373,413 (C. A. 27, 3015).

**Processing coffee.** Patrick T. Clary. U. S. 2,017,892, Oct. 22. After roasting coffee beans, moisture is added (as by spraying water on the roasted beans) and the resulting steam is confined in contact with the roasted coffee beans, which also may be treated with added substances such as salt. App. and various operative details are described.

**Coffee substitute.** Eduard Jalowetz and Max Hamburg. Fr. 784,172, July 22, 1935. A cereal is steeped and aerated, then heated in a current of steam until the temp. in each grain reaches the value necessary for dextrinization or transformation of the starch to paste. The water in the grain is then evap'd. and the grain roasted.

**Cereal beverages.** Ronald B. McKinnis (to Continental Can Co., Inc.). Brit. 431,804, July 16, 1935.

Cereals are prep'd for ready extn. by subjecting the roasted cereal tissues mixed with saccharine material to mech. pressure for rupturing the cells without completely disintegrating them. Thus roasted wheat, rye, corn, etc., may be mixed with molasses and passed between hot rollers.

### 13—CHEMICAL INDUSTRY AND MISCELLANEOUS INDUSTRIAL PRODUCTS

(PLASTICS, RESINOIDS, INSULATORS, ADHESIVES, ETC.)

#### HARLAN S. MINER

**Chemical research and the chemical industry.** Charles H. MacDowell. J. Western Soc. Engrs. 40, 163–72 (1935).—A general review of the history of chemistry and research with particular reference to the development in the packing industry.

**Chemical industries in Finland.** Heikki Soim. Suomen Kemistilehti 8A, 80–9 (1935).—The present state of the industry and the possibilities for future development are discussed.

**Advances in the field of recovery of volatile solvents.** H. L. Piatti. Nitrocellulose 6, 47–9, 65–6 (1935), cf. C. A. 28, 2429<sup>a</sup>.—A review.

**Chemical warfare and its defense.** Ugo Zanini. Scienza farm. [2], 3, 90–4, 133–41, et seq. (1935).—General and descriptive.

**The initiation and development of the defense against gas, 1915–1918.** II. Evolution of the defense. Paul Murphy. Ind. Chemist 11, 353–6 (1935), cf. C. A. 29, 4476<sup>a</sup>.

**Possibility of using carbon monoxide as a war gas.** Mielenz. Gasschutz und Luftschutz 5, 263–7 (1935).—A discussion of Hanne's article on the same subject (cf. C. A. 29, 6324<sup>a</sup>). A large part of Hanne's article is quoted. M. disagrees with Hanne in the latter's conclusion that CO will occupy a prominent place as a war gas in future wars.

**Protection against toxic gases and dusts.** P. R. Weldon. Australian Chem. Inst., J. & Proc. 2, 220–36 (1935).

**Influence of the breathing resistance of a gas mask on the capacity of the mask wearer for performing work.** Heinrich Herbst. Chem.-Ztg. 59, 823–4 (1935).—Modern gas masks with smoke filters have breathing resistances of 15–18 mm. water compared to the 5–6 mm. of the older masks. H. feels that manufacturers must reduce this for industrial use of the masks, as the efficiency and capacity of the wearer is too low. According to H.'s work he classes masks with a breathing resistance of 20 mm. and more and 10% or less capacity for work of the wearer as unserviceable,

with 11 to 18 mm. resistance and 54–15% capacity for work as bad, with 7–10 mm. resistance as av., and 4–6 mm. resistance as good. Masks having 15 mm. resistance cut down the work capacity of the wearer after 5 hrs. to 1/2 that of the wearer of a 5-mm.-resistance mask.

**Engineering control of occupational diseases.** J. J. Bloomfield. Am. J. Pub. Health 25, 1196–1204 (1935).

**Bakelite's anniversary.** James A. Lec. Chem. & Met. Eng. 42, 540–3 (1935).

**Synthetic resins from hydrocarbons.** H. I. Waterman, J. J. Leendertse and H. L. Ligteneberg. Chem. Weekblad 32, 342–3 (1935).—A light cracked gas-oil distillate, initial b. p. 25°, 80% at 110°, contg. 40% crude benzene, Br no. 88,  $n_D^{20}$  1.4625,  $d_4^{20}$  0.8051, mol. wt. 75, was kept for 4 to 5 hrs. at room temp. with 3%  $AlCl_3$ . A 30–40% yield of resin was obtained; by distn. 40–50% light benzene distillate was recovered after a little  $H_2O$  had been added to the reaction product for hydrolysis.

Stable spongy emulsions can be made of up to 75% resin from the product by addn. of ammoniacal water. These emulsions can be worked up with ammoniacal rubber latex (75% rubber) and ppt'd. together by AcOH. The mixts. can contain up to 83% resin. From the powd. dried resin mixed with powd. wood (up to 75%) pressed objects are made. The resin is sol. in benzene, ether, turpentine and pyridine, not in EtOH, Me<sub>2</sub>CO or H<sub>2</sub>O. The results confirm work of Thomas and Carmody (C. A. 26, 6163).

**Electrical temperature control in alkyl-resin manufacture.** C. S. Ferguson. Am. Paint J. 19, 11, Convention Daily (Oct. 29, 1935).

**Frequentite, an insulating material for high-frequency technic.** E. Albers-Schonberg and J. Gringold. Siemens-Nachr. No. 10, 3–13 (1933). Referatkarites Silikalliteratur No. 537 (1934).—Frequentite, V282, is a ceramic material with a base of pure magnesium silicate. Details as to its properties and characteristics are given.

Mineral wool and vermiculite as insulation. John A. Schaeffer *Ind Eng Chem* 27, 1298-1303 (1935) — The relation of thermal insulation to air conditioning, which is in a rapid state of development, is discussed. The greatest advance in insulating materials has been in the use of mineral wools which are Ca silicates or Ca Fe silicates artificially changed to the fibrous state. They are unique in combining the properties of low cond., chem. stability, and ability to withstand high temp. The use of mineral vermiculite that has been expanded by heat as an insulator is increasing, especially in industrial installations. A table of the d and cond. of common insulating materials is given, also a three-dimensional chart showing cond. of mineral wool as a function of d. and mean temp.

R. H. Baechler  
Determination of the heat conduction in insulating materials. Bertil Stalham Tid Värme-, Ventilations- och Sanitetstekn. 6, 5-9 (1935). — A Ni wire is used in the detn. of the heat conduction of the material. The wire is electrically heated and the variation of the resistance of the wire is observed. With the aid of some theoretical calculus it is possible to det. the heat conduction. It is detd. for sand, concrete, cork, masonite and other materials with different water contents.

J. W. Holst  
A "soldering" paste and general cement. H. E. Watson *School Sci. Rev.* 17, 137 (1935). — Make collodion varnish by dissolving celluloid in Am acetate and stir in aluminum powder. The mixt. sets quickly to a fairly hard mass, and since it is plastic in the intermediate stage, it can be molded and worked neatly.

O. Reinmuth  
Summary of the most important literature references on wetting agents in chronological order. Kurt Brass and A. Beyrodt *Monatsh. Textil-Ind.* 50, 247 (1935). — A list of references covering the years 1913-35.

L. S.  
The age of mineral utilization. John W. Finch. *Mining Congr. J.* 21, No. 10, 10-11 (1935). — A statistical survey of the economic importance of metals and other mineral products in modern life.

A. L. Kaye  
Considerations in developing a mineral-wool industry. Charles F. Fryling and Orval White *Chem & Met Eng* 42, 550-3 (1935).

E. H.  
Detection of the break point in the determination of retentivity of activated charcoal. Franz Krczil *Z. ges. Schweiß-Sprengstoffwiss.* 30, 318-20 (1935). — Tests previously used are described and their limitations are discussed. The lower limits of detectability by odor or irritation of eyes or mucous membranes are given for a no. of solvents, industrial gases and war gases. An app. is described in which the increase in weight of an absorption vessel, caused by the absorption of the first traces of gas which pass the charcoal being tested, produces an elec. contact which warns the operator of the break point. The sensitivity of the app. can be varied over wide limits by varying the length of the cantilever spring.

A. L. Kibler  
A study of the mineral composition of mine dust. T. L. Walker *Can. Mining J.* 56, 470-8 (1935). — The proportions of the common minerals, particularly of quartz and muscovite, were detd. Various means of checking the minerals present is given. The particles consist mostly of those with a diam. of less than 3 microns. The percentage of quartz has been detd. by means of the x-ray. Also in *Univ. Toronto Studies, Geol. Ser.* No. 38, 5-11 (1935).

W. H. Boynton  
Stability of bleaching powder in packages, and of its preparations designed for use in war gas attacks. C. A. Royahn and K. Zietan *Pharm. Ztg.* 80, 958-60 (1935). — Numerous data are presented showing the behavior of bleaching powder, Dakin's soln., bleaching powder salves and other preps. in relation to war gas attacks.

W. O. L.  
Comparative x ray and dilatometric investigations on dental filling materials. K. Endell and U. Hoffmann *Zahnärztl. Rundschau* No. 37 (reprint), 6 pp. 1934; *Referat. Kartei. Zahnärztliche Literatur* No. 1017 (1935). — Filling materials used for teeth can be divided into 2 groups: (1) those which show a comparatively high expansion at 700°, but which practically do not expand on setting; (a) quartz filler with a 0.7% expansion at 700°, (b) quartz masses having

0.9-1.1%; (c) cristobalite masses with an expansion of 1.3% at 700°. (2) Filling materials with thermal and setting expansions: these materials have a total expansion of about 1.6%. Chem. analysis does not show in what crystal form SiO<sub>2</sub> is present, and only x rays show whether the filling contains quartz or cristobalite. The expansion of filling materials owing to setting and heating are shown in curves.

M. V. Kondody  
A nonflammable pyrethrum spray for use in airplanes. C. L. Williams and W. C. Dreessen *U. S. Pub. Health Repts.* 50, 1401-4 (1935). — A mixt. of 1 part pyrethrum ext. in kerosene (contg. 2% pyrethrins) and 4 parts CCl<sub>4</sub> (contg. no pyrethrins) killed 100% of the mosquitoes (*Aedes aegypti*) with 5 min. exposure. By ordinary tests this mixt. is nonflammable. Kerosene ext. contg. 0.4% pyrethrins is about as effective against *Aedes aegypti* as one contg. 2%.

J. A. Kennedy  
Red squill investigations. M. G. O'Connor, R. E. Buck and C. R. Fellers *Ind. Eng. Chem.* 27, 1377-8 (1935). — Red squill is an ideal and specific raticide. The chem. nature of the poison is unknown, so a biol. assay must be used for standardization. A simplified method is described. Rats preferred baits prepd. from meat, fish or cereal, and the addn. of various essential oils did not increase the amt. consumed. Canned red squill baits were found convenient and effective in field tests.

E. R. Rushton  
Effects of fumigants on paper. Charles G. Weber, Merle B. Shaw and P. A. Back *J. Research Natl. Bur. Standards* 15, 271-5 (1935) (Research Paper No. 828). — Normal fumigation with HCN gas, CH<sub>3</sub>CH<sub>2</sub>O, CCl<sub>4</sub>, CS<sub>2</sub>, CH<sub>3</sub>CH<sub>2</sub>O CO<sub>2</sub>, or HCOOMe-CO<sub>2</sub> had no significant

effects on representative book and writing papers. These effective fumigants, all of which are commercially available, can be safely used for ridding valuable books and documents of destructive insects.

W. J. Peterson  
Fire prevention and protection in chemical industries. Bernard F. Flood *Ind. Eng. Chem.* 27, 1303-8 (1935). G. G.

Polynuclear phenols and nitration and sulfonation products [in making resins and as wetting, dispersing or emulsifying agents] (Brit. pat. 431,945) 10 Acetone carballic esters [as plasticizers or modifying agents with synthetic resins] (U. S. pat. 2,017,882) 10 Cellulose ester films, plastic compps., etc. (Brit. pat. 432,404) 21

Gas masks. Reginald P. Howes. Brit. 431,671, July 12, 1935.

Plastic compositions. Studien- und Verwertungs-G. m. b. H. Ger. 618,182, Sept. 3, 1935 (Cl. 39b 22). A carbonaceous fuel, e. g., coal, brown coal, peat or lignin is treated at a temp. up to about 80° with a phenol and (or) an amine, e. g., PhNH<sub>2</sub>, with the addn. to the mixt., or to the product, of 3-5% of rubber, calcd. on the product. The product is then molded under heat and pressure, with or without addn. of fillers, etc. The rubber may be taken as a soln. or emulsion, and may contain a vulcanizing agent. Sp. processes are described.

Plastic compositions. Studien- und Verwertungs-G. m. b. H. (Franz. Ischer and Otto Horn, inventors) Ger. 618,231, Sept. 4, 1935 (Cl. 39b 22). A carbonaceous fuel, e. g., coal, brown coal, peat or lignin, is treated at about 80° with an org. base, e. g., PhNH<sub>2</sub>, (C<sub>2</sub>H<sub>5</sub>OH)<sub>2</sub>, or pyridine, and the product is molded under heat and pressure, with or without the addn. of fillers, etc. Sp. processes are described.

Plastic composition. John P. Henharen. Fr. 781,902 May 24, 1935. A compn. for making linoleum, oilcloth, siccatives, wall coverings, insulating compps., etc., is made by heating ordinary resin to about 140° until the AcOH is liberated, adding a catalyst and continuing the heating to about 282°, introducing rubber and agitating and heating until the rubber is dissolved. The catalyst is Co resinate or other appropriate linoleates or resins.

Films from plastic materials. I. G. Farbenind. A.-G. Fr. 784,283, July 22, 1935. The plastic material (cellulose deriv., casein, synthetic resin, etc.) is applied in powder form to the inner face of an endless band which passes round a molding cylinder, so that the powder is molded under high pressure and at any desired temp. to form a film which is removed from the band after molding.

Seasoning sheet pyroxylin plastics, etc. John H. Clewell and Lloyd V. Wemple (to DuPont Viscoloid Co.) U. S. 2,019,115, Oct. 29. For removing a water-sol. volatile solvent such as alc. from a material contg. nitrocellulose of a N content of about 10.4-12.2% and also contg. a plasticizer such as camphor, the material is subjected to a preliminary air treatment, then to a water treatment, and then to a final air treatment (various details of the treatments being described).

Artificial resins. Aug. Nawack A.-G. and Richard Hessen. Brit. 432,190, July 19, 1935. This corresponds to Fr. 763,580 (C. A. 28, 5639).

Synthetic resins. Victor H. Turkington and Wm. H. Butler (to Bakelite Corp.) U. S. 2,017,877, Oct. 22. A resinous reaction product which is compatible with linseed oil, etc., is formed from  $\text{CH}_3\text{O}$  and a hydroxybiphenyl such as *o*- or *p*-hydroxybiphenyl. Further details may be used.

Synthetic resin. Geo. W. Seymour (to Celanese Corp. of America) U. S. 2,017,993, Oct. 22. A synthetic resin which is compatible with org. cellulose derivs. is prep'd by treating  $\text{PhOH}$  100 with  $\text{SCl}_2$  175 parts at temps. below  $20^\circ$  and then removing uncombined  $\text{SCl}_2$  (as by evapn.) at temps. not substantially above room temps. and hardening the resin by fusing it.

Synthetic resin. Richard D. Kite (to General Elec. Co.) U. S. 2,019,349, Oct. 29. An aq. colloidal suspension of an alkyl resin which may be used as a binder with mica, etc., is obtained by heating in an aq. medium an alkyl resin which has not been converted to the final infusible form in the presence of clay.

Synthetic resins. Sydney L. M. Saunders. Brit. 431,951, July 18, 1935. Resins are prep'd by dissolving a fusible phenol-aldehyde condensation product in a partial ester of a monobasic org. acid with a polyhydric alc. and condensing the product with a polybasic org. acid or anhydride. The partial ester, which contains at least 2 OH groups, is derived from acids such as  $\text{AcOH}$  or  $\text{BzOH}$  or the acids derived from fats, vegetable oils or naturally occurring resins. The products, which are sol. in vegetable oils, may be used in the manuf. of varnishes and coating comps. In examples, a resin derived from *o*-cresol or *p*-tert-butylphenyl and  $\text{CH}_3\text{O}$  in the presence of an alk. catalyst is dissolved in glyceryl monolinoleate and the product condensed with phthalic anhydride.

Synthetic resins. Sydney L. M. Saunders. Brit. 432,103, July 22, 1935. Resins are prep'd by the condensation, in the presence of a heat-treated vegetable oil, of a polybasic org. acid or anhydride with a partially esterified polyhydric alc. contg. at least 2 free OH groups, the acid constituent being a monobasic acid derived from an oil or a natural resin, which resin, oil or partial ester may be heat-treated prior to the condensation. The products are sol. in heat-treated vegetable oils. Natural resins or their esters and dyes or pigments may be added. In an example, the monoglyceride of linoleic acid is condensed with phthalic anhydride in the presence of heat-treated wood oil to yield a product that may be dild. with a hydrocarbon and mixed with a drier to give a varnish.

Synthetic resins. Sydney L. M. Saunders. Brit. 432,158, July 22, 1935. Divided on 432,103 (preceding abstr.). Resins are prep'd by condensing a partially esterified polyhydric alc. contg. at least 2 free OH groups, the acid constituent being a monobasic acid, e. g.,  $\text{AcOH}$ ,  $\text{BzOH}$ , or an acid derived from a vegetable oil or a naturally occurring resin, with the product obtained from the incomplete condensation of a polybasic org. anhydride and a polyhydric alc. in the presence or absence of a vegetable oil, which may be heat-treated, a natural resin or an esterified resin. The partial ester may be heat-treated before condensation or it may be made from a heat-treated oil.

The products, in conjunction with pigments, dyes, nitrocellulose or cellulose acetate may be used in the manuf. of lacquers. In an example, a monoglyceryl ester of linoleic acid is condensed with an initial condensate of glycerol and phthalic anhydride and the product may be dissolved in a hydrocarbon with the addn. of a drier.

Synthetic resins. Wilhelm Kraus. Brit. 432,350, July 25, 1935. Condensation products are prep'd, by causing novolacs to react with condensation products derived from  $\text{CH}_3\text{O}$ ,  $(\text{CH}_3)_2\text{N}$ , and urea or mixts. of urea and other comps. that can react with  $\text{CH}_3\text{O}$ . The novolacs, and also the urea-aldehyde condensation products, are prep'd under acid conditions. The products may be mixed with filling or fibrous materials, e. g., cellulose, at any stage of the process of manuf. and then molded. In examples, a novolac prep'd from  $\text{PhOH}$ ,  $\text{CH}_3\text{O}$  and  $(\text{COOH})_2$  is mixed and caused to react with a condensation product of (1) urea (with or without the addn. of thionurea),  $\text{CH}_3\text{O}$  and  $(\text{COOH})_2$ , that has been subsequently mixed with  $(\text{CH}_3)_2\text{N}$ , or (2) urea,  $\text{CH}_3\text{O}$ ,  $(\text{CH}_3)_2\text{N}$ , and  $(\text{COOH})_2$ , with or without the addn. of urethan or acetamide, to yield resinous products. Cf. C. A. 29, 2261.

Synthetic resin. Soc. pour l'ind. chim. A. Bâle. Swiss. 175,914, June 1, 1935 (Cl. 41). Addn. to 172,079 (C. A. 29, 5543). A resin suitable for making lacquers, synthetic masses, etc., is obtained by condensing  $\text{PhNH}_2$  with linseed oil, subjecting the product to the action of anhyd.  $\text{CH}_3\text{O}$ - $\text{PhNH}_2$  and treating the resulting product with  $\text{CH}_3\text{O}$ . An acid condensing agent may be present.

Synthetic resins. Glasurit-Werke M. Winkelmann A.-G. (Richard Weithöner, inventor). Ger. 618,310, Sept. 6, 1935 (Cl. 12a 26 02). The manuf. of resins, by heating glycerol and phthalic anhydride with a drying oil and a monobasic org. acid, is improved by pretreating the mixt. with a small proportion of a halogen, e. g., 1-2%, calc'd on the oil. Alternatively, the oil may be pretreated with the halogen. The latter may be used in admixt. with an inert gas. Examples are given.

Synthetic resin molding compositions. Allgemeine Elektricitäts-Ges. Ger. 618,343, Sept. 7, 1935 (Cl. 39b 22). See Brit. 390,354 (C. A. 28, 6641).

Synthetic resins from aromatic amines and formaldehyde. Allgemeine Elektricitäts-Ges. (Hermann Burmeister, inventor). Ger. 618,110, Sept. 2, 1935 (Cl. 12g 9). Addn. to 598,444 (C. A. 28, 5694).  $\beta$ -Naphthylamine is used as the amine in the process of Ger. 598,444.

Resins from cracked tar and petroleum pitch. Clarence R. Wise and David F. Edwards (to Standard Oil Development Co.) U. S. 2,018,771, Oct. 29. The initial material is treated with  $\text{H}_2\text{SO}_4$ , anhyd.  $\text{AlCl}_3$ , anhyd.  $\text{FeCl}_3$ , or anhyd.  $\text{ZnCl}_2$ , the sludge is removed to obtain an acid tar, the latter is treated with solid adsorbent material such as decolorizing clay and is then subjected to vacuum distn. until a solid resin is obtained as a distn. residue. Cf. C. A. 29, 5897.

Resinous urea-formaldehyde products. Arthur M. Howard (to Toledo Synthetic Products, Inc.) U. S. 2,019,453, Oct. 29. An initial product is formed by heating urea with an excess of  $\text{CH}_3\text{O}$  in a slightly acid soln., the soln. is rendered slightly alk., addn. urea is added, and the soln. is conc'd by evapn. The resulting product is suitable for making molded articles.

Resinous condensation product of urea and formaldehyde. Barnard M. Marks (to Dupont Viscoloid Co.) U. S. 2,019,354, Oct. 29. A resinous compn. which is suitable for making molded articles is produced by causing urea 1 to react with  $\text{CH}_3\text{O}$  1.0-2.5 mols., with the compn. formed by reaction of  $\text{H}_2\text{S}$  with an aldehyde such as  $\text{CH}_3\text{O}$  in aq. soln. and having a  $\text{pH}$  of about 4.0-7.5 until a reaction product contg. combined S is obtained, and with addn. urea 1 mol. per each 1.0-2.5 mols. of the compn. Various details and modifications are described. Cf. C. A. 29, 6609.

Resinous coating compositions. Horace H. Hopkins (to E. I. du Pont de Nemours & Co.). U. S. 2,018,557, Oct. 22. A polyhydric alc.-polybasic acid resin or phenol- $\text{CH}_3\text{O}$  resin is dissolved in a solvent distd. from petroleum



refined by a cracking process, b. about 60-200° and having an aniline point of from about -5° to about 60° and a dist. value of about 15 to 35. Such solns are suitable for coating furniture, etc.

Composition of rubber and alkyl resin Herman L. Grupe and Roy H. Kiemle (to General Elec. Co.). U. S. 2,118,432, Oct. 22. See Can. 347,518 (C. A. 29, 3574).

Insulating coatings. I. G. Farbenind. A.-G. Fr. 784,770, July 22, 1935. Polyvinyl chloride having a Cl content of 62-5% is used for coating metal wires or other conductors.

Insulating oils N. V. de Bataafsche Petroleum Maatschappij. Fr. 784,540, July 22, 1935. Oils for cables, condensers and transformers are made by adding to appropriate mineral oils unsatd. aliphatic hydrocarbons (10%), particularly those obtained by cracking or dehydrogenating mineral oils or products from mineral oils.

Producing insulation on conductors Siemens & Halske A.-G. Brit. 432,407, July 25, 1935. Wires and similar elec. conductors are insulated by extruding a bundle of fine threads or bands of polystyrene, a cellulose deriv. or similar substance from a press, carrying the bundle and the conductor through a stranding nipple or analogous device and thence over a draw-off device to a winding appliance, and stranding the threads or bands together around the conductor by the relative rotation of the draw-off and winding devices with respect to the extrusion orifices.

Heat- and sound-insulating articles Soc. italiana Pirelli. Ger. 616,930, Aug. 8, 1935 (Cl. 80b, 9.20). Addn. to 541,477 (C. A. 29, 1994). See Brit. 414,934 (C. A. 29, 6704).

Sound-absorbing and heat reflecting insulating material suitable for refrigerator cars, etc. Geo. A. Nicol, Jr. (to Geo. A. Nicol Corp.). U. S. 2,019,233, Oct. 29. A relatively thick layer of flexible, compressible, cellular sound-absorbent material such as hair felt is used with a layer of metal foil such as Al foil and with an intervening layer of adhesively secured material such as paper.

Insulating coverings for pipes, etc. Frederick W. King (to Cape Asbestos Co. Ltd.). U. S. 2,019,417, Oct. 29. Various mfg. details are described for making insulation from a fibrous material such as asbestos and a binder such as Na silicate.

Insulating and cooling composition suitable for use in transformers, etc. Frank M. Clark (to General Elec. Co.). U. S. 2,019,229, Oct. 29. A mineral oil is used with about 0.5% of maleic, succinic, malonic or adipic acid, which serves to inhibit sludge formation.

Mineral wool heat insulation Howard J. O'Brien (to Johns-Manville Corp.). U. S. 2,019,021, Oct. 29. A moisture-resistant material comprises loose mineral-wool fibers thinly coated with a mixt. of an oleaginous liquid such as fuel oil or paraffin oil and a normally solid water-repellant fatty acid compd. such as Zn stearate, etc.

Weatherproofing textile-insulated conductors Leslie P. Lamplough and Curtis E. Plass (to Western Elec. Co.). U. S. 2,018,494, Oct. 22. In a continuous operation (in a described app.), the conductor is first impregnated with a heated asphaltic compd., then slightly cooled and treated with a coating of heated liquid wax, and then treated with "anti-sticking" material such as powdered mica.

Dielectric composition suitable for use in transformers, capacitors, switches, etc. Frank M. Clark (to General Elec. Co.). U. S. 2,019,238, Oct. 29. A chloroolefin such as a chloroethylene and a chloropolyphenyl such as pentachlorophenyl or the like are used together. Cl. C. A. 29, 4104.

Dielectric material suitable for use in capacitors, etc. Frank M. Clark (to General Elec. Co.). U. S. 2,019,237, Oct. 29. A semi-rigid nitrochlorophenyl having a dielectric const. of about 7 to 8 is obtained by subjecting a mixt. of isomers of halogenated polyphenyls to a nitrating mixt. of HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> for 2 to 15 hrs. at a reaction temp. of about 100-110°.

Adhesive sheets Minnesota Mining and Manufacturing Co. Brit. 431,500, July 10, 1935. Divided on 427,653 (C. A. 29, 6330\*) and addn. to 405,247 (C. A.

28, 4191\*). An adhesive for use in prep. the adhesive transparent or translucent sheets or films described in 405,247 consists of a mixt. of a vinyl resin, e. g., the polymerization product of vinyl chloride and vinyl acetate, and a PhOH-ald-hyde resin. The resins are dissolved in, e. g., ethylene glycol or diacetone alc. and may be mixed with nitrocellulose soln. and a plasticizer, e. g., dibutyl phthalate. The coated surface is made adhesive by application of heat or of a solvent, e. g., gasoline, CCl<sub>4</sub>, CCl<sub>2</sub>, ethylene dichloride.

Transparent adhesive tape. Urtam Combault and Société Industrielle de la Cellulose (S. I. D. A. C.). Brit. 431,201, July 1, 1935. This corresponds to Belg. 392,738 (C. A. 29, 5493) but excludes the use of natural or synthetic resins contg. a plasticizer.

Casein products. The Casein Manufacturing Co. of America, Inc. Fr. 784,298, July 22, 1935. Casein is mixed with a small amt. of water or other appropriate plasticizing agent and an alkali or salt or one or more other solubilizing agents. The mass is mixed under a high pressure, with or without heat until a modification of the casein takes place. The mass is extruded, dried and ground to a powder which is easily sol. in water and may be used for making glue and other products.

Patterning artificial leather, oil cloth or the like Karl Fischer (to Oelrich Vazirsh. Corp.). U. S. 2,012,853, Oct. 22. A light-sensitive emulsion such as one contg. AgBr and gelatin is dried, powdered and mixed with a varnish, the mixt. is applied to the surface to be patterned or decorated, exposed to light and then is photographically developed and fixed in the presence of a varnish solvent and then dried.

Condensation product Soc. pour l'ind. chim. à Bâle. Swiss 174,963, Apr. 16, 1935 (Cl. 41). Addn. to 172,089 (C. A. 29, 5548\*).

A low-mol. nitrogenous product sol. in low-boiling solvents is obtained by condensing polymethylphenol (obtained by condensing PhOH with CH<sub>3</sub>I) and PhNH<sub>2</sub>, about 1 mol. of PhNH<sub>2</sub> being used to each PhOH residue, and the condensation being stopped when a test shows that the product is no longer clearly sol. in alc. The product is freed from volatile constituents by a stream of inert gas at 70°. The product is a resin sol. in acetone or in an alc.-C<sub>6</sub>H<sub>6</sub> mixt.

Condensation products of urea or its derivatives and formaldehyde. Soc. pour l'ind. chim. à Bâle. Ger. 618,427, Sept. 7, 1935 (Cl. 12a, 17.05). Processes in which urea or its derivs. and CH<sub>2</sub>O are condensed in the presence of active C are improved by using forms of active C which are capable of adsorbing acid electrolytes. Wood charcoal activated with steam at 800-1000°, and certain varieties of bone black, are suitable. The C may alternatively be added to the solns. of the reagents and removed before the solns. are mixed. Cl. Brit. 233,094 (C. A. 29, 2539).

Urea derivatives Imperial Chemical Industries Ltd. and Henry A. Piggott. Brit. 432,279, July 22, 1935. Urea is condensed with 2 or more (up to 55) mol. proportions of ethylene oxide (I) and the products are acylated with an acid derived from a natural fat or fatty oil. The products are useful as *tearful assistants* particularly as *teeling and emulsifying agents*. Among examples, urea is heated with 30 times its wt. of I and the product is heated with stearic acid, the acylated product may be added to an indigo vat, whereby slightly greener dyeings, much faster to rubbing, are obtained.

Synthetic materials Kurt Albert G. m. b. H. Chemische Fabrik Fr. 784,158, July 22, 1935. Condensation products of amides and aldehydes, cellulose derivs., proteinoplasts, amine-aldehyde condensation products, "novolacs," natural and synthetic resins, etc., or mixts. of aliphatic or aromatic amines and hardenable condensation products contg. phenols and carbonyl, are hardened by heat until a gel is formed which may be cut. The gel is cut into thin flakes or fragments which are fused together and molded with heat and pressure or simply heat.

Coloring polymerization products I. G. Farbenind. A.-G. (Walther Lütkenhoff, Hans Fikentscher and Heinrich Hopff, inventors). Ger. 618,000, Aug. 30, 1935

(Cl. 39b, 4 02). The polymerization products of compds. contg. one double bond, e. g., vinyl esters, acrylic esters, or acrylic nitrile, are colored by treatment with aq. suspensions of water-insol. or sparingly sol. dyes known to be suitable for dyeing cellulose esters. Aminoanthraquinones, nitroaromatics and aminoazo compds. are among the classes of dyes specified. Products obtainable by polymerizing mixts. of the olefinic compds. with other compds., or the products obtainable by after-treating the polymerization products, e. g., by chlorination or reaction with aldehydes, may also be colored. The products may be treated in the form of films, filaments, sheets, tubes, etc. Sp. processes are described.

**Emulsifying and other agents.** *Chemische Fabrik vorm. Sandoz.* Swiss 175,866-870, July 1, 1935 (Cl. 36a). Adds to 171,359 (C. A. 29, 5548<sup>1</sup>) A cleaning, washing, emulsifying, dispersing or softening agent is made by treating olive oil with a mixt. of  $H_2SO_4$  and the glycerol ester of  $H_2SO_4$  (175,866). The olive oil may be replaced by coconut oil, (175,867), castor oil, (175,868), coconut oil fat acid, (175,869), or naphthene acid (175,870).

**Emulsions.** Reinhold O. Bratke. Brit. 431,642, July 8, 1935.  $H_2O$  is dispersed in oils, fats, resins, pitches, etc., by adding to the  $H_2O$ , prior to or during the mixing, about 0.01% of the principal substance of aq. alkali, e. g.,  $NaOH$ ,  $KOH$ ,  $NH_4OH$ , having dissolved therein aromatic hydrocarbon derivs. or their salts sol. in alkali, e. g.,  $BzOH$ ,  $Na$  salicylate,  $o$ -,  $m$ - or  $p$ -resol. The products may have pigments or solid substances incorporated therewith for use as paints, color varnishes, printing inks and lubricants. Among examples, 350 g  $H_2O$  contg. 0.015 g.  $NaOH$  and 0.0015 g.  $BzONa$  are stirred at  $30^\circ$  into 1000 g. olive oil, the product may be used as salad oil. Cf. C. A. 28, 5163<sup>1</sup>.

**Wax emulsions.** I. G. Farben und A.-G. Tr. 784,614, July 22, 1935. Wax emulsions such as polishes and cleaning compds. are made stable as regards color and appearance by adding during or after their prepn a colorless substance liberating  $O$  easily, such as peroxides, persalts, alkali salts of nitrobenzoic, nitrophthalic or nitrobenzenesulfonic acids.

**Compositions containing collagen products.** Wilfred G. Dewsbury and Arnold Davies. Brit. 431,360, July 5, 1935. Glue or gelatin is treated with a mineral, animal or vegetable oil at  $130^\circ$  until a mass of fibers seps., the fibers are sepd and then dispersed in an aq. medium. The aq. dispersion may be mixed with glue as a size or to increase its setting time, with ice cream to control the formation of ice crystals, as a protective colloid with latex to check coagulation, e. g., on conen or on addn of asbestos fiber, as a stabilizer with emulsions, e. g., oil-in- $H_2O$  emulsions contg. pigments, with cream for whipping or with preserves, jams or meat exts., with the fillers or colloidal  $S$  in rubber, or with the  $H_2O$  used in wet-grinding, e. g., of graphite to be used in the impregnation of fibers to be used as elec. resistances, or of  $S$  for vulcanization.

**Colloidal dispersions.** Richard Werner. Ger. 618,269, Sept. 4, 1935 (Cl. 12e, 4 01). For the manuf. of colloidal dispersions of silicates, tungstates and other poor elec. conductors by the elec. dispersion method, use is made of electrodes in which one or more metal cores are surrounded by the poor conductor, which is itself enclosed in an outer metallic sheath.

**Reversible gels.** Soc. pour l'ind. chim. à Bâle. Swiss 177,270, Aug. 1, 1935 (Cl. 41). Carbamides and  $CH_3O$  are condensed, the condensation not being allowed to proceed beyond the hydrophile stage, in a neutral medium in the presence of a solvent at temps. in the region of  $100^\circ$ . The resulting soln. is dried to give a water-sol. powder capable of forming a reversible gel. In the example, urea is condensed with  $CH_3O$  in the presence of active C and water.

**Jelly-forming materials** such as carob-kernel flour, gum acacia, etc. Rudolf G. Schulz (to Henkel & Cie. G. m. b. H.). U. S. 2,019,303, Oct. 29. To prevent jelly-forming org. substances of various kinds from forming lumps when swelling or dissolving, the finely pulverized material is heated to about  $141-180^\circ$  in the presence of

about 3% of moisture for a sufficient time to form coarser, porous aggregates, without effecting any substantial gelation and without forming a soln. Boiling xylene may be used as a heating medium.

**Cleaning composition.** Paul Antenen and Arnold Brunner. Swiss 175,610, May 16, 1935 (Cl. 15c). Oil of turpentine, sulfonate, cyclohexanol,  $NH_4OH$ ,  $AcOAm$ ,  $MgO$  and water are mixed together.

**Use and cleaning of dry-cleaning liquids.** Wm. H. Alton (to R. T. Vanderbilt Co.). U. S. 2,018,507, Oct. 22. Dirty dry-cleaning liquid is mixed with a filtering material such as pyrophyllite of graded sizes and the resulting mixt. is passed to a filter where the larger sizes of the graded material build up a filtering foundation supporting the finer particles to insure a fine straining action on the passing liquid.

**Detergents.** Robert D. Grant. Brit. 432,140, July 22, 1935. A cleansing prepn is composed of soft soap, a gritty or abrasive substance, e. g., sawdust, ground wood or cork, an animal or vegetable fatty oil, a perfume and  $H_2O$  to form a paste.

**Washing composition.** Gottfried Held, *Chemische Fabrik.* Swiss 176,622, July 1, 1935 (Cl. 24d). A compn. as above contg. a ferment contains at least one protective colloid which does not arrest the action of the ferment, and in such quantity that it exerts an emulsifying and washing action. In an example, the compn. consists of  $Na$  laurate,  $NaCl$ , calcined soda and pancreatin.

**Washing and other agents.** *Chemische Fabrik vorm. Sandoz.* Swiss 174,511 to 174,514, Apr. 1, 1935 (Cl. 36a). Adds to 171,359 (C. A. 29, 5548<sup>1</sup>) A cleaning, emulsifying, dispersing and softening agent is obtained by treating sperm oil with a mixt. of  $H_2SO_4$  and glycerol- $H_2SO_4$  (174,511). The sperm oil may be replaced by spermaceti (174,512). Spermaceti may be treated with fuming  $H_2SO_4$  in the presence of a mixt. of monoxylglycerol ether isomers (174,513). Finally, bees wax may be treated with a mixt. of  $H_2SO_4$  and glycerol- $H_2SO_4$  (174,514).

**Coating compositions.** Colloidal Colours Ltd. and Reginald Graham Durrant. Brit. 431,288, July 4, 1935. Colored materials for coating surfaces, e. g., of slates, tiles, glass, porcelain, concrete, brickwork, are made by prep. sols from hydroxide of Fe and (or) hydroxide of Cr and incorporating therewith inorg. powders serving as coloring matters without acting as electrolytes or having any adverse effect upon the sols. The powders, e. g.,  $Fe_2O_3$ , green oxide of Cr, black oxide of Mn, ochre, smalt, may be incorporated with the liquid sol and the mixt., if desired, dried or they may be mixed with the already dried sol. For application, the mixt. is stirred with  $H_2O$  and, after application, the sol may be gelled by applying an electrolyte, e. g.,  $Na$  silicate soln. Cf. C. A. 29, 5244<sup>1</sup>.

**Coating articles with cellulose acetate.** Charles J. Nurse. Brit. 431,616, July 11, 1935. In molding coverings on knitting pins, dress buckles, etc., the cellulose acetate is placed around the article which is then heated in an oven until the cellulose acetate becomes plastic, the heated aggregate being then transferred to and pressed between dies at a lower temp. than that of the oven so that the cellulose acetate at its inner faces, which are to be joined, is more plastic than at the faces in contact with the dies. App. is described.

**Coating metals.** The British Thomson-Houston Co. Ltd. Brit. 431,816, July 16, 1935. A metal, e. g., steel plate, core, foundation or reinforcement, is united with a covering layer composed of textile fibers united by an artificial resin, e. g., a phenolic condensation product, by applying and baking onto the metal a coating of enamel such as used in enameling wire, applying the textile material and uncured artificial resin and curing the resin. The textile fibers may be loose or batted or in the form of threads or woven or felted fabric. A steel wire enameled by applying and baking on several coatings consisting mainly of linseed oil, china-wood oil, coal-tar oil and a solvent such as kerosene is passed through a soln. of uncured artificial resin and wound with cotton thread, which may be impregnated with uncured artificial resin. Further layers of resin and thread may be applied, and the wire

baked to cure or to cure partly the resin. Wire thus coated may be used as a reinforcement in the construction of a spinning pot for rayon from fibrous textile material and artificial resin.

Transparent or translucent sheet material. See also Crystall. Ir. 784,213, July 22, 1935. Textile fibers are impregnated with a 30% soln of polystyrene, and, after evapn of the solvent, are treated in a 2nd hot bath contg 50% of pure polystyrene resin. The product is dried and compressed while heated under 25-40 kg per cm<sup>2</sup> to form smooth sheets.

Rust-preventing composition. I. Bernhard Wurbs. Swiss 176,041, June 17, 1935 (Cl. 37g). The compn consists of an alk. reagent, Fe powder and at least one other powdered metal of the same electromotive series as Fe. The alkalinity of the compn is about equiv. to a 0.25 to 0.005 N acid soln. A varnish may be added to the compn. In an example, the compn. consists of Pb powder, Fe powder, K<sub>2</sub>CO<sub>3</sub>, white lead and varnish.

Plywood. Geo. R. Meyercord and Charles E. Rozema (to Reconstruction Finance Corp.). U. S. 2,018,733, Oct. 29. A layer of thermostetting resinous material such as a phenolaldehyde product and a layer of non-resinous adhesive material such as dried blood is placed between each ply and the next inner ply, the proportion of resinous material to the non resinous material decreasing from the outer faces of the assembly toward the center, and the materials are subjected to heat and pressure.

Wood ply. George H. Osgood and Russell G. Peterson. U. S. 2,019,036, Oct. 29. Wood plies are united by a vegetable hemicellulose-contg. adhesive which contains a sufficient excess of caustic alkali and CS<sub>2</sub> to soften the wood fibers along the glue line so that the natural acids of the wood react on the cellulosic materials to form a waterproof bond.

Gluing panels of wood veneer or the like under pressure. Charles B. Norris (to Reconstruction Finance Corp.). U. S. 2,018,736, Oct. 29. A pressure equalizing pad for use in a hot press is formed of a casing such as thin sheet steel and filled with metal of low m. p. such as an alloy of Pb and Bi.

Fiber container with metallic ends suitable for holding oils, greases, etc. Pierre Heyndrickx. U. S. 2,019,412, Oct. 29. An oil-resisting coating such as casein hardened with formal is used on the inner walls and ends.

Shoe counter material. Fred L. Ayers (to Brown Co.). U. S. 2,018,245, Oct. 22. A partially shaped counter is formed essentially of a felted fibrous base impregnated with rubber (various mfg. details being described).

Foamed slag. Dorman, Long & Co. Ltd. and John S. Lewis. Brit. 431,647, July 9, 1935. H<sub>2</sub>O, in the form of a jet or the like, is delivered into contact with the under surface of molten slag falling through space to a collecting device. App. is described.

Composite mica tubes suitable for resisting high temperatures. Willis A. Boughton and Wm. R. Mansfield (to New England Mica Co.). U. S. 2,017,943, Oct. 22. A sheet of flexible mica and a binder such as Na metaphosphate soln is rolled into a cylinder, the cylinder is provided with interior and exterior coatings of carboniz-

able material such as kraft paper and with a plant non-combustible outer covering such as Cu foil and is heated on a noncombustible core such as a steel rod to carbonize the carbonizable material and render the tube readily separable from the core and from the outer covering.

Stencil sheets. Wm. G. D. Orr (to A. B. Dick Co.). U. S. 2,018,501, Oct. 22. A backing sheet is used with a type-impressible sheet impregnated with a colored material such as one which is dark blue and a homogeneous sheet of elastic material such as cellophane having a waxy color-bearing coating on each side, the coating on one side being colored to yield offset copies of applied impressions and the coating on the other side being colored to give contrast between the stenciled impression and the undisturbed portions of the type impressible sheet.

Friction-testing apparatus suitable for testing brake linings, etc. Sydney G. Tilden (to Raybestos-Manhattan, Inc.). U. S. 2,018,688-0, Oct. 29. Mech. features.

Photomechanical printing processes. Bekk & Kaulen chem. Fab. G. m. b. H. Ger. 615,773, July 12, 1935 (Cl. 57d, 2 03). Addn. to 605,298 (C. A. 29, 1184). A chromate or dichromate of a nonvolatile alkali, e. g., Na or K, is used as the sensitizing agent in the process of Ger. 605,296.

Sterilizing and proofing bristles. The Pro-phy-lac-tic Brush Co. Brit. 431,780, July 5, 1935. Toothbrushes, etc., or the bristles from which they are made, are rendered self-sterilizing and mold-proof by impregnation with a soln. of a phenylmercuric salt, e. g., the nitrate or chloride, in H<sub>2</sub>O or other solvent, e. g., dioxane, AcOAm, EtOH, Me<sub>2</sub>CO or AcOEt. Before impregnation, the bristles may be treated with an oxidizing agent, e. g., H<sub>2</sub>O<sub>2</sub>, and, after impregnation, they may be treated with a stabilizing agent, NaCl or NaI. Alternatively, the bristles may be immersed in a bath contg. 2 or all 3 of the treating agents. The bristles may also be treated with a waterproofing agent, e. g., paraffin, ceresin, carnauba wax or an ester or ether of cellulose.

Lamp shades, etc. Deutsche Gasglühlicht-Auer-Gesellschaft m. b. H. (to Decca A-G (Auergesellschaft)). Brit. 432,145, July 22, 1935. A lamp screen, shade or reflector is provided with a coating contg. a Nd compd. or is made of a transparent or translucent material, other than glass, contg. a Nd compd. Among several methods described, a translucent paper, cardboard or fibrous material is soaked in Nd NH<sub>4</sub> nitrate and then coated on both sides with lacquer, or the soaking soln. may be mixed with the binder, e. g., wheat starch, turkey gum or gelatin, and the Nd salt then deposited in solid form by evapn.

Preserving cut flowers. I. G. Garbend A-G. Ir. 784,350, July 22, 1935. A soln. is used contg. a food for the flowers, such as cane sugar, and a substance preventing putrefaction such as org. compds. of N contg. at least one aliphatic radical of high mol. wt. attached to N or substitution products thereof, e. g., dodecylamine-HCl, dimethylbenzyl dodecylammonium chloride and dimethyl dodecyltrichlorobenzyllammonium chloride.

Fireproofing paints or glues. Marcel Flinaux and Paul Kuhler. Fr. 784,649, July 22, 1935. These are made fireproof by the addn. of Wassy cements.

## 14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

Mineral waters of Castrocara. Maria Bacchini. *Scienza farm.* [2], 3, 107-18(1935).—Analyses of waters from the different mineral springs of the Castrocara region are given.

City of Manchester Rivers Department. Ann. Rept. for year ending March 31, 1935. S. P. Dawson. 44 pp., 1935.—The sewage-purification works have functioned satisfactorily. The extensions at the Davyhulme Works have been completed and the new activated sludge units are now in full commission.

Edward Bartow  
Travelogue-depositing waters near Lexington, Virginia. Edward Steidtmann. *Science* 82, 333-4(1935).—The

waters are supersatd. with Ca(HCO<sub>3</sub>)<sub>2</sub> throughout the year, the excess ranging from about 68 to 76 parts CaCO<sub>3</sub> per million. Adjustment resulting in deposition of calcite is hastened by rise in temp., aeration and the presence of calcite. Excess of deposition of CaCO<sub>3</sub> over CO<sub>2</sub> suggests that much of the CaCO<sub>3</sub> which appeared to be in soln. was really in a cryst. colloidal state.

P. S. Roller  
The water supplies of German large and middle-sized cities. E. O. Stein. *Gesundh.-Ing.* 58, 600-602(1935), cf. C. A. 28, 7385.—Data, mostly of economic significance, for 27 cities are reported in tabular form and discussed.

M. G. Moore

Regime of salts in waters of the *Alexandretta sandjak* Vladimir Frolow. *Comp. rend.* 201, 613-15(1935); *J. C. A.* 27, 2308.—The elec. resistance of the waters of 21 springs or streams in the sandjak of Alexandretta (Syria) has been detd. at various times during 1932-4. The results show close connection with the rainfall and the nature of surrounding rocks. C. A. Silberrad

Acid mine drainage control on upper Ohio River tributaries. E. S. Trindle and E. W. Lyon. *J. Am. Water Works Assoc.* 27, 1181-88(1935).—Recently a mine-sealing program went into effect (C. A. 29, 1900) and after 9 months is considered successful. Two hundred and fifteen mines were closed and after 9 months 75% of the acid has been removed from their drainage. D. K. French

Chemical hazards in water-works plants. M. C. Smith, et al. *J. Am. Water Works Assoc.* 27, 1225-48(1935).—The handling and utilization of Cl and adverbial first and only is considered. Care in handling is stressed. Containers should be stored standing vertically and prevented from falling over. First aid and treatment for irritant gas poisoning, etc., is discussed. Twelve precautionary recommendations in regard to Cl handling are given. D. K. French

The relation of activated carbon to water purification. C. H. Billings. *Sanitation Water Works J.* 17, No. 4, 11-13 (1935).—Tests are given on 6 activated carbons. The best C for water treatment should remain longest in suspension and have a high phenol removal. Detailed methods of testing are given. O. M. Smith

Methods for determining traces of heavy metals in mineral waters. K. Heller, K. Kuhla and F. Machel. *Mikrochemie* 18, 193-222(1935).—According to Mikolok, C. A. 28, 2957, 3153, 3214, the heavy metal content of a mineral water serves to identify the probably source of the spring. A scheme of analysis is outlined for detg. as little as 0.01 mg. of Cu, Pb, Cd or Zn per l. These ions were concd. by shaking with  $\text{CCl}_4$  + dithione (cf. Fischer and Leopoldi, C. A. 28, 2953) and the final detns were made by the polarographic method (cf. Heyrovsky, C. A. 27, 5270) with a dropping Hg electrode. W. T. H.

Determination of traces of lead in drinking water. J. F. Reith and J. de Beus. *Z. anal. Chem.* 103, 13-27 (1935).—See C. A. 29, 5303. W. T. H.

The use of sodium pyrophosphate combined with metaphosphate in the determination of lead in drinking water. P. Karsten. *Chem. Weekblad* 32, 391-3(1935).—To avoid difficulties in Fe pptn. by pyrophosphate, metaphosphate is added to give the correct pH condition for Pb detn.; per 80 cc. water contg. less than 20 mg. Ca, 10 cc. 10% Na metaphosphate is added, then 1 g. Na pyrophosphate, 10 cc. ammoniacal  $\text{NH}_4\text{Cl}$  soln., and 2 drops of  $\text{Na}_2\text{S}$  soln. Cu is eliminated if necessary by adding 4 drops of 10% KCN soln. to the above. This procedure allows simple colorimetric detn. of Pb even in very hard water and in the presence of up to 20 mg. Fe per l. B. J. C. van der Hoeven

Step-photometric determination of manganese in drinking water and in service water. R. Baril. *Mikrochemie* 18, 250-55(1935).—Schmidt's method (C. A. 22, 1202) which depends upon the formation of a reddish violet compd. when quadrivalent Mn is treated with dimethyl-p-phenylenediamine is suitable for photometric measurement. The color, to be sure, is not sp. for Mn but it is easy to avoid interference by other oxidizing agents likely to be present in water. Full directions are given for carrying out the test with 100 cc. of water. W. T. H.

Step-photometric determination of free chlorine in chlorinated water. L. Goldenberg. *Mikrochemie* 18, 255-49(1935).—The colorimetric method of Ellms and Hauser (C. A. 8, 880, 2900) with an acid soln. of o-toluidine as reagent was studied with the photometer. A table was prepd. showing the Cl corresponding to each 0.1 in the drum readings ( $\text{Cl} = 0.54 - 0.077 \text{ mg. per l.}$ ) and possible interference studied. W. T. H.

Colorimetric method for the determination of dissolved oxygen. F. Wellington Gikreas. *J. Am. Water Works Assoc.* 27, 1166-77(1935).—A colorimetric method is

described; amido which is considered a valuable addn. to methods of water and sewage analysis is used. Only 2 reagents and a limited amt. of equipment are necessary. Certain oxidizing and reducing substances interfere, and the test while relatively accurate will probably never be as accurate as the standard Winkler method. D. K. F.

Results of algal activity, some familiar, others obscure. W. C. Purdy. *J. Am. Water Works Assoc.* 27, 1120-33 (1935).—Pure dissolved O is the result of algae growth.

This is produced only in sunlight, and is sufficient to support bacterial growth. In the absence of algae, bacteria soon exhaust the dissolved O, but in mixts. O production always exceeded depletion. Oxygen so produced has a vapor pressure about 5 times that of atm. O. D. K. French

Determining the bacteriological quality of drinking water. J. K. Hoskins and C. T. Butterfield. *J. Am. Water Works Assoc.* 27, 1101-9(1935).—The use of larger portions than 10 cc is considered desirable. The need for a more dependable method than the dilute plate method seems reasonable. D. K. French

Limiting standards of bacterial quality for sources of purified water supplies. H. W. Streeter. *J. Am. Water Works Assoc.* 27, 1110-19(1935).—Where waters require filtration the average *Es. coli* index should in no case exceed 5000 per 100 cc. and should not exceed 20,000 per 100 cc. during more than 5% of the period, as for instance a year. Where waters are simply chlorinated the av. *Es. coli* index should not exceed 50 per 100 cc. and should not exceed 400 per 100 cc. more than 5% of the time. D. K. F.

Preparation of fine anthracite coal for use in filter plants. F. L. Buller. *J. Am. Water Works Assoc.* 27, 1181-5 (1935).—Heavy refuse is first dropped out by water and then by shakers. There is also some ash removal. Singing follows. D. K. French

Corrosion control with a marble filter. Henry Ryan. *Water Works and Sewerage* 82, 360-1(1935).—Filtration through coarse crushed marble (99%  $\text{CaCO}_3$ ) will raise the pH of a soft water, high in  $\text{CO}_2$ , and materially reduce "red water" difficulties. G. H. Young

Progress report of committee on tentative methods for testing zeolites. Charles P. Hoover, et al. *J. Am. Water Works Assoc.* 27, 1178-83(1935).—Methods are advanced for the detn. of grain size and percentage of fines, weight per cu. ft. as received, resistance to mech. wear, rates of  $\text{Al}_2\text{O}_3$  to  $\text{SiO}_2$  and exchange value. D. K. French

Assistants for working with hard water. Fritz Ohl. *Spezial u. Wasser* 53, No. 33, 8-11(1935).—For industrial purposes water is softened either in special installations or by the use of washing softeners. The 2nd method is less expensive and hence more suitable for the *laundry industry*. These water softeners are (1) soda, (2) alkali bases, (3) mixts. contg. soda, water glass or other silice acid compds., (4) the same plus soap; (5) the same plus borax,  $\text{Na}_2\text{PO}_4$ , etc., with and without soap, (6) Na phosphates or alkali salts of silicic or phosphoric acids. The use of all of these softening agents is discussed. The following com. powders of German manuf. used as water softeners have these compos.: *Berol-Ursoda* is soap, soda and a mixt. of cyclic alcs. *Herby* is soda (free from Cl) and water glass. *Suro* is soap, soda, borax and water glass. *Trimalgon* washing soda is soap, soda and water glass. *Tyfo* is soda and water glass (it consists of ammonia soda,  $\text{Na}_2\text{PO}_4$ , and water glass). Various phases of water softening are discussed. Leopold Scheffan

Water softening on steamships. An interesting plant for the "Queen Mary." *Am. Water and Water Eng.* 37, 537-8(1935).—A combined lime and base-exchange plant is used. W. A. Moore

Diesel-engine exhaust used in water softening. Paul Stegeman. *Water Works Eng.* 88, 1182-4(1935).—The exhaust, contg. approx. 9%  $\text{CO}_2$ , is used for recarbonation. By this means a pH of 8.2 can be maintained in the treated water. No objectionable odors or tastes are imparted to the water nor is it necessary to scrub the gas before use. W. A. Moore

Routine analysis of boiler feedwater sources. S. H. Coleman. *Paper Plant Eng.* 39, 577(1935).—Rooting

analyses should be made not only of the water in the boiler but of the water in various parts of a system and raw water sources. Such analyses may show variations in the raw water or leaks in a circulation system that, when found, can be corrected. Edward Bartow

Mottled enamel Crawford A. McMurray. *Southwest Water Works J.* 17, No. 4, 18-19 (1935) —Ninety-eight of 400 children between the ages of 6 and 13 years developed mottled enamel by using water exclusively that contained 4.4 p.p.m. of F. O. M. Smith

Chemical sewage treatment in America. Imhoff. *Gesundh.-Ing.* 58, 599-600 (1935); cf. C. A. 29, 5208.

M. G. Moore

Features of the new northside sewage-treatment works of Durham, N. C. Wm. M. Platt. *Water Works and Sewerage* 82, 337-43 (1935). G. H. Young

Experiments in chemical sewage treatment at Liberty, N. Y. W. A. Hardenbergh. *Public Works* 66, No. 10, 31-2 (1935) —Original experiments with chem. application 1 1/2 miles upstream to effect mixing and coagulation were unsatisfactory, an air mix tank installed at the plant site gave satisfactory results with chem. treatment, as follows:

(1) "Ferrisul" and chlorinated coppers gave satisfactory effluents but necessitated lime and Cl application at the upstream dosing point, (2) "Blackalum" gave satisfactory results without lime-Cl dosage upstream, and the final Cl dosage could be materially reduced, (3) application of activated C direct to the sludge digesters (a) increased digestive activity, (b) gas production, (c) removed sludge odors and (d) gave a faster drying sludge. A combination of 2 and 3 became standard practice. G. H. Young

The new sewage treatment plant of Walden, N. Y. Newell L. Nussbaumer. *Water Works and Sewerage* 82, 355-8 (1935). G. H. Young

State Hospital sewage-disposal works. Henry Ryon. *Public Works* 66, No. 10, 33 (1935) —Operating statistics from the Poughkeepsie (N. Y.) State Hospital plant.

G. H. Young

The problem of sewage treatment at Brunn. Friedrich Frodl. *Tech. Gemeindeblatt* 38, 222-4 (1935) —The need for treatment in view of the very low dilution at times afforded by receiving streams and proposed types of treatment are discussed. *Ibid.* 245-52 —Graphical and tabular data important in the planning of a sewage-treatment plant are discussed, including the amt. of sewage flow, the phys. and chem. character of the sewage, the variation of these factors with the time of day and year, industrial wastes (which here are not of a troublesome nature) and the possible utilization of sewage sludge, etc.

M. G. Moore

Chemical mechanical treatment of sewage. IV. (a) Mechanical filtration, (b) sludge treatment. Philip B. Streaender and Michael J. Blew. *Public Works* 66, No. 10, 15-16 (1935), cf. C. A. 29, 75404\* —Under (a) the authors discuss (1) advantages of filtration, (2) filter types, (3) filtration rates and influencing factors, under (b) they discuss treatment and disposal, with factors influencing (1) sludge vol. and (2) concn. G. H. Y.

Sewage dangers. Marcel Berthier. *Tech. sanit. munic.* 30, 228-34 (1935) —The very toxic nature of H<sub>2</sub>S gas in sewers and its frequent occurrence are noted. A subacute intoxication is possible at exposures of several hrs. at concns. of 5-20 p.p.m.; or 5-10 min. at concns. of 50-60 p.p.m. The irritation first is noticed in the eyes and mucous membranes and in severe cases results in pulmonary edema. Anaerobic putrefactive fermentations in sewers form large quantities of the gas CO<sub>2</sub> and CH<sub>4</sub>, are also found in sewers but are less important in a public health sense than H<sub>2</sub>S. Measures of security are outlined. C. R. Fellers

Experience with chlorination of activated sludge. L. F. Smith. *Water Works and Sewerage* 82, 362-4 (1935) —Soln. feed chlorination effectively controlled bulking difficulties at the Lima (Ohio) activated sludge plant, in addn., reduction of compressed air requirements as a result of chlorination were such that an estd. annual saving of \$320-\$1500 can be realized. G. H. Young

The biological treatment in water of settled sewage.

F. E. Giesecke and P. J. A. Zellar. *Southwest Water Works J.* 17, No. 6, 11 (1935) —An exptl. lake of about 14 acres was constructed at College Station, Texas, to handle 300,000 gals. daily of settled sewage with a biochem. O demand of 475 p.p.m. The lake effluent had a biochem. O demand of 80 p.p.m., thus supplying 75 lb. of O per acre per day. O. M. Smith

The manufacturing, testing and use of diffuser media. Frank C. Roe. *Southwest Water Works J.* 17, No. 4, 14-15 (1935). —See C. A. 29, 66791. O. M. Smith

Stream pollution and textile wastes. C. D. Blackwelder. *Mech. Eng.* 57, 631-2 (1935) —See C. A. 29, 4114\*. E. H.

Bacteriological examinations of oysters and water from Narragansett Bay during the winter and spring of 1927-28. L. M. Fisher and J. L. Acker. *U. S. Pub. Health Repts.* 50, 1449-75 (1935) —The quality of the water is better in winter and early spring than it is in the late fall. The quality of the oysters also tends to improve at these times. This tendency on the part of the oysters is perhaps more marked but results are likely to be more erratic. There is a general tendency in northern oyster-growing areas during the marketing season for oyster scores to increase as water scores increase, and vice versa, when viewed broadly. In individual comparisons, differences may be extreme. A marked improvement in the quality of oysters occurs within about 1° of the f.p. This improvement was much more consistent at these lower ranges of temps. than at other cold-weather ranges. Also in *Sewage Works J.* 7, 727-41 (1935). J. A. Kennedy

The efficacy of various insecticidal sprays in the destruction of adult mosquitoes. J. A. Sinton and R. C. Watts. *Records Malaria Survey India* 5, 275-306 (1935) —Adult mosquitoes in houses were effectively controlled by spraying the atm. with a mixt. of 1 part of Pyrethroid 20 (a proprietary concd. ext. of pyrethrum) and 19 parts of kerosene with the addn. of either oil of citronella 5, oil of sassafras 0.5-1.0 or oil of pine 4-5%. The efficacy of the spray seemed to increase with a rise in temp. and to decrease with a rise in relative humidity. The spray seemed to be less effective against certain culicines than against certain anophelines. At a concn. of 1 cc./225 cu. ft., the spray was highly toxic to mosquitoes in 30 min. When the spray was used at a concn. of 1 cc./100-150 cu. ft., persons in a closed room experienced in about 5 min. a distinctly oppressive feeling which developed into a mild headache with occasionally a slight irritation of the nasal mucosa. In concns. of approx. 1 cc./200 cu. ft., except for a slight oppressive feeling, the symptoms were slight, while in concns. of approx. 1 cc./350-400 cu. ft., very little unpleasant effect was noted in a closed room up to 30 min. K. D. Jacob

Larvicides and a method for temporary protection from adult mosquitoes in limited areas. Joseph M. Ginsburg. *New Jersey Mosquito Extermination Assoc., Proc. 22nd Ann. Meeting* 1935, 147-51; cf. C. A. 29, 30807\* —A larvicide suitable for use on hard and salt waters is prepd. by mixing 6 lb. Gardonol W. A. Concentrated with 50 gal. water and then thoroughly agitating with 100 gal. kerosene contg. 2-3 lb. wool grease and enough pyrethrum ext. to equal 100 lb. flowers (contg. at least 0.9% pyrethrins). For spraying, the stock soln. is dild. with 10 parts water. The spray is effective against mosquito pupae and gives temporary control of adult mosquitoes when it is applied to grass, shrubs, etc. K. D. Jacob

Mosquito suppression work in Canada in 1934. Arthur Gibson. *New Jersey Mosquito Extermination Assoc., Proc. 22nd Ann. Meeting* 1935, 77-81 —Tray tests were carried out with the larvae and pupae of common species of *Aedes*. With oils applied at the rate of 1.5 gal./acre the kills of larvae in 18 hrs. were for kerosene 70, stove oil 63, fuel oil 48, crude petroleum 33 and used crankcase oil 9%. The prepn. of satisfactory oil emulsions was facilitated by the addn. of 0.25-6.00% of pine oil but the killing efficiency of the mineral oils was not improved thereby. When it was used at the rate of 1.5 gal./acre, an emulsion prepd. by emulsifying equal parts of fuel oil and 5% soap soln. and dild. with 20 parts of water killed

an av. of 83% of the larvae. The larvicidal value of fuel oil-soap emulsions, with or without the addn. of pine oil, was materially increased by the addn. of derris powder (0.25 lb./gal.). The larvicidal value of oil emulsions was also increased by the addn. of pyrethrum powder (1 lb./gal.). The oil emulsions were completely effective against mosquito pupae. Sulfite liquor (1 part/240 parts water) was ineffective against larvae and pupae, and lime S (1.100 and 1-200) was not harmful to larvae in 20 hrs. A 1:8000 soln. of 40% nicotine sulfate gave 100% kill of larvae in 17 hrs. but had no effect on pupae. A soln. of laundrey soap 1 in water 1600 parts gave 100% kill of larvae and 17% kill of pupae in 17 hrs. Dusts prep'd by mixing powd. derris (5% rotenone) 1 and infusorial earth 100 parts and applied at the rate of 1 lb. derris per acre gave complete control of larvae and pupae in 60 hrs. Infusorial earth impregnated with oil gave excellent results in destroying larvae and pupae when used in dosages per acre as little as 3-4 gal. oil. The use of oil exts. of pyrethrum instead of oil alone in prep'g the dusts gave markedly better results. Mosquito repellents for application to the clothing and skin are described. K. D. Jacob

Hygiene in lino-type rooms. A study of working conditions. W. Vaje and H. H. Weber. *Schriften Gesamth. Ges. d. Gewerbe 100*, 44, 1-44 (1935). *Arch. Reichsgesundh.* 68, 247-92 (1935).—A special portable app. for det'n. of Pb in air is described. The min. dangerous concn. (presumably 1 mg. per 4000 l.) was not found in any parts of the rooms. However, precautions are advised

in connection with collected dust on floors, etc. The CO<sub>2</sub> content did not exceed 0.5% and was considered without influence. The highest CO content recorded was 0.013%, which was considered insufficient to cause acute symptoms. R. P. Walton

Paints and preservatives for engineering structures [water or sewage-treatment plants] (Dodd) 26.

Base-exchange agents. Soc. anon. d'ite "Mutosol" Soc. Holding Luxembourg. Swiss 175,664-5, May 16, 1935 (Cl. 36g). A method of causing deposition of salts in soln. in water consists in having at least two base exchangers each with a different cation sep'd. diagonally in a container. Thus, in prep'g (AcO)<sub>2</sub>Pb, zeolite is sat'd. with Pb(NO<sub>3</sub>)<sub>2</sub> and a 2nd quantity of zeolite is sat'd. with (AcO)<sub>2</sub>Ca. The exchange of bases yields (AcO)<sub>2</sub>Pb.

Filter. Heinz Diekmann. Swiss 176,637, July 1, 1935 (Cl. 43). Details of a filter tower for sewage water are given.

Cleaning sewage water. Heinz Diekmann. Swiss 176,638, July 1, 1935 (Cl. 43). Sewage sludge is dried, pow'd. and used as an adsorption filter for cleaning sewage water. The dried sludge may be mixed with peat or lignite. Cl. C. A. 29, 59531.

Automatic tide gate and sewage regulator for sewage systems. Edwin R. Schofield. U. S. 2,018,383, Oct. 22. Various structural, mech. and operative details.

## 15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

M. S. ANDERSON AND K. D. JACOB

Ceylon soils. V. Soils associated with limestone. A. W. R. Joachim and S. Kandiah. *Trop. Agr. (Ceylon)* 85, 67-77 (1935). Cl. C. A. 29, 81931.—Chem. and phys. data relative to the classification of these soils are given. VI. Some forest soils of the wet low-country. A. W. R. Joachim and D. G. Pandittesekere. *Ibid.* 146-56.—Profile characteristics and analytical data on 7 forest areas are given. John O. Hardesty

The soils of Tadzhikistan. M. A. Pankov. *All-Union Sci. Research Cotton Inst.* (Tashkent, U. S. S. R.) 1935, 1-128.—A series of chem. analyses on the chernozem-like soils, dark and light gray soils, chestnut soils, alluvial and solonchak complexes. J. S. Joffe

A soil survey in the valley of the Great Stour, Kent. W. O. Sharp. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 183-4 (1935).

Relationships of roots, soil profile and irrigation in the Sudan. F. E. Kenchington. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 135-82 (1935).—The chem. and phys. properties of Sudan soils are discussed with particular reference to the occurrence, origin, nature and effects of soil salts and org. matter. K. D. Jacob

[Report on] biochemistry. N. Craig. Mauritius Dept. Agr., *Sil. Ann. Rept. Sugarcane Research Sta.* 1934, 21-39; Cl. C. A. 29, 22711.—Base-exchange capacity of soils.—The base-exchange capacity of the inorg. absorbing complex varied greatly in different types of soils. In the mature soils the clay from the most highly laterized samples had an extremely low capacity for base exchange, the capacity increased as the degree of laterization decreased. The exchange capacity of the org. absorbing complex remained practically the same in all the mature soils. In the highly laterized mature soils the clay exerted a very minor influence on the base-exchange properties of the whole soil, the predominating influence being that of the org. matter, which, wt. for wt., absorbed approx. 20 times more bases than did the clay. With mature soil of low degree of laterization the exchange capacity of the clay was more than twice that of the clay in the highly laterized zones; in these soils the inorg. fraction was as important as the org. fraction in exchange relationships. The base-exchange capacity of the org. fraction of the mature soils, the immature soils and the gray soils was

fairly const., around 250 milliequiv. % org. matter. In the mature soils, passing from the wet to the dry regions, the base-exchange capacity of the clay fraction increased from about 13 to 25 milliequiv. % clay and in the immature soils from about 21 to 70 milliequiv. %, while for the Médine gray soil it amounted to 90 milliequiv. %. Phosphate status of Mauritius soils.—When the org. complex in immature highly leached soils was destroyed by heating the sample at 550° for 8 hrs. the P ext'd by Truog's reagent was approx. the same as that ext'd. by 1% citric acid from the untreated soils; Truog's reagent ext'd. less P from the untreated soils than did 1% citric acid. The evidence indicates that Truog's reagent exts. only inorg. easily available P whereas 1% citric acid also exts. org. soil P. There was evidence that in the more acid soils, where exchangeable bases are not very plentiful, there is a tendency for P to be fixed in the org. form, but in the more neutral soils, where exchangeable bases are plentiful, the P tends to be associated, with these bases. Data are given on the relation between moisture and the phys. properties of Mauritius soils and on the nitrifying power of Mauritius soils. Admixt. of 10% of subsoil with the surface soil had no deleterious effect upon the soil microorganisms and the nitrification process was not suppressed in mixts. contg. 50% of subsoil. Mauritius subsoils are not toxic to soil microorganisms, but merely sterile, and the depression of the nitrification process is due to diln. of the active surface soil with the sterile subsoil. K. D. Jacob

Soil reaction and plant growth. James Hendrick and Walter Moore. *Trans. Highland & Agr. Soc. Scotland* 47, 34-49 (1935).—Under conditions in the north of Scotland the optimum pH ranges for the growth of ordinary crops are: sugar beets and peas 6.0-7.5, barley 5.8-7.5, red clover 5.5-7.5, wild white clover 5.5-7.1, wheat 5.5-7.0, turnips 5.2-6.5, oats 4.5-6.2, potatoes 5.0-6.2 and swedes 4.8-6.0. The soils of the northeast of Scotland usually have pH values of 5.5-7.0. The results of plant-growth tests on soils of different pH values are given.

Chemical nature of organic matter or humus in soils, peat bogs and composts. Selman A. Waksman, J. *Chem. Education* 12, 511-19 (1935).—H<sub>2</sub>O-sol. carbohydrates, starches and simple N compds. are the first

plant constituents to decompose in soils or composts, followed by free proteins, pentosans and cellulose. The lignins contain hemicelluloses, notably polyuronides and resins. These are resistant and tend to accumulate. Fresh wood products such as oak leaves, pine needles and cypress wood are high in lignin and polysaccharides but are low in N. Cereal straw and corn stalks are high in carbohydrates, and low in N. Leguminous plants are high in N and low in lignin. Humus constituents of decomposed plant remains consist of a varying proportion of lignin or lignin derivs and protein accompanied by a less stable group of resistant carbohydrates, fatty and waxy substances. Low moor peat contains nearly 70% of the ligno protein group compared with 36% in the original plant material. Corresponding values for high moor peat are 36 and 13%, resp. The humus in chernozem soils is characterized by its content of Ca and Mg. In arid soils the humus is high in protein and has a narrow C/N ratio. Acid humus is considered to differ primarily from mild humus in the nature and extent of base satn. of the exchange complex. The function of humus in plant nutrition is discussed.

Irvin C. Feustel

The organic matter content and the carbon nitrogen ratio of South African soils of the winter rainfall area. W. I. Isaac. *Trans. Roy. Soc. S. Africa* 23, 205-30 (1935).—Soil N was detd. by the Kjeldahl method, soil org. C by the  $\text{SO}_2$  reduction method of Robinson, and org. matter by C X 1.724. The estn. of soil org. matter by the formula  $\text{N} \times 20$  was shown to be entirely unsuitable for the soils investigated. Five soil groups were studied: Somerset West, Dwaarsriviers Hock, Egin District, Kistenbosch and Bredasdorp District. The C/N ratios of these soils ranged from 11.2 to 22.9 with an av. for the 12 soils of 16.6, and only 2 soils had ratios of the order 10-12.1. A C/N ratio of 15.1 is regarded as typical of the winter rainfall region soils (cultivated) of the South-west Cape. In passing from a soil to subsoil, with one exception (Krom River Farm of the Egin District) there was found a marked decrease of org. C and N, and this was accompanied by a narrowing of the C/N ratio. The av. of the 6 subsoils studied was 14.7. On the whole, the org. matter content and the C/N ratio of the cultivated soils are lower than those of the virgin soils. No correlation was found between the av. org. matter content and the C/N ratios of the soil groups and the av. annual rainfall.

K. C. Beeson

The organic matter content and the carbon nitrogen ratios of some semiarid soils of the Cape Province. W. I. Isaac and B. Gershl. *Trans. Roy. Soc. S. Africa* 23, 245-54 (1935).—Three soil groups, Whitehill, Karroo Poort and Ouberg Pass, were studied. An av. C/N ratio of 11.7 was found on 6 soils. By eliminating a soil from the Whitehill group that receives more moisture than normal for the region the av. is reduced to 10.0. The av. percentage of org. matter in the soil was lower than that of the winter rainfall soil series. The org. matter varied from 0.55 to 1.51% and averaged 0.96%, while the C/N ratio varied from 5.0 to 13.5. The soils were neutral to slightly alk. in reaction. Investigation of a Whitehill soil showed that a reduction in org. matter from 0.75% to 0.4% occurred in the subsoil. This was accompanied by a decreasing C/N ratio.

K. C. Beeson

Research at Rothamsted of importance in horticulture. Mary D. Glynn. *Sci. Horticulture* 3, 215-221 (1935).—The N value of dried poultry manure was somewhat inferior to the equiv. of  $(\text{NH}_4)_2\text{SO}_4$  in the first year of the expt. In expts with green manures, neither tares nor mustard ploughed in was able to maintain N fertility for the following wheat crop. A fertilizer trial on peas showed a definite yield increase from application of 224 lb. Nitrochalk per acre while  $\text{K}_2\text{SO}_4$  gave a small depression on gravel soil. Potash produced a striking improvement in Brussels sprouts on a heavy medium soil. Expts with kale demonstrated the capability of leafy crops to utilize large doses of quick acting N up to 672 lb. Nitrochalk per acre. A no. of tests on root crops showed that intensive cultivation when a crop is growing has a somewhat depressing action after the land has been freed from weeds.

Investigations on the decomposition of plant materials showed that the fungus tissue itself is a readily available source of N. Inoculation of plants with certain mild viruses appears to protect the plant from effects of more virulent strains. A mixt. of some mild viruses with certain other viruses produces a disease more severe than either component. Incorporation of antioxidants with tale pyrethrum and kieselguhr-pyrethrum dusts retards loss of activity as a poison due to light and air exposure. Tannic acid, pyrocatechol, resorcinol, hydroquinone and pyrogallol offer some protection.

I. C. Feustel

Organic soils and epinastic response. W. Neilson Jones. *Nature* 136, 554 (1935).—From certain infertile org. soils a gas may be exd. (by aeration, centrifuging or heating) which produces on tomato plants epinastic curvatures of the petioles similar to those brought about by ethylene. Exts. from such soils when added to water culture soils produce similar epinastic curvatures on cuttings and influence the rooting of the cuttings and the manner of root growth of cuttings and seedlings. The intensity of the effects may be seasonal.

Philip D. Adams

The apple-growing soils of Tasmania. I. A general investigation of the soils. C. G. Stephens. *Australian Council Sci. Ind. Research Bull.* 92, 7-31 (1935).—The 10 soil types in all the main apple growing areas of Southern Tasmania are classified and described. With the exception of the alluvial and Woodbridge soils, all are of a podsollic nature. The mech. analyses and reaction of these soils are given in an appendix. These soils can be divided roughly into 3 groups, the first having a mean  $\text{pH}$  value between 5.7 and 5.9 for surface soils, the second  $\text{pH}$  4.8 and the third  $\text{pH}$  6.3 to 6.5. Except for a few of the more acid and heavier samples it appears that these soils readily respond to moderate dressings of  $\text{CaO}$ . An examn. of 5 profiles shows that the replaceable base content for the surface soils is reasonably good. In some cases there is a relatively high proportion of Na. K is generally low but apparently sufficient for tree requirements. Relatively high proportions of Ca in the alluvial soil emphasize its superiority. In general, there is a very fair amt. of N and better tree growth is usually characterized by higher N values.  $\text{P}_2\text{O}_5$  is generally low although in sufficient quantity or of little significance in the trees' requirements. The  $\text{K}_2\text{O}$  figures show a definite correlation between  $\text{K}_2\text{O}$  content and condition of the trees. The soil problems and tree growth are discussed briefly. II. A soil survey of part of the Huonville District. J. K. Taylor and C. G. Stephens. *Ibid.* 32-55.—A brief discussion and location of the Huonville District, the appendix contg. mech. analyses and reaction data (see above) and a soil survey map.

J. R. Adams

Iodine contents in the soils in Japan. II. Influence of natural conditions and fertilizing on the iodine contents. Arai Itano and Yasuhiko Tsuji. *Ber. Ohara Inst. Landw. Forsch. Japan* 7, 103-14 (1935). *cf.* *Ch. A. 29, 1502*.—Virgin soils from the Takahashi River basin near the sea contained 20 times more I than those from the locality of the upper stream. With arable soils the difference was not so marked. There was no definite relation between Cl and I contents, but the influence of sea  $\text{H}_2\text{O}$  was similar in both cases. There was no marked variation in the I contents of soil samples taken at various elevations on a hillside up to 100 m. With regard to surface soils and subsoils, there was more I in the surface soil of the dry-farm and virgin fields, while in the paddy field the opposite was true. The I content was increased by the application of inorg. fertilizers. The same was true to a greater extent with org. fertilizers, and those from the animal source were more effective than those from the plant source. Of the total I content of the soil 1.6 to 16.4% was  $\text{H}_2\text{O}$ -sol. There was no definite relationship between the  $\text{H}_2\text{O}$ -sol. I and the nature of the soil.

John O. Hardesty

Determination of manganese and magnesium in soils and silicate rocks. L. A. Dean and E. Truog. *J. Ind. Eng. Chem., Anal. Ed.* 7, 333-5 (1935).—A method for Mn and Mg was developed in which both are pptd. as the phosphate and weighed or titrated together after which the Mn present is detd. by the bismuthate method and the

Mg is obtained by difference. The procedure was tested on a known soil contg. only Mn and Mg and on another soil. contg. also Fe, Al and Ca and on a soil. Results for Mn and Mg on both known soils were correct to within a few hundredths of a mg. Pptn. of Mn with Br or persulfate gave high results by the bismuthate method. The new procedure gave 0.48 mg. of Mn on a sample of pyroxene while the direct detn. of Mn gave 0.61 mg. These results agree within exptl. error. C. W. Whittaker

**Determination of nitrogen in soils by oxidative digestion.** C. R. Hariharan Iyer and R. Rajagopalan. *J. Soc. Chem. Ind.* 54, 341-2T (1935).—A crit. survey of the problem of hastening digestion and avoiding acid fumes in the detn. of N in soils was undertaken. Continuous heating of the soil with dichromate or  $\text{CrO}_3$ ,  $\text{H}_2\text{SO}_4$ , and  $\text{H}_2\text{O}$  gave lower results than the Kjeldahl method. Minute amts. of  $\text{NH}_4$  dichromate are formed if the oxidizing agent is added to the cold or moderately warm mixt. of soil and acid. On heating, part of the dichromate decomposes with loss of elemental N. This is avoided by adding the oxidizing agent to the boiling mixt. of soil and acid. Traces of  $\text{HNO}_3$  formed may be completely retained by use of an air- or water-cooled condenser. The following procedure is recommended: Weigh 10 g. of soil into a suitable flask, add 2 g.  $\text{H}_2\text{O}$  or  $\text{H}_2\text{SO}_4$  and 15-20 cc. of water and shake thoroughly. Add with shaking 30-40 cc. concd.  $\text{H}_2\text{SO}_4$ , connect a condenser to the flask and boil 5 min. Remove flame, add 5 g. dichromate to the hot mixt. and immediately resume heating. Heat for 30 min., allow to cool for 5 min., dil. to 300 cc. and treat with pure powd.  $\text{Na}_2\text{SO}_3$  (about 7 g.) until the dichromate is all reduced (this is indicated by color change and by odor of  $\text{SO}_2$  when the sulfite is in excess). Heat to boiling, add 2 g. pure Zn powder and continue boiling until most or all of the Zn dissolves. Cool and distill with excess alkali in the usual way. Results obtained on 6 soils were uniformly higher than those by the official Kjeldahl method but agreed nearly perfectly with the wet digestion procedure of Greenivasan and Subrahmanyan (cf. *C. A.* 28, 553<sup>9</sup>).

**Determination of nitrate in soils.** R. E. Stephenson. *Chemist Analyst* 24, No. 4, 16 (1935).—Shake 4 g. of soil and a little pure  $\text{CaSO}_4$  with 20 cc. of water. Allow the solid to settle. With a pipet, transfer 1 drop of the liquid to a spot plate and add 4 drops of diphenylamine soln. (0.05 g. solid reagent in 25 cc. of concd.  $\text{H}_2\text{SO}_4$ ). After a few min. compare the color with that obtained from definite vols. of standard  $\text{KNO}_3$  soln. W. T. H.

**Influence of temperature on the carbon nitrogen ratio of soils.** N. R. Dhar and S. K. Mulherji. *J. Indian Chem. Soc.* 12, 436-40 (1935); cf. *C. A.* 29, 6695<sup>4</sup>.—Detns. of the C/N ratio of soils have been made by estg. the C and N content according to the method of Robinson, McLean and Williams (*C. A.* 23, 5531). Results obtained from soils collected in Bengal, Behar, the United Provinces of Agra and Oudh, and the Punjab show ratios of 9.13, 9.17, 11.3 and 14.37 against ratios for England, the Sudan and the Transvaal of 10, 12.6 and 14.4, resp. These results show that the ratio in temp. climates is lower than in tropical. The proteins present in the soil of a warm country are oxidized more readily than those exposed to a temperate climate. It appears that soil oxidations take place in accordance with the same laws as those prevailing in animal metabolism and that the C/N ratio in soils is not altogether dependent on the microbial activity as has been hitherto believed.

**Rapid determination of amount of water in soil.** Fernand Obaton. *Compt. rend.* 201, 845-6 (1935).—The sample is placed in an ebullite tube 2.63 cm.  $\times$  2.54 cm., one end of which consists of a fixed C plate, the other also of C is attached to a piston whereby the contained soil is subjected to a pressure of 40 kg. The resistance of this cylinder of soil is then detd. by a c. (800 cycles). A curve relating resistance to percentage of  $\text{H}_2\text{O}$  is given.

**Determination of organic carbon in soil.** G. Pichard. *Bull. soc. chim.* [5], 2, 1591-4 (1935).—Heating the sample

in a current of  $\text{O}_2$  by the method of Danstedt is satisfactory for the detn. of org. C and if the temp. is not allowed to rise above  $440^\circ$  any  $\text{CaCO}_3$  present is not decomposed. W. T. H.

**Potash in Massachusetts soils and its availability for crops.** Fred W. Morse. *Mass. Agr. Expt. Sta. Bull.* 324, 2-16 (1935).—Mass. soils are derived from glacial drift which consists principally of material from K-bearing rocks. The lighter plain soils required K but heavier soils showed less response. The percentage of K in Mass. soils is influenced largely by the texture of the soil. The finer the soil, the more K is dissolved by the acid used in its detn. Characteristic samples of important soil series were sepd. into sands and silt-clay. The av. percentage of K (by fusion) in sands was 2.07 and in the silt-clay 2.13. The lowest percentage of K was 1.27 in the sands overlying a thorite ledge. The soil particles yield an important part of the K required by crops. Clay presents more surface to the soil soln. than sand and also holds more water. Therefore, clay soils supply more available K than sandy soils of similar origin. C. R. Fellers

**Further work with the Cunninghamella plaque method of measuring available phosphorus in soil.** A. Mehlich, E. B. Fred and L. Truog. *J. Am. Soc. Agron.* 27, 820-32 (1935), cf. *C. A.* 29, 2277<sup>1</sup>.—A description is given of a special clay culture dish for conducting the *Cunninghamella* test for available P of soils. The results obtained with this new dish and slight modifications of the method (smaller quantities of soil and nutrient soln.) agree quite satisfactorily with crop yield in the field and with the results of the Neubauer and chem. methods. *C. blakesleeana* showed better growth on calcareous soils than *C. elegans* and can be successfully employed in testing calcareous soils without pre-treatment with acid. P need is indicated when the diam. of growth is less than 16 mm. This will vary somewhat with the plant grown. J. R. Adams

**The biological effect of available phosphorus in Hawaiian soils.** A. Lloyd Heck. *J. Am. Soc. Agron.* 27, 847-51 (1935).—In the presence of available energy material, biol. activity in Hawaiian laterites is greatly stimulated by the presence of available P, when measured by assimilation of nitrate N by microorganisms. In the combination with energy material, P helps prevent leaching of mineral N and also helps build up a larger biol. balance in the soil, which increases the amt. of P and the N held in the org. form, thus increasing the availability of the P.

**The effect of dilution on the solubility of soil phosphorus.** Hugh Dukes. *J. Am. Soc. Agron.* 27, 760-3 (1935).—Concn. of P in aq. exts. of a no. of soil types increased with diln. up to a certain point for each soil, and upon further diln. decreased. P reaches its max. concn. at the point where Ca first disappears from the leachates. J. R. Adams

**A study of the effects upon growth and development of an upland rice of varying the moisture content of soil in pots.** Guillermo O. Palis. *Philippine Agr.* 24, 393-412 (1935).—Best results were obtained with soil contg. 80% of satn. with moisture. At 20% of satn. the crop was a total failure. A. L. Mehring

**Comparative nutritional experiments on some difficult soils.** Herbert Schorstem. *J. Landw.* 83, 219-22 (1935).—Seven soils were examd. for P and K according to the König-Hasenbäumer (*C. A.* 24, 4574), Neubauer (*C. A.* 26, 3320) and *Aspergillus* methods. The values obtained by the micromethod of Sekera are included in a qual. comparison of the results. There was considerable variation among the values obtained by different methods on the same soil. John O. Hardesty

**The clay ratio as a criterion of susceptibility of soils to erosion.** Geo. J. Bouyoucos. *J. Am. Soc. Agron.* 27, 728-41 (1935).—The ratio of  $\frac{\text{sand and silt}}{\text{clay}}$ , clay ratio, is suggested as a criterion of the erosiveness of soils.

The general agreement between this ratio and the ratio proposed by Middleton, cf. *C. A.* 26, 5306, indicates the value of this clay ratio for judging the relative susceptibilities of soils to erosion. J. R. Adams



The improvement of glasshouse soils W. Corbett. *Sci. Horticulture* 3, 148-62 (1935).—Yield of tomatoes and other crops grown continuously on the same soil gradually diminishes. Decrease is common to both fertilized and unfertilized plots. Examin showed that root decay starts earlier each successive year. When 5 crops had been grown the av crop wt. from fertilized plots showed a decrease of 10.5 tons per acre over the initial crop. The corresponding decrease on the unfertilized plots was 12.7 tons per acre. The decrease is ascribed to loss of green residue and fiber which stimulate bacterial activity and provide proper soil aeration. Beneficial effects were secured from straw placed almost vertically in the soil.

Irvin C Feustel

The temporary injurious effect of excessive liming of acid soils and its relation to the phosphate nutrition of plants W. H. Pierre and G. M. Browning. *J. Am. Soc. Agron* 27, 742-59 (1935).—With one exception, all soils gave considerably lower yields of alfalfa when limed to  $pH$  values slightly above 7.0 than where limed to lower  $pH$  values, the av decrease in yield being 46%. This was considerably more pronounced for the first than the second and third cuttings and, with 5 of the 9 soils, overliming injury to alfalfa disappeared after the first year. Addns of  $MnSO_4$ ,  $MgSO_4$ ,  $FeSO_4$ , iron humate or large amts. of KCl had no effect in reducing liming injury to corn grown on acid Dekalb loam which had been limed to  $pH$  values of approx. 6.5 and 7.5. Large amts of  $CaH_2(PO_4)_2$ ,  $KH_2PO_4$ , or silica gel overcame the injury. The substitution of increasing amts of  $MgCO_3$  for  $CaCO_3$ , up to 75%, improved growth and produced normal leaf color where the largest amt was added. Materials that overcame liming injury materially increased  $H_2O$  sol  $P_2O_5$  in the soil and ext. In a comparison of 4 liming materials added in sufficient amts to bring an acid Dekalb loam to  $pH$  7.0, corn and rape made better growth with dolomitic limestone or  $CaSiO_3$  than with  $Ca$  limestone or  $CaCO_3$ . In the latter cases the plants showed symptoms of  $P_2O_5$  deficiency. When used in amts to bring the soil to  $pH$  5.8, no liming injury was obtained. Corn plants from the soil limed to 7.0 had a higher ash content and a lower percentage of Mg and P on the ash basis than plants grown at  $pH$  5.8. With 1 exception, some evidence was obtained of a relationship between injury from excessive liming and the Ca/P ratio of plants. The temporary overliming injury obtained in these expts is due to a disturbed  $P_2O_5$  nutrition.

J. R. Adams

The waste products of horticulture and their utilization as humus Sir Albert Howard. *Sci. Horticulture* 3, 213-14 (1935).—The Indore process is described as used for increasing the humus supply in coffee, sugar, rubber and other plantations in India and Ceylon. A mixt of vegetable wastes (C:N ratio of about 30:1) is transformed into humus (C:N ratio of 10:1) by means of fungi and bacteria, combined N in the form of manure and urine, phosphate and potash and a base such as wood ashes or lime. Considerable fixation of N accompanies the later stages of decomposition. Weed seeds, eggs of noxious insects and harmful spores of fungi are destroyed.

Irvin C Feustel

Use of sugar-beet petioles as indicators of soil fertility needs Robert Gardner and D. W. Robertson. *Colo. Agr. Expt. Sta. Tech. Bull* 14, 3-16 (1935).—A comparison of sugar-beet petiole analysis with the  $K_2CO_3$  soil test in a sugar beet plat expt. showed the petiole test to be much more efficient in detecting the differences in available P due to the fertilizer treatments than the  $K_2CO_3$  soil test. The petiole test was sufficiently accurate to give a reliable indication of the P needs of the soil. Treble superphosphate and fertilizer were compared as to their effects on the compn of plant exts. Fertilizer was considerably more efficient in increasing the available P. The petiole test is also applicable to the detn of the nitrate and K needs of plants.

C. R. Fellers

Immediate effects of fertilization upon soil reaction C. B. Clevenger and L. G. Willis. *J. Am. Soc. Agron* 27, 833-40 (1935).—Detns were made on 4 soils of the early neutralizing effect of org. ammoniates such as cottonseed meal and urea, in comparison to that of dolomitic limestone

used at rates calcd. to produce a non-acid-forming fertilizer. On mixing the fertilizers with the soil, a drop in the  $pH$  of the soil ranging from 0.0 to over 1.0 took place immediately. This is due to salt effect. The reaction trends of the various fertilizers for the soils used are very similar except for the Coxville soil where the magnitude of change is smaller because of a high content of org. buffering material. In the unlimed soils without dolomitic supplements, the reaction trends for fertilizers contg. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and NaNO<sub>3</sub> show a slight rise in  $pH$  following the initial decrease. Fertilizers contg. cottonseed meal and urea showed significant rises in  $pH$  which were proportional to the amts. of these materials in the fertilizers. With dolomitic limestone supplements (unlimed soil), the (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> fertilizers gave a greater  $pH$  rise than that of NaNO<sub>3</sub> or cottonseed meal fertilizers and this was exceeded only by the  $pH$  rise for fertilizers contg. the larger increments of urea. Relative to the  $pH$  values of unfertilized soil, none of the fertilizers gave evidence of as great a neutralizing effect in the limed soil as in the corresponding soil unlimed, nor was the range in  $pH$  values produced by org. ammoniates without dolomite as great as in unlimed soils. The max.  $pH$  values reached by the fertilizer in which all the N was in the form of cottonseed meal without dolomite was approx. the same as that found in the corresponding treatment where urea was the source of half the N. For a period after application of fertilizers the org. ammoniates can serve as neutralizing agents almost as extensively as a dolomitic supplement.

J. R. Adams

The microorganisms in profiles of certain virgin soils in Manitoba M. I. Timonin. *Can. J. Research* 13, C, 32-46 (1935).—Twelve profiles of 5 different kinds of virgin soils of Manitoba were sampled, described and critically examd. for soil organisms, H-ion concn and the moisture and org. matter present. Usually the A horizon showed the highest count of each group of microorganisms and the C horizon the lowest, although the greatest no. of bacteria were present in the B horizon of 1 soil in the month of May. The proportion of anaerobic bacteria and fungi to total nos increased with the depth of the horizon. Moisture content of the soil was not found to exert any consistent effect upon the nos. of microorganisms present. Fungi were most abundant in the wooded and peat soils, bacteria more so in soils of the meadow-prairie phase. In the wooded soils the microbiol. horizons appeared to coincide with the morphological horizons.

J. W. Shipley

Microbiological studies of Appalachian upland podsol soils I. Effects of physical and chemical treatments P. H. H. Gray and H. J. Atkinson. *Can. J. Research* 13, C, 115-26 (1935).—A study has been made of the effects of fertilizers and limestone, of fallowing, and of deep ploughing, upon certain aspects of microbial activity in representative Appalachian podsol soils at 3 farms in the Eastern Townships region of Quebec Province. The results show that the evolution of CO<sub>2</sub> and nos. of bacteria and actinomycetes were not altered by any simple fertilizer applied annually for 2 years. Limestone, at the rate of 6 tons per acre, increased the nos. of microorganisms during the 2 years of the expt. Deep ploughing reduced both CO<sub>2</sub> and bacterial nos. in soils ordinarily ploughed to a normal depth. Studies have also been made to det. the effects of some other chem. treatments upon the soil microflora. CO<sub>2</sub>, bacterial nos. and nitrification of soil N were increased in field plots after treatment with CaO, Na<sub>2</sub>CO<sub>3</sub>, and NaOH, alone or in combination; the effects lasted through 2 seasons.

J. W. Shipley

Decomposition and movement of herbicides in soils, and effects on soil microbiological activity and subsequent crop growth II. J. D. Newton and A. D. Paul. *Can. J. Research* 13, C, 101-14 (1935); cf. C. A. 27, 1707.—Further expts. at Edmonton to det. the effects on soils and on subsequent crop growth of CuSO<sub>4</sub>, NaClO<sub>2</sub>, and NH<sub>4</sub>CNS are reported, together with new expts. with Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. The field plot expts. were limited to Edmonton black soil, but 3 typical Alberta soils, including Edmonton soil, were used in the lab. expts. CuSO<sub>4</sub> applied to a series

of fallow plots in 1931 did not affect the yields of wheat on these plots significantly in 1932, or the yields of wheat, oats, rye and flax on these plots in 1933. The soil was not appreciably injured, even temporarily, by the  $\text{CuSO}_4$ .  $\text{NaClO}_2$  was applied to one series of fallow plots in 1930, and to another series in 1931. The effect of the heaviest applications (1300 lb. per acre) lasted for 3 years in one series, but serious injury to crops from such heavy applications did not last for more than 2 years in either series.  $\text{Na}_2\text{Cr}_2\text{O}_7$  was applied to a series of fallow plots and a series of wheat plots in 1932. It reduced the wheat yields very much in 1932, but did not reduce crop yields the following year in either series, as it decomposed and lost its toxicity in the soil rather quickly.  $\text{NH}_4\text{CNS}$  was applied to a series of fallow plots and a series of wheat plots in 1932, and it reduced the wheat yields even more than  $\text{Na}_2\text{Cr}_2\text{O}_7$  in 1932. It retarded nitrification and did not decompose and lose its toxicity completely during the season of application, but even in the cases of the heavier applications (650 and 1300 lb. per acre) the toxic effect disappeared early in the following season. The total and straw yields of crops sown on these plots were generally increased by the lighter applications (160 and 325 lb. per acre) in 1933, but the grain yields were generally reduced by the heavier applications (650 and 1300 lb. per acre). Growth of certain annual weeds was considerably stimulated by this nitrogenous weed killer in 1933 and 1934, in the plots to which the heavier applications had been made. Lab experiments showed that the thiocyanate may be leached out of a soil with water, that it decomposes fairly rapidly in soils under favorable conditions of moisture and temperature and more rapidly in fertile soil rich in organic matter than in poorer soil, and that nitrification in soils is depressed for a time by the  $\text{NH}_4\text{CNS}$ . J. W. Shipley

The response to fertilizers and the changes observed in the nutrient economy of the soil during wet and dry years. K. Opitz. *Ernähr. Pflanze* 31, 341-9 (1935).—Field experiments on the utilization of nutrients by various crops showed that the natural fertility of the soil and the intensity of fertilizing influenced the nutrient reserves in the soil to a greater extent than did the rainfall. J. O. H.

Effect of plant nutrients, soil reaction and light on gardenias. Harold E. White. *Mass. Agr. Expt. Sta. Bull.* 315 (*Ann. Rept.* 1934), 58-9 (1935).—N and P greatly increased vegetative growth in gardenias; K had no effect. Low N caused a uniform chlorosis of the foliage and dwarfing of the plants. Interveinal chlorosis was severe on plants fed with  $\text{Ca}(\text{NO}_3)_2$  as a source of N, such plants became green after spraying with  $\text{FeSO}_4$  soln. N in the form of  $\text{NH}_4$  salts produced less chlorosis than  $\text{Ca}(\text{NO}_3)_2$ . Soil reaction alone is not a safe basis on which to judge the possibility of interveinal chlorosis in gardenia plants, since exptl. chlorosis occurred from pH 5.0 to 6.1. Gardenias are very sensitive to Fe deficiencies and require more Fe than the av. ornamental plant. C. R. F.

Efficiency of ammoniated superphosphates for cotton. J. T. Williamson. *J. Am. Soc. Agron.* 27, 724-8 (1935).—Increases in yield due to P were greatest on the Highland Rim and the Appalachian Plateau soil groups and least on the Greenville soil group of the Coastal Plain. With superphosphate as a basis, the relative increases due to different sources of P were: superphosphate, 100; ammoniated superphosphate (2% N), 100; ammoniated superphosphate (4% N), 90; and pptd.  $\text{Ca}_3(\text{PO}_4)_2$ , 85. Ground  $\text{CaCO}_3$  used with the complete fertilizer contg. superphosphate produced an av. increase of seed cotton per acre over 3 times that obtained when used with the complete fertilizer contg. 4% ammoniated superphosphate. J. R. Adams

Phosphate fertilizer investigations in sand cultures. E. A. Mitscherlich. *Phosphorsäure* 5, 517-26 (1935).—Increased applications of N or K without the application of P caused a reduction in the  $\text{P}_2\text{O}_5$  content of the plant. Application of 0.060 g. of  $\text{P}_2\text{O}_5$  with from 0.000 to 5.000 g. of N caused an increase in the amt. of  $\text{P}_2\text{O}_5$  taken up from 15.3 to 57.9% of the total added. A similar K-P series caused an increase in the  $\text{P}_2\text{O}_5$  taken up of from 45.2 to 57.2% of the total. K. C. Beeson

Decomposition of phosphate fertilizers. P. Kötting. *Phosphorsäure* 5, 551-7 (1935).—Five hundred-g. quantities of soil were treated with Thomas meal or superphosphate at the rate of 60 kg. per ha. Total acids and total bases were detd. at the end of 1 and 7 days by the electro-ultrafiltration method. The acid and base values at the end of one day were higher when superphosphate had been added than when Thomas meal had been added, but at the end of 7 days, the soly. of the acids and bases in the superphosphate-treated soil had decreased, and the soly. of the acids dropped below that in the soil treated with Thomas meal. In the soil treated with Thomas meal, the soly. of the acids increased after 7 days, but the soly. of the bases decreased to equal that in the superphosphate-treated soil. K. C. Beeson

The most suitable nutritive ratio in Nitrophoska (containing 83% E. Blanck and W. Heukeshoven. *J. Landw.* 83, 203-18 (1935), cf. C. A. 28, 6451\*.—The grain and straw of oats from 2 different soils which had received similar fertilizer treatments were examd. for the utilization of N, P and K. Favorable nutrition of the plant is not only dependent on the applied fertilizer but is also influenced by the phys. and chem. characteristics of the soil. John O. Hardesty

[Effect of liming on the phosphate status of soil.] F. Hardy. *Proc. Sugarcane Investigation Committee (Trinidad)* 4, 336-7 (1934).—Addn. of ground limestone to holes in soils increased the available P (Truog method) 39-67%. The limestone, supplied by quarries in Trinidad, contained 0.023-0.031%  $\text{P}_2\text{O}_5$  and 2.99-6.29%  $\text{MgCO}_3$ . The increase in the available P content of the soils was not due solely to the P content of the added limestone. K. D. Jacob

Some effects of lime on ryegrass. Alex. F. R. Nisbet. *Scottish J. Agr.* 18, 349-53 (1935).—The no. of surviving seedlings of ryegrass is usually increased by adding lime to the soil. The ordinary hay type of perennial ryegrass, while developing 70-80 seedlings per 100 viable seeds on light acid sandy soil (pH 4.98 and lime requirement 3 tons/acre), may give over 90 seedlings when the soil receives its lime requirement and almost every viable seed may develop a plant when heavy liming is given. The indigenous "Kentish" perennial ryegrass develops 50-80 seedlings per 100 viable seeds on light acid soil and 80-90 at the lime requirement rate, but the nos. are not increased with very heavy liming. Both types on light acid soils tend to tiller earlier and produce a greater no. of tillers when heavy liming is given. Both types produce more total growth on neutral clay loam than on acid peaty soil (original pH 4.27 and lime requirement 10 tons/acre) receiving its lime requirement, but less growth with fewer tillers than on acid sandy soil similarly treated. K. D. Jacob

Effect of fertilizers on the longevity of mowings. A. B. Beaumont, R. W. Donaldson and M. E. Snell. *Mass. Agr. Expt. Sta., Bull.* 322, 2-8 (1935).—The quality and yield of grass on Merrimack fine sandy loam were greatly improved by fertilizer mixtures high in N and K. Little or no benefit was derived from superphosphate. A fertilizer having a ratio of N,  $\text{P}_2\text{O}_5$  and  $\text{K}_2\text{O}$  of about 3:1:2 is recommended for old mowings on soils of this type. Mowings deteriorate after 6-8 years regardless of fertilization, and should be reseeded  $\text{NaNO}_3$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{CaCN}_2$  and urea were compared as top dressings at the 50 lb. per acre level (calcd. to N). The crop increase was slightly over 100%. No significant advantage for any source of N was obtained. Chem. analyses showed that total N of grass decreased as maturity advanced. In the earliest stage of growth the N fertilizers increased the N content of grass from 25 to 50%. In the last stages there was little effect on compn. as a result of fertilization. C. R. F.

Pasture fertilization. F. D. Gardner, S. I. Bechdel, P. S. Williams, C. F. Noll, J. W. White, E. S. Erb, E. B. Coffman, G. C. Fuller and C. R. Enlow. *Pa. Agr. Expt. Sta., Bull.* 323, 3-24 (1935).—This is a preliminary report after 5 years of a projected 10-year expt. designed to show the effect of different fertilizer treatments on the botanical compn. of the turf, yield of clippings and hay, and yield

of digestible nutrients, as determined by chemical analyses and by grazing cattle. The use of grazing animals to measure the carrying capacity of fertilized pasture plots necessitates a long term investigation to eliminate experimental error. To date, the results indicate that dairy cattle can be satisfactorily used to measure the carrying capacity of pastures under different systems of fertilization. Conclusions are withheld until the termination of the experiment. C. R. F.

The influence of potash fertilization on the value and efficiency of fodder. F. König. *Landw. Jahrb.* 81, 829-83 (1935).—The fluctuations in the nutritional value of both hay and beets from year to year were greater than any difference noted due to fertilization. Thus, the N-free extract of the beets varied from 5.88% in 1931 to over 9% in 1934, but those beets fertilized with 200 kg./ha. of K<sub>2</sub>O had 1.06% of N-free extract while those not fertilized showed 0.7%. Fertilization with potash increased the ash, K, and Cl contents of hay markedly. Smaller increases were found in the P content. In general no larger increases were obtained with 200 kg./ha. of K<sub>2</sub>O than with half that quantity. Na and Mg showed a tendency to decrease with increased potash fertilization while Ca decreased materially except in the fourth year of fertilization when a slight increase was noted with the application of 20 kg./ha. of K<sub>2</sub>O. A study of the mineral balance in animals showed that there was a greater retention of Ca and P from potash fertilized fodder than from the unfertilized material. Calorimetric determination showed no clear differences between the energy content of unfertilized and fertilized fodder. A calorimetric nutritional balance showed for both fertilized and unfertilized fodders an efficiency of about 58%. K. C. Beeson.

The effect of the addition of nutrient salts on the root secretions of various plants. Paul Solberg. *Landw. Jahrb.* 81, 891-917 (1935). *cf.* C. A. 29, 819<sup>6</sup>.—In unfertilized plots acid secretions were found from the roots of beans, corn and lupine. With beans and corn the difference between the root zone and outer zone in each case was about 0.5 pH unit, while the difference with the lupine was about 1 pH unit. The addition of a physiologically acid salt mixt. where the N was supplied from (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> decreased the pH about the root zone of beans from 5.23 to 4.60, corn 5.06 to 4.69 and lupine 4.90 to 4.79. When NaNO<sub>3</sub> was substituted as the source of N to give a physiologically alk. mixt., the increase in acidity was very small, and the difference in the zone was less in some cases than when no fertilizer had been added. K. C. B.

Comparative fertilizer investigations with Thomas meal and Alger phosphate on a high-moor sand-mature culture eighty years old. Brune. *Phosphorsäure* 5, 337-50 (1935).—No significant difference was noted in the yields obtained with Thomas meal and Alger phosphate. Application of either of the phosphates in increased quantities caused equal increases in the P and Ca contents of the plants. K. C. Beeson.

How to introduce mineral fertilizers for cotton during the vegetation period. D. V. Khar'kov. *Bor'ba za Akkolyot* 1935, No. 5-6, 54-9.—Kh. presents data and discusses the problem of fertilizer placement for cotton under conditions of irrigation. J. S. Joffe.

Progress report of fertilizer studies with Jonathan apples upon Ephrata fine sandy loam. F. L. Overlier and F. L. Overholser. *Wash. Agr. Expt. Sta., Bull.* 319, 14 pp. (1935).—See C. A. 29, 487<sup>2</sup>. C. R. F.

Fertilizer treatments for sweet corn. W. A. Huelsen and M. C. Gilbre. *Ill. Agr. Expt. Sta., Bull.* 417, 353-455 (1935).—As a result of 6 years' field work, treatments of single fertilizer salts were usually ineffective. Mixtures of 2 sal. s. contg. N and P or P and K gave good results when the proper ratios were used. When N was omitted, 80 lb. of a 0-16-3 fertilizer gave best results. Mineral fertilizers applied without N should not contain more than 1% K<sub>2</sub>O. Of all the 63 treatments investigated, an application of 40 lb. of 0-16-12 fertilizer supplemented by 50 lb. of side-dressed NaNO<sub>3</sub> per acre gave best yields. NaNO<sub>3</sub> should be applied 3-60 days after the corn is planted. In every case fertilizer treatments hastened maturity of the sweet corn. P had the greatest effect in

advancing maturity. K used either alone or in combination with P retarded maturity. C. R. Fellers.

Fertilizing of bulbs. Gordon W. Gibson. *Ns. Hort. culture* 3, 174-83 (1935).—Most of the important bulb-growing areas are observed as centering on soil having a low clay fraction and a high fine sand content. Fertilizer is generally applied to preceding crops; thus bulbs are allowed to benefit from residues. Lime, basic slag and bone meal are helpful to tulips. Deep cultivation and a suitable rotation are highly important for narcissus but soil may be acid or slightly alk. Recommended fertilizers contain approx. 6% N, 10% water-sol. P<sub>2</sub>O<sub>5</sub>, 10% insol. P<sub>2</sub>O<sub>5</sub>, and 7.5% potash. A compost of seaweed, weeds, manure, etc., is beneficial to bulbs either as a top dressing or when planting. Presence of Fe and abundance of K assist in giving good color to flowers. Irvin C. Feustel.

Nitrification of ammoniated peat and other nitrogen carriers. R. O. E. Davis, R. R. Miller and Walter Scholl. *J. Am. Soc. Agron.* 27, 724-37 (1935).—Nitrification of ammoniated peat prep'd at 180° and 300° showed approx. the same max. value of conversion to nitrates, 24%, in 1 series of tests, while in another series the 180° sample showed 31% conversion and the 300° sample gave only 13.4%. The nitrification of the 300° material was longer in starting and the rate was slower than with the 180° material. The H<sub>2</sub>O-sol. N compounds in both the 180° and 300° material were quite easily nitrified. It appears that the extent of nitrification of this portion is about the same as with (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. The soil portion from ammoniated dextrose gave a max. conversion of 40% after 18 weeks. The leached residues from ammoniated peat gave results varying from 6 to 17% conversion to nitrate. Ammoniated lignin and its leached residue gave nitrification values corresponding closely to those of ammoniated peat. Whole ammoniated starch gave about the same value as raw peat and somewhat less than the H<sub>2</sub>O-leached residue from ammoniated peat. The H<sub>2</sub>O-leached residue from ammoniated dextrose gave 8.2% of the total N as nitrate after 8 weeks. Ammoniated peat and dextrose residues from exhaustive extractions with several solvents were nitrified somewhat more in each case than raw peat. Ammoniated peat H<sub>2</sub>O-insol. residue with NaNO<sub>3</sub> added gave a nitrification value of 13.4% after 6 weeks as contrasted with 17% for a similar residue alone. Three ammoniated peat H<sub>2</sub>O-insol. residues, with active N of 50, 80 and 87%, all gave low conversion to nitrate, approx. 12, 10 and 11%, resp. Mg NH<sub>4</sub> phosphate nitrified at about the same rate as (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. After 12 weeks guanidine carbonate had a conversion value of only 6%. The insol. N of ammoniated peat becomes available as nitrate at about the rate of the N in the natural occurring soil org. matter. J. R. Adams.

Influence of certain sulfate-hydrated lime mixtures on transpiration. J. D. Wilson and H. A. Rummels. *Ohio Agr. Expt. Sta., Bi-monthly Bull.* 175, 143-f (1935).—CuSO<sub>4</sub> in combination with hydrated lime and water (Bordeaux mixt.) was not alone effective in increasing the transpiration rate of plants since NiSO<sub>4</sub>, MnSO<sub>4</sub>, and FeSO<sub>4</sub> caused nearly as great an increase when applied to *Coleus* as did Bordeaux mixt. Mixts. contg. ZnSO<sub>4</sub>, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and K<sub>2</sub>SO<sub>4</sub> were considerably less effective. The ppts. of these various mixts. settled in the reverse order of their effectiveness in increasing the transpiration rate, e. g., the mixt. contg. CuSO<sub>4</sub> increased the water loss most, but its ppt. settled most slowly, whereas that of the one contg. K<sub>2</sub>SO<sub>4</sub> settled most rapidly, and the mixt. caused the smallest transpirational increase. C. R. Fellers.

Composts and fertilizers in relation to greening. VI. Basic slag. T. W. Evans. *J. Board Greenkeeping Research* 4, 40-2 (1935). *cf.* C. A. 29, 309<sup>3</sup>. K. D. J.

Salinity of irrigation water and injury to crop plants. Frank M. Eaton. *Calif. Citregraph* 20, 392, 322, 224 (1935), 354, 392-5 (1935).—Under conditions in the irrigated districts of Calif., the contents of B, Ca, Mg, Na, Cl, carbonate and sulfate are usually several times higher in the displaced soil salts than in the irrigation waters. In irrigation practice the ratio of milliequivalents of Na to

miliequiv. of total bases is usually of more importance than is the content of total sol. salts in the water, except when the sol. salt content is excessively high, when the ratio is 0.7 or higher the water is definitely unsuited for irrigation purposes. In nutrient soils, the root growth and water uptake of corn and tomatoes are depressed by the presence of 10 miliequiv. of NaCl; Na<sub>2</sub>SO<sub>4</sub> has a similar effect. Salt injury to plants is not accompanied by characteristic symptoms other than reduced growth. There seems to be no three-fold concentration below which chloride-sensitive crops are unimpaired by NaCl. With contents of Na<sub>2</sub>SO<sub>4</sub> above those supplying the amt. of S necessary for plant growth, each successive increment of sulfate causes a depression in plant growth. K. D. Jacob.

Onions in the Connecticut Valley. A. B. Beaumont, M. E. Srell, W. L. Doran and A. I. Bourne. Mass. Agr. Expt. Sta., *Bull.* 318, 3-31(1935).—From 1 to 2 tons of lime every 2-3 years is recommended for onions. Greatest fertilizer response was obtained with P though K and N were also important in the production of max. yield. The optimum  $\frac{P}{K}$  of the soil for the growth of onions is 6.0-0.5. A 4-12-5 fertilizer must carry  $\frac{1}{2}$  of its N in org. form gave better results with onions grown from seed than did another of the same grade but containing N only. KCl was fully as satisfactory as K<sub>2</sub>SO<sub>4</sub> as a carrier of K. High analysis fertilizer must. gave as satisfactory results as dil. ones. Deferring application of  $\frac{1}{2}$  of the N until the top. were  $\frac{1}{2}$  developed gave beneficial results with seed onions only. A no-till-plant spray proved satisfactory over a period of years for the control of onion thrips. Since reinfestation of plants is very rapid, frequent follow-up sprays are necessary. C. R. F.

Date growing in Arizona. D. W. Albert and R. H. Hilleman. *Ann. Agr. Expt. Sta., Bull.* 149, 231-80 (1935).—The date palm has a relatively high tolerance for alkali soils and does better in the light dry soils than in the heavy clay types. Cultural methods are detailed. The use of org. matter, N and P, is advantageous in most Arizona soils. It is essential that the fruits have a high percentage of sugar accumulated before they are packed from the palm if a high-quality product is to result from artificial ripening. Thus the Khadrawi variety when green contained 20% total sugar, when 50-70% translucent, 30%; and when fully translucent, 42%. When semi-dried the sugar content of ripe dates reaches 70-80%. Rain damage is serious as it causes swelling and breaking of the outside, the absorption of water by the fruit, and finally rotting or spoilage. The fruit should be fumigated with HCN, or ethylene oxide-CO<sub>2</sub> before it is packed into wholesale or retail packages to insect infestation may be serious. For good keeping quality, Arizona dates should have their moisture content reduced to about 25%. Soft dates must be dehydrated or sun-dried will occur. In dehydration chambers the temp. is kept at approx. 55-60° while in the maturation chamber the temp. is 35-50° and the relative humidity 70-80%. Dates may be successfully held in cold storage at temps. of 0-2° at relative humidities of 65-70%. A description of the principal varieties is given. C. R. Fellers.

The way to independence by nitrogen economy in agriculture. A. J. Vanden. *Jaarboek Kemischheid* 54, 9-83(1935).—N fixation by leguminous plants is discussed. A I.V. stage has an important place in conserving soil N as well as being a valuable feed. E. E. Jekkels.

Some fundamentals of nutrition of horticultural crop plants. H. Hill. *Sci. Agr.* 16, 21-4 (1935).—With strawberries grown in sand culture, the withdrawal of K resulted in a marked reduction in total carbohydrate; this reduction appeared in reducing sugars, sucrose and starch. The omission of K salts resulted in increased N accumulation. In K-starved plants, nitrate is not reduced nor is protein formed. The importance of a balanced N:K ratio cannot be over-emphasized. Ash analyses indicate a definite antagonistic relation between K and Ca; as one falls the other tends to increase. In plants, low K symptoms were obtained with excess Ca treatment. A neg. correlation was found between P and K. Deficient K

tended to increase P accumulation and deficient P resulted in a tendency toward an increase in K.

C. R. Fellers.

Some responses of Yellow Transparent apple trees in Delaware to various nitrogen treatments. F. S. Lagasse. *Del. Agr. Expt. Sta., Bull.* 195, 3-42(1935).—A 3-yr. study showed that applications of N increased the circumference and terminal growth of trees but had no significant influence on yield. The use of N did not change the biennial bearing characteristic of the trees to annual bearing, nor did it affect firmness of the fruit or its keeping quality. N treatment of Yellow Transparent apple trees did not greatly affect the moisture content of the fruit or seeds, but each treatment did significantly increase the percentage of N in the flesh of the fruits. The percentage of N in the seeds was not increased. C. R. Fellers.

Tree injection—investigation by the injection of fertilizers. Progress report. W. A. Rouse. *East Malling Research Sta., 2nd Ann. Rep.* 1934, 155-8, *et. C. A.* 29, 20-7. — Cox's Orange Pippin apple trees in a cone plantation were injected with a soln. containing K<sub>2</sub>HPO<sub>4</sub> 0.2% and urea 0.25% at the rate of 0.655-0.167 lb. tree or 10-30 lb. acre. The amt. of shoot growth, as judged by the wt. and no. of prunings, was nearly doubled as a result of the injection. The increase was greater in the trees absorbing the larger amt. of fertilizer. Attack by apple leaf hopper and red spider was markedly less severe on the injected trees than on the controls. Just before and just after picking, the fruit from the trees absorbing the larger amt. of fertilizer was inferior in color and taste to that of the control trees. Although the fertilizer was applied in a single hole (0.25 m. in diam.) drilled diametrically through the main stem, all branches of the tree were uniformly affected. The diagnosis and cure of chlorosis in a peach tree. Progress report. *Ibid.* 19-41.—Lime-induced chlorosis in a peach tree was diagnosed and cured by the injection of a 0.35% soln. of FeCl<sub>3</sub> through 0.125-m. holes bored diametrically through the 2 main stems. The treatment had no ill effect on the tree and within 2 weeks the leaves had become a healthy green color. Injection of a 0.1% soln. of FeCl<sub>3</sub> caused about 50% of the leaves to fall but the remainder rapidly recovered from the chlorosis. K. D. Jacob.

Maintenance of adequate nitrogen for citrus trees. Willard G. Babcock. *Calif. Compost* 20, 212, 218-20 (1935).—In 23 well fertilized citrus groves the av. concn. of nitrate N in the 1st 6 m. of soil decreased from approx. 530 p. p. m. in Oct. to less than 50 p. p. m. in Jan. and Feb.; the concn. then gradually increased to 530 p. p. m. in the following Oct. In the next 30 m. of soil the av. concn. decreased from about 50 p. p. m. in Oct. to 150 p. p. m. in Jan. and then gradually decreased to nearly zero in the following Sept. The highest concn. of nitrate N in the root zone occurred at the time of min. activity on the part of the trees. Nitrogenous fertilizers applied to citrus trees during the summer months should be readily sol. in water, and must be immediately available for use by the tree and capable of readily penetrating to the root zone. K. D. Jacob.

Some aspects of citrus decline in Arizona. W. T. McGeorge. *Calif. Compost* 20, 186, 214-15 (1935).—A discussion of the effects of alkali soils and high soil  $\frac{P}{K}$  values on citrus trees. K. D. Jacob.

Progress report on vegetable diseases. VI. L. O'Brien, R. O. Mullen and P. W. Bruen. *Univ. Bristol Agr. Hort. Research Sta., Ann. Rep.* 1934, 173-90, *et. C. A.* 28, 1917. — In a 3-yr. expt., a considerable reduction in the incidence of *periderm-rot* disease on *Fraxinus ssp.* grown in a hum. soil of  $\frac{P}{K}$  5.2, was obtained during the 3rd year by annual applications (in January) of either ground burnt lime or hydrated lime at the rate of 5-4 tons/acre. Annual applications (in April) of 3-4 cwt. of CaCN<sub>2</sub> reduced the disease only slightly. *Fraxinus* rot lesions (caused by *Marasmius pomorum*) were not controlled by treating the soil with Carbide dust (4 lb. acre) shortly before the plants were set out or by digging the plants in Riverdax must. previous to planting out. The treatments had a deleterious effect on the plants which appeared

bronzed and stunted in the spring following the treatment (about 4 months after planting). Good control of mild rust (caused by *Puccinia psoralea*) on *Alnus incana* was obtained by watering the plants on January 5th with a 7.5% tar oil emulsion contg. 1%  $\text{CuSO}_4$ ; the treatment occasionally caused a substantial reduction in the no. of marketable shoots. It is not of course (caused by *Sclerotinia cepivorum*) was not controlled by treating the seed with formalin (1 pint to 16 gal.) at the time they were drilled into the ground. K. D. Jacob

The effect of calcium cyanamide and of formalin on pea sickness. Progress report. C. L. Walton, L. Ogilvie and B. O. Mulligan. Univ. Bristol Agr. Hort. Research Sta., *New Rep.* 1934, 158-61—See C. A. 29, 5070.

Raan or boron deficiency in swedes. D. G. O'Brien and R. W. G. Dennis. *Swedish J. Agr.* 18, 326-34 (1935).—The symptoms of the Raan disease of swedes appear to be restricted to the older zones of the root portion of the bulb and do not extend to the stem portion. A cross section of a bulb severely affected with the disease shows a clearly defined zone mottled with brownish areas, arranged according to no definite pattern. In less severe cases the area involved may be merely a small patch or arc of tissue having a water soaked appearance and usually situated halfway between the circumference and the center of the bulb. In a longitudinal section the brown areas are seen to be elongated, converging toward the base and maintaining a fairly constant zone of healthy tissue between themselves and the root. In very severe cases small cracks occur, marking the outer limits of the diseased zone. As compared with healthy bulbs, diseased bulbs of the same variety of swedes contained more fiber and less soil carbohydrate, the amt. of sugar in the juice being reduced as much as 12%. Application of Mn compds. to the soil had very little effect on the incidence of the disease, but the disease was completely controlled by application of borax at the rate of 20 lb./acre. In water culture solns. without B, turnip seedlings remained very small, had a pale yellowish green appearance and died after about 3 months, at this time they weighed only about 5% as much as plants grown in complete soln. contg. 0.003 g. borax/l. In the absence of B fibrous root development did not occur.

The boron status of fruit and leaves in relation to "internal cork" of apples in the Nelson district. Preliminary report. H. O. Adew. *New Zealand J. Sci. Res.* 17, No. 1, 388-91 (1935).—The B content of apples affected with "internal cork" was found to be 3-6 p. p. m. Unaffected apples contained 10-30 p. p. m. Leaves from unaffected trees contained 9-11 p. p. m. B, leaves of healthy trees contained 17-18 p. p. m. "Internal cork" of apples is attributed to a B deficiency of the soil.

"Potato sickness" on allotments at New Romney. S. G. Jary and S. J. Travers. *J. South-Eastern Agr. Soc. New. Kent* No. 36, 100-2 (1935).—Attack of the yellowing (*Hierodera schachtii* Schmidt) on potatoes was reduced by digging drained creosote salts (6 cwt./acre) into the soil 3 weeks before planting the potatoes.

Important diseases of pepper in Georgia. B. B. Higgins. *Ga. Agr. Expt. Sta., Bull.* 186, 20 pp (1934).—Semesan, a con. org. Hg prep., was the most effective seed disinfectant used for the sterilization of pepper seed. For the control of fungus diseases in the growing plants at least 2 sprayings with Bordeaux mixt. should be used. The concn. of the first spray should be 3-4-50 and the second, 4-6-50. If plant lice are present, nicotine sulfate should be added to the Bordeaux mixt. C. R. F.

Boron superphosphate and Bor-Am-Sup Ka against heart and dry rot of beets. K. Schumann. *Dest. Ind. Pr.* 62, 171 (1935); *Rev. Appl. Microb.* 14, 613—Heart and dry rot of beets required only 5 kg. of B per 0.34 hectare for control when a well-balanced fertilizer was used.

Control of bacterial fruitlet rots of the pineapple in the Philippines. F. R. Serrano. *Philippine J. Sci.* 57,

29-62 (1935).—There are at least 4 factors favoring the maladiers: incomplete closing of the eyes, fewness of shoots, high temp. and low acidity of the fruit. Potash application in the form of  $\text{K}_2\text{SO}_4$  caused the greatest reductions in the total infection of the fruit. The  $\text{K}_2\text{O}$  of fruit fertilized with  $\text{K}_2\text{SO}_4$  was 3.8 as compared with 3.9 when no potash was used and with 4.0 for the controls. Bordeaux mixt. of a concn. between 3-4-50 and 4-5-50 was superior as a spray for the control of fruitlet rot to lime sulfur,  $\text{CuSO}_4$ ,  $\text{HgCl}_2$  and Semesan. K. C. Beeson

Dry application of chlorates. H. W. Hulbert and L. V. Benjamin. Idaho Agr. Expt. Sta., *Circ.* 74, 3-8 (1935).— $\text{NaClO}_3$  gave excellent control of quack grass (*Alopecurus repens*), wild morning glory (*Convolvulus arvensis*), leaf spurge (*Euphorbia esula*), and Canada thistle (*Cirsium arvense*) when applied in the dry state in the autumn. The rate of application was 3-5 lb. per sq. rod. The dust was as effective as the liquid spray. Two sprayings or dustings, spaced several weeks apart, are recommended for max. results in destroying weeds. Both cost and fire hazards are reduced by the use of  $\text{NaClO}_3$  in dry form.

The tung-oil tree. Wilmon Newell, Harold Morrill, R. M. Barnett, A. F. Camp and R. D. Dickey. *Fla. Agr. Expt. Sta., Bull.* 280, 5-67 (1935).—The tung-oil tree, *Alseodaphne fordii*, thrives in Fla. on well-drained soils of acid reaction. Excesses of lime and phosphate in the soil are detrimental to growth. The use of  $\text{ZnSO}_4$  to prevent bronzing is recommended. Complete fertilizers contg. N,  $\text{P}_2\text{O}_5$  and  $\text{K}_2\text{O}$  in the ratios of 5 S 4 give the best results in tree growth and yield of nuts. C. R. F.

Final summary of the research into the origin of superphosphate. Max Speter. *Superphosphate* 8, 141-51, 161-8, 181-90 (1935); of C. A. 29, 5582. K. D. J.

History of guano. W. J. Copenhafer. *Farming S. Africa* 10, 221, 226 (1935).—A brief discussion is given of the history of the guano industry in Peru and in the Ichnaboe Islands on the southwest coast of Africa.

An apparatus for determining purity of marls. L. M. Turk. *Mich. Agr. Expt. Sta., Quarterly Bull.* 18, 29-32 (1935).—A known wt. of marl is treated with 1.5 HCl and the resulting vol. of  $\text{CO}_2$  is detd. by means of a gas buret. The rapid method requires only 3-5 min. and is accurate to 3% on a 3-g. sample. The value of a marl as a soil neutralizer depends on its content of  $\text{CaCO}_3$ .

Agricultural value of blast furnace slag. J. W. White. *Pa. Agr. Expt. Sta., Bull.* 320 (4th Ann. Rept.), 13 (1935).—From 5 to 10 million tons of basic slag is produced in Pa. annually. It possesses 40% the value of ground limestone for correcting soil acidity. When compared on the basis of equal units of Ca oxide, of the same degree of fineness, slag gives results superior to limestone.

Bagasse and paper mulches. O. C. Magistad, C. A. Farden and W. A. Baldwin. *J. Am. Soc. Agron.* 27, 813-25 (1935).—Bagasse mulch was distinctly better than paper which was in turn better than plain soil mulch in conserving moisture. The first 6 in. of soil contained less moisture but more nitrate than soil at greater depths. Paper mulch treatment maintained a higher nitrate content in the soil than either the bagasse or soil treatment.

Ultraviolet light as an aid on the farm, with special reference to fertilizers and feeding stuffs. Julius Grant. *Fertilizer, Feeding Stuffs & Farm Supplies* J. 20, 674-5 (1935).—Under ultraviolet light bone meal has a bright blue color and superphosphate is colored a dull violet. Basic slag does not fluoresce but raw phosphate rock usually appears brown or yellow. The method will detect 10% of phosphate rock in a mixt. and its sensitivity is increased if a 10%  $\text{HCl}$  ext. is examd. instead of the solid material. The method can be used to det. the thoroughness of distribution of fertilizers in soil. As little as 0.5% of  $\text{ZnO}$ , which gives a yellow color, can be detected in flour and cattle foods.

Diseases and insect pests of rhododendron and azalea

Richard P. White and C. C. Hamilton. N. J. Agr. Expt. Sta., *Circ.* 350, 3-23(1935).—These plants require acid soils. The use of 1 lb. of  $Al_2(SO_4)_3$  18H<sub>2</sub>O per 18 sq. ft. in ordinary garden soils will furnish adequate acidity. Chlorosis can be effectively controlled by spraying with a soln. of 0.25%  $FeSO_4$ . Spider mites, *Tetranychus telarius*, are serious pests and are best controlled by the repeated applications of either fine S dust or pyrethrin-soap emulsion. The emulsion should contain at least 0.005% pyrethrins and 0.5% soap. The control of *Cercospora* leaf spot caused by *Cercospora rhododendri* is attained by spraying at 2-week intervals with a 2-2-50 soln. of Bordeaux mixt. Black vine weevil, *Brachyrhinus sulcatus* Fab., can be poisoned in June by an attractant bait consisting of 5 lb. of wheat bran, 1 oz. of Ca arsenate, 1 pint of molasses and 2 quarts of water. Another useful attractant and poison consists of apple pulp impregnated with 3.5%  $Na_2SiF_6$ . C. R. F.

The control of the insect pests of basket willows, with special reference to the use of combined insecticidal and fungicidal washes and to methods of application. H. G. H. Kearns. Univ. Bristol Agr. Hort. Research Sta., *Ann. Rept.* 1934, 120-16.—A discussion of the properties and use of nicotine, derris, pyrethrum and Cu dusts and sprays and Pb arsenate sprays in relation to the control of insect and fungus pests of basket willows. K. D. J.

The control of the brassy willow beetle (*Phyllocolpa vitellinae* L.) with special reference to the use of dusts. H. P. Hutchinson and H. G. H. Kearns. Univ. Bristol Agr. Hort. Research Sta., *Ann. Rept.* 1934, 147-9.—The beetle was controlled by dusting the willow stools either with a proprietary derris prepn. contg. 0.18% rotenone or with a dust of the same rotenone content in which kaolin was used as a diluent. Under dry weather conditions the dust retained sufficient effective toxicity to kill or repel wandering beetles for at least 3 days after it was applied to the stools. For efficient control the dust should be applied just after the time of bud burst and before the shoots have attained a length of 18 in. K. D. Jacob.

The control of *Phylloperthia horticola* L. in grassland. C. L. Walton. Univ. Bristol Agr. Hort. Research Sta., *Ann. Rept.* 1934, 150-7.—See C. A. 29, 6977.

K. D. Jacob. Control of dothiorella rot on avocado fruits. W. T. Horne and D. P. Palmer. Calif. Agr. Expt. Sta., *Bull.* 594, 16 pp.(1935).—The most effective spray for the control of dothiorella rot consists of 16 lb. of 4-4-50 Bordeaux mixt., 6 lb. of wettable S, 6 oz. of blood albumin spreader, and 100 gals. of water. This spray is best used when the fruits attain a size of 1.5 in. in diam., with a second application 6-8 weeks later. S applied alone as a liquid with spreader is also a very effective fungicide against this disease. Liquid fungicides are more effective than dry dusts.  $CuSO_4$  is somewhat more efficient than  $ZnSO_4$ . The addn. of S to a Cu fungicide greatly increases its effectiveness. Fumigation of the fruits with  $NCl_3$ , with or without vacuum, was of little value in controlling the rot. C. R. Fellers.

Control of tobacco wildfire. W. S. Beach. Pa. Agr. Expt. Sta., *Bull.* 322, 3-29(1935).—Leaf-spot damage to tobacco is caused mostly by wildfire, *Phytophthora tabaci*. The importance of making early application of fungicides in seed beds, especially Bordeaux mixt. and Cu-lime dust, is emphasized. A spray of milk powder with water is as effective as Bordeaux mixt. in the prevention of wildfire in seed beds. HgCl causes some stunting of tobacco seedlings, or a delay in the maturity of the crop, by preventing the development of secondary roots near the soil surface. C. R. Fellers.

Experiments in the control of tobacco wildfire with chemical preparations. K. Böning. *Prakt. Bl. Pflanzensch.* 13, 60-7(1935); *Rev. Applied Mycol.* 14, 650.—In the seed bed and field beneficial effects of regular applications of Bordeaux mixt. (1 or 2%) against tobacco wildfire (*Phytophthora tabaci*) are apparent. In the seed bed satisfactory results were also obtained by various other Cu-contg. mixts. including the dusts Cusua and Cupulvit.

Cusua and Nosperit were also generally effective in the field tests in which, however, the outcome of the different treatments varied according to the year. Lime sulfur was not found to be altogether reliable. O. E. S.

Effects of modifications of the potato-spray program. E. O. Mader and F. M. Rodgett. N. Y. (Cornell) Agr. Expt. Sta., *Bull.* 621, 31 pp.(1935), cf. C. A. 29, 7667.—A summary of expts. carried on for 5 years shows that spraying and dusting are profitable practices even when late blight, caused by *Phytophthora infestans*, does not occur. 1lea beetles, leaf hoppers and early blight, caused by *Alternaria solani*, were well controlled by either Cu-lime dusts or by Bordeaux mixt. Cu has a distinct stimulative influence on potato foliage growth. The kind of lime used in prep. these dusts and sprays was of little significance, though slightly better results were obtained with high-Mg limes. In general, more Cu must be added per acre in the form of dust than as spray. Heavy applications of Cu spray or dusts late in the season resulted in tubers of uneven size and shape and a lower yield of No. 1 potatoes. C. R. Fellers.

Potato blight—a new method of control by chemical spraying. R. K. MacDowall. *Scottish J. Agr.* 18, 213-9(1935).—The infection of potatoes with blight about the time of harvesting caused by the presence of the fungus, *Phytophthora infestans*, on the haulm, was reduced by destroying the diseased green parts with a spray contg. about 16%  $H_2SO_4$  (15 gal. brown oil of vitriol per 100 gal. of spray). The treatment killed chickweed, charlock and other weeds and facilitated the mech. digging of the crop, it had no detrimental effect on the tubers themselves. K. D. Jacob.

A contribution to the knowledge of the chemical constitution of wart-infested potato soils. A. Němec. *Phytopath. Z.* 8, 301-5(1935), *Rev. Applied Mycol.* 14, 650-1.—Chem. analyses of some potato soils infested by wart disease (*Synchytrium endobioticum*) in northern Czechoslovakia indicated a general abundance of humus and an excess of  $IIPO_4$  and more or less markedly acid reaction. Exchangeable Ca and Mg was low. Similar conditions prevailed in other localities except for a deficiency of  $IIPO_4$ . The results do not permit one to draw conclusions as to the effect of soil constitution on the distribution of the disease but it is thought that the excessive use of stable manure may possibly be a contributory factor to its spread. Oden E. Sheppard.

Scale control by fumigation. G. Peters. *Indag.* 8, 208-10(1935).—A review of the properties of  $Ca(CN)_2$  and its use in the fumigation of citrus trees. K. D. J.

Studies on *Dytiscus tomentosus* Fabr. V. Experiments on the control of the raspberry and loganberry beetle, 1934. W. Steer. East Malling Research Sta., *2nd Ann. Rept.* 1934, 101-3, cf. C. A. 29, 2619.—A single application of a dust contg. 5-10% derris, applied in May to control the adult stage of the beetle, failed to give satisfactory results on raspberries, even when the rate of dusting was about 3 cwt./acre and the crude rotenone content of the dust as high as 0.36%. On loganberries derris gave as good results in controlling the larval stage of the beetle when it was incorporated in a spray of lime S and sulfite lye or of "colloidal Cu" and sulfite lye as when used with soft soap. The combined sprays gave an appreciable control of the loganberry cane spot disease caused by *Flasnoe veneta*. Satisfactory control of the beetle on blackberries was obtained by a single application of a soap-derris spray. K. D. Jacob.

Mexican bean beetle and its control. Ray Hutton. Mich. Agr. Expt. Sta., *Quarterly Bull.* 18, 7-9(1935).—The Mexican bean beetle, *Epiplatna corrupta*, is well controlled early in the season by a spray of 2 lb. of Mg arsenate in 100 gal. of water. From 25 to 30 lb. per acre of derris dust contg. 0.5% rotenone dusted on the infested plants also gave good control. Pyrethrum dusts were slightly less effective than derris. C. R. Fellers.

Further investigations on the control of wireworms by applications of potassium salts. Werner Subklewe. *J. mähr. Pflanz.* 31, 381-3(1935); cf. C. A. 28, 6917.—*Agriotes obscurus* L. from peat soils were more resistant to 0.2%

KCl soln. than those found in loamy sand soils. The differences in physical constitution of the larvae do not appear to be hereditary, but depend on the type and compn. of the soil in which the larvae occur. John O. Hardesty.

Earthworm control without the aid of water. R. B. Dawson and R. B. Ferro. *J. Board Greenkeeping Research* 4, 15-22 (1935).—Solid Pb arsenate, applied at the rate of 1.5-2.0 oz./sq. yd. or 4.00-5.25 cwt./acre, gave very effective control of earthworms in medium and medium-heavy soils. Pb arsenate, at the rate of 1.5 oz./sq. yd., did not give satisfactory results in a soil that was covered by a peaty mat. Under favorable conditions a marked reduction in worm activity was obtained in 11-28 days after application of Pb arsenate, but full results as regards worm control and improvement in quality of turf were not apparent until after a growing season. Pb arsenate was effective on medium soils for at least 4 years and possibly longer. It had no detrimental effect on the grass even at such heavy rates as 16 cwt. on the surface and 32 cwt./acre under the turf. K. D. Jacob.

Life history and control of the gladiolus thrips, *Taeniothrips gladioli* M. and S., in California. Howard L. McKenzie. *Calif. Agr. Expt. Sta., Circ.* 337, 1-16 (1935).—Treatment of infected bulbs by a 1:1000 HgCl<sub>2</sub> soln. for 17 hrs. is effective. Fumigation with Ca(CN)<sub>2</sub> with 2 oz. of the chemical per 100 cu. ft. with an exposure of 4 hrs. is effective. Approx. 1 lb. of naphthalene flakes per lb. of bulbs gives satisfactory control. An overdose of the naphthalene causes no injury. Weekly or biweekly sprays with a mixt. consisting of Mn arsenate 0.25 lb., brown sugar 4.12 lb. and water 6.25 gal. is recommended for control of the insects on the growing plants. K. D. Jacob.

Helminthosporium diseases of barley and their control. M. Mitra and R. D. Bose. *Indian J. Agr. Sci.* 5, 449-54 (1935).—The diseases were best controlled by treating the seed for 10 min. with 0.01 soln. of HgCl<sub>2</sub>. Treatment with either S, formalin, Uspulun or Ceresan was less effective. None of the treatments gave complete control of the diseases. K. D. Jacob.

A mealy bug new to Egypt (*Pseudococcus brevipes* Chitt.) on roots of Phoenix sp. and its control by the application of chemicals to the soil. M. Hosni and M. Shafik. *Ministry Agr. Egypt, Tech. Sci. Service Bull.* 159, 8 pp. (1935).—The insect was controlled by treating the soil around the plant with either *p*-dichlorobenzene (2-10 g.), *o*-dichlorobenzene (2-10 cc.), naphthalene (2-10 g.) or HgCl<sub>2</sub> (0.5 oz./gal. H<sub>2</sub>O). HgCl<sub>2</sub> had no deleterious effect on the plants and its action on the insects was more rapid than that of the other compds. K. D. Jacob.

The spittle insect or froghopper. Byrley F. Driggers and B. B. Pepper. *N. J. Agr. Expt. Sta., Bull.* 593, 1-4 (1935).—Of the 5 species of froghoppers, *Philaenus spumarius* Linn. is most abundant in N. J. Spraying exper. showed that nicotine sulfate and soap, used as a spray, and pyrethrum dust were ineffective in controlling the insect. Derris dusts carrying 0.75% rotenone gave satisfactory control of the nymphs on strawberries and sweet clover. C. R. Fellers.

The caterpillar of *Agrotis segetum* Schiff and its extermination. Josef Pazlar. *Livst. Cukurova* 54, 1-6 (1935).—Baits prepd. from 25 kg. dry wheat middlings mixed with 0.5 kg. Swinburn green were moistened with dil. molasses and placed as baits between the rows of beets. Other toxic agents used were NaF and Na fluosilicate in 0.5 kg. quantities. Sprays or powder preps. applied to the beet greens were effective only during the early life of the caterpillars. For the moths, shallow trays on posts 1 m. high spread over the fields contg. molasses with 0.5-1.0% Na arsenate were effective instruments of elimination. Frank Marech.

Combined washes—progress report. H. G. H. Kearns, R. W. Marsh and H. Martin. *Univ. Bristol Agr. Hort. Research Sta., Ann. Rept.* 1934, 100-25, cf. C. A. 28 2481.—No significant differences were observed in the degree of apple sawfly control obtained with sprays contg. nicot-

ine, lime S and spreader in which the spreaders (Agnal N, Isepon, Leithalite Weiting Isepon and Sulfonated Loral) were used at the rate of 5 oz./100 gal., the results were practically the same as when sulfite lye (60° Tw.) at 6 pints/100 gal. was used as spreader. Crude Ca-sulfonate at 24 oz./100 gal. also proved to be an effective spreader. Refined petroleum oil (1%) and lime-S (1%) combinations were applied post-blossom to a no. of coal varieties of apple without causing spray damage. Addition of 5 lb. cryst. FeSO<sub>4</sub> per gal. of lime-S concentrate reduced the formation of soot. As in combination sprays of lime S and Pb arsenate, obviated sludge production, rendered the spray deposit more clearly visible and increased its adherence. Combinations of refined petroleum oil (4.25%) and lime S were successfully applied to black currants at the pre-blossom stage for the simultaneous control of bag bud and caped bug. Excellent control of caped bug on black currants was obtained by the application of combinations of either semirefined or half-white petroleum oil (4.25%) and strained anthracene oil (4.25%) as dormant sprays. The substitution of oleic acid for Agnal W. B. as emulsifier increased slightly the efficiency of the spray. K. D. Jacob.

Studies on the ovicidal action of winter washes—1934 trials. M. D. Austin, S. G. Jary and H. Martin. *J. South-Eastern Agr. Coll. Wye, Kent No.* 36, 86-94 (1935), cf. C. A. 28, 69122.—Emulsions of asphaltic, paraffinic and naphthemic base oils were prepd. at 2 and 4% concns. by the 2-soln. oleic acid method with 0.6% oleic acid and 0.1% NaOH as emulsifier. In lab. exper., oils falling within the following limits appeared to be equally efficient as ovicides (when used at 4% concns.) against *L. psyllaeus* on red currants: (a) viscosity between 12<sup>nd</sup> and 80<sup>th</sup> Redwood I at 70°F. and (b) unsaponified residue 60-100% by vol. In general, the oils were not effective at 2% concns. Emulsions contg. 2-6% of either dibutyl phthalate, methylcyclohexanyl stearate or dimethyl tartrate were deficient in ovicidal properties as compared with petroleum oils of similar boiling range. Under field conditions an emulsion contg. strained anthracene oil 4 gal., semirefined petroleum oil 6 gal., oleic acid 1 gal. and NaOH 1.5 lb. per 100 gal. of spray was an effective ovicide against *L. psyllaeus* on red and black currant bushes; the spray retarded foliage development on the Tay's Prolific variety of red currant. K. D. Jacob.

Bordeaux mixture-nicotine combinations against aphids and apple scab. M. D. Austin, S. G. Jary and H. Martin. *J. South-Eastern Agr. Coll. Wye, Kent No.* 36, 95-9 (1935).—The sprays used were (1) ordinary Bordeaux mixt. (8.12/100) plus 6 oz. nicotine per 100 gal.; (2) Bordeaux-sulfite lye prepd. by dilg. 6 pints sulfite lye (60° Tw.) with water, adding to a suspension of 12 lb. hydrated lime in 80 gal. water, then adding 8 gal. 10% CuSO<sub>4</sub>, 5H<sub>2</sub>O soln. and 6 oz. nicotine and dilg. to 100 gal.; and (3) cottonseed oil-Bordeaux mixt. prepd. by adding 6 pints cottonseed oil and 5 gal. 10% CuSO<sub>4</sub>, 5H<sub>2</sub>O soln. to a suspension of 6 lb. hydrated lime in 90 gal. water and then adding 4 oz. nicotine, followed by vigorous stirring and dilg. to 100 gal. The modified sprays showed a fungicidal efficiency equal to that of ordinary Bordeaux mixt. The sprays contg. sulfite lye and cottonseed oil gave better control of aphid (*Amuraphis rosae*) than did the straight Bordeaux-nicotine spray but the results were inferior to those normally obtained with tar distillate sprays. The cottonseed oil Bordeaux spray caused less foliage damage and fruit (apple) russeting than did ordinary Bordeaux mixt. or the Bordeaux-sulfite lye spray. Similar amts. of Cu were deposited on the leaves by the 3 sprays. K. D. Jacob.

The control of apple scab. Allington Phipps and Newton Wonder. 1934. W. Goodwin, N. H. Pizer, E. S. Salmon and W. M. Ware. *J. South-Eastern Agr. Coll. Wye, Kent No.* 36, 55-61 (1935), cf. C. A. 28, 69187.—Fully effective control of the scab was obtained by spraying the trees with either ordinary Bordeaux mixt. (CuSO<sub>4</sub>, 5H<sub>2</sub>O S, hydrated lime 12 lb., water 100 gal.) or an emulsion of cottonseed oil and Bordeaux mixt. (6 pints of cottonseed oil and 4 gal. of 10% CuSO<sub>4</sub>, 5H<sub>2</sub>O soln., added simultane-

ously to 95 gal. water contg. 6 lb. hydrated lime).

**Special sulfur dust versus lime sulfur for apple-seab control.** John W. Hall *Scottish J. Agr.* 18, 254-9 (1935).—S dusts and lime-S sprays were equally effective in controlling apple seab. The leaves were not injured by lime-S spray (1:20) in the pre-blossom stage but were very susceptible to injury with a spray of this strength in the post-blossom stage.

**Preliminary experiments on the control of apple surface-eating tortricid [*Cacoccia podana* Scop.] larvae.** F. J. D. Thomas. Last Malling Research Sta., 22nd *Ann. Rept.* 1934, 205-7.—A 50% reduction of the damage caused by the insect was obtained by spraying with derris 2 and soft soap 5 lb/100 gal approx. 9 weeks after petal fall; the spray contained 0.0075% of crude rotenone. A spray contg. BaSiF<sub>6</sub> 4 and Agral N 1 lb/100 gal. was somewhat less effective.

**Observations on woolly aphid (*Eriosoma lanigerum* Hausm.) in 1934.** R. M. Greenslade. Last Malling Research Sta., 22nd *Ann. Rept.* 1934, 236-8; cf. *C. A.* 29, 2906.—Attack of the aphids on cankers and pruning wounds of apple trees was prevented by painting the parts with a prep. composed of castor machine oil (a heavy machine oil with a proportion of special soap) 1, water 3 quarts, nicotine (95-98%) 1 fluid oz. and flour 0.5 lb. The flour and water were boiled to a paste and the nicotine was added. The oil was stirred in and the whole shaken in a closed container to a smooth creamy emulsion.

**Field spraying and dusting trials on the control of apple blossom weevil [*Anthonomus pomorum* L.] and of apple sawfly [*Hoplocampa testudinea* Klug.] in 1934.** W. Steer and J. D. Thomas. East Malling Research Sta., 22nd *Ann. Rept.* 1934, 194-204; cf. *C. A.* 29, 2648.—Blossom capping, due to apple blossom weevil, was reduced approx. 50% by a derris dust (finely ground derris 10 and china clay 90%) contg. 0.36% rotenone applied to prevent oviposition. A BaSiF<sub>6</sub> dust (BaSiF<sub>6</sub> 72, Na<sub>2</sub>AlF<sub>6</sub> 8 and inert material 20%) was ineffective. The eggs were not affected by nicotine sprays. The eggs of the apple sawfly were very susceptible to nicotine; addn. of nicotine to the petal-fall, lime-S seab spray gave good control. Sawfly damage was also considerably reduced by the addn. of derris to the lime-S spray. Derris dusts gave good control of the migratory larvae. Addn. of a wetting agent to a petal-fall spray of lime-S and nicotine was not necessary if the trees were heavily sprayed. As a petal-fall spray, Pb arsenate, either alone or in combination with lime S, was less effective than lime S plus derris and a wetting agent.

**Apple blossom weevil experiments in 1934.** Impregnation of tree banding materials. R. M. Greenslade, A. M. Macsee and F. J. D. Thomas. Last Malling Research Sta., 22nd *Ann. Rept.* 1934, 180-4.—The best catches of weevils (*Anthonomus pomorum* (L.) Curt.) were obtained with corrugated cardboard bands treated with a 3% soln. of "Seckay." Wax A XIII in trichloroethylene. There were indications that the treatment attracted weevils to the bands. Promising results were also obtained with bands treated with a 7% soln. of tetrachlorobenzene in trichloroethylene and with a 3% soln. of "Seckay." Wax A 123 in trichloroethylene, resp. None of these treatments caused any mortality of the weevils collected in the bands and they were not injurious to the bark of the trees. The weevils seemed to be repelled by bands treated with either 30% solns. of "Seckay." Wax A 68 or A 65 in trichloroethylene, 10% soln. of "Seckay." Wax A 68 in *o*-dichlorobenzene, soln. contg. 10% "Seckay." Wax A 68 and 50% *p*-dichlorobenzene in trichloroethylene or 20% of "Seckay." Wax in creosote oil. The "Seckay." waxes are a series of chlorinated naphthalenes prep'd. by Imperial Chemical Industries.

**Resistance of the apple to fungal invasion.** A. S. Horne and P. Webb. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 165-76 (1934).—Tests were carried out at East Malling (1933) and in Northern Ireland (1932) on the relationship between resistance to fungal

invasion and chem. compn. of fruit from trees subjected to various fertilizer treatments. The results at the 2 stations were quite different, and in some cases diametrically opposite, indicating the complexity of the problem.

**Lime-sulfur sprays for the combined control of purple scale and rust mites.** W. L. Thompson. 11a. Agr. Expt. Sta., *Bull.* 282, 4-38 (1935).—Three lime-S applications made at intervals of 6 weeks or longer reduced purple-scale infestations and controlled rust mites for a period of 7-10 months. Two lime-S applications at an interval of 2 weeks gave results comparable with 1 emulsion application in the control of purple scale when applied after Bordeaux mixt. The effectiveness of liquid lime-S and dry lime-S against purple scale and rust mite was increased by the addn. of either wettable S or bentonite S.

**Removal of arsenic and lead residues from apples.** O. C. Roberts and J. K. Shaw. Mass Agr. Expt. Sta., *Bull.* 315 (*Ann. Rept.* 1934), 73 (1935).—Apples which had been sprayed with Pb arsenate were washed 3-5 min. in a specially designed washer contg. 1 1/2% HCl at 13-15° and were practically freed from As or Pb residues without apparent injury to the fruit.

**Field inspection work (on arsenic and lead on sprayed citrus fruits).** Gray Singleton. *Fla. Quart. Bull.* 42, No. 2, 11-20 (1933).—The Gutzeit test as simplified for use in field tests is described. Marked differences were found in the acidity of sprayed as compared with unsprayed citrus fruits. The acidity was always decreased in some cases where As was applied as dust it could not be detected on the foliage after 3-4 weeks but could be found on the rind of the fruit. Where As was applied as a spray with lime S it could be found on the leaves for several months. If there was Bordeaux mixt. spray residue on the trees when the As was applied, the As persisted as long as the leaves remained on the tree. If Bordeaux was applied after the As spray, and while the As was still on the leaves, the As became fixed. The effect of As, both on the fruit and on the tree, is stopped by the application of Bordeaux mixt. or other Cu spray or dust. That is, CuSO<sub>4</sub> prevents damage to the trees and fruits from As sprays.

**Relative amounts of arsenic found on the surface and in the tissues of celery plants which had been sprayed with lead arsenate or other arsenical poisons.** L. Longfield-Smith. *Fla. Quart. Bull.* 42, No. 2, 48-52 (1933).—In the analysis of 508 samples of celery and cabbage for As residues, a concn. of 5% H<sub>2</sub>SO<sub>4</sub> by vol. was preferable to 3% boiling HCl for the extrn. of As from the plant material. The heat of soln. aids the extrn. Boiling acid makes the tissues very soft and causes considerable org. matter to go into soln. This org. matter makes the evolution of AsH<sub>3</sub> much slower and results in a smaller amt. of As being obtained. It is desirable to wait for at least 3 hrs. before reading the strip. When the vegetable residue after extg. the As 2-3 times with 5% H<sub>2</sub>SO<sub>4</sub> was digested with H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> to destroy all trace of org. matter, and the remaining As detd., a large quantity, frequently 50-60% of that extd. by plain acid wash, was obtained. When the amt. of As detd. by acid wash was added to that obtained by subsequent digestion, the total As was invariably greater in amt. than the quantity which could be obtained by 1 digestion. This difference may be as much as 60%. Numerous data are given showing that large amts. of As are found on celery and cabbage which have been sprayed with As sprays.

**Enforcement of the arsenical spray law.** Report of the Chemist. L. Longfield-Smith. *Fla. Quart. Bull.* 42, No. 2, 27-33 (1933); cf. *C. A.* 28, 5555.—Over 2000 samples of fertilizers, soils, citrus fruits, twigs and miscellaneous vegetables were exam'd. for As content. Fla. phosphate rock varied from a trace to 0.004% As<sub>2</sub>O<sub>3</sub>. Nitrophoska averaged 0.076% As<sub>2</sub>O<sub>3</sub>. Tankage, raw bone meal, castor pomace, cottonseed meal, German kaimie, KNO<sub>3</sub> and NaNO<sub>3</sub> (Chilean) contained only traces of As. Synthetic NaNO<sub>3</sub> contained 0.031% of As<sub>2</sub>O<sub>3</sub>. Twelve colored figures showing As standards by the Gutzeit test



are given. A detailed description of the improved Gutzzeit method is presented. C. R. Fellers

Influence of various sulfur-containing fungicides on transpiration. J. D. Wilson and H. A. Runnels. Ohio Agr. Expt. Sta., *Bimonthly Bull.* 175, 146-8 (1935); cf. C. A. 29, 6353<sup>2</sup>.—Eighteen S-contg spray materials were applied to *Coleus* plants to det. their transpiration rates. Liquid lime-S plus Ca(OH)<sub>2</sub> and Kolofof, which contains a high percentage of bentonite, were the only materials tested which caused an increase in transpiration comparable with that of Bordeaux mixt. The transpiration increases were approx. 1/3 to 1/2 as great as those caused by Bordeaux. Dry lime-S and liquid lime-S caused an increase in transpiration over the untreated checks of more than 50% during the night period. Any injury resulting from the application of S-contg. materials to plant tissue cannot be due to excessive increase in transpiration. C. R. Fellers

Action at a distance of metals on some species of fungi. E. Corneli. *Riv. Pat. reg.* 24, 397-406 (1934), *Rev. Applied Mycol.* 14, 646.—When spore suspensions of *Penicillium glaucum* in hanging-drop cultures were exposed to a lead disk in hermetically sealed glass containers only a small percentage of the spores germinated, the figure being highest when the Pb was farthest away (3.5 mm.) and lowest when it was nearest (1 mm.). When the spores were removed to normal conditions in the absence of the metal, growth was resumed, and was more rapid than in controls. The radiation effect was less marked on spore masses than on single spores, and when open containers or Cu and Ag disks in closed ones were used no appreciable effect of the radiation resulted. The distance of the metal from the fungus was less important than the area of the disk and the vol. of the container, the effect of a disk of given size at a given distance increasing as the vol. of the container decreased. *Ascochyta pisi* and *Tricothecium roseum* were less susceptible to the radiation than was *P. glaucum*, the uredospores of *Uromyces betae* remained unaffected. C. believes that germination was reduced only in sealed containers because of the fact that in these there was a constantly increasing accumulation of secondary radiation or a progressively more complete ionization of the atm. Oden E. Sheppard

Contact insecticides from fatty alcohols. E. W. Bousquet, P. L. Salzberg and H. F. Dietz. *Ind. Eng. Chem.* 27, 1342-4 (1935).—The higher fatty alcs were found to be effective against aphids and do not react with hard water. Numerous long-chained alkyl derivs were synthesized and tested. The rhodanates were found to be particularly effective and satisfactory (cf. C. A. 29, 2658<sup>1</sup>). A max. insecticidal effect was found for rhodanates contg 12 C atoms. The normal, primary compds were used, as branch-chain rhodanates were not promising. No one phys. property was correlated with this peak in toxic action, and it is probably the resultant of several properties. The 12-C homolog is safe on many kinds of plants, in the proper dispersion medium. E. R. Rushton

Insect and allied pests of cultivated mushrooms. V. Control of flies and mites. M. D. Austin and S. G. Jary. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 167-10 (1935).—Sprays contg nicotine (98%) at a diln of 1:1600 were ineffective in killing eggs of *Sciara fenestrata* in lab expts. The addn of Sulfonated Loral (1:4000) did not increase the efficiency of the spray. Under the most favorable exptl conditions a 60% mortality of the larvae of *S. fenestrata* was obtained with nicotine sprays. Good control of the adult flies was obtained by spraying the mushroom beds with nicotine (98%) at the rate of 1 oz./10 gal water or by fumigating the cellars with nicotine (0.75-1.00 oz. per 1000 cu. ft. of space). The larvae were not affected by spraying with 1:1600 solns of NaCl. The wetting agents, Sulfonated Loral and Na γ-sulfonate, were harmless to growing mushrooms when they were applied as sprays at 1:2000 diln. A proprietary oil emulsion, when carefully mixed and lightly sprayed on growing mushrooms, checked the injury caused by *Tyroglyphid* mites; a light application, when the temp of the liquid

was 104°F., produced no injury to mushrooms. Warm water, applied at a temp. of 110°F., also caused no injury to mushrooms. K. D. Jacob

The effect of antioxidants on highly concentrated pyrethrum extracts. C. B. Gnadinger, C. S. Corl and C. A. Clark. *Soap (Sanitary Products Sect.)* 11, No. 10, 95, 97, 99 (1935).—Kerosene (K) exts of low pyrethrum content (2.5%) were quite stable over periods of 4-9 months but concd exts (10-15%) were unstable during prolonged storage. In order to prep concd pyrethrum exts. which would not have the antioxidant (A) present in K or other mineral oils, decahydronaphthalene (D) was used as a solvent. Neither D nor K interfered with the Cu reduction method for analyzing total pyrethrum content (cf. C. A. 23, 5546). Before proceeding with the analysis the exts are first dild. with petr ether, chilled overnight and filtered to eliminate altered or oxidized pyrethrins. At 35° concd pyrethrum ext. in D or K contg no A had 12-26% pyrethrum content in 2-4 months. Of 19 A tried on concd. D-pyrethrum ext. held at 35°, one proprietary material at 0.1% prevented any loss for 60 days but the loss was 9.2% after 81 days, and α-naphthylamine (0.5%) and thymol (0.5%) had similar effect. Conc'd K exts. contg. the same proprietary A were quite stable at 6° for 94 days but lost up to 10.4% at 35° storage. Henry H. Richardson

Fused needle of species of *Pinus*—progress report. H. E. Young. *Queensland Agr. J.* 44, 286-98 (1935).—The condition is manifested by a resinosis of the terminal buds and a twisting and adhesion of each needle in the fascicle. There was no relation between the chem. compn of the soil and the occurrence of the disease. Treatment of individual trees with the essential elements and also with B, Zn, Cu and Al gave neg results. Chem. analyses of material collected from diseased and healthy trees showed no great dissimilarities. K. D. Jacob

A new method for precision testing in the laboratory of the toxicity of lime sulfur and of Bordeaux mixture as protective fungicides. Progress report. H. B. S. Montgomery and M. H. Moore. *East Malling Research Sta., 22nd Ann. Rept.* 1934, 217-22.—Mark individual glass slides with a ring 15 mm. in diam. cut into the glass with a diamond by means of a lathe. Clean the slides very carefully by successive treatment with warm dild. HNO<sub>3</sub>, CrO<sub>3</sub> cleaning soln. and ether, with intervening washing with water. Store the slides in abs. alc. and flame and cool just before using. Transfer 0.015 cc. of the spray fluid to the marked-off area of the slide from a graduated fine-glass tube. Spread the spray fluid to the edge of the groove with a finely pointed glass rod, dry the deposit in the lab and store the slide for 1 day in an unheated water oven at approx. 15-18°. Support the slide over a pool of distd water for several hrs. before application of the spore suspension. Apply 0.05 cc. of the spore suspension (spores of *Venturia aequalis* Aderh. were used by the authors) and spread it in the groove. Incubate the slide in an individual moist chamber for 24 hrs. and det. the degree of germination by counting under the high power field of the microscope. At least 10 test slides and 10 control slides are recommended for each spray fluid. Under these conditions germination of the spores was almost completely inhibited by lime S at a concn. of 1:50 and free germination was entirely prevented by Bordeaux mixt contg 0.025% CuSO<sub>4</sub> in which hydrated lime was used at the rate of 3 parts to 2 parts CuSO<sub>4</sub>. Methods of raising large supplies of viable spores of *V. aequalis* in pure culture are discussed. K. D. Jacob

Preliminary laboratory tests of bactericides on the plum bacterial canker organism [*Pseudomonas mors prunorum*]. H. Wormald. *East Malling Research Sta., 22nd Ann. Rept.* 1934, 151-5, cf. C. A. 29, 2051<sup>1</sup>.—Growth of the organism was completely prevented by either phenol 0.1, EtOH 8.0, CH<sub>3</sub>O 0.01, quinosol 0.005, CuSO<sub>4</sub> 0.2, CuCl<sub>2</sub> 0.01, ZnSO<sub>4</sub> 0.005 or HgCl<sub>2</sub> 0.0005%. In culture media that reacted only slightly with CuSO<sub>4</sub>, the organism was killed by contact for 10 min with a 0.01% soln of CuSO<sub>4</sub>. Under these conditions the bactericidal action of CuSO<sub>4</sub> was much more rapid than that of ZnSO<sub>4</sub>.

Tar oil at 1% concn killed the organism in 10 min., though at 0.1% it did not kill in 1 hr. K D Jacob

Laboratory trials of wetters against woolly aphids, *Eriosoma lanigerum* (Hausm.). R. M. Grainside, Last Malling Research Sta., 22nd Ann Rept 1934, 185-90.—Of a no. of wetting agents tested with 0.025% nicotine soln., Agral II was the only one that showed any promise of increasing the effectiveness of the nicotine. At 0.5% concn, with nicotine it gave results as good as those obtained with nicotine plus 1% of soft soap, but at this concn Agral II may cause damage to the fruit (apples). At 1% concn Agral II completely wetted tufts of woolly aphid "wool" in 40 min., while none of the other wetting agents showed signs of wetting before 1 hr. Soft soap at 0.5% concn was a much better wetting agent than Agral II at 0.1% concn. None of the preps had any apparent solvent action on the wax threads of the "wool." The strawberry tarsonemid mite was not controlled by spraying the strawberry plants with either 3% lime S plus 1% of a no. of different wetting agents, lime S alone (1.14%), petroleum emulsion (2-5%), paraffin emulsion (2.50%) or a spray composed of liver of S 30 oz and soft soap 10 20 lb per 100 gal. K D Jacob

Effect of molds and other seed-borne fungi on germination. F A McLaughlin, Mass Agr Expt Sta., Control Bull 77, 58-60 (1935).—Thirty-five lots of fungus-infected sweet-corn seeds were divided into 3 lots and 1 lot was dusted with Et Hg phosphate, 1 was treated with 1 1000 HgCl<sub>2</sub> for 10 min., and the last lot was left untreated. The seeds were germinated and the seedlings noted for fungus diseases. The org Hg prep and the HgCl<sub>2</sub> were equally effective in destroying fungi on the seeds. *Penicillium* and *Rhizopus*, 2 of the most abundant seedling fungi, are well controlled by seed treatment. C R Fellers

A field spraying trial of combined fungicide-contact insecticide sprays in 1934. Progress report. M H Moore and H B S Montgomery, East Malling Research Sta., 22nd Ann Rept 1934, 208 16, of C A 29, 1202.—Fairly good control of apple scab (*Venturia inaequalis* Aderh.) on Cox's Orange Pippin was obtained by 1 pre-blossom (pink-bud) followed by 2 post-blossom applications of lime S (1:30 pre-blossom and 1 100 post-blossom) to which, at petal fall, nicotine was added, with either Sulfonated Loral, "Lethalate" Wetting Prep or sulfite lye as spreader. Very good control of sawfly (*Hoplocampa testudinea* Klug) and red spider (*Oligonychus ulmi* Koch) was obtained with all spray treatments. There was some evidence that "Lethalate" Wetting Prep was the most efficient of the wetting agents tested. K D Jacob

Tolerance of cabbage seedlings to insecticide dips for the control of aphids and cabbage worms. Harry G Walker and Lauren D. Anderson, Va Truck Expt Sta., Bull. 86, 1203-10 (1935).—Expts. showed that the dipping of cabbage plants infected with aphids and cabbage worms in 1-400 concns. of pyrethrum or derris for 30 sec. destroyed these insects and caused only slight reduction in plant survival. If only aphids were present, dipping in a soln. of 0.7 lb. of nicotine sulfate or Black Leaf 40 in 50 gal. of water was also effective. Pyrethrum and derris lost their activity rapidly in aq. solns. and only enough soln. should be made up to last 1 day. C R F

Fish-poisoning vines, a native insecticide in Kwangsi Chin-Pi Chen, Science (China) 19, 1405-30 (1935).—*Millettia parhyrcarpa* Benth., commonly known as "fish-poisoning vines" in China, is widely distributed in the mountains of Kwangsi province, China. C. finds this plant contains a large amt. of saponin (C<sub>24</sub>H<sub>40</sub>O<sub>11</sub>) and possibly also considerable rotenone. Mixts of this plant with soap or tea oil serve not only as good insecticides, but also as contact poison and stomach poison, the efficiency for the latter purposes being not inferior to derris, though far cheaper. C. L. Tseng

Some plants with insecticidal properties. A. Barcellos Fagundes, Bol. ministerio agr. e com. (Brazil) 24, Numbers 1-3, 69-75 (1935).—A list of 89 toxic or insecticidal

plants with their uses and bibliography of 24 references. C. W. Whittaker

The applicability of hydrogen peroxide as a seed disinfectant. F. Pichler, Phytopath. Z. 8, 245-51 (1935); Rpt. Applied Mycol. 14, 619-20.—Lab. and field expts using up to 3% H<sub>2</sub>O<sub>2</sub> for wheat and barley and 4-5% for oats failed to give control of wheat bunt (*Tilletia caries*), covered smut of barley (*Ustilago hordei*) and loose smut of oats (*U. avenae*). These results are contrary to those of Kresser and Porthem (C. A. 29, 1571). O. E. S.

Investigations on machinery used in spraying II. Nozzles. Cornelius Davies and G R B Smyth-Home-wood, J. South Eastern Agr. Coll. 11, 32, Kent No 36, (2 85) (1935).—The surface area of trees covered by spray fluid was detd. by a method previously described (C. A. 28, 6214). The results obtained in the field under com. conditions showed an av. cover of about 78%. An analysis of the uniformity and degree of atomization of the spray deposit on the trees used was also made and the "spray cover efficiency" (based on area covered, atomization and uniformity) averaged 62%. In field expts under various pump and nozzle conditions the cover ranged from 60 to nearly 92%. K D Jacob

Crab grass control on lawns. Howard B Sprague, N J Agr Expt Sta., Circ 354, 1-4 (1935).—The use of 5-10 lbs fertilizers at the rate of 15-25 lb per 1000 sq ft encourages desirable grasses which will overgrow the crab grass, *Digitaria sanguinalis*. The use of such org. fertilizers as bone meal and tankage alone benefits crab grass more than desirable lawn grasses. Highly acid soils should be rendered only slightly acid by the use of Ca(OH)<sub>2</sub>. C R Fellers

Plant fish poisons as insecticides. Roland M Whittaker and Arthur L Whittaker, Poultry Sci 14, 351-4 (1935).—Body lice on badly infected chickens were completely killed in 18 hrs by dusting the birds, by the pinch method, with small amts of powd. derris contg 1% rotenone. In expts on the toxicity of rotenone and derris to chickens, no visible effects were obtained by the oral administration of either 0.2 g. of derris dust or 0.02 g. of pure rotenone in gelatin capsules fed twice in 12 hrs to cockerels 4 weeks old; a single dose of 0.1 g. of rotenone had no effect. The egg-laying ability of hens was not affected by the ingestion of rotenone. K D Jacob

A homemade cattle spray. C B Dibble, Mich. Agr. Expt. Sta., Quarterly Bull 18, 5-7 (1935).—A good easily prep'd cattle spray is made by mixing 0.5 lb. of ground pyrethrum flowers with 1 gal. of either oil or mineral spirits. Shake occasionally for 48 hrs, strain out the residue and use the clear liquid as a spray. Specifications for oil or mineral spirits are: flash 104°, sp. gr. 47.6, color 26; no S, initial b. p. 305°; 20% distils at 330°, 50% at 346° and 90% at 386° with the end point at 423°. C R Fellers

Weed killers. R. M. Woodman, The Horticultural Education Assoc. Yearbook 2, 77-83 (1933).—A report on the observation of various investigators indicates that an application of approx. 200 lb. per acre of NaClO<sub>2</sub> broadcasted in autumn cleared very weedy land of couch and creeping bent grasses, crowfoot and shallow-rooted perennial weeds, but did not seriously affect deep-rooted weeds. Five and 15% solns. with a spreader gave excellent control of most weeds. NaClO<sub>2</sub> was not efficient against aquatic weeds. Two to 5% solns. of NaAsO<sub>2</sub> are excellent for clearing land in autumn. Four applications each of a mixt. contg. 150 lb. of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 50 lb. calcined FeSO<sub>4</sub>, and 1/2 ton of soil per acre of old lawn turf completely eradicated white clover, daisy, mouse-ear, chickweed, moss, pearlwort, etc. The NH<sub>4</sub><sup>+</sup> ion appears to have a selective toxic action on weeds. An application of 800 lb. of cyanamide per acre immediately prior to active growth of lawn is said to destroy most weeds. A soln. of 1 lb. of As-O<sub>3</sub> in 8 gals. of H<sub>2</sub>O applied at 240 gal. per acre controlled all weeds except yarrow, dandelion, dock and sorrel, as effectively as the same vol. of 2.5% NaClO<sub>2</sub>. Wheat has been safely sown without ill effects after spraying soil with 1% NaClO<sub>2</sub> but with more concd solns. sowing should be left until the following season.

KCl soln than those found in loamy sand soils. The differences in physiol. constitution of the larvae do not appear to be hereditary, but depend on the type and compn of the soil in which the larvae occur. John O. Hardesty.

Earthworm control without the aid of water. R. B. Dawson and R. B. Ferro. *J. Board Greenkeeping Research* 4, 58-72 (1935).—Solid Pb arsenate, applied at the rate of 1.5-2.0 oz./sq. yd. or 4.00-5.25 cwt./acre, gave very effective control of earthworms in medium and medium heavy soils. Pb arsenate, at the rate of 1.5 oz./sq. yd., did not give satisfactory results in a soil that was covered by a peaty mat. Under favorable conditions a marked reduction in worm activity was obtained in 11-28 days after application of Pb arsenate, but full results as regards worm control and improvement in quality of turf were not apparent until after a growing season. Pb arsenate was effective on medium soils for at least 4 years and possibly longer. It had no detrimental effect on the grass even at such heavy rates as 16 cwt. on the surface and 32 cwt./acre under the turf. K. D. Jacob.

Life history and control of the gladiolus thrips, *Taeniothrips gladioli* M. and S., in California. Howard L. McKenzie. *Calif. Agr. Expt. Sta., Circ.* 337, 1-16 (1935).—Treatment of infected bulbs by a 1:1000 HgCl<sub>2</sub> soln for 17 hrs is effective. Fumigation with Ca(CN)<sub>2</sub> with 2 oz. of the chemical per 100 cu. ft. with an exposure of 4 hrs is effective. Approx. 1 lb. of naphthalene flakes per lb. of bulbs gives satisfactory control. An overdose of the naphthalene causes no injury. Weekly or biweekly sprayings with a mixt. consisting of Mn arsenate 0.25 lb., brown sugar 4.12 lb. and water 6.25 gal. is recommended for control of the insects on the growing plants. C. R. Fellers.

Helminthosporium diseases of barley and their control. M. Mitra and R. D. Bose. *Indian J. Agr. Sci.* 5, 449-54 (1935).—The diseases were best controlled by treating the seed for 10 min. with 0.01 soln of HgCl<sub>2</sub>. Treatment with either S. formalin, Uspulun or Ceresan was less effective. None of the treatments gave complete control of the diseases. K. D. Jacob.

A mealy bug new to Egypt (*Pseudococcus brevipes* Kell.) on roots of Phoenix sp. and its control by the application of chemicals to the soil. M. Hosni and M. Shafik. *Ministry Agr. Egypt, Tech. Sci. Service Bull.* 159, 8 pp. (1935).—The insect was controlled by treating the soil around the plant with either *p*-dichlorobenzene (2-10 g.), *o*-dichlorobenzene (2-10 cc.), naphthalene (2-10 g.) or HgCl<sub>2</sub> (0.5 g./gal. H<sub>2</sub>O). HgCl<sub>2</sub> had no deleterious effect on the plants and its action on the insects was more rapid than that of the other compds. K. D. Jacob.

The spittle insect or froghopper. Byrley F. Druggers and B. B. Pepper. *N. J. Agr. Expt. Sta., Bull.* 593, 1-4 (1935).—Of the 5 species of froghoppers, *Philaenus spumarius* Linn. is most abundant in N. J. Spraying expts. showed that nicotine sulfate and soap, used as a spray, and pyrethrum dust were ineffective in controlling the insect. Derris dusts carrying 0.75% rotenone gave satisfactory control of the nymphs on strawberries and sweet clover. C. R. Fellers.

The caterpillar of *Agrotis segetum* Schiff and its extermination. Josef Pazler. *Listy Cukrovár* 54, 1-6 (1935).—Baits prepd. from 25 kg. dry wheat middlings mixed with 0.5 kg. Swimbrod green were moistened with dil. molasses and placed as baits between the rows of beets. Other toxic agents used were NaF and Na fluosulfate in 0.5 kg. quantities. Sprays or powd. prepn. applied to the beet greens were effective only during the early life of the caterpillars. For the moths, shallow trays on posts 1 m. high spread over the fields contg. molasses with 0.5-1.0% Na arsenate were effective instruments of elimination. Frank Mareš.

Combined washes—progress report. H. G. H. Kearns, R. W. Marsh and H. Martin. *Univ. Bristol Agr. Hort. Research Sta., Ann. Rept.* 1934, 109-25, cf. C. A. 28, 2487.—No significant differences were observed in the degree of apple sawfly control obtained with sprays contg. micro-

time, lime S and spreader in which the spreaders (Agrisol N, Igepon, Lethalate Wetting Prepn and Sulfonated Lorol) were used at the rate of 8 oz./100 gal.; the results were practically the same as when sulfite lye (60° Tw.) at 6 pints/100 gal. was used as spreader. Crude Ca γ-sulfonate at 24 oz./100 gal. also proved to be an effective spreader. Refined petroleum oil (1%) and lime-S (1%) combinations were applied post-blossom to a no. of com. varieties of apple without causing spray damage. Addition of 5 lb. cryst. FeSO<sub>4</sub> per gal. of lime-S concentrate reduced the formation of soot. As in combination sprays of lime S and Pb arsenate, obviated sludge production, rendered the spray deposit more clearly visible and increased its adherence. Combinations of refined petroleum oil (4.25%) and lime S were successfully applied to black currants at the pre-blossom stage for the simultaneous control of big bud and capsid bug. Excellent control of capsid bug on black currants was obtained by the application of combinations of either semirefined or half-white petroleum oil (4.25%) and strained anthracene oil (4.25%) as dormant sprays. The substitution of oleic acid for Agral W. B. as emulsifier increased slightly the efficiency of the spray. K. D. Jacob.

Studies on the ovicidal action of winter washes—1934 trials. M. D. Austin, S. G. Jary and H. Martin. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 86-94 (1935); cf. C. A. 28, 6422.—Emulsions of asphaltic, paraffinic and naphthenic base oils were prepd. at 2 and 4% concns by the 2-soln. oleic acid method with 0.6% oleic acid and 0.1% NaOH as emulsifier. In lab. expts., oils falling within the following limits appeared to be equally efficient as ovicides (when used at 4% concns) against *L. pabulinus* on red currants: (a) viscosity between 120° and 800° Redwood I at 70°F and (b) unsulfonated residue 60-100% by vol. In general, the oils were not effective at 2% concns. Emulsions contg. 2-6% of either dibutyl phthalate, methyleyclohexanyl stearate or diamyl tartrate were deficient in ovicidal properties as compared with petroleum oils of similar boiling range. Under field conditions an emulsion contg. strained anthracene oil 4 gal., semirefined petroleum oil 8 gal., oleic acid 1 gal. and NaOH 1.5 lb. per 100 gal. of spray was an effective ovicide against *L. pabulinus* on red and black currant bushes; the spray retarded foliage development on the Fay's Prolific variety of red currant. K. D. Jacob.

Bordeaux mixture-nicotine combinations against aphids and apple scab. M. D. Austin, S. G. Jary and H. Martin. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 95-9 (1935).—The sprays used were (1) ordinary Bordeaux mixt. (8.12.100) plus 6 oz. nicotine per 100 gal.; (2) Bordeaux-sulfite lye prepd. by dilg. 6 pints sulfite lye (60° Tw.) with water, adding to a suspension of 12 lb. hydrated lime in 80 gal. water, then adding 8 gal. 10% CuSO<sub>4</sub> 5H<sub>2</sub>O soln and 6 oz. nicotine and dilg. to 100 gal.; and (3) cottonseed oil-Bordeaux mixt. prepd. by adding 6 pints cottonseed oil and 5 gal. 10% CuSO<sub>4</sub> 5H<sub>2</sub>O soln to a suspension of 6 lb. hydrated lime in 90 gal. water and then adding 4 oz. nicotine, followed by vigorous stirring and dilg. to 100 gal. The modified sprays showed a fungicidal efficiency equal to that of ordinary Bordeaux mixt. The sprays contg. sulfite lye and cottonseed oil gave better control of aphus (*Anuraphis rosae*) than did the straight Bordeaux-nicotine spray but the results were inferior to those normally obtained with tar distillate sprays. The cottonseed oil-Bordeaux spray caused less foliage damage and fruit (apple) russeting than did ordinary Bordeaux mixt. or the Bordeaux-sulfite lye spray. Similar amts. of Cu were deposited on the leaves by the 3 sprays. K. D. Jacob.

The control of apple scab. Allington Phippin and Newton Wonder. 1934. W. Goodwin, N. H. Fizer, E. S. Salmon and W. M. Ware. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 55-61 (1935); cf. C. A. 28, 6918.—Equally effective control of the scab was obtained by spraying the trees with either ordinary Bordeaux mixt. (CuSO<sub>4</sub> 5H<sub>2</sub>O 8, hydrated lime 12 lb., water 100 gal.) or an emulsion of cottonseed oil and Bordeaux mixt. (6 pints of cottonseed oil and 4 gal. of 10% CuSO<sub>4</sub> 5H<sub>2</sub>O soln. added simultane-

only to 0.5 gal. water contg. 6 lb. hydrated lime)

K. D. Jacob

Special sulfur dust versus lime sulfur for apple-scab control. John W. Hall. *Scottish J. Agr.* 18, 234 (1935).—Sulfur dusts and lime-S sprays were equally effective in controlling apple scab. The leaves were not injured by lime-S spray (1:20) in the pre-blossom stage but were very susceptible to injury with a spray of this strength in the post-blossom stage.

K. D. Jacob

Preliminary experiments on the control of apple surface-eating tortricid [*Cacoreia podana* Scop.] larvae. J. D. Thomas. East Malling Research Sta., 22nd Ann. Rept. 1934, 205-7.—A 50% reduction of the damage caused by the insect was obtained by spraying with derris 2 and soft soap 5 lb./100 gal. approx. 4 weeks after petal fall; the spray contained 0.06% of crude rotenone. A spray contg.  $\text{BaSiH}_4$  4 and Agral N 1 lb./100 gal. was somewhat less effective.

K. D. Jacob

Observations on woolly aphid (*Eriosoma lanigerum* Hausm.) in 1934. R. M. Greenblade, 1st Malling Research Sta., 22nd Ann. Rept. 1934, 236-8, cf. C. A. 29, 29904.—Attack of the aphids on cankers and pruning wounds of apple trees was prevented by painting the parts with a prep. composed of castor machine oil (a heavy machine oil with a proportion of special soap) 1, water 4 quarts, nicotine (85-94%) 1 fluid oz. and flour 0.5 lb. The flour and water were boiled to a paste and the nicotine was added. The oil was stirred in and the whole shaken in a closed container to a smooth creamy emulsion.

K. D. Jacob

Field spraying and dusting trials on the control of apple blossom weevil (*Anthonomus pomorum* L.) and of apple sawfly [*Homocidus testudinea* Klug.] in 1934. W. Steer and I. J. D. Thomas. East Malling Research Sta., 22nd Ann. Rept. 1934, 191-204; cf. C. A. 29, 29449.—Blossom capping, due to apple blossom weevil, was reduced approx. 50% by a derris dust (finely ground derris 10 and china clay 90%) contg. 0.30% rotenone applied to prevent oviposition. A  $\text{BaSiH}_4$  dust ( $\text{BaSiH}_4$  72,  $\text{NaAlF}_6$  8 and inert material 20%) was ineffective. The eggs were not affected by nicotine sprays. The eggs of the apple sawfly were very susceptible to nicotine; addn. of nicotine to the petal-fall, lime-S scab spray gave good control. Sawfly damage was also considerably reduced by the addn. of derris to the lime-S spray. Derris dusts gave good control of the migratory larvae. Addn. of a wetting agent to a petal-fall spray of lime-S and nicotine was not necessary if the trees were heavily sprayed. As a petal-fall spray, Pb arsenate, either alone or in combination with lime S, was less effective than lime S plus derris and a wetting agent.

K. D. Jacob

Apple blossom weevil experiments in 1934. Impregnation of tree banding materials. R. M. Greenblade, A. M. Mawer and I. J. D. Thomas. East Malling Research Sta., 22nd Ann. Rept. 1934, 180-4.—The best catches of weevils (*Anthonomus pomorum* (L.) Curt.) were obtained with corrugated cardboard bands treated with a 3% soln. of "Seckay" Wax A XIII in trichloroethylene. There were indications that the treatment attracted weevils to the bands. Promising results were also obtained with bands treated with a 7% soln. of tetrachlorobenzene in trichloroethylene and with a 33% soln. of "Seckay" Wax A 123 in trichloroethylene, resp. None of these treatments caused any mortality of the weevils collected in the bands and they were not injurious to the bark of the trees. The weevils seemed to be repelled by bands treated with either 50% solns. of "Seckay" Wax A 68 or A 83 in trichloroethylene, 10% soln. of "Seckay" Wax A 68 in *o*-dichlorobenzene, soln. contg. 10% "Seckay" Wax A 68 and 30% *p*-dichlorobenzene in trichloroethylene or 20% of "Seckay" Wax in creosote oil. The "Seckay" waxes are a series of chlorinated naphthalenes prep. by Imperial Chemical Industries.

K. D. Jacob

Resistance of the apple to fungal invasion. A. S. Horne and P. Welsh. Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 165-76 (1934).—Tests were carried out at 1st Malling (1933) and in Northern Ireland (1932) on the relationship between resistance to fungal

invasion and chem. compn. of fruit from trees subjected to various fertilizer treatments. The results at the 2 stations were quite different, and in some cases diametrically opposite, inheriting the complexity of the problem.

A. Lapieneu-Coutin

Lime-sulfur sprays for the combined control of purple scale and rust mites. W. L. Thompson. 11st Agr. Expt. Sta., Bull. 282, 1 38 (1935).—Three lime-S applications made at intervals of 6 weeks or longer reduced purple-scale infestations and controlled rust mites for a period of 7-10 months. Two lime-S applications at an interval of 2 weeks gave results comparable with 1 emulsion application in the control of purple scale when applied after Bordeaux mixt. The effectiveness of liquid lime-S and dry lime-S against purple scale and rust mite was increased by the addn. of either wettable S or bentonite S.

C. R. Fellers

Removal of arsenic and lead residues from apples. O. C. Roberts and J. K. Shaw. Mass. Agr. Expt. Sta., Bull. 315 (Ann. Rept. 1931), 73 (1935).—Apples which had been sprayed with Pb arsenate were washed 3-5 min. in a specially designed washer contg. 1 3/4% HCl at 17-15° and were practically freed from As or Pb residues without apparent injury to the fruit.

C. R. Fellers

Field inspection work (on arsenic and lead on sprayed citrus fruits). Gray Singleton. Fla. Quart. Bull. 42, No. 2, 11 26 (1933). The Gutzelt test as simplified for use in field tests is described. Marked differences were found in the acidity of sprayed as compared with unsprayed citrus fruits. The acidity was always decreased in some cases where As was applied as dust it could not be detected on the foliage after 3-4 weeks but could be found on the rind of the fruit. Where As was applied as a spray with lime S it could be found on the leaves for several months. If there was Bordeaux mixt spray residue on the trees when the As was applied, the As persisted as long as the leaves remained on the tree. If Bordeaux was applied after the As spray, and while the As was still on the leaves, the As became fixed. The effect of As, both on the fruit and on the tree, is stopped by the application of Bordeaux mixt or other Cu spray or dust. That is,  $\text{CuSO}_4$  prevents damage to the trees and fruits from As sprays.

C. R. Fellers

Relative amounts of arsenic found on the surface and in the tissues of celery plants which had been sprayed with lead arsenate or other arsenical poisons. I. Longfield-Smith. Fla. Quart. Bull. 42, No. 2, 48 52 (1933).—In the analysis of 10% samples of celery and cabbage for As residues, a concn. of 5%  $\text{H}_2\text{SO}_4$  by vol. was preferable to 3% boiling HCl for the extrn. of As from the plant material. The heat of soln. and the extrn. Boiling acid makes the tissues very soft and causes considerable org. matter to go into soln. This org. matter makes the evolution of  $\text{AsH}_3$  much slower and results in a smaller amt. of As being obtained. It is desirable to wait for at least 3 hrs. before reading the strip. When the vegetable residue after extr. the As 2-3 times with 5%  $\text{H}_2\text{SO}_4$  was digested with  $\text{H}_2\text{SO}_4$  and  $\text{HNO}_3$  to destroy all trace of org. matter, and the remaining As detd. a large quantity, frequently 50-60% of that extrd. by plain acid wash, was obtained. When the amt. of As detd. by acid wash was added to that obtained by subsequent digestion, the total As was invariably greater in amt. than the quantity which could be obtained by 1 digestion. This difference may be as much as 60%. Numerous data are given showing that large amts. of As are found on celery and cabbage which have been sprayed with As sprays.

C. R. Fellers

Enforcement of the arsenical spray law. Report of the Chemist. L. Longfield-Smith. Fla. Quart. Bull. 42, No. 2, 27-33 (1933); cf. C. A. 28, 55845.—Over 2000 samples of fertilizers, soils, citrus fruits, twigs and miscellaneous vegetables were examd. for As content. Fla. phosphate rock varied from a trace to 0.001%  $\text{As}_2\text{O}_3$ . Nitrophoska averaged 0.078%  $\text{As}_2\text{O}_3$ . Tankage, raw bone meal, castor pomace, cottonseed meal, German kainite,  $\text{KNO}_3$  and  $\text{NaNO}_3$  (Chilean) contained only traces of As. Synthetic  $\text{NaNO}_3$  contained 0.031% of  $\text{As}_2\text{O}_3$ . Twelve colored figures showing As standards by the Gutzelt test

are given. A detailed description of the improved Gutzert method is presented. C. R. Fellers

Influence of various sulfur-containing fungicides on transpiration. J. D. Wilson and H. A. Runnels. Ohio Agr. Expt. Sta., *Bimonthly Bull.* 175, 146-8 (1935); cf. C. A. 29, 6353<sup>1</sup>.—Eighteen S-contg spray materials were applied to *Coleus* plants to det. their transpiration rates. Liquid lime-S plus Ca(OH)<sub>2</sub> and Kolofog, which contains a high percentage of bentonite, were the only materials tested which caused an increase in transpiration comparable with that of Bordeaux mixt. The transpiration increases were approx.  $\frac{1}{2}$  to  $\frac{1}{3}$  as great as those caused by Bordeaux. Dry lime-S and liquid lime-S caused an increase in transpiration over the untreated checks of more than 50% during the night period. Any injury resulting from the application of S-contg. materials to plant tissue cannot be due to excessive increase in transpiration. C. R. Fellers

Action at a distance of metals on some species of fungi. E. Corneli. *Riv. Pat. veg.* 24, 397-406 (1934). *Rev. Applied Mycol.* 14, 646.—When spore suspensions of *Penicillium glaucum* in hanging-drop cultures were exposed to a lead disk in hermetically sealed glass containers only a small percentage of the spores germinated, the figure being highest when the Pb was farthest away (3.5 mm.) and lowest when it was nearest (1 mm.). When the spores were removed to normal conditions in the absence of the metal, growth was resumed, and was more rapid than in controls. The radiation effect was less marked on spore masses than on single spores, and when open containers or Cu and Ag disks in closed ones were used no appreciable effect of the radiation resulted. The distance of the metal from the fungus was less important than the area of the disk and the vol. of the container, the effect of a disk of given size at a given distance increasing as the vol. of the container decreased. *Ascochyta pisi* and *Tricothecium roseum* were less susceptible to the radiation than *P. glaucum*, the uredospores of *Uromyces betae* remained unaffected. C. believes that germination was reduced only in sealed containers because of the fact that in these there was a constantly increasing accumulation of secondary radiation or a progressively more complete ionization of the atm. Oden E. Sheppard

Contact insecticides from fatty alcohols. E. W. Bousquet, P. L. Salzberg and H. F. Dietz. *Ind. Eng. Chem.* 27, 1342-4 (1935).—The higher fatty alcs were found to be effective against aphids and do not react with hard water. Numerous long-chained alkyl derivs were synthesized and tested. The rhodanates were found to be particularly effective and satisfactory (cf. C. A. 29, 2656<sup>1</sup>). A max insecticidal effect was found for rhodanates contg 12 C atoms. The normal, primary compds were used, as branch-chain rhodanates were not promising. No one phys. property was correlated with this peak in toxic action, and it is probably the resultant of several properties. The 12-C homolog is safe on many kinds of plants, in the proper dispersion medium. E. R. Rushton

Insect and allied pests of cultivated mushrooms. V. Control of flies and mites. M. D. Austin and S. G. Jary. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 107-10 (1935).—Sprays contg nicotine (98%) at a diln of 1:1600 were ineffective in killing eggs of *Sciarra fenestralis* in lab. expts. The addn of Sulfonated Lorol (1:4000) did not increase the efficiency of the spray. Under the most favorable exptl. conditions a 60% mortality of the larvae of *S. fenestralis* was obtained with nicotine sprays. Good control of the adult flies was obtained by spraying the mushroom beds with nicotine (98%) at the rate of 1 oz./10 gal. water or by fumigating the cellars with nicotine (0.75-1.00 oz. per 1000 cu ft of space). The larvae were not affected by spraying with 1:1600 solns of NaCl. The wetting agents, Sulfonated Lorol and Na  $\gamma$ -sulfonate, were harmless to growing mushrooms when they were applied as sprays at 1:2000 diln. A proprietary oil emulsion, when carefully mixed and lightly sprayed on growing mushrooms, checked the injury caused by *Tyroglyphid* mites; a light application, when the temp. of the liquid

was 104°F., produced no injury to mushrooms. Warm water, applied at a temp. of 110°F., also caused no injury to mushrooms. K. D. Jacob

The effect of antioxidants on highly concentrated pyrethrum extracts. C. B. Gnadinger, C. S. Corland and C. A. Clark. *Soap (Sanitary Products Sect.)* 11, No. 10, 93, 97, 99 (1935).—Kerosene (K) exts of low pyrethrin content (2-5%) were quite stable over periods of 4-9 months but concd exts (10-15%) were unstable during prolonged storage. In order to prep. concd pyrethrum exts which would not have the antioxidant (A) present in K or other mineral oils, decahydronaphthalene (D) was used as a solvent. Neither D nor K interfered with the Cu reduction method for analyzing total pyrethrin content (cf. C. A. 23, 5546). Before proceeding with the analysis the exts are first dild with petr. ether, chilled overnight and filtered to eliminate altered or oxidized pyrethrins. At 35° concd pyrethrum ext. in D or K contg. no A lost 12-26% pyrethrin content in 2-4 months. Of 19 A tried on concd D-pyrethrum ext. held at 35°, one proprietary material at 0.1% prevented any loss for 60 days but the loss was 9.2% after 81 days, and  $\alpha$ -naphthylamine (0.5%) and thymol (0.5%) had similar effect. Conc'd K exts. contg. the same proprietary A were quite stable at 6° for 94 days but lost up to 10.4% at 35° storage. Henry H. Richardson

Fused needle of species of *Pinus*—progress report. H. C. Young. *Queensland Agr. J.* 44, 286-98 (1935).—The condition is manifested by a resinosis of the terminal buds and a twisting and adhesion of each needle in the fascicle. There was no relation between the chem. compn. of the soil and the occurrence of the disease. Treatment of individual trees with the essential elements and also with B, Zn, Cu and Al gave neg. results. Chem. analyses of material collected from diseased and healthy trees showed no great dissimilarities. K. D. Jacob

A new method for precision testing in the laboratory of the toxicity of lime sulfur and of Bordeaux mixture as protective fungicides. Progress report. H. B. S. Montgomery and M. H. Moore. East Malling Research Sta., *22nd Ann. Rept.* 1934, 217-22.—Mark individual glass slides with a ring 15 mm. in diam. cut into the glass with a diamond by means of a lathe. Clean the slides very carefully by successive treatment with warm dil. HNO<sub>3</sub>, CrO<sub>3</sub> cleaning soln. and ether, with intervening washing with water. Store the slides in abs. alc. and flame and cool just before using. Transfer 0.015 cc. of the spray fluid to the marked-off area of the slide from a graduated fine-glass tube. Spread the spray fluid to the edge of the groove with a finely pointed glass rod, dry the deposit in the lab. and store the slide for 1 day in an unheated water oven at approx. 15-18°. Support the slide over a pool of distd. water for several hrs. before application of the spore suspension. Apply 0.05 cc. of the spore suspension (spores of *Venturia inaequalis* Aderh. were used by the authors) and spread it in the groove. Incubate the slide in an individual moist chamber for 24 hrs. and det. the degree of germination by counting under the high-power field of the microscope. At least 10 test slides and 10 control slides are recommended for each spray diln. Under these conditions germination of the spores was almost completely inhibited by lime S at a concn. of 1:50 and free germination was entirely prevented by Bordeaux mixt. contg. 0.025% CuSO<sub>4</sub> in which hydrated lime was used at the rate of 3 parts to 2 parts CuSO<sub>4</sub>. Methods of raising large supplies of viable spores of *V. inaequalis* in pure culture are discussed. K. D. Jacob

Preliminary laboratory tests of bactericides on the plum bacterial canker organism [*Pseudomonas mors-prunorum*]. H. Wormald. East Malling Research Sta., *22nd Ann. Rept.* 1934, 151-5; cf. C. A. 29, 2651<sup>1</sup>.—Growth of the organism was completely prevented by either phenol 0.1, EtOH 0.1, CH<sub>3</sub>O 0.01, quinosol 0.005, CuSO<sub>4</sub> 0.2, CuCl<sub>2</sub> 0.01, ZnSO<sub>4</sub> 0.005 or HgCl<sub>2</sub> 0.0005%. In culture media that reacted only slightly with CuSO<sub>4</sub>, the organism was killed by contact for 10 min. with a 0.01% soln. of CuSO<sub>4</sub>. Under these conditions the bactericidal action of CuSO<sub>4</sub> was much more rapid than that of ZnSO<sub>4</sub>.

Tar oil at 1% concn. killed the organism in 10 min, though at 0.1% it did not kill in 1 hr K D Jacob

Laboratory trials of wetters against woolly aphid, *Eriosoma lanigerum* (Hausm.). R M Greenslade East Malling Research Sta, 22nd Ann Rept 1934, 185-90.—Of a no of wetting agents tested with 0.025% nicotine soln, Agral II was the only one that showed any promise of increasing the effectiveness of the nicotine. At 0.5% concn with nicotine it gave results as good as those obtained with nicotine plus 1% of soft soap, but at this concn Agral II may cause damage to the fruit (apples). At 1% concn Agral II completely wetted tufts of woolly aphid "wool" in 40 min, while none of the other wetting agents showed signs of wetting before 1 hr. Soft soap at 0.5% concn was a much better wetting agent than Agral II at 0.1% concn. None of the preps. had any apparent solvent action on the wax threads of the "wool". The strawberry tarsonemid mite was not controlled by spraying the strawberry plants with either 3% lime S plus 1% of a no of different wetting agents, lime S alone (1-14%), petroleum emulsion (2-5%), paraffin emulsion (2-50%) or a spray composed of liver of S 30 oz and soft soap 10-20 lb per 100 gal K D Jacob

Effect of molds and other seed-borne fungi on germination. F. A. McLaughlin Mass Agr Expt Sta, Control Bull 77, 58-60 (1935).—Thirty-five lots of fungus-infected sweet-corn seeds were divided into 3 lots and 1 lot was dusted with Et Hg phosphate, 1 was treated with 1 1000 HgCl<sub>2</sub> for 10 min, and the last lot was left untreated. The seeds were germinated and the seedlings noted for fungus diseases. The org Hg prep. and the HgCl<sub>2</sub> were equally effective in destroying fungi on the seeds. *Penicillium* and *Rhizopus*, 2 of the most abundant seedling fungi, are well controlled by seed treatment C R Fellers

A field spraying trial of combined fungicide-contact insecticide sprays in 1934. Progress report. M H Moore and H. B. S. Montgomery East Malling Research Sta, 22nd Ann. Rept 1934, 208-16, cf C A. 29, 1202.—Fairly good control of apple scab (*Venturia inaequalis* Aderh.) on Cox's Orange Pippin was obtained by 1 pre-blossom (pink-bud) followed by 2 post-blossom applications of lime S (1-30 pre-blossom and 1 100 post-blossom) to which, at petal-fall, nicotine was added, with either Sulfonated Lorol, "Lethalate" Wetting Prepn or sulfite lye as spreader. Very good control of sawfly (*Hoplocampa testudinea* Klug.) and red spider (*Oligonychus ulmi* Koch) was obtained with all spray treatments. There was some evidence that "Lethalate" Wetting Prepn. was the most efficient of the wetting agents tested K D Jacob

Tolerance of cabbage seedlings to insecticide dips for the control of aphids and cabbage worms. Harry G. Walker and Lauren D. Anderson Va Truck Expt Sta, Bull. 86, 1205-10 (1935).—Expts showed that the dipping of cabbage plants infected with aphids and cabbage worms in 1 400 concns. of pyrethrum or derris for 30 sec destroyed these insects and caused only slight reduction in plant survival. If only aphids were present, dipping in a soln of 0.7 lb. of nicotine sulfate or Black Leaf 40 in 50 gal. of water was also effective. Pyrethrum and derris lost their activity rapidly in aq. solns and only enough soln should be made up to last 1 day C R F

Fish-poisoning vines, a native insecticide in Kwangsi. Chm-Pi Chen. Science (China) 19, 1405-30 (1935).—*Melissia panchyrrarpa* Benth., commonly known as "fish-poisoning vines" in China, is widely distributed in the mountains of Kwangsi province, China. C finds this plant contains a large amt. of saponin (C<sub>25</sub>H<sub>40</sub>O<sub>8</sub>) and possibly also considerable rotenone. Mixts of this plant with soap or tea oil serve not only as good insecticides, but also as contact poison and stomach poison, the efficiency for the latter purposes being not inferior to derris, though far cheaper. C. L. Tseng

Some plants with insecticidal properties. A. Barcellos Fagundes. Bol. ministerio agr. e com. (Brazil) 24, Numbers 1-3, 69-75 (1935).—A list of 89 toxic or insecticidal

plants with their uses and bibliography of 24 references C W. Whittaker

The applicability of hydrogen peroxide as a seed disinfectant. F. Pichler. Phytopath. Z. 8, 245-51 (1935), Rev. Applied Mycol. 14, 619-20.—Lab. and field expts using up to 3% H<sub>2</sub>O<sub>2</sub> for wheat and barley and 4-5% for oats failed to give control of wheat bunt (*Tilletia caries*), covered smut of barley (*Ustilago hordei*) and loose smut of oats (*U. avenae*). These results are contrary to those of Kissner and Portheim (C A. 29, 1571) O E S

Investigations on machinery used in spraying. H. Nozzles. Cornelius Davies and G. R. B. Smyth-Home-wood J. South Eastern Agr. Coll. Wye, Kent No. 36, (2-85) (1935).—The surface area of trees covered by spray fluid was detd. by a method previously described (C A. 28, 6021). The results obtained in the field under com. conditions showed an av. cover of about 78%. An analysis of the uniformity and degree of atomization of the spray deposit on the trees used was also made and the "spray cover efficiency" (based on area covered, atomization and uniformity) averaged 62%. In field expts under various pump and nozzle conditions the cover ranged from 60 to nearly 92% K D Jacob

Crab grass control on lawns. Howard B. Sprague. N J Agr Expt Sta, Circ 354, 1-4 (1935).—The use of 5-10-5 fertilizers at the rate of 15-25 lb per 1000 sq ft encourages desirable grasses which will overgrow the crab grass, *Digitaria sanguinalis*. The use of such org fertilizers as bone meal and tankage alone benefits crab grass more than desirable lawn grasses. Highly acid soils should be rendered only slightly acid by the use of Ca(OH)<sub>2</sub>. C R Fellers

Plant fish poisons as insecticides. Roland M. Whittaker and Arthur L. Whittaker. Poultry Sci. 14, 351-4 (1935).—Body lice on badly infected chickens were completely killed in 18 hrs by dusting the birds, by the pinch method, with small amts of powd. derris contg 1% rotenone. In expts on the toxicity of rotenone and derris to chickens, no visible effects were obtained by the oral administration of either 0.2 g of derris dust or 0.02 g of pure rotenone in gelatin capsules fed twice in 12 hrs to cockerels 4 weeks old; a single dose of 0.1 g. of rotenone had no effect. The egg-laying ability of hens was not affected by the ingestion of rotenone K D Jacob

A homemade cattle spray. C B Dibble Mich. Agr. Expt Sta, Quarterly Bull. 18, 5-7 (1935).—A good easily prepd. cattle spray is made by mixing 0.5 lb. of ground pyrethrum flowers with 1 gal. of either oleum or mineral spirits. Shake occasionally for 48 hrs, strain out the residue and use the clear liquid as a spray. Specifications for oleum spirits are: flash 104°, sp gr 47.6, color 26; no S, initial b. p. 305°; 20% distils at 330°, 50% at 346° and 90% at 386° with the end point at 423° C R Fellers

Weed killers. R. M. Woodman. The Horticultural Education Assoc. Yearbook 2, 77-83 (1933).—A report on the observation of various investigators indicates that an application of approx 200 lb per acre of NaClO<sub>2</sub> broadcasted in autumn cleared very weedy land of couch and creeping bent grasses, crowfoot and shallow-rooted perennial weeds, but did not seriously affect deep-rooted weeds. Five and 15% solns with a spreader gave excellent control of most weeds. NaClO<sub>2</sub> was not efficient against aquatic weeds. Two to 5% solns of NaAsO<sub>2</sub> are excellent for clearing land in autumn. Four applications each of a mixt contg 150 lb of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 50 lb calcined FeSO<sub>4</sub>, and 1/4 ton of soil per acre of old lawn turf completely eradicated white clover, daisy, mouse-ear, chickweed, moss, pearlwort, etc. The NH<sub>4</sub><sup>+</sup> ion appears to have a selective toxic action on weeds. An application of 800 lb. of cyanamide per acre immediately prior to active growth of lawn is said to destroy most weeds. A soln of 1 lb. of As<sub>2</sub>O<sub>3</sub> in 8 gals. of H<sub>2</sub>O applied at 240 gal per acre controlled all weeds except yarrow, dandelion, dock, and sorrel, as effectively as the same vol of 2.5% NaClO<sub>2</sub>. Wheat has been safely sown without ill effects after spraying soil with 1% NaClO<sub>2</sub> but with more concd solns. sowing should be left until the following season

depending on the nature of the soil, climatic conditions, etc. For doses up to 12 lb. per acre it is stated no injury to germination occurs. **Irvin C Feustel**

**Use of a wetter in weed spraying** **I L Ingledand and R M Woodman J Ministry Agr (Engl) 42, 663 b (1935)**—A spray soln composed of 2 lb of  $\text{NH}_4$  sulfate, 1 lb of soft soap and 2 gal of water readily killed young seedlings of *Polygonum aviculare* (pig weed) and of *Trifolium stratum*, but did not kill grass. Without the soap this spray failed to adhere to the waxy coating of the cotyledons and seedling leaves. Concentrated soap and  $\text{NH}_4$  sulfate solns should be made separately and mixed with each other and the necessary water just before using.

**C W Whittaker**  
**The extermination of weeds along railway tracks and in courtyards with sodium chlorate** **F Neuwirth Lusty Cukrovar 53, 477 80(1935)**—Travex (contg 97.80%  $\text{NaClO}_3$ ) in a 2% concn and in doses of 2.1 per sq m sprayed on controlled plots along railroads and in yards about sugar factories was considered satisfactory in its action, although the results were not abs. Most of the vegetation on the plots was exterminated, and only a few growths occurred after a single treatment. An equivalent quantity of dry, powdered  $\text{NaClO}_3$  sprayed on plots was not as effective as the wet spray. Iron sheets dipped in Travex and exposed to the atm. gained 0.8% in weight, those dipped in distilled  $\text{H}_2\text{O}$  under the same conditions gained 0.4% in weight. Clothes, wood, etc., impregnated with Travex become highly inflammable. **Frank Mareš**

**The extermination of weeds on railroad tracks and in factory yards by means of sodium chlorate** **Otakar Král Lusty Cukrovar 53, 490(1935)**—Instead of Travex (cf. preceding abstr.) K. has been using a 2%  $\text{NaClO}_3$  soln (2.1 per sq m) during a 3-yr. period. Since  $\text{NaClO}_3$  is half as expensive as Travex, the  $\text{NaClO}_3$  sprays can compete with manual procedures for removing weeds. Although the effects of  $\text{NaClO}_3$  are not absolute, they remain satisfactory for an entire summer, they appear on the day after the spray, and they are particularly effective in crevasses between stones, ties, etc., where manual efforts are useless. The corrosive action of the  $\text{NaClO}_3$  under actual conditions of application is negligible and can be ignored. **Frank Mareš**

**Detn. of nitrates [application in soils]** (**Bennett, Hartwood**) 7. **Drying malt sirups, other sirups, vegetable juices, molasses residues, etc., for use in feeds or fertilizers, etc.** (**U. S. pat. 2,018,797**) 12. **Transforming hydrocarbons [product as a fertilizer]** (**Brit. pat. 432,368**) 22. **Polynuclear phenols and nitration and sulfonation products [as insecticides]** (**Brit. pat. 431,945**) 10.

**Fertilizer** **Chemische Fabrik Uetikon vorm Gebrüder Schnorr** **Swiss 172,740, Apr. 1, 1935 (Cl. 42)** A rich fertilizer contg N and P is obtained by treating crude phosphate with a mixt. of  $\text{H}_2\text{SO}_4$  and  $\text{HNO}_3$  without sep. the gypsum formed. The excess of acid may be neutralized after disintegration of the crude phosphate. The  $\text{N}_2\text{O}$  gases evolved may be led back into the fertilizer mass.

**Fertilizer** **Fischer Wyss Maschinenfabrik A.-G. Swiss 177,273, Aug. 1, 1935 (Cl. 42)** Vegetable waste matter such as waste grain, fruit, parings, flowers, etc., are worked up to form a fertilizer by pulping while hot, and evap. the pulp to dryness.

**Fertilizers** **I G Farbenindustrie A.-G. 1: 784,196, July 22, 1935** A mixt. of crude phosphate and carbonaceous materials such as lignite or peat is treated with oxides of N, Cl or  $\text{SO}_2$ , or a mixt. of more than one of these, water or aq. liquid being supplied to the treated material during the decompn. in amt. sufficient to replace that consumed.

**Insecticides** **Frank J. De Rewal (to Atmospheric Nitrogen Corp.) U. S. 2,019,121, Oct. 29** Comps. of metals such as Cr, Pb and Cu with  $(\text{CH}_3)_4\text{N}_4$  are used in insecticides suitable for treating plants.

**Insecticides and fungicides** **John F. Littooy and Frank F. Lindstedt (to Hercules Glue Co.) U. S. 2,018,681, Oct. 29** A dry mixt. is formed comprising a toxic substance such as Pb arsenate, together with a smaller proportion of a nonmetallic oleaginous material such as mineral oil which is present in an amt. (suitably about 0.25-10% of the wt. of the toxic substance) insufficient materially to augment or modify the insecticidal effect of the toxic material but which serves to improve the compn. for use as a spray on fruits, etc.

**Sulfur mixture for use as an insecticide** **Ludwig J. Christmann and David W. Jayne, Jr. (to American Cyanamid Co.) U. S. 2,019,443, Oct. 29** Powder  $\text{S}$  is used in admixt. with pine oil and liquorice root.

**Protecting vegetable matter such as plants against injurious animal life such as insects** **Philip T. Sharples (to Sharples Solvents (Corp.) U. S. 2,019,275, Oct. 29** Triamylamine is used (suitably in an oil emulsion).

**Apparatus for drying or wiping films to remove spray residues** **Douglas C. Morris and Leonard O. Cockrill (to Trust Packers Supply and Equipment Co.) U. S. 2,019,087, Oct. 29** Structural, mech. and operative details.

**Seed disinfectants** **Schering Kahlbaum A.-G. Brit. 431,172, July 2, 1935** See Fr. 769,840 (C. A. 29, 5427).

**Disinfectants containing mercury** **I G Farbenindustrie A.-G. Fr. 784,329, July 22, 1935** Insol. disinfectants, particularly for treating seeds, are made by causing sol. organometallic compds. of Hg to react with compds. of Si capable of reaction. Thus, compds. are prepd. from ethylmercuric nitrate and Na orthosilicate, methoxyethylmercuric acetate and sol. glass, ethoxyethylmercuric acetate and sol. glass, *o*-hydroxyphenylmercuric chloride and sol. glass, dimethylaniline-mercuric acetate and sol. glass, etc.

**Plant-rust preventives** **I G Farbenindustrie A.-G. Brit. 431,331, July 1, 1935** Preps. to be applied to the soil or to plants to prevent or treat rust diseases consist of an amide of an aromatic sulfonic acid, e. g., *p*-toluene sulfamide or its Na salt, *o*-toluene sulfamide, benzene sulfamide, xylene sulfamide, dichlorobenzene sulfamide, together with an inert solid diluent and (or) a fertilizer, e. g., talc, prep. chalk, Thomas slag,  $\text{NaNO}_3$ , K salts.

## 16—THE FERMENTATION INDUSTRIES

### C N FRY

**Material economy in the alcohol industry.** **L. Kowalczyk. Przemysł Chem. 19, 158-60(1935)**—This industry presents a closed cycle. There are no by-products. From the viewpoint of consuming all the materials it is nearly perfect. **A. C. Z.**

**Butyl and acetone fermentation** **I Intermediate products in the butanol-acetone fermentation** **Konrad Bernhauer and Karl Kurschner Biochem. Z. 280, 379-87 (1935)**—The explt. evidence points to butyric acid as the mother substance from which butanol is formed, with butyraldehyde probably as an intermediate product in the reduction. The butyric acid is not formed from either acetaldehyde or  $\beta$ -hydroxybutyric acid but probably from

crotonic acid. Since the transformation acetaldehyde  $\rightarrow$  butanol takes place over acetaldehyde, although the latter is not actually utilizable, it is concluded that acetaldehyde changes to butyric acid only when it is in the nascent state. The acetone fermentation proceeds by way of  $\text{AcOH} \rightarrow$  acetoacetic acid. **S. Morgulis.**

**Cider-making trials for the season 1933-34** **P. T. H. Pickford Univ. Bristol Agr. Hort. Research Sta., Ann. Rept. 1934, 191-208, cf. C. A. 28, 6929**—Analyses are given of numerous samples of cider prepd. from a wide variety of apples. **K. D. Jacob.**

**The role of pectin in cider-making processes** **I Introduction and historical** **Vernon L. S. Charley**

Univ. Bristol Agr. Hort. Research Sta., *Ann. Rept.* 1934, 217-26.—A review with 13 references. II. Pectin changes associated with maceration and fermentation and the effects of the addition of pectin solutions to dry ciders *Ibid.* 227-45. Apple juices macerated with once-pressed pomace dissolved considerable quantities of sol pectin. The favorable flavor changes in ciders made by this process could not be correlated directly with pectin data, as the fermented products contained no sol pectin, this having been removed during fermentation. Ciders fermenting in cask, lost most of their pectin (as given by Ca pectate) before fermentation ceased. In a few cases of filtered juices which had been 6 months in bottle, the Ca pectate figure was extremely small, but most of the ciders gave no ppt. at all. The alc ppt method gave very much higher results for pectin than were obtained by pptn as Ca pectate. Samples of sterile pasteurized apple juices fermented separately with 10 different pure yeasts showed no loss of pectin over a 60-day period. In a similar series, not subjected to pasteurization, approx 60% of the pectin was removed. The amt of decompn varied with the yeast used. Complete removal of the pectin in 1 case was correlated with the occurrence of mold growth in the flask. A dry cider to which pectin was added in the form of cold and hot water pomace exts and as citrus pectin was found to contain no pectin after a few weeks in bottle.

K. D. Jacob.

The biochemical characteristics of the barleys of the Soviet Union. N. N. Ivanov. *Bull. Applied Botany, Genetics, Plant Breeding* (U. S. S. R.) Ser. III, No. 7, 1-174, in English 175-88 (1935). Data on the chemical characteristics of barley from the Soviet Union are presented from the standpoint of its beer-brewing qualities. It is pointed out that the high-protein barleys may also be just as good as the low-protein for beer brewing. It was found that the quality of the starch of the high-protein barley is, in some respects, superior to that of low-protein barley. The starches are more sol. and more easily converted into sugars by diastase. These also contain a more active amylase.

J. S. Joffe.

"Cats'-tails," a new variety of hop. L. S. Silmon. *J. South-Eastern Agr. Coll. Wye, Kent* No. 36, 41-7 (1935). The new variety (OZ70) was obtained by the natural crossing of the wild hop of America (*Humulus americanus* var. *neo-mexicanus*) with an English male hop (*H. lupulus*). Analyses of the new variety, English com. varieties and the richest samples of American hops obtainable on the London market, during the period 1930-31, showed a acid 5.30-8.85, 5.07-7.81, 6.23-9.26;  $\beta$ -fraction 7.71-12.60, 7.21-9.47, 7.00-13.35, estd. antiseptic substances 8.41-12.14, 7.66-10.22, 10.52-12.55%, resp. OZ70 is considerably richer in preservative properties than any of the English com. varieties and occupies a position intermediate between the best English com. varieties and the richest American hops.

K. D. Jacob.

Colloid chemistry of the brewing industry in the last ten years. K. Silbereisen. *Kolloid-Z.* 73, 104-11 (1935). Conclusion of the review (cf. C. A. 29, 82237). Fifty references.

Oscar T. Quimby.

Fermentation of wort in the presence of malt diastase. M. Hamburg and S. Pockholz. *Brau. Malzind.* 35, 63-6 (1935).—Addn. to sterile wort of a pasteurized or unpasteurized aq. ext. of diastatic malt had no influence on the biol. condition of the resulting beers; pasteurization, however, reduced the diastatic power of the infusion by 6 and 30% when kept for  $\frac{1}{2}$  hr. at 55° and 60°, resp. Addn. of unpasteurized diastase soln. to wort in pitch-lined glass fermentation vessels had no effect on the acidity, total and formal N of the fermented product. With increasing amts. of added diastase, the nonfermentable ext. decreased, the maltose content remained approx. const., while the attenuation increased from 74.6 to 82.7-84.7% and the alc. content from 3.55 to 3.92-4.07%. Similar fermentations in Al vessels were less pronounced with regard to increase in attenuation and alc. content.

S. Laufer.

Substances which can stop alcoholic fermentation. L. Genevois. *Ann. fermentations* 1, 80-100 (1935).—A re-

view of the properties and action toward yeast of the 3 classes of substances which can stop alc. fermentation. (1) "narcotics," which arrest fermentation by, and respiration and growth of yeast, but which exert an action that is strictly reversible provided it does not act for too long a period, (2) antiseptics, which kill the cells; (3) specific fermentation inhibitors which, under certain conditions, stop cellular fermentation without affecting respiration or even growth.

A. Papinian-Couture.

Experimental work on cucumber fermentation. VIII. Genuine dill pickles. A biochemical and bacteriological study of the curing process. F. W. Fabun and L. J. Wickerham. *Mich. Agr. Expt. Sta., Tech. Bull.* 146, J 20 (1935), cf. C. A. 29, 5534.—There is a definite sequence of bacterial population in dill pickle fermentation. At first, Gram-pos. cocci predominate, followed by short acid-producing rods which are Gram-pos. Finally, the latter give place to long, acid-producing, Gram-pos. rods which are responsible for the marked rise in acid production after 8-10 days. The addn. of 2 lb. of sugar per bbl. of pickles greatly increases the no. of bacteria and insures a more rapid production of acid. Hard water did not significantly delay fermentation. Addn. of 2 lb. of mustard per bbl. did not appreciably retard the fermentation. Washing the cucumbers in Cl soln. before packing in brine had no influence on the course of the fermentation.

Normal amts. of soil or dirt adhering to cucumbers had no harmful influence on the fermentation. The addn. of sufficient acid such as AcOH to dill pickle brine to produce an initial pH of 4.5 is desirable.

C. R. Teller.

Vinification in California wineries. E. M. Brown and Victor Del Henriquez. *Ind. Eng. Chem.* 27, 1235-40 (1935).

G. G.

Metals in wineries. Charles S. Ash. *Ind. Eng. Chem.* 27, 1213-4 (1935).—Ni, Cr steel and Duriron are very resistant to the corrosive action of fruit juices and wines. Cu, monel, brass and bronzes corrode more or less but are usually satisfactory for use in wineries. Al and Al alloys pit and corrode rapidly. Pb, Sn and Zn are rapidly dissolved by grape juice and wine. Cast Fe gives variable results. In some cases the corrosion rate increases with time. Wrought Fe and steel are suitable for use in places where they become coated with insol. Fe tartrate provided the coating is not removed by mech. action as it is with moving parts.

L. F. Gilson.

Pasteurization of New York State wines. Carl S. Peterson, Harry L. Goresline and E. A. Havens. *Ind. Eng. Chem.* 27, 1257-65 (1935).—Various types of wines of low  $\text{FeO}$  content can be effectively stabilized by pasteurizing at 130°F. for 20 min., and this pasteurization can be carried out, under controlled conditions, in bulk, in closed bottles or in open bottles. The microorganisms which occasionally survive the heating at this temp. are not of the spoilage type and apparently have little significance. The addn. of small quantities of  $\text{SO}_2$  to the wine (25-50 p. p. m.) had an appreciable effect in lowering the temp. (approx. 5°F.) or shortening the time necessary. Methods and app. are described.

L. E. Gilson.

Effect of filter aids and filter materials on wine composition. L. G. Saywell. *Ind. Eng. Chem.* 27, 1245-50 (1935); cf. C. A. 29, 82227.—The use of cellulose and asbestos filter pads did not cause any significant increase in Fe or Ca. Diatomaceous earth and porous silica candles gave excellent results. Certain (California) wines became cloudy and deposited a sediment as a result of the presence and growth of certain microorganisms. After filtration through diatomaceous earth the wine was free from such microorganisms and remained brilliant for long periods. App. and methods are described.

L. E. G.

Volatile acids of wine. Mark M. Morris. *Ind. Eng. Chem.* 27, 1250-2 (1935).—Many volatile acids reported by earlier workers were not found in the various types of Calif. wines examined. Young wines contained practically no volatile acids except AcOH. Sound old wines contained AcOH and traces of  $\text{FeCO}_2\text{H}$ . Diseased wines contained AcOH, traces of  $\text{HCO}_2\text{H}$  and little or no lactic acid. Twenty-eight references.

L. E. Gilson.

Estimation of citric acid in wine. A. Heiduschka and J.



Sommer *Pharm Zentralhalle* 76, 593-5(1935).—After commenting on the errors inherent in the methods heretofore employed, recourse to the following procedure is suggested. The citric acid is first pptd as Ba salt which is thereupon treated with concd  $H_2SO_4$ , whereby the citric acid is made to yield acetonedecarboxylic acid or AcMe. The latter is distd off and then treated with  $KMnO_4$ , the resulting AcH being estd iodometrically via Messinger.

W O C

Turbidity due to reduction in bottled white wines Jules Flamand *Bull assoc élèves inst sup. fermentations Gand* 36, 209-16, 227-32(1935).—A review of the causes and remedies of this defect, and of the detn of the liability of a wine to suffer from it.

A Papineau-Couture

Effect of temperature on the precipitation rate of cream of tartar from wine G L Marsh and M. A. Joslyn *Ind Eng Chem* 27, 1252-7(1935).—The ptn of cream of tartar from new wines (California) is hastened by cold storage. The ptn is more rapid during freezing storage.

L E Gilson

Selected yeasts in wine making L. Moreau and F. Vinet *Ann fermentations* 1, 101-7(1935).—Attention is drawn to the complexity of the problem of using selected yeast cultures in wine making, which renders their use for this purpose much more difficult and the results obtained less certain than in the brewing industry.

A P C

Pure yeast for fruit and berry wine production F. Wrede *Suddeut Apoth Ztg* 75, 731-5(1935).—The necessity for employing pure yeast in fruit wine production is emphasized, in connection with suggestions for suitable manipulation of the must and containers therefor and the finished products.

W O C

Errors in the production of fruit and berry wines F. Wrede *Suddeut Apoth Ztg* 75, 742-3(1935).—Supplementary to the foregoing article, W. points out new certain precautions to be observed in the treatment, bottling and aging of the finished products.

W O C

Composition of brandy G L Marsh *Fruit Products J* 15, 42-3(1935).—A review.

E H

Manufacture of champagne and sparkling burgundy F M Champlin, H F Goresline and D K Tressler *Ind Eng Chem* 27, 1240-3(1935).

G G

Beer quality improved by newer methods for hops removal Geo Defren *Food Ind* 7, 578-9(1935).—Rapid and continuous straining of the hot wort direct from the brew kettle through an enclosed unit with relatively small filtering area reduces the time of contact of wort with hops, and prevents loss of volatile hop aromatics. The process also avoids contact of hot wort with the  $O_2$  of the air, increases the yield of wort per 100 lb of hops, and reduces the quantity of hops necessary to give the desired flavor and aroma. The moisture content of the spent hops is reduced by approx 85%.

C R F

The determination of glutathione in beer G Weller *Ann fermentations* 1, 108-14(1935).—A review of the literature on the detection and detn of glutathione, more particularly in yeast, with 28 references. The method of Binet and Weller (*C A* 28, 4095'; 29, 1117') is briefly described.

A. Papineau-Couture

Effect of the degree of oxido reduction of beer on pasteurization Jacques Segard *Bull assoc élèves inst sup fermentations Gand* 36, 243-51(1935).—A study of  $r_H$  of beer pasteurized under various conditions indicated that optimum results as regards taste and stability are obtained when pasteurization is carried out at an  $r_H$  value of about 10. This result can possibly be obtained by a combination of as complete elimination of air as possible and the use of small amts of Na hyposulfite.

A P C

The storage of beer J Raux *Brasserie & malterie* 25, 225-32, 241-8(1935).—A Papineau-Couture.

Stabilization of beer by means of "Astro" short period plate heater H. Schneegg and H. Kipphan *Z ges Brauw* 58, 41-6(1935); cf Richter and Damm, *C A* 27, 5887.—Further expts were carried out on pasteurization of beer with this app. Loss of  $CO_2$  at pressures above 5 atm was found to be due to bending of some plates and to rapid deterioration of rubber washers caused by steam sterilization of the app. before use. These defects were

remedied by strengthening the plates with ribs, and by using water at 80° for sterilization according to a procedure which is described. Twenty-four tests were carried out over a period of 9 months with normal beers which have been in an artificial manner heavily infected with various organisms, such as culture and wild yeast, sarcina, lactic and acetic acid bacteria. The pasteurizing temps employed varied from 55° to 75°. The treated samples were incubated at 25° for 4 weeks and observed for growth development. A pasteurization temp of 65° was found sufficient in all cases, while for moderately infected beers even a lower temp of 62° gave satisfactory results. Pasteurization below 70° had no detrimental effect on flavor and other properties such as head formation and protein haze development on prolonged storage at 25°, as compared with normal unpasteurized beers.

S Laufer

Investigations of the yeast assimilable nitrogen content of beer wort Niels Nielsen *Compt rend trav lab Carlsberg* 21, 113-38(1935).—The yeast-assimilable N and total N of Pilsner, Lager and Porter worts from the Carlsberg breweries were detd. In making these detns the worts were dild with a N-free sugar and salt-contg nutrient soln, inoculated with a small quantity of pure yeast, and resulting yeast crops were harvested daily over an incubation period of a no of days. The weight of the yeast crops and the N content of the yeast gave a measure of the amt of N assimilated. Nielsen concludes that of the total N content of beer worts 40 to 50% is assimilable by yeast although in brewery practice only about 30% is assimilated. The temp at which the yeast is cultivated (10-20°) has no influence on its ability to assimilate the N compds of wort. Microorganisms vary in their N-assimilating ability. *Rhizopus sinensis*, e g, can assimilate about 60-70% of the N of the wort, whereas *Klosteria apiculata* can assimilate only 40%. The greater portion of the assimilable N is assimilated very easily whereas a lesser portion is assimilated more slowly and with greater difficulty.

G W Kirby

Butyl alcohol and acetone by fermentation Deutsche Hydrierwerke A-G *Ger* 618,424, Sept 9, 1935 (Cl 68 162). See *Fr* 669,650 (*C A* 24, 1931).

Yoghurt Société d'études et applications industrielles Swiss 174,626, Apr 1, 1935 (Cl 28c). Milk powder is fermented. The yoghurt bacteria culture may be added to the milk powder in soln or in a dry state. In the latter case, solvent is added. The method is applicable to full or skim milk powder.

Apparatus suitable for condensing and separating carbon dioxide from fermentation gases Alvin H Baer (to Worthington Pump and Machinery Corp.) U.S. 2,018,594, Oct 22. Various structural, mech and operative details.

Apparatus for carbonating beverages such as beer in barrels or the like Fred J C Wiechmann U.S. 2,017,879, Oct 22. Various structural and operative details.

Apparatus for determining the volatile acidity of wines and other fermented liquids Cesare Iozzi *Fr* 784,569, July 22, 1935.

Malting-apparatus Oliver Sleeman Brit 430,923, June 27, 1935. The app comprises a closed vessel of cylindrical or oval section provided with a series of superposed malting floors that may be rotated or rocked, 1 or more portions of a floor being hinged to open downward so that the grain may be transferred from 1 compartment to another.

Aerating devices, especially suitable for use in yeast fermentation vats Wilhelm Vogelbusch Brit 431,586, July 11, 1935. Addn to 430,348 (*C A* 29, 7576').

Yeast N V Industriële Maatschappij voorheen Noury & van der Lande Brit 431,688, July 8, 1935. See *Fr* 766,555 (*C A* 29, 2658').

Yeast Ivan A Ffront and Adalbert Popper. *Fr* 784,127, July 22, 1935. Only a part of the liquid in the fermenting vat is submitted to strong aeration and this

part may be caused to rise in an open cylinder in the vat and flow over and down again or may be caused to flow to a 2nd vat in which the same action takes place to a 3rd vat and so on.

Yeast. Wirtschaftliche Vereinigung der Deutschen Heleindustrie. Ger. 618,021, Sept. 2, 1935 (Cl. 6)

## 17—PHARMACEUTICALS, COSMETICS AND PERFUMES

W. O. EMERY

Chemical examination of the roots of *Aristolochia indica*. Linn. I. P. R. Krishnamurthy, B. L. Manjunath and S. Venkata Rao. *J. Indian Chem. Soc.* 12, 470-85 (1935).—*Aristolochia indica* is a twining perennial, native to the tropical portions and known locally as *ishkori bera* whose roots taste very bitter and have a characteristic aromatic odor. Though supposedly valuable against snake and insect bites it has recently been shown that the plant has no antidotal or therapeutic effect against cobra venom. Percolation of 75 kg. of crushed roots with alc. and distn. and steam distn. of the concd. ext. gave 130 g. of pale yellow oil which had the characteristic odor of the roots. The aq. layer was decanted from the semi-solid residue which yielded, on extn. and recrystn. from alc., 0.4 g. of white cryst. material (II),  $m. 285-90^\circ$  (Ac der.,  $m. 102-3^\circ$ ), hydrolyzed by HCl in  $\text{AmOH}$  to a phytosterol,  $m. 146^\circ$ , and a reducing sugar. I is probably a glucoside of the type of ipuranol. The aq. layer was freed from resins by the addn. of 5%  $\text{Na}_2\text{CO}_3$  and the filtered liquid was extd. with  $\text{CHCl}_3$ . The dried  $\text{CHCl}_3$  ext. was extd. with 1% HCl. On concn., this HCl ext. yielded a HCl salt,  $m. 208^\circ$  (decompn.), which liberated a well-defined individual alkaloid termed *aristolochine*,  $m. 215^\circ$ , mol. compd. with toluene,  $m. 150^\circ$  (decompn.). The aq. layer was treated successively with  $\text{Pb}(\text{OAc})_2$  and  $\text{Pb}(\text{OH})_2\text{OAc}$ . After removal of the minute ppt. and excess Pb by passing in  $\text{H}_2\text{S}$ , the filtered liquid was evapd. and extd. with  $\text{AmOH}$ . The concd. ext. gave a reddish syrup possessing reducing properties. The aq. soln. was concd. to a thick syrup contg. glucose and hydrolyzable sugars. A  $\text{MeOH}$  soln. of the syrup yielded a crop of large prismatic crystals of allantoin,  $m. 232^\circ$  (decompn.). The alc. ext. from the original roots, after sepn. from the phytosterol, was concd. and taken down to dryness with 2 g. of crushed root. This material was then exhaustively extd. with petr. ether, ether,  $\text{CHCl}_3$ ,  $\text{AcOEt}$  and finally  $\text{EtOH}$ . The petr. ether ext. yielded 0.2 g. of I and 1.23 kg. of a dark brown oil, consisting of the glycerides of palmitic, stearic, lignoceric, cerotic, oleic and linoleic acids. A considerable amt. of cholesterol, a small quantity of the glucoside of phytosterol,  $m. 146^\circ$ , and some ceryl alc. were isolated from the unsaponifiable matter. As the extn. with  $\text{EtO}$  proceeded these sepd. from the solvent a microcryst. yellow solid (II),  $\text{C}_{27}\text{H}_{48}\text{NO}_2$ ,  $m. 275^\circ$ . This bitter principle is similar to and isomeric with aristolochic acid isolated by various workers from different species of *Aristolochia* and has been named *isovarilochic acid*. II has no  $\text{MeO}$ ,  $\text{OCH}_3$  or enolic groups but contains 1 active H atom. Attempts at acetylation were unsuccessful and II does not react with  $\text{H}_2\text{NOH}$ ,  $\text{H}_2\text{NNHPh}$  or  $\text{H}_2\text{NCONH}_2$  and does not react with  $\text{MeI}$ . Benzoylation gave *benzoyl-aristolochic acid*,  $\text{C}_{29}\text{H}_{48}\text{NO}_4$ ,  $m. 170-1^\circ$ . *Me isovarilochic acid*,  $\text{C}_{27}\text{H}_{48}\text{NO}_4$ ,  $m. 267^\circ$  (decompn.), is tasteless and unaffected by refluxing with  $\text{N KOH}$  in  $\text{MeOH}$  for 4 hrs., proving it to be an ether and demonstrating the lack of a  $\text{COH}$  group in II. Oxidation of II with alk.  $\text{H}_2\text{O}_2$  produced an acid,  $\text{C}_{27}\text{H}_{48}\text{NO}_6$ ,  $m. 164.5^\circ$ . Though Hesse (*Arch. Pharm.* 233, 634 (1895)) isolated 3 acids from the roots of *A. caryophyllata*, no other acids similar to II could be obtained from the roots of *A. indica*. II. The essential oil. U. S. Krishna Rao, B. L. Manjunath and K. N. Menon. *Ibid.* 494-5.—The essential oil distillate (163 g.) was dried over anhyd.  $\text{MgSO}_4$  and gave a pale yellow oil,  $d_{20} 0.925$ ,  $n_D^{20} 1.5023$ ,  $[\alpha]_D^{20} -33.11^\circ$ , acid value 2.0, ester value 7.3 (after acetylation 22.5). The oil contains 3% carbonyl compds. but no substances

possessing the phenolic or the lower alkoxy groups. A small amt. of an oil with the odor of isovanillin was isolated. Sapon and a sepn. of Twitchell's method showed the presence of palmitic and oleic acids. The remaining oil (130 g.) was fractionally distd. under diminished pressure into a 1st fraction contg. a cryst. substance  $m. 170^\circ$  with the odor of camphor and 2 main fractions A (75 g.),  $b.p. 104-5^\circ$ , and B (15 g.),  $b.p. 128-30^\circ$ . Diets. of the colorless fraction A over  $\text{Na}$  gave a mobile colorless liquid sesquiterpene, *isovarrene* (I),  $\text{C}_{15}\text{H}_{24}$ ,  $b.p. 104-5^\circ$ ,  $d_{20} 0.9227$ ,  $n_D^{20} 1.5035$ ,  $[\alpha]_D^{20} -42.37^\circ$ , liquid mono-HCl deriv.,  $b.p. 128-30^\circ$ ,  $d_{20} 1.0230$ ,  $n_D^{20} 1.5107$ . I reacted attempts to dehydrogenate it by heating it with  $\text{Se}$  for 48 hrs. at  $240-70^\circ$ . The highly viscous pale green oil constituting fraction B gave indications of ketonic structure. A soln. of 60 g. of B in excess 95% alc. was refluxed with excess  $\text{H}_2\text{NCONHNH}_2\text{HCl}$  and  $\text{AcONa}$  for 2 hrs. and yielded 30 g. of a semicarbazone,  $\text{C}_{15}\text{H}_{24}\text{N}_2\text{O}$ ,  $m. 240^\circ$ , which, on decompn., formed a colorless sesquiterpene ketone, *isovarrene* (II),  $\text{C}_{15}\text{H}_{20}\text{O}$ ,  $b.p. 118-20^\circ$ ,  $d_{20} 0.9290$ ,  $n_D^{20} 1.5122$ ,  $[\alpha]_D^{20} -46.47^\circ$ , *p-nitrophenylhydrazone*,  $m. 186.5^\circ$ , *2,4-dinitrophenylhydrazone*,  $m. 167.5^\circ$ , *isovarrene*,  $m. 133^\circ$ . The residues from fraction B (30 g.) after removal of II were distd. and the distillate was fractionated. Residn. of the middle fraction gave a pale yellow viscous *sesquiterpene*, *isovarrene*,  $\text{C}_{15}\text{H}_{20}\text{O}$ ,  $b.p. 126-8^\circ$ ,  $d_{20} 0.9926$ ,  $n_D^{20} 1.5098$ ,  $[\alpha]_D^{20} -7.29^\circ$  which did not react with  $\text{PhNCO}$  but was partially acetylated on boiling with  $\text{AcONa}$  and  $\text{AcO}$  for 3 hrs.

C. R. Addinall.  
Sodium (propylmethylcarbinyl)allylbarbiturate, a short-acting hypnotic. Edward E. Swanson. *Proc. Soc. Exptl. Biol. Med.* 32, 1565-6 (1935). C. V. Bailey.  
Canadian vs. American fine-cured tobacco—comparison of chemical constituents and primary prices. John W. Symons. *Can. Chem. Ind.* 19, 1259-61 (1935).—If everything is taken into consideration the Canadian tobacco is chemically equiv. to the American tobacco which sells at twice the price. The percentage compns. of 5 Canadian and 4 American tobaccos are tabulated.

W. H. Boynton.  
Chemistry of the new antimalarials. Konrad Funke. *Pharm. Monatsh.* 16, 129-31 (1935).—A review.

H. M. Burlage.  
Galenical studies. I. Creosote pills. Berta Suiko. *Pharm. Monatsh.* 16, 131-3 (1935).—The drug/naturation of pills and disintegrating agents are discussed. Eleven formulas are offered and comments made on the same.

H. M. Burlage.  
Detection of alkaloids in ergot and ergot extracts. Dragutin Barkovic. *Pharm. Monatsh.* 16, 154-5 (1935).—

Drag.—Treat 0.2 g. of the powder in a test tube or a small flask with 20 cc.  $\text{Et}_2\text{O}$  contg.  $\text{CH}_3\text{CHO}$  (2-3 drops per 10 cc.) (I), 1 cc.  $\text{H}_2\text{O}$  and 1-2 drops of ammonia (II) or  $\text{Na}_2\text{CO}_3$  soln. (10%) (III). Shake the mixt. vigorously for 10 min. and allow the  $\text{Et}_2\text{O}$  layer to sep. completely. Draw off carefully 10 cc. of the clear  $\text{Et}_2\text{O}$  layer ( $\approx 0.1$  g. of drug) and shake strongly for  $\frac{1}{2}$  min. with 2 cc.  $\text{AcOH}$  (30%). After clearing sep. the acid alkaloid soln. and underlay carefully with 3-3.5 cc.  $\text{H}_2\text{SO}_4$  (80%). After a short time a blue-violet ring should appear at the zone of contact. If alkaloids are absent this zone is colorless or not more than pale yellow. Mix the layers and in a few min. if alkaloids are present a dark to pale blue-violet color appears; if no alkaloids are present the mixt. is colorless or not more than pale yellow. *Fluxionect.*—Add to 0.4 g. 10 cc.  $\text{H}_2\text{O}$  and 20 cc. of (I) and make alk.

with 1 2 drops of (II) or (III). Shake the mixt for some min vigorously and allow the  $\text{Et}_2\text{O}$  to sep. completely, draw off 10 cc of the clear  $\text{Et}_2\text{O}$  (= 0.2 g fluid ext.) and treat as above. **Extract**—Dissolve with titration 0.2 g of the extract, in 10 cc  $\text{H}_2\text{O}$ , add 20 cc (I) and make alk with 1 2 drops of (II) or (III). Shake vigorously for some min and treat the  $\text{Et}_2\text{O}$  layer as before. The test will detect 0.05% alkaloids in the drug and ext. and 0.025% in the fluid extract. H. M. Burlage.

**Suncucha**. The magic drink causing oblivion. Victor A. Reko. *Pharm. Monatsh.* 16, 155-7 (1935).—This Mexican drink is identified with 3 distinct plants: (1) *Hernia salicifolia* (var. *mer.*, Link), the leaves of which contain chlorophyll, fats, tannins, dyestuffs, gums, glucose, starch, traces of a volatile oil and the usual salts but apparently no ingredients which give the characteristic reactions (these ingredients seem to be formed after fermentation), (2) the kidney-shaped seeds of *Rhynchospora praeacornia* D. C., (3) the root-bark of *Piscidia erythrina*, L. When 1 3 g is taken as a tea it serves as a good diaphoretic, larger amts produce a deep sleep. The seeds contain besides resins, fats and a glucoside, an insol substance, piscidin, which is not an alkaloid and is responsible for the action of the drug. H. M. Burlage.

**Ultraviolet capillary analysis of pharmaceutical drugs**. Paul Ernst and Alfred Stueber. *Pharm. Monatsh.* 16, 171-6 (1935).—Me<sub>2</sub>CO exts. of the drugs of the Austrian Pharm are prep'd as follows. Ext. 0.2 g of the drug with 25 cc pure Me<sub>2</sub>CO (sp. gr. 0.79 at room temp.) 24 hrs with occasional shaking. To 7 cc of the liquid obtained by decantation add 1 4 cc N-NH<sub>4</sub>OH; to a 2nd portion add 1 4 cc  $\text{H}_2\text{O}$ , to a 3rd portion add 1 4 cc NAcOH and place in cylindrical tubes with diams of 2 cm a sufficient amt of these mixts to occupy heights of 3.2 cm. Dip in the liquids strips of filter paper (1/4" x 28 cm) to a depth of 2 cm and allow to stand for 1 hr., remove, dry and exam under the lamp. In a table the capillary pictures of the 3 types of solns for 141 drugs are described which might serve as a means of detg. the identity and purity of these drugs. H. M. Burlage.

The evaluation of the medicinally used tannin bearing drugs. O. Dalfert and M. Fleischer. *Pharm. Monatsh.* 16, 185-90 (1935).—A review of the methods proposed for the qual. and quant. examn. of these drugs. One hundred and one references. H. M. Burlage.

**Pharmaceutical action of aromatics and volatile oils**. Josef Augustin. *Riechstoff-Ind.* 10, 157-8 (1935).—The action and use of fenchone, benzyl alc., fenchyl alc., amine acid, C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>, amine oil, anethole, menthol, mustard oil, angelica root oil, apiole, amber, anthranilic acid and its derivatives, Atlas cedar oil and Am. rosenlammate in pharmaceuticals are discussed. H. M. Burlage.

**New procedures in the chemistry of aromatics**. A. Lewinson. *Riechstoff-Ind.* 10, 160-3 (1935). cf. C. A. 29, 82284.—A review dealing with aldehydes. H. M. Burlage.

**History of cosmetics in recent times**. A. Hauenstein. *Riechstoff-Ind.* 10, 171-2 (1935); cf. C. A. 29, 82283.

**Chemical and physiological investigation of the alkaloids of Aconitum napellus** L. W. A. van Bronhorst. *Pharm. Weekblad* 72, 1036-68 (1935).—Physiol. standardization of aconite does not check with the chem. detn. of total alk., since aconitine is more active than aconine. A chem. method is proposed which is based on detn. of Br<sub>2</sub>OH liberated from the aconitine by sapon. of the mixed alkaloids. Deterioration of aconite, however, cannot be detd. by this method since the spontaneous decomp. does not proceed beyond the benzoylaconine stage. Tinctures of aconite are more stable if the alc. is first acidified with HCl so that the final percolate has a pH of 5.2. A. W. Dor.

**Our Dutch Pharmacopoeias**. W. F. Daems. *Pharm. Weekblad* 72, 1078-1101 (1935).—Historical. A chronological list is given of national Pharmacopoeias from the Danish of 1772 to the Jugoslav. of 1933, and of the local Dutch Pharmacopoeias from that of Amsterdam in 1629 to that of Groningen in 1729 and their subsequent editions. Some obsolete recipes are quoted from the old Amsterdam

Pharm., and the naive dedicatory verses of the Leiden Pharm. are given. A. W. Dor.

**Communications from the laboratory of the Dutch Society for the Advancement of Pharmacy**. No. 52. Camphoras hexamethylenetetrammi. H. J. van Giffen. *Pharm. Weekblad* 72, 1153-8 (1935); cf. C. A. 29, 26391.—"Amphotropin" is shown to be the 1 2 and not the 1 1 salt of camphoric acid and (CH<sub>3</sub>)<sub>6</sub>N<sub>4</sub>. Directions are given for prep. solns. for ampoules. A. W. Dor.

**Oleum citronellae**. P. A. Rowaan. *Pharm. Weekblad* 72, 1158 (1935). cf. C. A. 29, 63579.—Of 14 samples of Ceylon citronella oil purchased in retail drug stores 7 failed to meet the "Schimmel test" of complete soly. in 80% EtOH. This supports R.'s contention (C. A. 29, 6359) that Ceylon oil is inferior to Java oil. A. W. Dor.

**Spectrographic investigation of cinchona alkaloids**. C. G. van Arkel and P. van der Wielen. *Pharm. Weekblad* 72, 1194-1208 (1935).—Curves of absorption spectra in the ultraviolet are given for total alkaloids of cinchona bark, for the tartrates of quinine, cinchonidine, quinidine and cinchonine, and for various mixts of quinine and cinchonidine. The curves are so similar that a detn. of quinine in the presence of one or more of the other alkaloids is not feasible spectrographically. Total alkaloids from the leaves, however, give a different curve from those of the bark. A. W. Dor.

**Phenobarbital injections**. C. J. Blok. *Pharm. Weekblad* 72, 1221-5 (1935).—Phenobarbital-Na in aq. soln. decamps spontaneously with sepn. of cryst. PhEtCHCO-NHCONH. A stable soln. suitable for ampoules can be prep'd by dissolving 10 g phenobarbital and 2 75 g Et<sub>3</sub>NH in a mixed solvent contg. 26 g EtOH and 31 g glycerol dild. to 100 cc. This soln. can be heated to 60-5° without decompn. A. W. Dor.

**Preparation of infusions, decoctions and macerations in accordance with the D. A. B. VI**, by the aid of the "Sintax" apparatus of Schott & Gen., Jena. Aumüller. *Apoth. Ztg.* 50, 1264-5 (1935).—An illustrated article showing the use of this app. when working condurango, ipecac, cinchona, etc. W. O. E.

**Examination of sterilized surgical dressings**. Ernst Deussen. *Apoth. Ztg.* 50, 1403-4 (1935).—A commentary on the D. A. B. specifications. W. O. E.

**German drug plant in the past and present**. Hubert Vollmer. *Deut. Heilpflanze (Apoth. Ztg.)* 50, 1366 12, 89-91 (1935).—An address. W. O. E.

**Scheele Addendum I**. O. Zekert. *Pharm. Monatsh.* 16, 119-20 (1935).—A personal record of Scheele, confirmed by Jacob Berzelius, dealing with the essential acids of tartar. H. M. Burlage.

**Modern viewpoints and methods for the examination of tinctures**. A. Mayrhofer. *Pharm. Monatsh.* 16, 109-18 (1935).—A review with 34 references. H. M. Burlage.

**Experience and accomplishment in drug testing**. Benno Schwenke. *Pharm. Zentralhalle* 76, 529-33, 545-53 (1935).—An address. W. O. E.

**Preparation of gray mercury salve**. W. Poethke and K. H. Bauer. *Pharm. Zentralhalle* 76, 533-5 (1935).—The explanation of Burgess (C. A. 28, 5609) relative to the reactions taking place in producing this prep'n. is criticized by P. and B. to the effect that what actually takes place in making the salve involves superficial oxidation of the Hg by the H<sub>2</sub>O<sub>2</sub> followed by conversion of the HgO thus formed into a fatty acid salt of Hg, the latter thereupon functioning as emulsifier. W. O. E.

**Detection of eucalyptus oil in pine-needle extract**. B. Stempel. *Pharm. Zentralhalle* 76, 547 (1935).—With adulterated samples any notable increase in d. as content, or particularly isolation of eucalyptol (cineol) from the steam-dist. product and formation of the dibenzide, C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>, therefrom are strongly indicative of the addn. of eucalyptus oil to the ext. W. O. E.

**Baum's**. Curt Lockow. *Pharm. Zentralhalle* 76, 164-5 (1935).—The development of this system of areometric measurement is discussed in connection with the inaccuracies inherent therein, and certain suggestions are offered for improvement of the system. W. O. E.

**Use and detection of saponin in German pharmaceutical**



add 3 cc of distd  $H_2O$ , heat over a small hole in the steam bath with const stirring with a glass rod for at least 10 min, replacing with a small amt of addnl water that which was evapd. This heating will soften the alkaloidal residue and allow the water to take up the strychnine and just so much of the cinchona alkaloids. In this manner the ratio of strychnine to cinchona alkaloids is boosted to nearly equality of each. Cool and filter through a very small paper filter placed in a 1-in glass funnel, having the filter paper when folded cut down to a length of not over one half in. Insert in a funnel, wet with water and pour through this the alkaloidal soln, allowing it to go into a 70 cc Pyrex beaker. Wash the filter paper with 2 cc of distd water and evap the soln to dryness in the steam bath by inserting into a hole of the bath by means of beaker tongs. After evapn the residue will usually be pure white and cryst. If any strychnine is present a small amt of this cryst material will readily show up by any of the principal color tests for strychnine. The following is an example of a digestion and extn. of strychnine from colloidal protein material into 100 cc of horse saliva which was of a very thick gelatinous consistency were incorporated by heating with alc and then driving off the alc before digestion, the following drugs: one 1/100 grain strychnine sulfate hypodermic tablet, 0.00017 g morphine alkaloid, 0.3 gram of caffeine alkaloid, 0.12 gram of quinine sulfate. The entire amt was digested for 6 hrs with 15% by wt of  $H_2SO_4$  by strong vigorous refluxing. The strychnine, caffeine and quinine were recovered by the usual extn processes for these drugs, the caffeine and quinine being extd completely, as put in the soln

W G Gaessler

Determination of the carbonyl group in camphor, menthone, pulegone, citral and fural with 2,4-dinitrophenylhydrazine L Socas Vinals *Anal cal nac farm* (Madrid) 1935, 1, *Anales farm. bioquim*, Supl 6, 84 (1935).—The hydrazones form readily and are very insol in water-alc mixts. The reagent is especially suitable for the quant analysis of perfumes and similar preps.

Pharmaceutical and commercial chaulmoogra oils A Chalmeta and C Chalmeta *Farm. mod* 44, 63(1935); *Anales farm. bioquim*, Supl 6, 75(1935).—Pharmacopea descriptions should include Dymock's reaction for identification and  $n_D^{20} = 1.4842-1.4898$ . The upper limit for  $[\alpha]$  and the lower limit for free acid should be removed.

Chaulmoogra oil and its antileprosy products Andrea Gandini *Scienza farm.* [2], 3, 64-71, 97-106, et seq (1935).—Practically all the known compds and derivs of the chaulmoogra oil acids are described.

Pharmaceutical preparations of equisetum Cristoforo Masino *Scienza farm* [2], 3, 72-8(1935).—Fluidextracts of equisetum contain little or no Si. The expressed juice and infusions of the fresh plant contain sol. Si compds, probably mon.

Bismuth compounds in therapy. Tomaso Cessi *Scienza farm* [2], 3, 78-99(1935).—A review, with 80 references.

Colloidal chemistry in pharmacy Giuseppe Corazza *Scienza farm* [2], 3, 119-32, et seq (1935).—The properties of various types of colloidal solns are discussed.

Analysis of turpentine liniment T McLachlan *Analyst* 60, 685(1935).—In distn with steam for the purpose of removing turpentine and camphor, frothing is likely to cause trouble but can be overcome by adding 0.5 ml of surphy  $H_2PO_4$  per 50 g of liniment.

Analysis of iodine ointments F W Edwards, E B Parkes and H R Nanji *Analyst* 60, 747-8(1935).—Weigh out 2 g of the well mixed ointment into an open capsule which can be prep'd by cutting off the bottom of a test tube. Add a little pumice powder and introduce the capsule and its contents into 100 ml of strong  $H_2SO_4$  in a 400-ml. distg flask which is connected with an air condenser fitted with a bulb-tube dipping under 50 ml of water in a tall cylinder. Heat and distl slowly. At first  $HI$  and  $SO_2$  come off chiefly. Gently boil for 3-4 hrs

which will cause all of the  $I$  to pass into the distillate. Rinse the condenser tube and filter the distillate and the washings to remove fatty acids. Make acid with  $HNO_3$  and ppt with  $AgNO_3$ .

Examination of rubbed spearmint W A N Markwell and A I Cross *Analyst* 60, 747-8(1935).—Spearmint, which has been dried and rubbed to pass through a sieve, is sold in packet form for culinary use. The adulteration with *Asianthus glandulosa* has been mentioned but it is shown here that the appearance of some portions of dried spearmint under the microscope is very similar to that of the striated epidemid of *Asianthus*. The analyst, therefore, as a result of microscopic tests, is likely to report adulteration when the sample is really pure.

Analysis of essence of cumum S Sakay and L Palfray *Ann chim anal chim appl* 17, 289(1935); *cf C A* 27, 5144.—The method previously described is unchanged. The reaction, however, does not take place directly. Cumaldehyde first reacts with benzyl alc. to form benzaldehyde and cumic alc. and it is the benzaldehyde, rather than cumaldehyde, which reacts with the  $KOH$  used in the analysis.

Critical solution temperature in alcohol of castor oil Doris Anderson *Soc Chem Ind. Victoria, Proc*, 34, 914-16(1935).—In the standard British methods for testing the purity of castor oil, the soly. in alc. is required. The test is a delicate one and the so-called critical soln temp varies greatly with slight variations in the d of the alc. used. As a result of this, some pure samples of castor oil will not pass this requirement of the British Pharm.

Fatty alcohol sulfonates in cosmetics Josef Augustin *Am Perfumer* 31, 79-81(1935).

Recent investigations of digitals glucosides A Stoll *Chem-Ztg* 59, 773-6(1935); *cf C. A.* 29, 7584.

Manufacture of ammoniacal smelling salts A G Arend *Perfumery Essent. Oil Record* 26, 331-3(1935).

Anethole. V A Vuishenskii *Bull Applied Botany, Genetics Plant Breeding* (U.S.S.R.), Ser. A, No 14, 173-6 (1934).—*Ileradum lehmannianum* Bge was analyzed and the etheral oils were found to contain 80% anethole. A hectare of this plant gives 10-30 tons of green material, contg 25-100 kg of oil per hectare or 20-80 kg of anethole. The plant residues may be used for sludge for animal feed.

The chemistry of the cardiac glucosides Robert C Filderfield *Chem. Rev.* 17, 187-249(1935).—A review.

Sternutators Kirby P Jackson. *Chem Rev* 17, 251-92(1935).—A review.

Odoriferous principle of lignum aloe Kinzo Kafuku and Nobutoshi Ichikawa *J Chem. Soc Japan* 56, 1155-63 (1935).—Powd lignum aloe obtained from *Aquilaria gallocha* Roxb is extd with alc.; the yield is 48%. Sapon of the ext and steam distn gave benzylacetone (I) and a monoketone (II),  $C_{11}H_{18}O$ , whose semicarbazone is 160-2°. From the residue of sapon of the alc. ext, the presence of hydrocinnamic acid and a cryst. acid,  $C_{11}H_{16}O_2$ , m 103°, was confirmed. A sesquiterpene alc. which gives the odor of lignum aloe is sepd. I and II are probably formed during the sapon.

Essential oil of Lantana camara L II and III. Kinzo Kafuku, Tessaku Ikeda and Chuta Hata *J. Chem. Soc Japan* 56, 1184-91(1935). *cf C. A.* 28, 4175.—The earlier report indicated that sesquiterpene fraction of the oil contained a caryophyllene type (I) and a cadinene type (II); the substance isolated from I is camerenene (III) and from that of II is isocamerenene (IV). II turns to IV, as it is unstable. The other substance isolated from cadinene type is micranene (V). Oxidation of III with  $O_3$  gives  $HCHO$  and acetone, indicating that III contains  $CH_3$  and  $Me_2C$  groups. The nonvolatile residue contains succinic acid. Oxidation of IV gives only  $HCHO$  but no acetone, this may be the change of the  $Me_2C$  group in III to  $Me(CH_2)_2C$  group in order to give an isomer IV. Consts ( $d_{20}^{20}$ ,  $d_4^{20}$ ,  $n_D^{20}$ ,  $M R$ ,  $\alpha_D^{20}$ ) of III and IV in

order named are: 263°, 253°; 121-2°, 110-11°; 0.9056, 0.8942; 1.500, 1.4925; 66.25, 66.41; +6.74°, -11.21°. V has no conjugated double bond as it is not reduced by Na and AmOH. Oxidation with O<sub>2</sub> gives small amt. of HC-HO and acetone. Nonvolatile residue gives Ag salt, C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>Ag. Oxidation with KMnO<sub>4</sub> gives hemihydro-malephonic acid, m 179-181.5°. These facts give a clue that the structure of V must be either VI or VII.



(VI)



(VII)

V b<sub>1</sub> 126-8°, d<sub>1</sub><sup>20</sup> 0.9155, n<sub>D</sub><sup>20</sup> 1.5050, M R 66.09, V-HCl m 105.5-6.5°, V-HBr m 123-4°, V-HI m 114-15°, V-NOCl m 94-6° K. Kutsa.

Newer information on local anesthetics Guy H. Ellingham. *Brit. Dental J.* 59, 198-205 (1935).—No anesthetic is of more general use than ethocaine (procaine). The synergizing effect of K sulfate and methylene blue should be investigated further. Anesthetic soles are usually acid and alkalization to a degree approximating that of living serum has been shown to be very desirable. A naturally alk. salt should be used whenever possible.

Frances Krasnow  
The constituents of Chinese drug, hsi-hsin, Asarum Sieboldi, Mei T. Q. Chou and J. H. Chu. *Chinese J. Physiol.* 9, 261-6 (1935).—The essential oil of this drug contains the following constituents: a small amt. of org. acid not identified, pinene, a phenol, m 110°, forming an acetate, m 129-130° and a benzoate, m 124°, a ketone whose semicarbazone m. 182°, and methyl eugenol. From the nonvolatile resinous matter obtained by extr. there has been isolated a well-cryst. neutral principle, having a mol. formula C<sub>14</sub>H<sub>18</sub>O, m. 124° and with a specific rotation -130°. It contains no methoxyl groups and does not react with Ac<sub>2</sub>O and phenylhydrazine.

L. A. Maynard  
Emetine camphosulfonate. Carlos A. Grau. *Bull. sci. pharm.* 42, 452-6 (1935).—The prep. of camphosulfonic acid is described. Emetine base should be prep. from the pure HCl salt. Most com. salts contain up to 3% cephaeline which is about 3 times as toxic as emetine. The camphosulfonate is prep. by mixing a soln. of 14.128 g. emetine with 13.67 g. camphosulfonic acid in EtOH and evap. The thick sirup is dild. to 1 l. and filled aseptically into ampoules. The effect in amebiasis is quite satisfactory; the toxicity is 1/3 of that of the HCl salt causing a far better tolerance.

A. E. Meyer  
The question of assaying ergotocine, the new ergot principle. Edward E. Swanson, Chester C. Hargreaves and K. K. Chen. *J. Am. Pharm. Assoc.* 24, 835-9 (1935).—Thirty-five lots of ergotocine were compared by 6 methods of testing: the isolated rabbit uterus, the U. S. P. cock's comb, the polarimetric, the colorimetric, the postpartum dog's uterus and the postpartum human uterus methods. With a few exceptions the results by the different procedures appear to confirm one another. Ergotocine may be assayed by the isolated rabbit uterus method, supplemented by the clinical method. Polarimetric and colorimetric tests may furnish preliminary information concerning the purity of the product, but should always be confirmed by physiol. and clinical expts. In the light of our present knowledge the U. S. P. cock's comb method is not sp. enough for the assay of ergotocine, and the Broom-Clark method is entirely useless for this purpose.

A. Papineau-Couture  
Drug extraction. V. The extraction of belladonna root with glyceric menstrua. Wm. J. Husa and Louis Magd. *J. Am. Pharm. Assoc.* 24, 839-40 (1935); cf. C. A. 29, 7582°.—Percolation expts. with glyceric menstrua indicated that glycerol retards the extrn. of alkaloids from belladonna root. The retardation is greater with increasing concn. of glycerol and with decreasing concn. of alc.

A. Papineau-Couture

1 Assay for phenol in official preparations. Glenn L. Jenkins and Melvin F. W. Dunker. *J. Am. Pharm. Assoc.* 24, 840-2 (1935).—The following method was studied: introduce a sample contg. about 0.04 g. PhOH into a glass-stoppered flask, dil., add 30 cc. of 0.1 N Koppe-schaar's soln., rinse down the neck of the flask, quickly introduce 5 cc. HCl, shake vigorously at intervals during 5-10 min., add 5 cc. 20% KI soln., taking care to avoid loss of Br vapors, shake vigorously for 3-5 min., add 1 cc. CHCl<sub>3</sub> and titrate with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. Glycerite of phenol and phenolated soln. of I can be assayed directly by this method after dild. without sepp. the PhOH. Camphorated phenol, phenolated oil and ointment of PhOH can be assayed satisfactorily after extrn. of the PhOH with H<sub>2</sub>O. The method of assay for ointment of PhOH proposed for inclusion in the U. S. P. XI is subject to variations due to the volatility of the constituents of the ointment base.

A. Papineau-Couture  
A chemical study of sulfur ointment. Lewis C. Britt. *J. Am. Pharm. Assoc.* 24, 854-6 (1935).—The following method has been devised and found workable: gently boil 1 g. of the ointment with 50 cc. of 10% KOH till the lard is saponified and the S converted in K<sub>2</sub>S (about 30 min.), add 50 cc. of H<sub>2</sub>O soln., digest 30 min. slightly below boiling, acidify slightly with SO<sub>2</sub>-free HCl, heat to boiling, cool, filter, wash and det. S in the filtrate by pptn. with BaCl<sub>2</sub>. Examin. of a no. of com. samples of S ointment indicated a tendency on the part of retail pharmacists to substitute petroleum for benzonated lard.

A. Papineau-Couture  
Report on (the assay of) drugs Arthur E. Paul. *J. Assoc. Official Agr. Chem.* 18, 509-15 (1935), cf. C. A. 28, 6527°.—A brief discussion of the reports of the associate referees.

A. P.-C.  
Report on (the determination of) tetrachloroethylene. G. M. Johnson. *J. Assoc. Official Agr. Chem.* 18, 519-20 (1935), cf. C. A. 28, 6528°.—The previously described method was studied collaboratively on a sample contg. 51.24% C<sub>2</sub>Cl<sub>4</sub> and approx. 25% each of liquid petrolatum and oleoresin aspidium. The results were in excellent agreement with the amt. actually present, and adoption of the method as tentative is recommended.

A. P.-C.  
Report on (the analysis of) mercurials. Wm. C. Cavett. *J. Assoc. Official Agr. Chem.* 18, 520-1 (1935); cf. Deal, C. A. 28, 6527°.—The ointment base interferes with the detn. of Hg in mild mercurial ointment according to U. S. P. directions. The following method, which was studied collaboratively, gave satisfactory results, and its adoption as tentative is recommended: mix the ointment thoroughly with a glass rod avoiding contact with metals, to 1 g. weighed into an Erlenmeyer flask add 20 cc. H<sub>2</sub>O and 20 cc. HNO<sub>3</sub>, heat gently over a small flame till red fumes cease to evolve, cool, decant the aq. soln. from the ointment base into a separator, wash the ointment base repeatedly with 50-cc. portions of H<sub>2</sub>O till all the Hg is removed, decanting the washings into the separator, shake with 50 cc. Et<sub>2</sub>O, transfer the aq. soln. to an Erlenmeyer flask, wash the Et<sub>2</sub>O with 3 X 10 cc. H<sub>2</sub>O till the Hg is removed, add the washings to the flask, add 3 cc. of ferric NH<sub>4</sub> sulfate indicator soln., and titrate with 0.1 N NH<sub>4</sub>-CNS.

A. Papineau-Couture  
Report on microchemical methods for (the identification of) alkaloids. K. K. Glycart. *J. Assoc. Official Agr. Chem.* 18, 521-3 (1935); cf. C. A. 28, 6527°.—A 5% AuCl<sub>3</sub> aq. soln. is a suitable microchem. reagent for the identification of homatropine, hyoscyamine and scopolamine, the test being carried out by adding 2 drops of reagent to 1 drop of approx. 1% soln. of the alkaloid, exam. microscopically and comparing with the crystals produced by known alkaloids. Atropine forms no cryst. ppt. with AuCl<sub>3</sub>.

A. Papineau-Couture  
Report on microchemical methods for (the identification of) synthetics. Irwin S. Shupe. *J. Assoc. Official Agr. Chem.* 18, 523-5 (1935), cf. C. A. 28, 6527°.—In preliminary work on acetanilide and acetophenetidine several reagents were found that produced crystals with PhNH<sub>2</sub>, sulfate and phenetidine sulfate, their products of hydrolysis, but the crystals showed considerable resemblance and did not serve to differentiate PhNH<sub>2</sub> from acetophenetidine.

A 5%  $\text{HgCl}_2$  aq. soln. gives fairly characteristic crystals with  $\alpha$ -naphthyl-ether, but they are rather similar to those given by antipyrine; Marine's reagent (3 g.  $\text{CdI}_2$  in 18 cc.  $\text{H}_2\text{O}$  contg. 6 g.  $\text{KI}$ ) produces more characteristic crystals with antipyrine in acid soln., but is not so sensitive as  $\text{HgCl}_2$  soln. The general microchem. reagents produce no rxns. with 2,4- $\text{C}_6\text{H}_3\text{O}_5(\text{NO}_2)_2$  on treating an alk. soln. of the compd. with  $\text{HCl}$  to liberate the free nitrophenol 4-branched compd. plates are formed. The limit of sensitivity is a concn. of about 1/500  $\text{C}_6\text{H}_3\text{O}_5(\text{NO}_2)_2$ , at which single rectangular plates are formed, but the best results are obtained at a concn. of 1/100 and with a 1%  $\text{HCl}$  soln. In every case comparison should be made with a control of the pure synthetic treated in the same way.

#### A. Papineau-Conture

Report on the determination of hypophosphites (in pharmaceutical preparations). Henry R. Bond, *J. Assoc. Official Agr. Chem.* 18, 525-6 (1935); cf. C. A. 28, 652<sup>1</sup>.—Further study of the detn. of hypophosphites by reduction of  $\text{HgCl}_2$  to  $\text{Hg}_2\text{Cl}_2$  showed that the method is unsatisfactory, the highest results obtained being only 90% of theoretical. Oxidation with standard  $\text{I}_2$  soln. and titration of the excess  $\text{I}_2$  with standard  $\text{Na}_2\text{S}_2\text{O}_3$  give inconsistent results. Collaborative study of the oxidation of hypophosphite to phosphate by boiling with  $\text{HNO}_3$ , with subsequent pptn. of the phosphate, supplemented by the detn. of Ca in Ca hypophosphite, gave satisfactory results, recoveries ranging from 99.42 to 102.56% (av. 100.57%) by  $\text{PbO}_2$  pptn. and from 99.26 to 101.20% (av. 100.17%) by Ca detn.

#### A. Papineau-Conture

Report on (the determination of) santonin. Harry J. Fisher, *J. Assoc. Official Agr. Chem.* 18, 520-22 (1935).—The method of Fernandez and Socas (C. A. 26, 4414, 5853) gives satisfactory results on pure santonin, for the detn. of santonin in chocolate mixts. it gives high results, that are not due to the presence of excess reagent remaining in the ppt. Correct results can be obtained by washing out the fat with petr. ether satd. with santonin and then extg. the santonin with  $\text{CH}_2\text{Cl}_2$ , evapg. the solvent, taking up the residue in alc. and treating with dimethylphenylhydrazine; the present tentative A. O. A. C. method gives slightly high results on chocolate mixts., but it is doubtful whether the increased accuracy obtainable by the modified Fernandez and Socas method is great enough to recommend its adoption in place of the present method which is much less time-consuming. The following method was developed for the detn. of santonin in chocolate: shake 6 g. of ground sample for 3 hrs. with 100 cc.  $\text{CH}_2\text{Cl}_2$ , filter, shake the filtrate vigorously for 5 min. with 25 cc. of 5%  $\text{Na}_2\text{CO}_3$  soln., discard the aq. layer, centrifuge the  $\text{CH}_2\text{Cl}_2$  soln., filter, evap. 50 cc. to dryness, take up in 60 cc. satd.  $\text{Ba}(\text{OH})_2$ , heat to boil and then for 10 min. on a steam bath, filter, cool, add 6 cc. dil.  $\text{HCl}$  (2 + 1), ext. with 25, 15, 10, 10 and 5 cc. of  $\text{CHCl}_3$ , filtering each portion through a pledget of cotton, evap. to dryness, dissolve in 25 cc. warm alc., add 50 cc. of 1% 2,4-dinitrophenylhydrazine in 10%  $\text{H}_2\text{SO}_4$  (by vol.), let stand 4 hrs. in a dark place, filter on a tared Gooch crucible, wash with 150 cc. of dil. (1 + 2) alc., dry 1 hr. at 100°, cool and weigh. Collaborative study of the method on chocolate and on a santonin-magwort mixt. of known santonin content showed that, while check detns. were satisfactorily close and the av. value for all collaborators was very near the true value, there was too wide a variation between the results of the collaborators with the santonica sample. The method, however, appears promising and should be studied further.

#### A. Papineau-Conture

Report on (the determination of) ether. W. F. Kunkle, *J. Assoc. Official Agr. Chem.* 18, 522-25 (1935), cf. C. A. 28, 652<sup>1</sup>.—The effect of essential oils (oil of eucalyptus, turpentine, Me. schavolate) on the detn. of  $\text{Et}_2\text{O}$  in alc. soln. was studied with a view to finding some selective absorption liquid that will retain the vapor of volatile substances and permit the  $\text{Et}_2\text{O}$  vapor to pass through. Of all the liquids tried, only the combination of a satd. soln. of  $\text{KOH}$  in alc. (mixed with the sample) and  $\text{H}_2\text{SO}_4$  and alc. (1 - 1 by vol.) in 2% chloroform quantitatively retains the volatile substances. This combination, however, al-

most completely retains  $\text{Et}_2\text{O}$ .  $\text{H}_2\text{SO}_4$ - $\text{H}_2\text{O}$ -alc. (1 - 1 + 2 by vol.) also retains  $\text{Et}_2\text{O}$  vapor. The results show that some progress was made in the search for the proper selective absorption liquid, but they do not justify recommendation of a method for  $\text{Et}_2\text{O}$  in a sample contg. an unknown amt. of essential oil or other volatile substance. When it is definitely known that the sample contains only a small quantity of a volatile substance when compared with the quantity of  $\text{Et}_2\text{O}$  present, the present tentative A. O. A. C. method for 1 : 1 : 0 gives fairly good results.

#### A. Papineau-Conture

Report on (the determination of) benzyl compounds (in pharmaceutical preparations). Joseph Callaway, Jr. *J. Assoc. Official Agr. Chem.* 18, 533-9 (1935); cf. C. A. 28, 652<sup>1</sup>.—When attempts were made to apply the previously devised method to benzyl benzoate, it was found that aq. alkali does not completely hydrolyze the compd., even after prolonged boiling. In alc. soln. it was found to be quickly hydrolyzed. The following formulae were worked out for calg. amts. of both benzyl and EtOals in the same soln. based on the d. and immersion refractometer readings:  $3.1\text{N} - 7.91\text{S} = 74.30\text{A} - 257.6\text{B}$ ,  $3.1\text{N} + 1.624\text{I} = 5 - 11.3\text{S}$ , in which  $\text{A} = \text{g. per 100 cc. of benzyl alc.}$ ,  $\text{I} = \text{g. per vol. of } \text{Et}_2\text{OH}$ ,  $\text{A} = \text{d. of the mixt.}$ ,  $\text{B} = \text{immersion refractometer reading at } 20^\circ$ . The method is described in detail and shown to give fairly accurate results for benzyl alc.

#### A. Papineau-Conture

Report on (the determination of) small quantities of morphine in syrup. F. O. Laton, *J. Assoc. Official Agr. Chem.* 18, 539-41 (1935); cf. C. A. 28, 652<sup>1</sup>.—Further collaborative study of the previously described method (modified by using larger quantities of  $\text{CHCl}_3$ -alc. mixt. to insure absence of emulsions) gave substantially similar results.

#### A. Papineau-Conture

Report on chemical assay for ergot alkaloids. C. K. Givart, *J. Assoc. Official Agr. Chem.* 18, 543-4 (1935); cf. C. A. 28, 652<sup>1</sup>.—The colorimetric assay of fluid extract of ergo. with both ergotamine ethanesulfonate (I) and ergotamine tartrate (II) as standards was studied collaboratively. With 2 exceptions, the results were in good agreement. The general av. of alkaloids was 0.0785 g. per 100 cc. with I and 0.0635 with II. One collaborator using a Lovibond tintometer instead of a colorimeter obtained results with I which were equal to the av. of all results.

#### A. Papineau-Conture

Report on (the determination of) nitrates in tablets. Frank C. Symon, *J. Assoc. Official Agr. Chem.* 18, 544-6 (1935); cf. C. A. 28, 652<sup>1</sup>.—A collaborative study of the  $\text{KClO}_4$  method on tablets composed of dried fluid extract of *Crataegus* fr., starch,  $\text{NaHCO}_3$  and  $\text{NaNO}_3$  gave very satisfactory results, the max. calcd. deviation being 1.9% from theoretical.

#### A. Papineau-Conture

Report on (the analysis of) ointments. W. F. Kunkle, *J. Assoc. Official Agr. Chem.* 18, 546-6 (1935); cf. C. A. 28, 652<sup>1</sup>.—The 2 methods studied previously for the detn. of free  $\text{I}$  and  $\text{KI}$ , though they appeared to be promising when used on relatively fresh ointments, were later found to be unsuitable, because of the following changes which occur after the ointment has stood 6-8 months: (1) a trace (about 0.1-0.2%) of the  $\text{I}$  is absorbed by the fat, (2) there is a slow decrease in the  $\text{I}$  content accompanied by a corresponding increase in water, the cause of which has not been ascertained. A method was developed (and is described in detail) for the detn. of total  $\text{I}$ , which is essentially as follows: 1 g. of ointment is carbonized at not over dull red heat in a Ni crucible in presence of 4 g.  $\text{K}_2\text{CO}_3$ , the *residue* is taken up with 100 cc.  $\text{H}_2\text{O}$  and treated with 2%  $\text{KMnO}_4$  to a permanent pink color is discharged with alc.; the soln. is made to definite vol., and a suitable aliquot is acidified with  $\text{H}_2\text{SO}_4$  treated with 1%  $\text{KI}$  soln. and titrated with 0.1  $\text{N}$   $\text{Na}_2\text{S}_2\text{O}_3$  of which 1 cc. = 0.002115 g.  $\text{I}$ .

#### A. Papineau-Conture

Report on (the determination of) acetophenone, acetrisalicylic acid and caffeine. L. F. Warren, *J. Assoc. Official Agr. Chem.* 18, 547-51 (1935); cf. C. A. 28, 652<sup>1</sup>.—A method for the sepn. of the 3 drugs is described. It is based on the removal of the medicinal ingredients from the mixt. in slightly acid soln. by means of  $\text{CHCl}_3$ .

before attempting to sep. the individual constituents Acetylcholine and is extd. from the  $\text{CHCl}_3$  soln. by means of  $\text{Na}_2\text{CO}_3$  soln. and detd. gravimetrically as tetraiodophenylmercurine. Caffeine is sep'd. from acetophenetidine by evap. the  $\text{CHCl}_3$  in the presence of  $\text{H}_2\text{SO}_4$ , dilz. to not over 20 cc. with  $\text{H}_2\text{O}$ , extg. with 3 X 50 cc.  $\text{CHCl}_3$ , distg. off the bulk of the solvent, evap. the last portions at room temp. and allowing to stand in the open to const. wt. Acetophenetidine is detd. by neutralizing the soln. from which caffeine was extd. by means of an excess of solid  $\text{NaHCO}_3$ , adding 5 drops  $\text{Ac}_2\text{O}$ , extg. with  $\text{CHCl}_3$ , evap. the solvent, removing any considerable excess  $\text{Ac}_2\text{O}$  by repeated addns and evapns of 1 cc.  $\text{CHCl}_3$  and a drop of alc., and allowing to stand in the open or over  $\text{CaO}$  in a vacuum desiccator till the final wt. differs from the preceding by not over 0.5 mg. A collaborative study of the method gave results that were far from uniform, the most consistent errors being the caffeine detn. The results were so far from theoretical that further work on the subject is considered to be unwarranted.

#### A. Papineau-Couture

Report on (the determination of) pyridum C. L. Clav. *J. Assoc. Official Agr. Chem.* 18, 531-2 (1935).—Pyridum base can be detd. in pyridum soln. by extg. a 0.1% soln. with  $\text{CHCl}_3$  in the presence of  $\text{NH}_4\text{OH}$ , evap. the solvent on the water bath and drying at  $100^\circ$  for 1 hr. Purity of the residue can be ascertained by detg. the  $\text{m.p.}$  which should be  $137-9^\circ$ , and by testing with 5%  $\text{KCNS}$  soln. in the presence of very dil.  $\text{HCl}$  which gives characteristic reddish brown crystals. The technique is described. The results tend to be slightly high. Prolonged heating of the residue at  $100^\circ$  produces a considerable loss.

#### A. Papineau-Couture

Report on (the determination of) gums (in drugs). J. H. Cannon. *J. Assoc. Official Agr. Chem.* 18, 532-4 (1935); cf. C. A. 28, 6329.—Collaborative tests were carried out on the seps. of Irish moss and arar (individually) from interfering substances in drugs and their identification, by use of the tests previously described and carrying them out successively in the order named. The results, while not entirely satisfactory, were encouraging, and the methods are considered capable of yielding satisfactory results.

#### A. Papineau-Couture

Report on (the assay of) resins and oleoresins—podyphyllum. L. E. Warren. *J. Assoc. Official Agr. Chem.* 18, 555-7 (1935); cf. C. A. 27, 5148.—A collaborative study of the previously described method (slightly modified) (described in detail in *J. Assoc. Official Agr. Chem.* 18, 80 (1935)) yielded results that showed that close duplication cannot always be obtained, even when the procedure is carried out with rigid attention to details. The method is somewhat empirical and requires that the technique be followed in meticulous detail. A. P.-C.

#### A. P.-C.

Report on (the testing of) psyllum. Henry M. Burdge. *J. Assoc. Official Agr. Chem.* 18, 557-60 (1935).—Clevenger (*Drug Market* 29, 27 (1931)) has proposed to det. the swelling factor of the seeds as follows (Method A): introduce 1 g. of seeds in a 10-cc. graduated cylinder (use a 50-cc. cylinder for *Lillemorhiza myrsinacea*), fill with water, shake frequently to facilitate the swelling of the seedlings, after 30 min. allow the seeds to settle and note the total vol. occupied by the swollen seeds. Youngkin has suggested that the following modification (Method B) permits the formation of more moisture and therefore serves as a better index of the moisture-forming capacity: place 1 g. of seeds in a 50-cc. graduated cylinder and add tap water to the 20-cc. mark (to the 50-cc. mark for *Lillemorhiza myrsinacea*), shake at intervals during a period of 24 hrs., allow to settle and note the total vol. occupied by the seeds. Collaborative study indicated that Method B gives considerably higher swelling factors than Method A because of longer period of exposure, and the use of a larger cylinder prevents packing and ensures more thorough agitation. Results obtained by both methods showed considerable variation and indicated that temp. plays a part in the detn. and that fermentation must be prevented if possible.

#### A. Papineau-Couture

Humectants and their use in cosmetics. R. Emphens.

*Parfums de France* 13, 218-26 (1935) (in French and English).—A review of the present state of the humectant question and of the use that can be made of this knowledge in dermatology.

#### A. Papineau-Couture

Oil of hyssop. Établissements Antoine Chiris. *Parfums de France* 13, 229 (1935).—The d. of oil of hyssop frequently exceeds 0.945 and can even reach 0.970. The ester value is generally low (1.4-13.0) but can at times be as high as 33.0.  $\eta_{sp}$  generally ranges from 1.476 to 1.482, but sometimes reaches 1.49, exceeding the max. 1.486 given by Gildemeister and Hoffmann (C. A. 22, 2812; 24, 620, 25, 1639, 3775). The free alc., detd. by cold formylation and expressed as  $\text{C}_{17}\text{H}_{35}\text{O}$ , range from 10.4 to 15.1%. Contrary to Gildemeister and Hoffmann, it is very seldom that the oil is sol. in 70% alc. A. P.-C.

#### Stiffen oil of myrtle. Établissements Antoine Chiris.

*Parfums de France* 13, 230 (1935).—Analysis of oils of myrtle produced in the Avola regions during the past 10 yrs. yielded the following results: d. 0.892-0.8912,  $\eta_{sp}$  24.93-28.48,  $\eta_{sp}$  1.46-1.49, and value up to 2.8, ester value 42-50.5, esters as  $\text{C}_{17}\text{H}_{35}\text{O}$ ; 14.95-20.86, ester value after acetylation 88.3-105.7, sol. in 0.2-1.5 vol. and more of 90% alc., except in a few rare instances where the more dil. solns are opalescent. These characteristics differ considerably from those published by Pellini and Morani (*Ann. chim. applicata* 7, 1923, cf. C. A. 18, 3682).

#### A. Papineau-Couture

Cesium tetraiodophenolphthalein—a new salt for gall-bladder visualization. Julian Johnson and Lewis H. Hittner. *J. Pharmacol.* 54, 328-9 (1935).—Cs tetraiodophenolphthalein produces Roentgen shadows in smaller amt., and is slightly less toxic intravenously in doses than the corresponding Na salt. In human beings the Cs salt is more effective when given orally (0.2 gr. 2-3 x) but Na phenoltetraiodophenolphthalein was more effective intravenously. T. H. Rader.

A study of a common plant used for popular medicine in Argentina: carqueja. Cleofe Crocco and Héctor Dastugue Basso. *Rev. sociedad médica* 33, 211-18 (1934). *Actas conc. cárm. Argentino* 23, 118 (1935); cf. C. A. 27, 3053.—The characteristics of the root, the pharmacological forms, the phys. and chem. characteristics, the uses and doses of the prep. are given for carqueja (*Carpaea*; *Genista filifolia*). E. M. Symmes.

The germicidal properties of solutions containing potassium permanganate and oxyanate of mercury. Ferenc Varga. *Magyar Orvosi Arch.* 35, 237-45 (1935).—The presence of oxyanate of Hg, boric acid and 0.01 N or 0.02 N  $\text{H}_2\text{SO}_4$  increases the germicidal property of potassium permanganate greatly. Henry Tauber.

Detn. of moisture in small quantities of substance (Erdős). Naphthol deriv. [as a drug] (Swiss pat. 175,307) 25.  $\alpha$ -Arylaminoamide and arylaminoamide sulfonic acid [as intermediate for pharmaceutical products] (U. S. pat. 2,015,515) 25.

Medicines. Chemische Fabrik vorm. Sandoz. Swiss 175,192, July 16, 1935 (Cl. 1154). The triacetanilamide salt of 3,5-dinitro- $p$ -aminobenzoic acid is obtained by treating the acid with monoacetylamine. The compd. is used in therapy.

Therapeutic preparation. Georg Penz. Ger. 616,566, July 31, 1935 (Cl. 12<sup>a</sup>, 17,10). Addn. to 613,222 (C. A. 29, 5021). A substance which counteracts the effects of histamine is recovered from exts. of the accessory thyroid gland or from placental exts. by a modification of the process of Ger. 613,222. The  $\text{H-OH}$  salt contg. the substance which reduces the Ca content of the blood serum, obtained in the last stage of the process of Ger. 613,222, is treated with about twice its vol. of  $\text{E-OH}$ . The Ca-reducing substance is thus precip., while the antihistamine substance remains in soln.

Sedative therapeutic compounds. Abbott Laboratories. Brit. 452,267, July 24, 1935. Compds. having the formula  $\text{RR'XCOON}^+\text{R}''$ , where R is a satd. alkyl group, R' is a secondary alkyl group contg. 4 or 5 C atoms,



X is H or a halogen, R' is H or an aliphatic hydrocarbon group and R'' is H, an aliphatic hydrocarbon group, CONH<sub>2</sub>, or CONHCOCH<sub>3</sub>, are prep'd. by treating a comp'd of formula RR'XCOCI, where R, R' and X are the same as above, with NH<sub>3</sub> or a primary or secondary aliphatic amine to form an amide, or condensing such comp'd with urea to form an acetylurea comp'd. Among examples, (1) ethyl-1-methylbutylacetic acid is converted into the corresponding acid chloride by SOCl<sub>2</sub> and slowly dropped into cold conc'd. NH<sub>4</sub>OH to form ethyl-1-methylbutylacetamide, (2) ethyl-1-methylbutylacetyl chloride is stirred with urea at 130° to form ethyl-1-methylbutylacetylurea Cf. C. A. 28, 6250<sup>5</sup>.

**Antaemic preparations** Wm. S. McElroy and Wm. F. Herron Brit. 431,196, June 27, 1935. The preps are obtained by the autolysis of animal tissue, especially liver and other glandular tissue, the autolysis being continued for 10-14 days in an aq. antiseptic acid soln. until substantial satn. of the soln. with amino acids. CHCl<sub>3</sub> is conveniently used as antiseptic and may be removed by concn. of the autolyzed product. Any solid matter is filtered off and a preservative, e. g., alc., is added to the concentrate.

**Ointments.** Fabrik chem.-pharm. Präparate Johann Trimmel and Hugo Weil Ger. 615,655, July 9, 1935 (Cl. 30k 9 04). Addn. to 587,142 (C. A. 28, 859<sup>9</sup>). Ointment bases are prep'd. by treating alkali silicate solns. with the NH<sub>4</sub> salts of aliphatic or hydroaromatic acids contg. more than 9 C atoms. Examples are given.

**Laxatives** James Henry Gates Brit. 430,960, June 27, 1935. A laxative confection is made by soaking agar (I) in cold H<sub>2</sub>O, sepg. the I and dissolving it in boiling dist'd H<sub>2</sub>O, adding sugar and heating to about 220°F., allowing to stand 2 1/2 hrs. and mixing with phenolphthalein. Soln. of the I is effected in a Cu steam-jacketed kettle with agitation. After addn. of the sugar, corn syrup may be added and the temp. raised to 238°F.

**Glycosides.** Chemische Fabrik vorm. Sandoz Swiss 176,255-7, June 17, 1935 (Cl. 116k). Addns. to 171,174 (C. A. 29, 5601<sup>9</sup>). A new active glycoside is prep'd. by treating the C glycoside of *Digitalis lanata* with Ca(OH)<sub>2</sub> in the presence of water. The product is deacetylated *D. lanata* glycoside C of formula C<sub>20</sub>H<sub>32</sub>O<sub>10</sub> and can be hydrolyzed to give digoxigenin, digitoxose and glucose (176,255). Similarly, acetyldigitoxin is treated with NaOH to give a glycoside identical with digitoxin (176,256). Also, a mixt. of *Digitalis lanata* glycosides A, B and C is treated with Ca(OH)<sub>2</sub> in the presence of water to give the deacetylated glycosides A, B and C which, on hydrolysis give 40-41% of aglucon, 31% digitoxose and 32-33% of the cryst. disaccharide C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>. The compds have a strong cardiac action and are used in therapy. Cf. C. A. 29, 5602<sup>9</sup>.

**Quinine salt of 2 phenylquinoline-4-carboxylic acid** R. & O. Weil, chem.-pharmazeutische Fabrik, Ger. 618,023, Aug. 30, 1935 (Cl. 12p, 12). Addn. to 563,457 (C. A. 28, 4540<sup>9</sup>). The CHCl<sub>3</sub> used as solvent in the process of Ger. 563,457 is replaced by another org. liquid in which the reagents are sol at 60-70°, e. g., benzene or a mixt. of alc. and ether.

**Preparations containing volatile constituents of plants** Madaus & Co. Fr. 784,616, July 22, 1935. Plants or parts of plants contg. volatile constituents are ground up with noncrystallizable or difficultly crystallizable sugars and dried. Dextrose, invert sugar or a mixt. of glucose and lactose may be used.

**Diamino alcohols** Chemische Fabriken Jochum Wiernik & Co. A.-G. Brit. 431,848, July 12, 1935. Divided on and addn. to 431,788 (C. A. 29, 8000<sup>9</sup>). Diamino alcs. of formula RCH(OH)CH(CH<sub>3</sub>)NHR', in which R is an aromatic radical of the C<sub>6</sub>H<sub>5</sub> series and R' is a dialkylaminoalkyl group, are prep'd. from diketones of formula RCOCOCOR' or keto alcs. of formula RCH(OH)COCH<sub>3</sub> by condensing these with aliphatic diamines contg. a primary amino and a dialkylamino group to form azomethine derivs. of formula RCOC(CH<sub>3</sub>)NR' or RCH(OH)C(CH<sub>3</sub>)NR', and subjecting these, simultaneously or subsequently, to reduction. In the examples, (1) 1-phenyl-

propane-1,2-dione and 1-amino-2-diethylaminoethane (II) are condensed in the presence of H<sub>2</sub>SO<sub>4</sub> and Zn dust to yield directly 1-phenyl-2-diethylaminoethylamino-1-propanol (II), which may be purified by way of its oxalate and then converted into its dihydrouide, (2) 1-phenyl-1-hydroxy-2-propanone and I yield an azomethine deriv. which is reduced, without isolation, by treatment with H in the presence of Pd catalyst; II is obtained. The products are of therapeutic value in the treatment of asthma Cf. C. A. 29, 6836<sup>9</sup>.

**Cyclic amines.** Soc. pour l'ind. chim. à Bâle. Swiss 176,830-1, July 16, 1935 (Cl. 116k). Addns. to 172,873 (C. A. 29, 5603<sup>9</sup>). Cycloheptadecanone thioxosime is treated with a reducing agent to give heptadecamethyl-

imine (CH<sub>3</sub>)<sub>17</sub>NH, m. 64-6° (176,830). Similarly, cyclooctanone thioxosime is reduced to give octamethyl-

enimine (CH<sub>3</sub>)<sub>18</sub>NH, m. 148-9° (176,831). The cyclic amines are used in therapy.

**Hydantoin** Soc. pour l'ind. chim. à Bâle. Swiss 174,461, Apr. 1, 1935 (Cl. 116k). Hydantoin contg. a furan ring is prep'd. by heating ethyl furyl ketone with a cyanide and (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> in the presence of water and CO<sub>2</sub> under pressure. Thus, ethyl furyl ketone, (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, KCN, water and EtOH are heated in a closed vessel with a CO<sub>2</sub> pressure of 10-15 atm. The product is 5,5-ethylfurylhydantoin which has therapeutic uses. The structural formula is given.

**Hydantoin derivative.** Chemische Fabrik vorm. Sandoz Swiss 176,827, July 16, 1935 (Cl. 116k). Addn. to 166,004 (C. A. 28, 2850<sup>9</sup>). The deriv. 3-methyl-5,5-phenylmethylhydantoin is prep'd. by treating 5,5-phenylmethylhydantoin with Me<sub>2</sub>SO. The comp'd m. 188-190° and is used in therapy. Its structural formula is given Cf. C. A. 29, 7343<sup>1</sup>.

**Gulonic acid derivative** Tadeus Reichstein. Swiss 175,347, May 1, 1935 (Cl. 36g). The methyl ester of 2-keto-4-gulonic acid is prep'd. by treating the acid with MeOH. The ester m. 158-9° and is used in the prep'n. of vitamin C.

**Nucleotide derivative.** Soc. pour l'ind. chim. à Bâle. Swiss 174,914-15, Apr. 16, 1935 (Cl. 116k). Addns. to 171,871 (C. A. 29, 5604<sup>9</sup>). An acetylating agent is caused to react on muscle adenylic acid (174,914). Alternatively, a deriv. may be obtained by the action of an acetylating agent on guanylic acid (174,915). The derivs. are used in therapy. Examples are given. Cf. C. A. 29, 6248<sup>7</sup>.

**Nucleotide derivative.** Soc. pour l'ind. chim. à Bâle. Swiss 176,258-9, June 17, 1935 (Cl. 116k). Addns. to 171,871 (C. A. 29, 5604<sup>9</sup>). Yeast adenylic acid is subjected to the action of a benzoylating agent to give the nucleotide deriv. benzoyladenylic acid (176,258). Also cytidylic acid is subjected to the action of an acetylating agent to give acetylcytidylic acid (176,259). The derivs. are used in therapy.

**Colloidal calcium malate.** The Drug Products Co., Inc. Brit. 431,375, July 5, 1935. An aq. dispersion of colloidal Ca malate for medical use by intravenous or intramuscular injection is prep'd. by pptg. Ca(OH)<sub>2</sub> and causing malic acid to react therewith in the presence of a dispersing agent, e. g., Na gluconate. NaOH is preferably added to a soln. of CaCl<sub>2</sub> to ppt. the Ca(OH)<sub>2</sub>. Cf. C. A. 29, 4901<sup>1</sup>.

**Heterocyclic mercury compounds.** Soc. pour l'ind. chim. à Bâle. Swiss 176,757, July 16, 1935 (Cl. 116k). The above are prep'd. by the action of a mercuric salt on pyridine-3-carboxylic acid allylamide. Thus, the above mentioned pyridine deriv. is treated with (AcO)<sub>2</sub>Hg to give pyridine-3-carboxylic acid allylamide-(AcO)<sub>2</sub>Hg. This is treated with KOH and evap'd. to give pyridine-3-carboxylic acid allylamide-Hg(OH)<sub>2</sub>, which decomposes at 120°. It forms water-sol salts which are used in therapy.

**Silver glycerophosphate preparation.** Karl Roth. Ger. 618,374, Sept. 6, 1935 (Cl. 12p, 15). Addn. to 617,153 (C. A. 29, 8239<sup>9</sup>). Freshly ppt'd. Ag<sub>2</sub>O is dissolved in an aq. soln. of glycerophosphoric acid and (CH<sub>3</sub>)<sub>2</sub>NH<sub>3</sub>, and the

soln. is filtered and evapd. *in vacuo*. The product is identical with that described in Ger. 617,153.

Complex compounds of alkali metal and alkaline earth metal halides with hydroxy alkyl amines. Walter Kropp (to Winthrop Chemical Co.). U. S. 2,017,976, Oct. 22. Cryst. compds. of basic character and suitable for use in prepg. therapeutic media are obtained by treating a secondary or tertiary amine at least once substituted by a hydroxy alkyl group, such as triethanolamine, diethanolamine, or *N*-benzyltriethanolamine with a halide such as  $\text{CaBr}_2$ ,  $\text{NaI}$ ,  $\text{CaCl}_2$ ,  $\text{LiCl}$  or  $\text{KI}$ , in a solvent such as alc. or water, or by reaction of a hydrohalide of the amines with an alkali or alk. earth metal hydroxide. Several examples with details of procedure are given.

Primary phosphates of amino acid esters. Schering-Kahlbaum A.-G. Brit. 432,155, July 22, 1935. See Ger. 609,997 (C. A. 29, 5224).

Vitamins. Tadeusz Reichstein. Swiss 174,028, Apr. 1, 1935 (Cl. 1164). Vitamin-C (*l*-ascorbic acid), is obtained by enolizing 2-keto-4-gulonic acid ester by an alk. reagent and treating the product with concd. acid. Thus, 2-keto-4-gulonic acid methyl ester is dissolved in hot anhyd. MeOH. The air is driven out by N and Na added. Pure HCl free from Fe is then added to give ascorbic acid.

Fat-soluble vitamins. Sophie Botcharsky (one-fifth to Solomon Testelbaum). U. S. 2,017,942, Oct. 22. A food material contg. these vitamins such as liver, kidney fat, egg yolk or vegetable material is minced and subjected to plasmolysis; any remaining whole cells are caused to burst by the addn. of a suitable quantity of deaerated hot water of such temp. that the resulting mixt. does not exceed 75°, the material is filtered under pressure, the residue is dried at a relatively low temp. under subatm. pressure and is cooled and crushed, extd. with a solvent for the fat-sol. vitamins of low b. p. such as "petr. ether" or 95% alc. and the ext. is filtered and the solvent is distd. off (all the steps being carried on in an inert atm.), to obtain a vitamin concentrate.

Vitamin A. Soc. pour l'ind. chim. à Bâle. Swiss 174,869, Apr. 16, 1935 (Cl. 366). Addn. to 168,135 (C. A. 29, 5529). Monocyclic unsatd. primary diterpene alc. of the vitamin A type are obtained by condensing  $\text{C}_{15}\text{H}_{22}$  with  $\alpha$ -dihydroionone, reducing the resulting acetylene carbinol to the corresponding vinyl carbinol, treating this with an inorg. acid halide and condensing the resulting primary halide with acetoacetic ester. The product is saponified by alkali and the resulting ketone condensed with  $\text{C}_2\text{H}_5$ . The resulting acetylene carbinol is reduced to the corresponding vinyl carbinol which is heated with an org. acid anhydride to form the ester. This is saponified to give the free primary diterpene alc. which b. 136-8°. The product is used in prepg. physiologically active compds. Examples and the structural formulas of the compds. arising at each stage of the prepn. are given.

Hormones. C. F. Boehringer & Soehne G. m. b. H. (Fritz Johannessohn and Erich Rabald, inventors). Ger. 618,165, Sept. 3, 1935 (Cl. 12p 17.10). As an adsorbent for recovering ovarian hormones from liquids contg. them in free or combined form, use is made of an  $\text{Al}(\text{OH})_3$  gel prepd. by treating a soln. of an Al salt with a concd. soln. of  $\text{NH}_3$ , and gradually heating the ppt. The gel A described by Willstätter and Kraut (C. A. 17, 3513) is suitable. The hormones are recovered from the gel by extn. with an org. solvent.

Follicle hormones. Schering-Kahlbaum A.-G. Brit. 432,435, July 25, 1935. Urine of pregnant individuals, or prepn. obtained from urine, are subjected to steam distn. to remove volatile phenols, the residue is treated with an alk. agent, e. g., alkali hydroxide or pyridine, the soln. is then caused to react with an acylating agent, e. g., *p*-toluenesulfonic acid chloride dissolved in  $\text{Me}_2\text{CO}$  or  $\text{BzCl}$ , and the acylation product is isolated and saponified to yield the hormones. When urine is used, the boiling with alkali is continued until all  $\text{NH}_3$  is expelled. Kieselguhr may be added to facilitate removal of the acylation product by filtration, or it may be extd. by means of  $\text{Et}_2\text{O}$ .

Sex hormones. Schering-Kahlbaum A.-G. Brit. 432,474, July 26, 1935. Divided on and addn. to 432,435 (preceding abstr.). The process of 432,435 is applied to other starting materials than urine, such as placenta ext., prepn. from ovaries and other organs, and exts. from vegetable matter prepd. in accordance with Brit. 271,492 (C. A. 22, 1654), 277,302 (C. A. 22, 2440) and 294,650 (C. A. 23, 1996).

Elastic woven bandages. Ludwig Flemmich, Wilhelm Flemmich and Otto Flemmich (trading as A. Flemmich's Sohne). Brit. 431,663, July 12, 1935. A binder or bandage for medical purposes, etc., comprises a warp of rubber threads, fast to boiling, interspersed with cotton, silk, wool or like inelastic warp threads, the rubber threads and some or all the other threads being woven with spaced inelastic weft threads. The rubber threads may be made by spraying rubber latex as described in Brit. 214,615 or may be "Lastex" or "Lactron".

Germicidal dressings. Deutsche Gold- und Silber-Scheideanstalt vormals Roessler. Brit. 431,656, July 12, 1935. See Fr. 780,917 (C. A. 29, 5938).

Dentifrice. Guy A. McDonald and Daniel Müller (to Victor Chemical Works). U. S. 2,018,410, Oct. 22. Communitated di-Ca phosphate is used with a small proportion (suitably about 1-4%) of a stabilizing agent such as a Mg phosphate, stearate or sulfate, etc.

Dentifrice. Rudolf A. Kuever (to Pepsodent Co.). U. S. 2,019,142, Oct. 29. Insol. Na metaphosphate is used in fine particles so as to act as a polishing and cleansing agent (suitably alone or with various other ingredients).

Cosmetics. Ernst Schnabel. Brit. 432,022, July 15, 1935. See Ger. 605,804 (C. A. 29, 1944).

Cosmetic creams. Bella Brisko (nee Margoline). Fr. 784,203, July 22, 1935. The cream contains sapon. spermaceti 10, soda lye 12, stearic acid 50, glycerol 200 kg. and water 1751.

Cleansing agent for the skin. Pierre P. Coustolle. Fr. 784,401, July 22, 1935. A cleansing agent comprises or contains a salt made by the combination with one or more mineral or org. acids, other than those forming soaps, of an amine derived from mono- or poly-hydric alcs, particularly triethanolamine. Examples of acids are  $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ , phosphoric acids, boric acids and glycerophosphoric acid.

Reducing the nicotine content of tobacco. Hall Tobacco Chemical Co. Brit. 431,596, July 11, 1935. See U. S. 1,902,145 (C. A. 28, 4842).

Denicotinizing tobacco. Dietrich Brumund. Ger. 618,359, Sept. 6, 1935 (Cl. 79c. 1). See Fr. 764,754 (C. A. 28, 5934).

## 18—ACIDS, ALKALIES, SALTS AND OTHER HEAVY CHEMICALS

R. M. SYMES

Theoretical considerations on the synthesis of ammonia by Fauser's method. L. Kowalczyk. *Przemysł Chem.* 19, 89-104 (1935).—Four different conditions of chem. reactions are recognized, in which the heat of reaction has different values, depending on the pressures under which the heat content of the reagents is calcd., (a) the first 2 of an imaginary van't Hoff model I for reservoirs and II for reaction vessel, and (b) for tech. app. III,

perfect app. in which the reaction attains an equil. and IV actual app. in which the equil. is not completely reached. In tech. app. the heat of reaction is about 6% lower than for reservoirs of the van't Hoff model. Factors involved in app. design are considered and a material balance is set up.

A. C. Zachlin  
[and hydrogen]. Ängel Julä Sauri. *Quim. e ind.* 12,

157-61(1935)—Several types of com. app. are described.

L. E. Gilson

**Analysis and commercial value of nitrates from Goiás.** Virgilio Lucas *Bol. Assoc. bras. farm.* 16, 313-17(1935)—The nitrates from the state of Goiás are made up almost exclusively of K (93.5 to 98.6%  $\text{KNO}_3$ ), while the Chilean nitrates contain no  $\text{KNO}_3$ . E. S. G. B.

**Cooling of salt solution in a cooling tower.** O. Mertens. *Kunstf. 6*, 26-29(1934)—In the process of KCl production in Solikamsk the brine is cooled to crystallize out KCl. The cooling of the brine in cooling towers vs. the cooling in Fe tanks is discussed. The savings of labor, space and time is emphasized. The problem of the troublesome KCl incrustations forming inside the cooling tower, which later interfere with the operation of the centrifuges, is solved by the following scheme: the cold brine from the tower contg. KCl crystals and broken-off incrustations is passed through a revolving screen. The fine crystals pass through with the brine into a settling tank. Large pieces of incrustations left in the screen are removed by a screw conveyor to a roller mill, crushed and let fall into the settling tank. The uniformly fine crystals obtained in this manner pass to the centrifuges. Pumps, piping and other equipment suitable for this service are discussed.

James Sorrell

**The reaction of sulfur dioxide with water under pressure.** L. L. Kuz'min and V. F. Postnikov. *J. Chem. Ind. (Moscow)* 12, 571-80(1935)—Thermodynamic calculations show that in the formation of  $\text{H}_2\text{SO}_3$  and S from  $\text{SO}_2$  and  $\text{H}_2\text{O}$ , pressures exceeding 300 atm need not be used. Expts. show that increased temp. favors the reaction. In a sealed tube at 320° an approx. 10% soln. of  $\text{SO}_2$  reacts completely in 12 hrs. A smaller vol. of gas space above the liquid is helpful. However, H<sub>2</sub> ions slow the reaction greatly, so that as  $\text{H}_2\text{SO}_3$  is formed the reaction rate falls sharply. This effect can be partially overcome by increasing the pressure of  $\text{SO}_2$  in the soln., but even when liquid  $\text{SO}_2$  is added to the tube the max. concn. of  $\text{H}_2\text{SO}_3$  which can be obtained is 25%. S and Se act as catalysts only at low temps., and  $(\text{NH}_4)_2\text{S}$  is little better. Thus, although the S which is formed is very pure, the reaction has com. limitations. A better method is to use a soln. of  $(\text{NH}_4)_2\text{SO}_3$  and  $\text{NH}_4\text{HSO}_3$ . These react smoothly to form  $(\text{NH}_4)_2\text{SO}_3$  and  $\text{H}_2\text{O}$  at relatively low temp. and in any concn. H. M. Leicester.

**Sulfur from sulfur dioxide.** Arthur J. Caddick. *Mining Mag.* 53, 213-16(1935)— $\text{H}_2\text{S}$  and elemental S are produced when  $\text{SO}_2$  is reduced by incandescent carbonaceous matter, but investigations show that COS and  $\text{CS}_2$  are also formed. Expts. were conducted to det. whether these last 2 could be made to deliver up their S in elemental form; this was found to be readily achieved if either gas were mixed with an excess of  $\text{SO}_2$  and passed through a tube contg. red hot pumice. The secondary reactions and general expt. data are discussed. Each vol. of  $\text{CS}_2$  requires twice its vol. of  $\text{SO}_2$ , and each vol. of COS half its own vol. for complete decompn. to yield elemental S.

A. W. Furbank

**Is there a profit in chlorine and nitrate for salt?** F. W. de Jahn. *Chem. & Met. Eng.* 42, 537-8(1935).

E. H.

**Extraction of germanium and gallium from germanite.** 1. Removal of germanium by the distillation of germanous sulfide. Warren C. Johnson, Laurence S. Foster, and Charles A. Kraus. *J. Am. Chem. Soc.* 57, 1828-31(1935)—A simplified process for the removal of at least 99% of Ge from germanite as germanous sulfide in the vapor phase is described. 2. Acid extraction of gallium. Laurence S. Foster, Warren C. Johnson and Charles A. Kraus. *Ibid.* 1832-5—Residues from the above removal of As and Ge from germanite are extd. with HCl. The ext. is treated with  $\text{H}_2\text{S}$  to ppt. the heavy metal sulfides, Ga and Al are then sepd. from Fe and Zn by boiling the soln. with  $\text{NH}_4\text{HSO}_4$ , leaving the Fe and Zn in soln. Al is sepd. from the Ga as  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$  from a ether-water mixt. satd. with HCl, in which Ga and traces of Fe remain dissolved. The Fe is removed and the Ga pptd. as hy-

drated oxide. The latter is dissolved in slight excess of KOH and electrolyzed, giving the metal Ga.

M. McMahon

**Columbium and tantalum.** Clarence W. Balke. *Ind. Eng. Chem.* 27, 1166-9(1935).—The treatment of tantalite and columbite and the sepu. of Ta and Nb by recrystn. of their double K fluorides are described. These metals are finding extensive uses because of their corrosion-resistant properties, low work function and low cost. Ta carbide is used in the manuf. of hard carbide compns. for cutting tools, wire dies and abrasion-resisting surfaces.

M. McMahon

**A feldspar producer in Colorado.** S. A. Ionides. *Eng. Mining J.* 136, 513-14(1935).—The Western Feldspar Milling Co. has 20 or more deposits, with 7 working.

W. H. Boynton

**Mechanism of hydration of langbeinite.** Tadeusz Pieschowicz. *Przemysł Chem.* 19, 105-110, 122-5(1935).—The process of hydration of langbeinite consists of 2 stages involving soln. of langbeinite to yield a supersatd. soln., which later crystallizes. Covering of the surface by hydrated products can be avoided by agitating the soln. The supersatd. soln. can, when sufficiently stable, be sepd. from the raw material by filtration or decantation and yields pure cryst. products.  $\text{K}_2\text{SO}_4$  free from insol. impurities was obtained from schönite treated by this method. The rate of soln. of langbeinite in satd. solns. is a function of temp. and is represented by curves with a max. at 25° (equil. of schönite- $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) or at 60° (schönite- $\text{K}_2\text{SO}_4$ ). A math. theory of continuous soln. is given.

A. C. Z.

**The activation of bleaching earths.** M. Krayčnové and E. Cerkovnikov. *Chem. Zitz* 59, 716-17(1935).—Samples heated between 250° and 300° showed a max. adsorptive capacity.

S. Bradford Stone

**Recovering solids from solns.** (Ger. pat. 618,107) 1.

**Anhydrous hydrogen fluoride.** Wm. S. Calcott and Lee C. Holt (to E. I. duPont de Nemours & Co.). U. S. 2,018,397, Oct. 22. For producing substantially anhyd. HF from a crude material contg. less water than the const.-boiling mixt., the material is fractionally distd. from a still the final temp. of which is brought to about the b. p. of the const.-boiling mixt., and through a fractionating device so controlled that the exit gas from it is at the temp. of the b. p. of anhyd. HF.

**Dearsenizing sulfuric acid.** Paul W. Bachman (to General Chemical Co.). U. S. 2,019,038, Oct. 22. Gaseous HCl, HF, Cl or F is continuously circulated in intimate contact with a dispersed stream of  $\text{H}_2\text{SO}_4$  at an elevated temp. (suitably about 60° or higher) so that an As halide is formed and passes off as vapor with the unreacted gas, the As halide is in large proportion condensed from the gas and sepd., and residual As halide is sepd. from the gas by the action of an adsorbent such as a carbon, silica gel or the like and the purified gas is then used for treating further portions of  $\text{H}_2\text{SO}_4$  to be purified. An arrangement of app. is described.

**Refining alkali and alkaline earth metals and their alloys.** Deutsche Gold- und Silber-Schneidanstalt vorm. Roessler. Ger. 618,492, Sept. 9, 1935 (Cl. 40a 15.20). A filtering app. is described through which the fused metal or alloy is passed at a temp. at which the impurities remain solid. Means for removing the accumulated impurities is provided. The app. may be operated continuously or intermittently.

**Alkali metal cyanides.** Hermann T. J. König (to N. V. Stikstofbindingsindustrie "Nederland"). U. S. 2,018,125, Oct. 22. For the production of high percentage alkali metal cyanide free from sulfide and chloride, an alk. earth metal cyanamide, such as that of Ca, is heated in the absence of chlorides at a reacting temp. above 500° with an alkali metal sulfide, such as that of Na, together with a C-yielding material, such as anthracite coal, and an excess of an alk. earth metal compd., such as  $\text{CaCO}_3$ , capable of reacting at the temp. involved with the

alkali metal sulfide to form alk. earth metal sulfide; the resulting mass is extr. with a solvent for the alkali metal cyanide, such as with MeOH or EtOH, in which the alk. earth metal sulfide formed is insol.

**Alkali nitrates.** Paul Kubelka. Fr. 784,225, July 22, 1935.  $\text{HNO}_3$  is caused to react on an aq. soln. of an alkali chloride in which is suspended  $\text{Cu}_2\text{O}$  or  $\text{CuO}$  and Cu in amt. at least equiv. to the Cl present. The  $\text{CuCl}$  formed is sepd. and the soln. of alkali nitrate is evapd. The  $\text{Cu}_2\text{O}$  is regenerated.

**Easily soluble alkali metal metasilicate hydrate.** Ernest R. Boller (to Grasselli Chemical Co.). U. S. 2,018,632, Oct. 22. A molten alkali metal metasilicate hydrate is mixed with an easily decomposable peroxide, such as  $\text{Na}_2\text{O}_2$  or  $\text{H}_2\text{O}_2$ , and the mixt. is solidified.

**Gas reactions such as ammonia oxidation.** Nikodem Caro, Albert R. Frank, Rudolf Wendlandt and Thomas Fischer. U. S. 2,018,219, Oct. 22. For assisting in temp. and pressure control, steam or  $\text{CO}_2$  or other gases are added which are easily condensed, absorbed or otherwise sepd. from the reaction gases (as in the form of an aq. soln.) after they have passed through the reaction zone; or water vapor and  $\text{CO}_2$  may be formed from H and CO added to the reaction gases, and in some instances  $\text{SO}_2$  may be added. Various examples with details of procedure are given, and app. is described.

**Catalyst for ammonia oxidation.** Geo. M. Hickey (to J. Bishop & Co. Platinum Works). U. S. 2,018,760, Oct. 29. A catalytic gauze is formed of an alloy of Pt with Rh and Co in small proportions and is annealed and acid washed.

**Catalysts.** I. G. Farbenind. A.-G. Fr. 784,503, July 22, 1935. See Brit. 429,410 (C. A. 29, 70309).

**Purifying zinc salt solutions.** Leon R. Westbrook (to Grasselli Chemical Co.). U. S. 2,017,030, Oct. 22. A soln. of a Zn salt, such as one of  $\text{ZnSO}_4$ , contg. as an impurity an alkali or alk. earth metal compd., such as  $\text{Na}_2\text{SO}_4$ , is treated with Zn fluosilicate, and the resulting ppt. is sepd.

**Aluminum chloride.** Claude G. Miner and Dudley Baird. Fr. 784,178, July 22, 1935. Aluminous materials are caused to react with a chloride of Cu, Pb, Fe or Zn and a reducing agent to produce  $\text{AlCl}_3$  and a metal. Thus  $\text{Al}_2\text{O}_3$  is caused to react with  $\text{FeCl}_3$  and C at  $1200^\circ$ , with  $\text{CuCl}$  and C at  $1000^\circ$  or with  $\text{ZnCl}_2$  and C at  $1450^\circ$ . Cf. C. A. 29, 18934.

**Recovery of beryllium sulfate from its mixture with aluminum sulfate.** Charles B. Sawyer and Bengt Kjellgren (to Brush Beryllium Co.). U. S. 2,018,473, Oct. 22. The  $\text{Al}_2(\text{SO}_4)_3$  in the mixt. is converted, with an alkali sulfate, such as that of  $\text{NH}_4$ , to an alum, and the concn. of the soln., with respect to Be sulfate, is adjusted to or sufficiently close to satn. to prevent the soln. from holding in soln. more than any predetd. small amt. of alum at the final temp. at which the alum is sepd.

**Monocalcium phosphate.** Wm. H. Knox, Jr. (to Victor Chemical Works). U. S. 2,018,449, Oct. 22. Strong  $\text{H}_3\text{PO}_4$  is caused to react with a substantially dry lime base, such as hydrated lime, to produce mono-Ca phosphate and water, the reaction being effected under a substantial vacuum, so that evapn. of water maintains the temp. of the reaction below  $100^\circ$ .

**Calcium sulfate.** Winfield W. Heckert and Gordon D. Patterson (to Krebs Pigment & Color Corp.). U. S. 2,018,955, Oct. 29. A finely divided  $\text{CaSO}_4$  suitable for use with pigments is obtained (suitably by reaction of  $\text{H}_2\text{SO}_4$  with  $\text{CaCl}_2$ ) in an aq. medium in the presence of a compd. of Ce, Th, trivalent Ti or U, which serves to restrain crystal growth.

**Dehydrating copper sulfate.** Hector A. M. Toledo. Brit. 431,859, July 9, 1935.  $\text{CuSO}_4$  contaminated with org. matter, e. g., that resulting from the low-temp. dehydration of an org. liquid, e. g., alc., by means of anhyd.  $\text{CuSO}_4$ , is sepd. by phys. means from adhering org. matter, preferably ground and then dehydrated at  $185\text{--}300^\circ$  with movement or agitation of the salt and removal of the liberated  $\text{H}_2\text{O}$  vapor.

**Magnesium chloride from dolomite.** Joseph S. Laird

(to Ford Motor Co.). U. S. 2,019,265, Oct. 29. Dolomite contg.  $\text{MgCO}_3$  and  $\text{CaCO}_3$ , in proportions deviating substantially from equimol. proportions, is calcined and hydrated with water to form a slurry of  $\text{Ca}(\text{OH})_2$  and  $\text{Mg}(\text{OH})_2$ , and  $\text{HCl}$  is then added to this slurry in an amount not materially exceeding that corresponding to the  $\text{Mg}(\text{OH})_2$  present; the resulting slurry of mixed hydroxides and chlorides is then carbonated to ppt.  $\text{CaCO}_3$ , and the latter is sepd., leaving  $\text{MgCl}_2$  in soln. An arrangement of app. is described.

**Nickel carbonyl.** I. G. Farbenind. A.-G. (Leo Schlecht and Max Naumann, inventors). Ger. 618,108, Sept. 2, 1935 (Cl. 12n 4). Metallurgical or other products which contain Ni and S, and have been obtained by a process involving fusion, are treated with CO (or gases contg. it) at a raised temp. and pressure, e. g.,  $200\text{--}275^\circ$  and 200 atm., without preliminary roasting and reduction. Good yields of  $\text{Ni}(\text{CO})_4$  are obtained. The starting material may be pretreated to increase its surface, e. g., by pouring the fused material into water or by granulation. The preferred starting materials contain, in addn. to Ni, other heavy metals capable of combining with S, e. g., Cu, Co or Fe. Sp. processes are described.

**Potassium sulfate.** Wintershall A.-G. Ger. 618,162, Sept. 3, 1935 (Cl. 12l 5). In the manuf. of  $\text{K}_2\text{SO}_4$  from kieserite by way of the double salt  $\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4$ , undried kieserite is ground after it has been treated with  $\text{KCl}$ -contg. sulfate mother liquor obtained in the process. The double salt thus produced is worked up in known manner. Cf. Ger. 577,031 (C. A. 28, 8657).

**Sodium aluminat.** Russell E. Cushing and Clarence W. Burkhart (to Pennsylvania Salt Mfg. Co.). U. S. 2,018,607, Oct. 22. See Brit. 427,439 (C. A. 29, 6375).

**Flaked sodium chloride suitable for use in household cleaning mixtures, etc.** Ernest R. Boller (to Grasselli Chemical Co.). U. S. 2,018,633, Oct. 22. A paste comprising  $\text{NaCl}$  particles and an org. adhesive such as gum arabic is spread into a film and the film is solidified and broken into flakes.

**Fusing materials such as sodium nitrate, etc.** Herman A. Bechhaus (to Atmospheric Nitrogen Corp.). U. S. 2,019,112, Oct. 29. For fusing a solid, fusible salt of an alkali metal and an oxy-acid of N, such as  $\text{NaNO}_3$ , a melt of the salt is brought directly into contact with a heating gas and the resulting heated melt then has the solid salt introduced into it. An app. for continuous operation is described.

**Zinc oxide.** Polenski & Zollner, Zweigniederlassung Berlin. Fr. 784,642, July 22, 1935.  $\text{ZnO}$  in ores is sepd. from other metal oxides, e. g.,  $\text{PbO}$ , by transforming the latter to sulfides or arsenides, e. g., by a neutral or reducing heating in admixt. with  $\text{FeS}$ . This may be followed by a flotation sepn.

**Sulfur dioxide.** Chemical Construction Corp. Fr. 784,147, July 22, 1935. Acid muds from the purification of hydrocarbons by  $\text{H}_2\text{SO}_4$  are decomposed by bringing them into contact with heated solid bodies, such as metal balls, pebbles, sand or coke. The  $\text{SO}_2$  is withdrawn from the heating zone.

**Cooling hot gases containing sulfur oxides.** Geo. A. Berry (to Calco Chemical Co.). U. S. 2,019,245, Oct. 29. The gases are cooled to temps. suitable for the production of  $\text{H}_2\text{SO}_4$  by the contact process by passing them in heat-exchange relation with liquid water maintained under a const. pressure such that its b. p. is below the temp. of the gases but above the dew point of the gases, so that heat is absorbed at const. temp. by the water and steam is evolved, and the gases are then passed in heat-exchange relation with the steam so generated, so that the steam is superheated and the temp. of the gases is lowered to the desired degree. App. is described.

**Lead recovery from chloride brine.** Niels C. Christensen. U. S. 2,018,438, Oct. 22. A relatively concd. chloride brine contg. Pb salts is treated with an amt. of  $\text{Ca}(\text{OH})_2$ , substantially less than that chemically equiv. to the Pb in soln. in the brine, but more than three-fourths of such equiv. to form a ppt. which may be converted into basic Pb carbonate with  $\text{CO}_2$ .

Hydrogen Fritz Haussing (to American Magnesium Metals Corp.) U. S. reissue 19,733, Oct. 22. A reissue of original pat. No. 1,886,919 (C. A. 25, 1074).

Purifying hydrogen for the hydrogenation of carbonaceous materials. International Hydrogenation Patents Co. Ltd. B-7 431,970, July 18, 1935. For the hydrogenation of carbonaceous materials, H is scrubbed with liquefied hydrocarbons that are gaseous at ordinary temps. and is subsequently washed with oils to remove traces of said scrubbing agents. Liquefied  $\text{CH}_4$ ,  $\text{C}_2\text{H}_6$ ,  $\text{C}_3\text{H}_8$ ,  $\text{C}_4\text{H}_{10}$ ,  $\text{C}_5\text{H}_{12}$ , or a mixt. of these is suitable. The H is preferably preliminarily purified to remove the greater part of the impurities such as hydrocarbons,  $\text{CO}$ ,  $\text{NH}_3$ , etc.

Treating blow-off gas from hydrogen production by the iron water vapor reaction Raymond Blondelle (to Societe des mines de Douerges) U. S. 2,018,118, Oct. 22. A cyclic process for obtaining a mixt. of H with  $\text{CO}$  and a small proportion of N and only traces of hydrocarbons involves first causing coke-oven gases to pass over Fe oxides at about 750° in H-producing app. of the "iron-water steam" type, then causing the resultant gases to pass continuously through a mass of coke, externally heated to about 1200° and collecting the gaseous mixt. passing from the mass of coke (the coke being of sufficiently small sized particles to offer high-contact surface without causing excessive loss of pressure in the app.). App. is described.

Nitrogen and hydrogen. Ammoniaque synthetique et derives Fr. 784,355, July 22, 1935. A mixt. of N and H suitable for the production of  $\text{NH}_3$  is made by gasifying carbon under pressure by a mixt. of steam and air so as to obtain a gas formed principally of H, N and  $\text{CO}$ , which is afterwards treated to remove  $\text{CO}$  and traces of  $\text{CO}$ ,  $\text{CH}_4$  and other impurities. App. is described.

Complex metal compounds of tartaric, gluconic, mucic, lactic and citric acids, etc. Hans Schmidt (to Winthrop Chemical Co.) U. S. 2,018,811, Oct. 29. New metallic complex compds are prep'd by reaction of a peroxide, such as  $\text{H}_2\text{O}_2$  or  $\text{MgO}_2$  with a complex heavy metal compd of a sat'd. aliphatic hydroxy carboxylic acid contg. at least one hydroxy group in a position to a carboxylic acid group, said heavy metal being present in a lower stage of valency. By this oxidation process the complex bound heavy metal is transformed from its lower into its higher stage of valency. Such heavy metals are, for instance, Sb, Sn, Fe, Co, Mn, As, Cr and V. The starting materials suitable for the purpose are obtainable quite generally by heating an aq. suspension of an oxide or hydroxide of a metal of the character described and an aliphatic hydroxy carboxylic acid or a salt thereof of the character described until a clear soln. has been obtained and, if desired, neutralizing the reaction mixt. and pptg. the complex compd formed by pouring the reaction mixt. into an org. precipitant, such as alc. or by evap. to a dry state. Some complex compds crystallize from their aq. solns., such as, for instance, antimonyl potassium tartrate. The starting complex compd is dissolved in water. To the soln. there is added at least the calcd. amount of a peroxide.

1 Preferably, a small excess is used. The reaction proceeds while self-heating and is complete after a short time. If the temp. increases too much, for instance, up to the b. p., cooling of the mixt. is advisable. Instead of using a substance yielding O, free O or gases containing O may be employed. After completion of the reaction, the soln. is neutralized by the addn. of an org. or inorg. base and evapd. to a dry state or pptd. by pouring the soln. into an org. precipitant, such as alc. or acetone.

2 Apparatus for activating carbonaceous materials in thin layers by gases. Albert A. Godel. Fr. 784,079, July 22, 1935.

3 Activating bleaching clays. Anton Wirmüller (to Bayerische A.-G. für chemische & landwirtschaftlich-chemische Fabrikate). U. S. 2,018,887, Oct. 29. Clay material to be activated is suspended in an aq. soln. of a salt such as  $\text{AlCl}_3$  and there is added to the suspension a quantity of free acid of the salt such as  $\text{HCl}$  and the acidified suspension is boiled until the acid is completely used, so that the acid treatment is effected entirely in the presence of salts of the acid.

4 Active charcoal. Albert A. Godel. Fr. 784,115, July 22, 1935. A hard charcoal of high volumetric capacity is made by prolonged maceration of liguorous substances in a soln. of  $\text{H}_2\text{SO}_4$  at appropriate concn. (25° Bc.) and temp. (70°), drying and carbonizing at 100° or higher and then activating by gases or chem. agents at a high temp.

5 Active charcoal. Oswald Heller. Fr. 784,139, July 22, 1935. Grams, dust or molded pieces of a carbonaceous material are activated mechanically in a reaction chamber, a reaction taking place between the material to be activated and the activating substances ( $\text{CO}_2$  and steam) on the one hand and the reaction products ( $\text{CO}$  and  $\text{H}_2$ ) and the means of reaction (O or air) on the other hand, so that by the latter reaction heat is liberated and activating gases are reformed.

6 Exchange and adsorptive material. N. V. Oetrooten Maatschappij "Activit." Fr. 784,348, July 22, 1935. A material having zeolitic and adsorptive properties is made by causing hygroscopic chem. substances capable of fixing water by chem. reaction to react on materials contg. C. Thus, coal, anthracite, sawdust, peat, lignite, starch or molasses is treated with  $\text{SO}_2$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{ZnCl}_2$ ,  $\text{CaCl}_2$ ,  $\text{HCl}$  or  $\text{H}_3\text{PO}_4$  at 60-250°. The product is called active or activated humus.

7 Refining mineral products. Franziska Bleibtreu (nee Stintzing), Helene Bleibtreu (nee Grefenberger), Anna Lahusen (nee Bleibtreu), Max Bleibtreu, Adelheid Bleibtreu (nee Schmidt) and Dorothea Bleibtreu. Ger. 618,022, Aug. 31, 1935 (Cl. 12g. 1.01). Kaolin, trachyte and like mineral products, contg. impurities sol. in hydrohalic acids, are powd. and mixed with (a) a halide of an alkali or alk. earth metal or Al and (b) a solid org. acid or an acid salt of an inorg. acid. All the components are taken in the air-dried state, but at least one of the components (a) or (b) must contain water of crystn. The mixt. is heated to about 130° and then lixiviated with water or dil. acid.

## 19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. E. BARTON AND C. H. KERR

Canada an important market for American glassware Charles H. Ducote. *Ceram. Age* 26, 163 (1935).

Effect of viscosity of the slip on porosity of glass pots produced by casting R. Moretti. *Ind. retro ceram.* 7, 321-5 (1934); *Referatserie Silikatliteratur* No. 866 (1934). —It was found that lowering the viscosity effects a decrease in porosity, it is unimportant whether viscosity is obtained by surplus of liqueficients or water. M. V. Kondodv.

The use of kyanite in crucibles for glass melting S. R. Scholes. *Bull. Am. Ceram. Soc.* 14, 335-7 (1935). —Satisfactory crucibles for glass melting expts. have been developed. They are made from 65% calcined kyanite

(60-mesh) and 35% of a refractory ball clay (known in the trade as "XX Sagger" clay). A slip is made with the addn. of 0.2% alkali by wt. (equal parts  $\text{Na}_2\text{CO}_3$  and Na silicate, brand "S"), and crucibles 2½-7 in. high are cast. Firing is to cone 4. C. H. Kerr.

Controlled luminous gas flame in glass melting J. B. Nealey. *Ceram. Ind.* 24, 273-4 (1935). H. S. W.

The transparency of colorless glasses to various radiations of the spectrum J. Escher-Desrivieres. *Ind. silicates ind.* 6, 254-6, 370-1, 381-3 (1935). —Several transmission curves are included with a general discussion. Herbert S. Wilson.

The question of the magnitude of powdered glass sur-

faces. E. Berger, W. Geffken and K. Stöcker. *Glastech. Ber.* 13, 301-25 (1935).—On the basis of theoretical considerations and exper. results, it is shown that the surfaces of powdered glass can be characterized by statistical values, which are readily reproducible if proper precautions are taken in the prepn. of the samples. They are, of course, not the abs. values and in general will vary with the method of measurements. Extn with HNO<sub>3</sub> and the absorption of iodocyan were the methods used here. Twenty-three references. J. F. Hyde.

The development of optical glasses. In memory of Otto Schott. W. West. *Angew. Chem.* 48, 677-8 (1935).—A historical sketch. Karl Kammermeier. Glass in building. R. A. Miller. *Ind. Eng. Chem.* 27, 1291-3 (1935). G. G.

Hygienic glass for sanatoriums. J. Polivka. *Tschekoslov. J. No. 4-5, 83-6 (1934)*. *Referatortitel: Substitutions- und No. 1327 (1935)*.—Glasses permeable to ultraviolet rays and those absorbing infrared rays are discussed and their properties pointed out. M. V. Kondody.

The determination of alkalis in silicates with special reference to high-alumina refractories. L. J. R. Read. *J. Am. Ceram. Soc.* 18, 341-6 (1935).—For precise analysis of high-Al<sub>2</sub>O<sub>3</sub> materials the J. Lawrence Smith method (*Am. J. Sci.* [2], 50, 269 (1871)). *Am. Chemist* 1, (1871)) is preferable to the HF method, provided the fusion temp. is high enough (1200°) to insure complete decomposition of the sample, and the top of the crucible is cooled sufficiently to prevent loss of alkalis by evapn. For routine analysis the results by the HF method are satisfactory, if it is remembered that the results are slightly low. If the sample can be decomposed completely by HF, that method is as good as the J. Lawrence Smith method and has the advantage of direct detn. of total alkalis. C. H. Kerr.

Italian kaolins. Fausto Pozzi. *Centro studi ceram.* 1, 36 (1934).—The properties of Italian kaolins are given.

Mining of kaolin and refractory clay in Italy. A. Lotti. *Ind. estr. min.* 7, 427-32 (1934); *Referatortitel: Substitutions- und No. 1182 (1935)*.—Italian kaolins contain a high amt. of Fe compounds, and fine quartz particles. The mining and refining of kaolin and clays are described.

M. V. Kondody. Mechanical method for the measurement of the plasticity of clays and mixtures of clays. J. W. Whittemore. *J. Am. Ceram. Soc.* 18, 332-9 (1935).—The app. used measures the penetration of a constantly loaded (15 lb.) hemisphere (3 in. diam.) into the plastic clay mass during a definite time period. The data indicate that  $d = c/t^2$  expresses the relationship between depth of penetration,  $d$ , and the corresponding time,  $t$ ,  $c$  and  $P$  being consts. Undoubtedly  $c$  is related to the load used, the diam. of the hemisphere and some of the properties of the clay.  $P$  is the measure of the plastic properties of the clay. The amt. of recovery of penetration of the hemisphere after the load is removed is a measure of the air and gas content of the plastic mass. Further work is urged.

C. H. Kerr. Plasticized sulfur as a jointing material for clay products. W. C. Rueckel and W. W. Duecker. *Bull. Am. Ceram. Soc.* 14, 329-32 (1935).—Excellent results with plasticized S as a bonding agent for paving brick suggests its use as a jointing material for sewer pipe, sewer brick, etc.

C. H. Kerr. The effect of the solid-liquid ratio on grinding a ceramic nonplastic. Karl Schwartzwalder and Paul G. Herold. *J. Am. Ceram. Soc.* 18, 350-2 (1935).—A ceramic nonplastic (sp. gr. 3.04) was wet-ground with solid-liquid ratios of 2.7-1 and 1:15 for various lengths of time. Grain-size distribution and surface area, as detd. by the Wagner turbidimeter method, were compared. The material ground with the least water gave a lower percentage retained on a 325-mesh screen, fewer particles between 10 and 60 microns, and more between 10 and 0 microns.

C. H. Kerr. Specific gravity as an index for slip control. J. R. Beatty. *Bull. Am. Ceram. Soc.* 14, 333-5 (1935).—The sp. gr. of a casting body should be controlled to within

±0.003. The slip must be well blunged to release air, and the sample for testing must be evacuated before making the detn. C. H. Kerr.

The international standardization of colors. A. Maerz. *J. Am. Ceram. Soc.* 18, 361-6; *Bull. Am. Ceram. Soc.* 14, 349-53 (1935).—The classification and nomenclature of colors have been based upon arbitrary numerical systems. No such system has found wide practical use. Names of colors are usually preferred, even in the industries and sciences. There should be international standardization. C. H. Kerr.

Recent revisions of high temperatures. L. Donald Morris and S. R. Scholes. *J. Am. Ceram. Soc.* 18, 339-60 (1935).—Data on recent detns. of certain fixed points for reference in pyrometry are collected here to be readily available to the ceramist. C. H. Kerr.

Screen analysis for plant control. Edgar W. Rugh. *J. Am. Ceram. Soc.* 18, 346-9 (1935). C. H. Kerr.

Pyrophyllite in San Diego County, Calif. (Richard) Coating compns. [for glass, porcelain and brickwork] (Brit. pat. 431,283) 13

Electric furnaces for melting glass. John Ferguson (to Fairmount Glass Works). U. S. 2,018,883-4, Oct. 29. Structural and operative details. U. S. 2,018,883-6 (no assignment noted) also relate to elec. furnaces suitable for melting glass.

Apparatus for molding glass articles such as bottles and jars. Victor E. Hofmann (to Owens-Ill. Glass Co.). U. S. 2,018,021, Oct. 22. Mech. features.

Apparatus for forming glassware such as bottles and jars. Frank O'Neill and Clarence C. Kinker (to Owens-Ill. Glass Co.). U. S. 2,018,030, Oct. 22. Mech. features.

Apparatus for forming hollow glass articles such as bottles. Wm. H. Watkins and Clarence C. Kinker (to Owens-Ill. Glass Co.). U. S. 2,017,998, Oct. 22. Mech. features.

Apparatus for manufacture of hollow glass articles. Frederick A. Harrison (to United Glass Bottle Manufacturers Ltd.). U. S. 2,018,735, Oct. 29. Mech. features.

Varietated glass articles. Henry T. Hellmers and Robert H. Donald (to Akro Agate Co.). U. S. 2,019,048, Oct. 29. Various details of app. and operation are described, for combining molten glasses of different colors and forming articles such as cups, jars, etc.

Chromium-plated glass-working apparatus. Rowland D. Smith (to Corning Glass Works). U. S. 2,018,814, Oct. 29. Devices such as molds, planers, punties and blow irons are formed of oxidizable metal such as steel or cast iron and coated with Cr.

Soft glass of high electrical resistance. Wm. C. Taylor (to Corning Glass Works). U. S. 2,018,816, Oct. 29. A glass contg. Pb oxide also contains boric oxide and F in a combined proportion of 1-6%, the boric oxide content being at least as great as the F content. U. S. 2,018,817 relates to glass such as may contain silica 53-60, Pb oxide 18-30, Na<sub>2</sub>O 2.7-3.5, K<sub>2</sub>O 8-10, Li<sub>2</sub>O 0.1-1.2 and Al<sub>2</sub>O<sub>3</sub> not more than 2%. C. H. Kerr.

Sheet glass. Wm. L. Monro (to American Window Glass Co.). Brit. 431,668, July 12, 1935. In drawing a sheet upwards from a bath of molten glass contained in a drawing pit and moving the sheet continuously in a vertical direction until the glass has been annealed and cooled, the surrounding temp. of the glass is so controlled that the annealing zone lies at least partly inside the drawing pit. The invention may be applied to an app. of the Fourcault type in which a sheet is drawn from a mass of glass through a debiteuse which is situated in a drawing pit, the sheet being annealed and cooled in a hear situated above the pit. Coolers are situated as usual adjacent the meniscus of the sheet and a 2nd set of coolers is provided, adjacent to the sheet and above the main coolers, to reduce the temp. of the rising glass close to, and preferably within, the annealing range. Air is blown through the base of the hear transversely to the sheet by the means and in

the manner described in Brit. 431,699 (following abstr.). U. S. C. A. 2,943,281.

**Making sheet glass.** Frederic L. Bishop (to American Window Glass Co.). Brit. 431,699, July 12, 1935. In drawing a sheet continuously upward from a bath of molten glass, means is provided for causing the air or other gas that is in contact with the rising sheet, particularly with its lower portion, to circulate transversely to obtain more uniform annealing. A Fourcault type of app. is described, where the required circulation of the gas may be effected by means of blower nozzles which are placed at the sides of the drawing pit and direct air blasts transversely to the length of the glass sheet. The circulation of air may take place either in the pit or in the lower part of the hear above the pit so long as it takes place adjacent to that part of the glass where the temp. is within or above the annealing range.

**Apparatus for cutting sheet glass.** Joseph P. Crowley and Victor Mamborg (to Libbey-Owens-Ford Glass Co.). U. S. 2,019,175, Oct. 29. Structural and mech. features.

**Apparatus for automatically cutting predetermined lengths of a strip of glass.** Pilkington Brothers Ltd. and Joseph Gaskell. Brit. 431,495, July 8, 1935.

**Apparatus for tempering sheet glass.** Pilkington Brothers Ltd. Ger. 618,283, Sept. 6, 1935 (Cl. 32a 30). See Brit. 438,548 (C. A. 28, 579).

**Hardening glass.** Fritz Eckert. Brit. 431,565, July 1, 1935. In hardening glass articles, the temp. to which the glass should be heated is decided by observing the change of some phys. property of the glass which varies with temp., a sep. detm. being made for each article that is hardened. The desired temp. can be detd. by measuring the rapid decrease in the elec. resistance that takes place as the temp. approaches the softening point, or by observing the softening of a specimen of glass that is placed in such a position in the furnace that it is somewhat hotter than the sheet which is being heated.

**Opacifying glass.** Geb. Pustler Glasfabrikumverw. G. m. b. H. Ger. 618,299, Aug. 31, 1935 (Cl. 33b 6). An irregular opacity is produced by treating glass with an alk. HF bath of moderate concn., e. g., 10-60%, to which added HF has been added in a proportion up to 15% by vol.

**Frosting glass bulbs.** Yukiochi Sakakura. U. S. 2,018,422, Oct. 22. A glass bulb is filled with a soln. of NaHF<sub>2</sub> and is vertically dipped in a heating liquid such as water so that convection currents are created and the bulb is frosted in gradation on its inner side and a transparent portion is left at the top of the neck.

**Tools for polishing and grinding glass.** Frederic B. Waldron (to Pilkington Bros. Ltd.). U. S. 2,019,204-5, Oct. 29. Structural, mech. and operative details.

**Compound glass.** Schlensker Spiegelglas-Manufaktur, Carl Tietz G. m. b. H. Brit. 431,254, July 2, 1935. An inspection glass, e. g., for indicating liquid levels in pressure and vacuum containers for corrosive liquids, comprises several glass pieces sep'd by a layer or layers of a non-dissolving material having approx. the same  $n$  as glass. The single glass pieces may be hardened and may differ in compn., e. g., the innermost piece may be a corrosion-resisting glass or have a low coeff. of expansion or may have a high Al<sub>2</sub>O<sub>3</sub> content. A suitable non-dissolving material is a mixt. of polymerized vinyl acetate with a diluent, e. g., castor oil, oil of turpentine, and, if desired, nitrocellulose, and may be applied as a soln.

**Laminated glass ("safety glass").** Wallace H. Carothers, Gerard J. Berthel and Ralph A. Jacobson (to E. I. du Pont de Nemours & Co.). U. S. 2,019,118, Oct. 29.

**Sheets of glass** are united by a film orig. a polymer of a vinyl-ethyl carboxyl. Several examples are given.

**Porous ceramic tiles** partially glazed and suitable for walls, etc. Nelson P. Corne (to Mosier Tile Co.). U. S. 2,018,201, Oct. 22. Structural details.

**Finishing siliceous articles.** Gaston Delpech (to Societe anon. des manufactures des glaces & produits chimiques de Saint-Gobain, Chassy & Cirey). U. S. 2,018,855, Oct. 22. The surfaces of a hot siliceous article are ground while at a temp. which is at least equal to the transforma-

tion point of cristobalite, the ground surfaces are heated while they are maintained at such temp. to glaze them, and the article is subjected to a reheating to at least 1000°. App. is described.

**Bricks for regenerator checkerwork.** Carl Otto. U. S. 2,018,223, Oct. 22. Structural details. U. S. 2,018,224 relates to regenerator chamber and checkerwork filler construction.

**Casting hollow articles or those with thin edges** such as tubes or containers of zirconia, silica, etc. Gaston Delpech (to Societe anon. des manufactures des glaces & produits chimiques de Saint-Gobain, Chassy & Cirey). U. S. 2,019,045, Oct. 29. App. and various operative details are described.

**Porcelain resistant to passage of x-rays.** Leslie Brown (to Lenox Inc.). U. S. 2,018,590, Oct. 22. A reactive Pb compd. such as Pb oxide, an acidic oxide such as SiO<sub>2</sub> and a relatively small amt. of a heat-decomposable alkali metal salt such as KNO<sub>3</sub> are heated together to form a vitrified calcine of relatively high m. p.; this calcine is added to a porcelain mix and the mixt., in finely divided form, is pressed to the desired shape and fired to cause vitrification. Numerous examples are given.

**Refractory materials.** Victor M. Goldschmidt. Ger. 618,094, Sept. 4, 1935 (Cl. 8b 8,08). Minerals consisting mainly of Mg orthosilicate and contg. no more than 10% of Fe, calc'd. as FeO, are worked into mixts. comprising mineral powder assocd. with granules or pieces of the mineral, and the mixts. are sintered without fusion in an oxidizing atm. and then, if desired, in a reducing atm. The mixts. may be melted before they are sintered. Olivine is the preferred mineral.

**Refractory product suitable for resisting spalling.** Henry N. Baumann, Jr., and Charles McMillen (to Carborundum Co.). U. S. 2,019,238, Oct. 29. Fused cast refractory articles are formed of alumina together with about 2-10% of magnesia, the microstructure of the casting consisting principally of interlocking crystals of mullite-spinel and crystals of alumina.

**Cast refractory articles** such as linings for glass tanks. Raymond C. Bonner and Henry N. Baumann, Jr. (to Carborundum Co.). U. S. 2,019,239, Oct. 29. Cast articles are formed of Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>, SiO<sub>2</sub>-2-14 and an alk. oxide such as Na<sub>2</sub>O or K<sub>2</sub>O 1-10%.

**Firing refractory blocks.** Hartford-Empire Co. Ger. 618,266, Sept. 4, 1935 (Cl. 8b 12 16). See Brit. 276,993 (C. A. 22, 2443).

**Electric furnace for heat-testing refractory products.** Daniel Petit. Fr. 784,105, July 22, 1935.

**Abrasive articles.** The Carborundum Co. Ltd. Brit. 431,592, July 1, 1935. In mfg. resin-bonded abrasive articles by moistening grains of abrasive material with a resin solvent and mixing with powd. resin, the mixt. is heat-treated while the grains are maintained sufficiently sep'd. to prevent agglomeration so that a coating or resin bond is formed around the individual abrasive grains. The article is subsequently formed by adding a small quantity of resin solvent, e. g., less than 1%, to the coated grains, pressing the mixt. and curing by heating. As a result of the previous heat-treatment of the coated grains, the resin bond is partially hardened and does not tend to flow between the grains during the pressing and heat-treatment of the article, which is thus open-pored and cut's freely.

**Recovering grain** such as silicon carbide from ceramic bonded articles such as scrap abrasive material. Alfred P. Thompson (to Carborundum Co.). U. S. 2,019,622, Oct. 29. Lumps of the material are subjected to disruptive forces such as by crushing and the disrupted masses are treated with chem. reagents such as NaOH soln. for further freeing the grain from the bond.

**Lead glazes, etc.** Detrich Harkort. Fr. 784,647, July 22, 1935. Ores of Pb contg. S as well as residues of the Pb industry contg. S are converted directly to glazes or frits instead of obtaining the metal. The Pb ore is introduced into a fritting furnace along with the constituents necessary to form the glaze and reducing agents and heated to fusion.

## 20—CEMENT AND OTHER BUILDING MATERIALS

J. C. WITT

A specification and test methods for cold-set cements. S. M. Phelps. *Am. Refractories Inst. Tech. Bull.* No. 60, 8 pp (1935).—The requirements are (1) 95% must pass a 28-mesh standard Tyler sieve, (2) the  $H_2O$  retention period must not be less than 2 min., (3) the av. strength of dry unfired joints must not be less than 100 lb per sq. in.; (4) the mortar must not flow out of the joint in the 1500° refractoness test. The procedure for tests is given. E. C. Petrie

The dependence of shrinkage of portland cement on physical and chemical influences. Hans Kühl and Dzung-Hsien Lu. *Tonind.-Zig* 59, 843-5, 844-6, 913-15, 1016-18, 1028-9 (1935).—The following conditions were studied: amt. of mixing water, humidity of air, compn. of cement, gypsum and  $CaCl_2$  addn. Shrinkage increased with increase in  $Al_2O_3$  modulus but was a min when the molar content of  $Al_2O_3$  and  $Fe_2O_3$  was equal.  $SiO_2$  and  $CaO$  contents had little effect. Forty references.

P. S. Roller

Magnesite lining for cement kilns. Gngor'ev Zlatan-So Ser. 79, No. 61, *Referatov Selskoihkhim. No. 587* (1934).—Expts. showed that the addn. of calcined  $MgO$  to cement does not affect the vol. stability of concrete, and is harmless when present in cement.  $MgO$  present in magnesite brick is dead-burned and therefore does not react with the constituents of the cement mix. Therefore magnesite brick is strongly recommended as lining for cement kilns. M. V. Kondoidy

The testing of cement plaster for water permeability. P. Mecke. *Tonind.-Zig* 59, 991-2 (1935).—An app. is described in which the pressure required to force water through a mortar specimen is measured. P. S. R.

Determining colloids in soil for rammed earth construction. Ralph L. Patty. *Agr. Eng.* 16, 275-6 (1935).—Data are given on the mech. compn. of soils in relation to their suitability for use in the construction of rammed earth walls. Soils contg. 40% of colloids are of doubtful value for pipe work. Soils contg. more than 40% of colloids are definitely undesirable. Addn. of sand does not improve the wall if the soil naturally contains more than 42% of colloids. The Bouyoucos hydrometer method is suitable for detg. colloids in soils for pipe work. K. D. Jacob

Tightness and standing of concrete structures. H. Rahti. *Teknillinen Aikakauslehti* 25, 270-83 (1935).—Small sand particles give tight concrete, the larger particles increase strength. The grain of a suitable sand for water-tight concrete is above the Fuller curve  $y = 100\sqrt{d/D}$ . The newer special cements have not yet been thoroughly tested. Addns. to plaster to increase tightness usually reduce its resistance to acids and its strength. These are chiefly org. substances. Inorg. addns. increase acid resistance, but have no influence on strength or tightness. E. E. Juikola

A new method for impregnation of wood. Bror Häger. *Trävarindustrien* 20, 281-4 (1935).—The logs are treated for some hrs. with a soln. of Na arsenate at 92-4°. At this temp. most of the air and water enclosed in the log are removed. The soln. is cooled to about 75-80° and left for 12 hrs. The soln. partly penetrates the log. The warm soln. of Na arsenate is pumped off, and a cold soln. of  $ZnCl_2$  is poured in and left for 12 hrs. The  $ZnCl_2$  also penetrates the log and reacts with the Na arsenate to form  $Zn$  arsenate, which is insol. in water. In this form the As is poisonous to all wood-destructive organisms, but is not poisonous to the human body. J. W. Holst

Experiments on the preservation of mine timber.—Progress Report No. 1. J. Bryan and N. A. Richardson. *Dept. Sci. Ind. Research, Forest Products Research Reports* No. 3, 10 pp (1935).—Pross of *Pinus sylvestris*, of domestic and Scandinavian origin, in lots of 50 were treated by the open-tank hot- and cold-bath process, with Wolman salts (Trolith),  $NaF$ ,  $ZnCl_2$  and  $NaCl$  in 2 concns. each and with coal-tar creosote and placed in colliery pits in England

and Scotland. After exposure for 5 years, which is too short a time to arrive at definite conclusions, the following results are reported: untreated controls are practically all destroyed; creosote, Wolman salts and  $NaF$  are showing best protection;  $NaCl$  even in high concns. (10%) is the least effective. Nevertheless  $NaCl$  treatment will probably double the life of mine timbers. The lower concns. of Wolman salts and  $NaF$  (2%) are sufficient for satisfactory protection. Alfred L. Kammerer

The life of untreated and impregnated oak timbers. Willy Kimberg. *Lästy Cukrovár* 53, 483-4 (1935), *Zuckerind. Cechoslovak Rep.* 59, 471-2.—Untreated oak ties in Germany and France serve 8-12 yrs. In Czechoslovakia such ties began to decay after 2 yrs and had a useful life of 5-8 yrs. The necessity for using sapwood and inferior timber, the cracking at extremities and the collection of moisture in crevasses were responsible for the shorter life. Oak ties impregnated with a mixt. of  $ZnCl_2$  and a tar oil served 15 yrs and had to be removed because of mech. disintegration, on sections of the railroad where traffic was light, the life of the impregnated ties ranged from 20 to 25 yrs. Frank Maresh

Machines for moistening cement raw material with water (Brit. pat. 431,352) 1. Coating compns. [for concrete] (Brit. pat. 431,380) 13

Cement Lennart Forsén. Brit. 430,781, June 25, 1935. See Fr. 770,747 (C. A. 29, 38039).

Cements. Aktiebolaget Vallevikens Cementfabrik (to Georg S. Lahn). Brit. 431,060, July 1, 1935. A cement having a rapid initial set, e. g., within 12 sec., and a sufficiently slow hardening speed to avoid the formation of cracks by the evolved heat comprises a mixt. of aluminous cement and portland cement or other Ca silicate cement free from gypsum or other retarding agent. The cement may be made by mixing 2 kinds of clinkers, e. g., in proportion from 1-3 to 3-1, and grinding.

Portland cements. Société des ciments français et des portland de Boulogne-sur-Mer et compagnie des portland de Desvres. Brit. 431,090, July 1, 1935. White cement clinker is obtained by removing the clinker directly from the hottest zone in the kiln and cooling it in the absence of any gas contg. O. The cooling may be effected in an atm. of water gas, producer gas or  $H_2O$  vapor in a closed vessel or by quenching with  $H_2O$  or by allowing the clinker to cool in a heap in the air, the surface layer being removed in this case. App. is described. Cf. C. A. 29, 12264.

Preparing raw materials for cement manufacture. Mikael Vogel-Jørgensen. Brit. 430,430, June 19, 1935. The raw materials are passed direct from 1 or more wash mills, without intermediate grinding, to 1 or more sieve machines which disintegrate the material and sep. the fine from the coarse particles. Each sieve machine comprises a sieve which surrounds the rotor carrying blades that project outward from the rotor and force the material against the sieve, the shape and inclination of the blades and speed of the rotor being so coordinated that the blades present advancing wall surfaces which receive all the material and distribute it substantially evenly in the axial direction of the sieve. The fine particles may be passed direct to a cement kiln or to a container for the finished slurry or may first be passed through 1 or more further sieve machines. The coarse particles, after being passed, if desired, through 1 or more further sieve machines, may be returned to the wash mill or mills or may be passed to a grinding mill and thence either back to the sieve machine or machines or direct to the container for the finished slurry.

Apparatus for cooling cement clinker. Harry Swadner Lee. Ger. 618,251, Sept. 6, 1935 (Cl. 80c. 14 20). This corresponds to Brit. 401,392 (C. A. 28, 2494).

Rotary kiln and associated apparatus suitable for cement, limestone or ore treatment. Johan S. Fastang



(to F. L. Smith & Co.). U. S. 2,019,179, Oct. 29 1  
Structural and operative details.

Shaft furnace for burning cement, etc., fired with gaseous, liquid or pulverulent fuel. Ernst C. Loesche. Ger. 618,207, Sept. 5, 1935 (Cl. 80x 12)

Molding fibrous cement slabs in rolling-machines. Lina Schneider (see Sturm). Brit. 431,103, July 1, 1935. Light fibrous cement slabs are produced by passing a band formed by stitching together wood fibers, straw, reeds, etc., through a slurry of magnesite or portland cement, cutting off lengths and placing them in molds to set. App. is described.

Machines for coating tubes or similar articles internally with asphalt, varnishes, etc. Alfredo Vallini. Brit. 431,413, July 8, 1935.

Coating surfaces. The British Mannesmann Tube Co. Ltd. and Arthur J. Hodgkiss. Brit. 431,562, July 11, 1935. An Fe or steel pipe or tube, while heated and containing a quantity of fluid bitumen in the bottom, is maintained substantially horizontal and rotated slowly, e. g., by rollers to lap or spread the bitumen in successive layers into a uniform coating, the speed of rotation being insufficient to subject the bitumen to any appreciable centrifugal force. A small quantity of filler, e. g., slate dust, kieselguhr, may be added.

Concrete and hydraulic cement. Dewey & Almy Ltd. Brit. 431,679, July 8, 1935. This corresponds to Fr. 763,952 (C. A. 28, 5623).

Colored concrete. Dewey & Almy Ltd. Brit. 431,680, July 8, 1935. Addn. to 431,679 (preceding abstr.). See Fr. 44,218 (C. A. 29, 3131).

Light weight aggregate suitable for use in concrete. Carrell W. Bowyer. U. S. 2,017,889, Oct. 22. A light-weight aggregate is prepared by rapidly cooling molten slag, reheating it to near the m. p., and gradually recooling it.

Insertible vibrating device for use with concrete before setting. Enoch B. Jorgensen (to Vibro Co. Ltd.). U. S. 2,018,789, Oct. 29. Structural and mech. details.

Apparatus and method for molding articles from concrete, etc. Eugene Frevsnet. Brit. 431,484, July 9, 1935.

Road materials. Société de recherches et de perfectionnements industriels. Brit. 431,781, July 8, 1935. See Fr. 762,692 (C. A. 28, 5204). Powder pitch may be used in place of the powder bitumen.

Road materials. Soc. anon. des ciments portland de Rombas. Fr. 784,582, July 22, 1935. Blast-furnace slag, containing a high proportion of lime, which has been quickly cooled in air and finely ground is mixed with bituminous road materials.

Road surfacing compositions. Société de recherches et de perfectionnements industriels. Brit. 431,474, July 8, 1935. A compn. that can be spread by app. usually used for spreading ordinary road tar comprises a tar bitumen or tar-pitch mixt. containing 65-85% of bitumen or pitch, and a carbonaceous filler, preferably finely powdered coal, comprising 25-40% by vol. of the total compn. Cf. C. A. 29, 6590.

Road surfacing and other compositions. Wm. F. Rees and Wm. F. Rees Ltd. Brit. 430,979, June 28, 1935. In material for surfacing roads, roofs, etc., of the type consisting of particles of aggregate coated with matrix and then mixed with aggregate of smaller particle size, the matrix is sufficiently soft to be the equiv. of bitumen having a penetration greater than 350. The matrix may be tar, resinous or asphaltic materials or petroleum or rosin distill. residues and the aggregate may be limestone, granite, slag or clinker. In an example, "1/4 in. dust" limestone is put through a 20 mesh sieve, the material retained on the screen is heated to 150°F., treated with asphaltic or creosote oil and mixed with 400-penetration bitumen at 120-50°F., i. e., flowable, the fines from the screen then being mixed in the cold. The quantity of fines added equals that of the coarse aggregate and the bitumen used amounts to 6% of the total aggregate.

Molded artificial stone. Charles H. Hagopian. U. S. 2,018,355, Oct. 22. A soln. formed of water, MgSO<sub>4</sub>,

H<sub>2</sub>SO<sub>4</sub>, MgCl<sub>2</sub>, chrome alum and MgF<sub>2</sub> is used for prepreg a mult. of cream-like consistency with a mixt. formed from casein, talcum, calcined magnesite, borax and sand, and China wood oil is added just before pouring the final mixt. into a mold.

Artificial marble. Ernesto Revelant. Brit. 430,948, June 27, 1935. See Fr. 768,156 (C. A. 29, 574).

Cementitious building tile, etc. August H. Serton (to Superior Cement Corp.). U. S. 2,018,192, Oct. 22.

A fluid mixt. of cementitious material of a cream like consistency is placed in a sheet metal mold having resilient side walls and the mold is vibrated to dispel air from the mixt. and to give it a hard smooth dense surface where it comes into contact with the surfaces of the mold after setting and hardening.

Building blocks. Heinrich Frank. Swiss 173,321, June 17, 1935 (Cl. 85). These are made by molding or pressing a mixt. of shavings, (AcO), Ca, NaOH lye, cement and water.

Sheet material suitable for various structural uses. Albert C. Fischer (to Philip Carey Mfg. Co.). U. S. 2,019,449, Oct. 29. Compressible and expandable strips are formed by mixing a mass of ductile waterproofing binder with vegetable reinforcing elements of relatively large surface areas such as corn husks and finely divided fibers and forming the material into a sheet between belts.

Constructional material suitable for partitions or panels. Philip B. Brill and Geo. H. Ellis (to Insulite Co.). U. S. 2,018,911, Oct. 29. Insulating material or the like is formed with a core of pulped hemicellulose material on the opposite sides of which are plates of metal united with the core by a casein cementing compn., kerf's extending along at least 2 edges of the composite material.

Preparing exfoliated Jeffersonite suitable for use in wall plasters, etc. James Keeth (to Universal Insulation Co.). U. S. 2,017,904, Oct. 22. Exfoliated Jeffersonite is finely comminuted in water to form a self-binding pulp and this pulp is mixed with a filling pulp formed by finely comminuting a vegetable fiber such as wood fiber in water, the resulting plastic compn. is dehydrated, and is then hydrated preparatory to use.

Porous masses. Pietro Panzera and Carlo Pellegri. Swiss 175,596, May 16, 1935 (Cl. 85). Porous masses for building or industrial purposes are made by soaking vegetable fiber in a soln. of CaCl<sub>2</sub> and mixing with a cement with a Ca base.

Hard masses. Géza Farkas. Swiss 174,661, Apr. 16, 1935 (Cl. 41). A solid vegetable filling material, alum, luhopone, size, gypsum and a vegetable drying oil are mixed to form a hard mass for making wall or floor blocks or plates, etc.

Composition board suitable for use on walls and carrying an asphalt layer and differently colored grts. Robert S. Maclean (to Mastic Asphalt Corp.). U. S. 2,018,216, Oct. 22. Structural details.

Synthetic lumber. Charles E. Hartford (to National Cornstalk Processes, Inc.). U. S. 2,019,452, Oct. 29. Pulp such as that of cornstalks is treated with chem. solns. such as a wax emulsion and fireproofing material and alum and a thick layer of the treated pulp is passed through a forming machine and press where excess of the solns. is removed (various operative details being described).

Wood. Hermann Gamber-Eggmann. Swiss 175,411, May 16, 1935 (Cl. 80A). Wood is toughened by impregnating it with a soap and size soln. and drying. The soln. may be applied while boiling. The wood may be pretreated with a boiling soln. of NaCl.

Ague wood. Jean Grusard and Félix Baptist. Fr. 784,378, July 22, 1935. Wood is artificially aged by heating it gradually and progressively to 160-220° at which it is maintained till the color is sufficiently deepened.

Apparatus for drying wood, etc. Emil Raess. Swiss 176,698, July 1, 1935 (Cl. 80A).

Proofing wood against fungi. Imperial Chemical Industries Ltd., Thomas Callan and Stephen H. Oakeshott. Brit. 431,344, July 1, 1935. Cut timber and wood products generally are protected against mildew or other fun-

gold growths by spraying or brushing with, or immersion in, a suspension or emulsion obtained by dispersing in  $H_2O$  a soln. of an arylamide of salicylic acid in an org. solvent and an admitted dispersing agent compatible with the org. solvent. In example, salicylamide is dissolved,

resp., in  $\beta$ -ethoxyethyl alc., MeOH and pine oil, the soln. being mixed with Turkey-red oil and dild. with  $H_2O$  for use. Other known compatible wood-preserving agents, e. g., creosotes, cresylic acids, may be added to the concd. mixts.

## 21—FUELS, GAS, TAR AND COKE

A. C. FIELDNER AND ALDIN H. EMERY

Fuel problems in central heating establishments  
A. W. Granberg. *Tekn. Foren. Finland Förel. 55*, 187-91 (1935).—Descriptions are given of various central heating plants which use garbage as fuel. An av. analysis of garbage is: moisture 40, ash 30, paper 10, coal and coke 0.2, wood shavings 2, food refuse 3, bone 4 S, glass 4 G, metals 3-4 and miscellaneous 2%. The performance on 3 municipal plants is 1 kg. steam per kg. garbage.

Liquid fuels. I. O. Erkkö. *Suomen Kemistilehti* 8A, 103-5 (1935).—A survey of mfg. methods. E. B. J. Hydrogenation of liquid fuels. M. Stenart. *Industrie chimique* 22, 642-5 (1935).—A brief discussion of the problem from the tech. and economic standpoints.

Alcohol and alcohol gasoline blends as fuels for automobile engines. III. Performance tests of alcohol, gasoline and alcohol gasoline mixtures as fuels for an eight-cylinder automobile engine. A. L. Teodoro. *Philippine Agr.* 24, 352-87 (1935).—Up to 50% of 100° proof EtOH was miscible with gasoline at all temps. above 21°. 100° proof denatured alcohol was used for mixts. contg. more than 50% EtOH. Mixts. contg. as high as 20% EtOH were more economical than gasoline at full load.

A new technic and apparatus for testing Diesel fuels. T. B. Hertz and P. H. Schweitzer. *Automotive Ind.* 73, 2-5 (1935). A. L. Kaye

A new method for the rapid and exact determination of the hydrogen content of solid and liquid fuels. G. Lambiris. *Angew. Chem.* 49, 679-83 (1935).—The method consists of a combination of the calorimetric bomb combustion ( $O_2$ ) and the detn. of the formed  $H_2O$  by means of soly. data of the system alc.- $H_2O$ -petroleum hydrocarbons (phase sepn.). The total  $H_2O$  formed in the combustion is calcd. by the formula,  $x = (d - n) \times (W' - w) / (100 + W) + d$ , where  $d$  is the wt. of (alc. +  $H_2O$  + ppt.) obtained by washing the bomb,  $n$  the amt. of  $Na_2CO_3$  added to the bomb (0.3-0.4 g. of  $Na_2CO_3$  for 0.5-1 g. of sample),  $W$  the  $H_2O$  in the bomb detd. by the phase sepn. (exclusive of the 100 g. of original alc. used),  $w$  the  $H_2O$  which the alc. may have absorbed during storage, detd. by phase sepn. like  $W'$ ,  $d$  the  $H_2O$  released as vapor when the bomb is brought to atm. pressure, calcd. as  $d = g \times V_s \times \ln p_1 / (g \times \text{vapor density} + V_s \times \text{vol. of bomb} + p_1 \times \text{initial gas pressure})$ . Detailed directions are given. Test results on chemically pure and technically pure substances and other pertinent data are presented. The variations of analytical results were 0.04-0.09% of  $H_2$ , and 0.1-0.2% when alc. denatured with petroleum (100:1 by vol.) is used.

Microchemical analysis of solid fuels. W. R. Kirner. *Ind. Eng. Chem., Anal. Ed.* 7, 294-9 (1935); cf. C. A. 28, 634, 634<sup>1</sup>.—The adaptation of Pregl's micromethods to the analysis of solid fuels is discussed and modifications are suggested necessitated by the unique and chem. characteristics of solid fuels. The practicability of some of these modifications has been demonstrated in the Coal Research Lab., Pittsburgh, Pa.

Comparative carbonization tests with vertical retorts and vertical chamber ovens and the correlation of tests with Geipert apparatus with plant operation. H. Köhler. *Gas-u. Wasserfach* 78, 805-10 (1935).—Operating results secured with vertical retorts and vertical chamber ovens on the same coal are compared. In the latter, the mixed gas yield (coal gas plus water gas obtained on steaming retorts) is increased at the expense of the tar yields, and

a lower amt. of fuel is required for heating. The Geipert app. was used to det. the amt. of coke and gas compn. for various coals. The analysis of gas from the G. app. showed higher illuminant and H contents and less methane than gas from com. carbonizing equipment, because of higher rate of carbonization in the G. app. The yield of coke could be correlated with practice by adding about 1.5% to the test results. The G. app. coke had a higher C content and lower volatile content than in plant practice.

R. W. Ryan. Microscopic and petrographic studies of certain American coals. Reinhardt Thiessen and G. C. Sprunk. *Bur. Mines, Tech. Paper No. 564*, 71 pp (1935).—The petrography and microscopy of the following 6 coals are described and illustrated by 42 figures, largely photomicrographs: Green River bed, Green River mine, Muhlenberg Co., Ky.; Upper Ireport bed, Wildwood mine, Allegheny Co., Pa.; Pittsburgh bed, Consolidation No. 61 mine, Marion Co., W. Va.; Pratt bed, Wylam No. 8 mine, Jefferson Co., Ala.; Sewell bed, Summerlee mine, Lavette Co., W. Va.; and Sewell bed, Cranberry mine, Raleigh Co., W. Va.

Alden H. Emery. Classification and selection of Illinois coals. Gilbert H. Cady. *Ill. State Geol. Survey Bull. No. 62*, 354 pp (1935).

Indiana coals—their classification and analysis. F. B. Place. *Combustion* 7, No. 4, 27-9 (1935); cf. C. A. 29, 60191. Joseph H. Wells

Coal-sampling methods of the Detroit Edison Company. A. W. Thorson. *Combustion* 7, No. 4, 10-14 (1935).—Automatic devices for sampling coal from bucket and belt conveyors are described. A special design of rifle box has been developed to eliminate clogging with wet coal.

Joseph H. Wells. The economics of water treatment in the colliery industry. F. Hamer and S. Stevens. *Colliery Guardian* 151, 617-20 (1935).

E. H. Carbonization of coal. Effects of variation of rate of heating during the carbonization of a typical coking coal. Wm. B. Warren. *Ind. Eng. Chem.* 27, 1330-4 (1935); cf. C. A. 29, 1229<sup>9</sup>.—Three series of carbonizations were made on Edenborn coal to a max. temp. of 700° in which 2 rates of heating—1.4° and 21.8° per min.—were used in several combinations. The thermal history up to and after the plastic range is important since no direct correlation could be made between the effects found and the rate of heating through the plastic range alone. The vol. of gas is const. for the 2 rates of heating in the preplastic range but the H content is higher for the slower rate. Coke hardness is detd. largely by the rate of heating through the plastic range, being greater for the slower rate of heating.

Joseph H. Wells. Coal carbonization—the plastic stage. James H. Lum and Harry A. Curtis. *Ind. Eng. Chem., Anal. Ed.* 7, 327-33 (1935).—Correlation of the gas pressure-temp. curve of the Foxwell procedure with the condition of the coal during carbonization was made and data on plasticity were obtained by another method. Variables affecting the results were assumed to be rate of gas flow through the charge, packing of the coal in the fusion tube, length of the coal charge, screen analysis of the coal used and the rate of heating the charge. Gas flow of 6 cc. per min. per sq. cm. cross section of the fusion tube gave reproducible results. Irregularities due to packing the charge were readily obviated and a length of 2.5 cm. of charge was found satisfactory. Wide variations in results were noticed depending on the screen analysis and these variations

were not removed by grinding, briquetting and regrinding fairly consistent values for the temps of initial softening and max gas pressure could be obtained by grinding to pass 10 mesh and keeping the amt passing 80-mesh below 35%. A rate of 2° per min in heating was found satisfactory. The max gas pressure varied in successive runs and no significance was found to attach to the slope of the gas pressure-temp curve as suggested by Layne (C. A. 19, 381, 21, 3117, 22, 859). No agreement was found between the height of the gas pressure temp curve as a measure of the degree of plasticity reached by a coal during carbonization and the degree of plasticity as indicated by the amt of swelling observed when small briquets of the coal were carbonized. The rate at which coal in the plastic condition and under pressure passes through a small orifice was detd and a correlation was found to exist between this rate, the temp of initial softening, and the temp of max gas flow/pressure. J. W. Shipley

Hydrogenation of coal V Sihvonen Suomen Kemistilehti 8A, 76-83 (1935).—Some of Germany's achievements in the utilization of lignite as a raw material in the chem industries are described. The chem reactions which appear in the manuf of water gas are explained by the combustion theory of S. E. B. Jukkola

Note on the determination of nitrogen in coal P. I. Hall Chem. Abstr. Mining Soc. S. Africa 30, 28-31 (1935).—Brett's modifications (cf C. A. 26, 6098, 29, 4154) of the Kjeldahl method for estn of N in coal and cokes are reviewed and further modifications in reagents and procedures are suggested. The method is accurate, quick and money saving. Alden H. Emery

Action of nitric acid on a bituminous coal B. Juetner, R. C. Smith and H. C. Howard J. Am. Chem. Soc. 57, 2322-26 (1935).—The action of  $NHNO_3$  in converting bituminous coal into sol products has been shown to be chiefly an oxidation. The yield of humic acids reaches a max in a few days, while that of the sol acids increases slowly with time. If the reaction is carried out in air, only about 25% as much  $HNO_3$  is consumed as in its absence. The residue from the  $CaH_2$  pressure extrn of this coal oxidizes more rapidly than the original coal. Oxidation of the cokes obtained by thermal decompn of the coal in vacuum shows no difference from the original coal with cokes up to about 440°. Cokes prepd above this temp show markedly greater resistance to oxidation. This temp coincides with that at which gas evolution becomes rapid. Details of the ultimate compn and functional groups on both types of acids formed in this oxidation show the regenerated humic acids contain markedly less HO than is reported for other humic acids, while the sol acids contain negligible amts. In the humic acids only approx half the  $O_2$  detd by ultimate analysis can be accounted for in functional groups, while in the sol acids only small amts of  $O_2$  are thus unaccounted for. The presence in the sol acids of  $C_6H(CO_2H)$  and  $C_8(CO_2H)$  was shown by isolation of their esters. Of the C of the coal which was oxidized to sol acids, approx 10% was recovered by decarboxylation as steam-volatile, aromatic hydrocarbons. The presence of acids more complex than the benzene carboxylics is indicated. C. J. W.

Volatile matter of Pennsylvania anthracite H. G. Turner and W. L. Keene Ind Eng Chem 27, 1373-6 (1935).—The compn of the volatile matter of Pennsylvania anthracite was detd on 36 samples considered to be representative of the area. The proximate and ultimate analyses of the coals are given. The samples were heated at temps ranging from 21° to 1036° in a nichrome tube connected to 12-15 l of space evacuated to less than 1 mm pressure. With increase in the volatile matter from 3.8% in the low-volatile group to 7.6 in the high volatile group, the gas yield increased from 5000 to 7200 cu ft per ton. The percentage of  $H_2$  is const for all the coals, and averages 83%. No illuminants were detected.

P. J. Wilson, Jr  
Control of forms of iron in the determination of fusion temperatures of coal ash W. T. Reid Ind Eng Chem, Anal Ed 7, 335-8 (1935), cf C. A. 27, 178.—The effect of varying percentages of ferrous and ferric iron on

the viscosity of slags was detd in a furnace operating in an atm of N. The standard of viscosity was chosen as 10 poises compared with castor oil and was detd by estg the fluidity by the ease of movement of a Pt rod inserted in the molten slag. The lowest temp at which no resistance is felt to vertical movement of the rod, horizontal movement is but slightly impeded and the slag flows immediately into the trough left by the passing rod, is taken as the flow temp of the slag. Flow temps were found to rise with the percentage of ferric iron present. With the iron about 25% in the ferric condition the flow temp was 1255°, while with about 64% ferric iron the flow temp was 1400°. J. W. Shipley

Gasoline from coal C. H. S. Tophole Ind Eng Chem, News Ed 13, 414-15 (1935). E. J. C.

Humic acids II. Walter Scheele, Werner Schulze and Hans Spandau Kolloid-Z 73, 84-90 (1935); cf C. A. 29, 7998.—Humic acid was isolated from 2 lignites mined in central Germany. After successive 8-hr extrns with  $CaH_2$  and  $Me_2CO$ , the humic acid was leached from the lignite by 14-day digestion with  $N NaOH$ ; the clear Na humate soln was then acidified with HCl and the insol humic acid sepd by centrifuging; the ppt was redissolved in aq  $NaOH$ , the soln filtered and the humic acid reprecipd with HCl; the humic acid was washed thoroughly, first with HCl soln and then with  $H_2O$ , and dried over  $P_2O_5$  in a vacuum oven at 80°. Both samples of humic acid were practically insol in cold aq. KF soln, contained no more than traces of alc-sol matter (hymatomelanic acid), and had an equiv wt of about 190. Boiling an aq. Na humate soln contg excess  $NaOH$  causes decompn with the production of acidic substances sol in aq. acidic solns. The decompn is not an autoxidation because the same result is obtained whether  $N_2$  or  $O_2$  is bubbled through the soln. This decompn also takes place at room temp, but requires a much larger excess of  $NaOH$ . Oscar T. Qumby

The structure of humic acid and its relation to lignin and coal I. Sedletskii and B. Brunovskii Kolloid-Z 73, 90-1 (1935).—X-ray diagrams for lignin, humic acid, lignite, anthracite and graphite suggest a close structural genetic relation between them. The d of these substances increases in the order given. Oscar T. Qumby

Practical solution to Salt Lake Valley smoke nuisance S. Clark Jacobson and George W. Carter Ind Eng Chem 27, 1278-83 (1935); cf C. A. 29, 4545.—The Salt Lake City area has a higher rate of soot fall than any other locality studied so far; it is due primarily to the use of bituminous coal in houses and small plants. Processing such coals by high-temp distn has not solved the problem, but when low-temp distn (300-750°) under pressure was used, secondary cracking of volatile products formed being used, the resulting C is not graphitic, but burns readily. Dust-free coals were treated in batch furnaces, at 538-740°, and surrounded by steam, in retorts as shown. Data on test runs, results obtained upon using such fuels in household heating devices and the marked improvements in smoke elimination are presented. W. C. Ebaugh

Manchester and District Junior Association Pres. dental address A. K. Colledge Gas J 212, 201-4 (1935).—Operating methods for reducing the costs in gas production are discussed. P. J. Wilson, Jr

Natural gas in 1933 R. B. Harkness. Ann. Rep. Ontario Dept. Mines 43, Pt. 5, 1-51 (1935). A. H. E.

The use of gas for industrial purposes F. L. Atkin Gas World 103, No. 2672 Ind. Gas Supply, 89-6 (1935). Gas J 212, 259-62.—Small, gas-fired ovens are briefly described. Heated air is continuously recirculated at a rate 50 to 100 times that of natural convection.

P. J. Wilson, Jr  
The chemist and works control Arthur J. Doran Gas World 103, 339-42 (1935); Gas J 212, 205-7.—The gas-plant chemist's work is outlined. P. J. Wilson, Jr

Fifty years of the Auer gas mantle, changes and developments Karl Quasebaart Gas u Wasserfach 78, 715-9 (Oct 12, 1935).—The discovery and development of the incandescent gas mantle are reviewed. R. W. Ryan  
The flash system of carbureting [gas] Threlk

Nagel. *Gas J.* 211, 704(1935); cf. *C. A.* 29, 6398<sup>1</sup>, 7016<sup>1</sup>. P. J. Wilson, Jr.

Purification of manufactured gas with active carbon. Kurt Blume. *Gas-u. Wasserfach* 78, 783-9, 810-4(1935).

—The theory of the adsorption of vapors on active C is reviewed, with diagrams. Active C adsorbs little water as compared with silica gel and will take benzene out of a benzene-water mixt., while silica gel removes the water. For this reason, the heat of adsorption is greater with silica gel when removing vapors from gas contg. water vapor. Adsorption isotherms are given for  $\text{CCl}_4$ , benzene, acetone and ether, on active C. In absorbing vapors from manifold gas, the active C first becomes sat'd with respect to  $\text{CS}_2$  and unsat'd hydrocarbons, and these then escape. The benzene satn. point comes much later. Consequently to secure quant. removal of  $\text{CS}_2$ , unsat'd hydrocarbons, etc., it is necessary to stop at about  $\frac{1}{2}$  of the benzene satn. point. The active C plant for recovering light oils at Bayreuth is described. Because of varying make, the rate of gas passage through the adsorbers varies widely, but uniformly good results are secured. The water condensate produced on steaming out the adsorbers has a very disagreeable odor due to cyanogen compds. Special valves on adsorber cooling and heating coils were required, because of very great temp. differentials. Steam requirements per gallon of light oil were approx. 3.8 cu. ft., while cooling water requirements were about 22 lb. of steam per hr., and labor requirements about  $\frac{1}{4}$  hr. for each steaming out period (the av. adsorption period was 3-4 hrs. for each adsorber). Based on the recovery of approx. 85 tons of light oil per year, operating costs were approx. \$18.70 per net ton (exchange at par). Capital charges, etc., increased this to approx. \$10.40 per net ton of light oil. When operating plant at  $\frac{1}{2}$  of the light oil satn. point so as to secure greater gas purification, costs were about 60% greater. The quality of the light oil recovered depends more on the type of carbonizing plant than the recovery system. However, the light oil must be redist'd. after addn. of Fe chloride soln. to remove low-boiling S compds.—thus removing yellow color and bad odor. Complete removal of naphthalene from gas by this process has caused a decrease in pressure drop in mains, but iron rust released on removal of naphthalene has collected at pockets or sharp turns in services, causing stoppages.

R. W. Ryan  
A new modification of the circular manifold type of gas-analysis apparatus. Minor C. K. Jones. *Am. Gas J.* 143, No. 4, 27-9, 110(1935).—The app. has been improved by the incorporation of the Huff pumping pipet.

Joseph H. Wells  
An electric gas generator. Ivar Hole. *Gas J.* 212, 271(1935).—See *C. A.* 29, 6733<sup>1</sup>. P. J. Wilson, Jr.

Flow of natural gas through high-pressure transmission lines. T. W. Johnson and W. B. Bierwald. *Bur. Mines Monograph No. 6*, 120 pp (1935).—A fundamental study.

Alden H. Emery  
The latest form of flexible pipe main for gas. P. H. Wilson. *Gas J.* 212, 256-8(1935); *Gas World* 103, 308-9. —The Stanton-Wilson self-adjusting joint for cast-iron pipe consists of 2 loose parts, a cast-iron locking ring and a joint ring of special rubber. The latter is special rubber, V-shaped in section, and provided with a Pb-sheath.

The locking ring is first placed on the spigot, the joint ring is put in front of it, and the end of the spigot is inserted in the bell. By rotating the locking ring, lugs on it engage with lugs on the bell.

P. J. Wilson, Jr.  
Benzene in natural gas. H. H. Schrenk, W. P. Vaut and S. J. Pearce. *Bur. Mines, Rept. of Investigations No. 3293*, 2 pp (1935).—The dry natural gas supplied to the Pittsburgh station of the Bur. of Mines contained 2.01 and 2.98 p. p. m. by vol. of  $\text{C}_6\text{H}_6$ ; a wet gas from a private well near Pittsburgh analyzed 17.5 and 17.9 p. p. m.

Alden H. Emery  
The age of the oil used in benzene recovery. G. C. Rico Avello. *Anales soc. esp. fis. quim.* 33, 713-20 (1935).—Wash oil, used for the recovery of  $\text{C}_6\text{H}_6$  from coke-oven gas, undergoes increase in viscosity with age,

which reduces markedly its absorptive properties. The reason for this is  $\text{C}_{12}\text{H}_6$  formation from the oil, a process insufficiently investigated. The effective, washed  $\text{C}_{12}\text{H}_6$  is always less than the recovered  $\text{C}_{12}\text{H}_6$ . P. M. Symmes.

Removal of nitric oxide from manufactured gas by dry purification material. K. Hunte, H. Brückner and G. R. Haas. *Gas-u. Wasserfach* 78, 751-7(1935).—In the course of dry purification a considerable decrease in the NO content of gas has been noted, and this has been attributed to formation of Roussin's salt ( $\text{R. S.}$ ) or nitroprusside compds. The black Roussin's salt,  $\text{M}_3\text{Fe}(\text{NO})_2\text{S}_2$ , is more stable than the red salt, and should be the one formed, but attempts to identify this salt by extg. spent purifying material with water, filtering, and extg. the filtrate with ether, failed to give the characteristic ether color. No nitroprusside could be found in the spent purifying material. By means of lab. expts. it was found that FeS was necessary for the formation of  $\text{R. S.}$ , and that FeS would not yield  $\text{R. S.}$  in ordinary dry purifier operation. O is added to ensure continuous reinvigoration, and this prevents  $\text{R. S.}$  formation as insufficient FeS is available. Under these conditions NO absorbed to a slight extent may be evolved as  $\text{NO}_2$ . In the absence of O, an  $\text{NH}_3$  odor has been noted after NO absorption. This is attributed to the following reactions:  $2\text{NO} + 6\text{H}_2\text{O} + 21\text{FeS} = 2\text{NH}_3 + 2\text{SO}_2 + 21\text{Fe}(\text{OH})_2$ ; and  $3\text{SO}_2 + 4\text{FeS} + 6\text{H}_2\text{O} = 7\text{S} + 4\text{Fe}(\text{OH})_2$ . In practice NO and  $\text{NO}_2$  may be removed almost quantitatively by using a small box of sulfided purifying material before the usual box series, and admitting O after this box. R. W. Ryan.

The recovery of sulfur from coal gas. O. W. Roskill. *Gas World* 103, 330(1935).—The present S capacity of Thylor process plants in Germany amounts to 6500 tons annually. Developments in S recovery from dry purification are discussed.

P. J. Wilson, Jr.  
Benzene synthesis from carbon monoxide and hydrogen under ordinary pressure. XXVI. Gas contraction and benzene synthesis. Shunzo Tsunokawa and Yoshio Murata. *Sci. Papers Inst. Phys. Chem. Research (Tokyo)* 28, 48-50(1935); cf. *C. A.* 29, 4551<sup>1</sup>.—No relation appears to exist between the total gas contraction and yield of benzene.

W. J. Peterson  
Free carbon in coal tar. I. rich Bierling. *Kolloid-Z.* 72, 92, 100(1935).—A crude coal tar from Knurow was dist'd. in the range 180° to 412° and the tar fractions were tested for free C (Berl and Schildwächter, *C. A.* 22, 4409), viscosity and oil, tar and coke content. Viscosity decreased with increase in free C. The latter existed in the coal tar in every state of subdivision from mol. disperse through colloidal to coarse and fully unsolvated. Heating the coal tar under pressure in the range 200° to 425° coagulated the C particles as shown by the viscosity curve and by no count. Plasticity of the pitch was decreased because of the coagulation. In distn. of coal tar a condensation of the dispersed C particles takes place along with a possible small cracking.

P. S. Roller  
Coke Oven Manager's Association. Presidential address. T. Westthorp. *Gas J.* 212, 316-18(1935); *Gas World* 103, No. 2074 Coking Sect., 109-11.—Research problems in the coke and gas industries are outlined.

P. J. Wilson, Jr.  
Process of formation of coke. M. Chorazy and J. Chmielewski. *Przemysł Chem.* 19, 113-22(1935).—Permeability of semicokes and cokes secured from coal at temp. from 450° to 1000° was investigated by a method worked out by the Coal Department of the Polish Chem. Research Inst. The app. is described briefly. Decidedly different permeabilities of cokes may be shown depending on the coal from which they are derived as well as the temp. at which they were obtained. The permeability of coke obtained at high temp. from coking coals rises to a max. value while that for cokes obtained at various temps. from gas-flaming caking coals possesses 2 distinct maxima corresponding with those of enhanced degassing of coal. Depending on the rate of heating not only different values of permeability of a given coal may be obtained but even the character of the permeability curve may be altered. The fundamental shape of the temp.-permeability curve

is not changed by varying the grain size of coke, but larger grains are likely to increase the permeability values at the given temps. The addn of bituminous substances leads to increased permeability of semicokes and cokes without changing the character of the curves, but the addn of debittuminized materials (semicoke, *pit-head coal slack*, and hard coals) not only increase permeability generally but also shift the temp. of max. permeability. When mixed with *nitra*, *durain* produces an increase in permeability but *fusain* has no significant effect.

A. C. Zachlin

Geological aspects of research on coal (Hickling) 8  
Purifying H for the hydrogenation of carbonaceous material (Brit pat 431,970) 18

Volatile combustible liquid Cruma soc anon Swiss 175,341, May 1, 1935 (Cl 366) The liquid, contg. ether, is obtained by first heating EtOH in the presence of a catalyst consisting of a metal oxide, and then heating the product with a catalyst consisting of a metal salt. Examples of the first catalyst are,  $Al_2O_3$ ,  $Fe_2O_3$  and  $Cr_2O_3$ , and of the second catalyst are,  $K_2S_2O_8$ ,  $(NH_4)_2SO_4$ ,  $MgCl_2$ ,  $CuCl_2$ ,  $CaSO_4$ , etc.

Elongated closed tank for containing liquefied gases such as liquefied propane for use as fuel. Paul S. Endacott (to Phillips Petroleum Co.). U. S. 2,019,004, Oct. 29. Various structural details.

Controlling furnace combustion of fuels such as fuel oil Ernest O. Scheerer. U. S. 2,018,925, Oct. 29. Various details of app. and operation.

Distilling solid carbonaceous materials Hastings J. Holford Brit. 431,397, July 5, 1935. Coal, peat, lignite, shale, *cannel coal*, etc., are carbonized in thin layers on shelves, the heating means comprising direct contact with superheated steam and a body of heated refractory material in proximity to the distn zone. App. is described.

Distilling and gasifying solid carbonaceous materials Jean Lefevre Brit. 431,896, July 17, 1935. Coal, etc., is fed from a hopper into the space in a preheated container above incandescent coke, the layer of fuel being distd. by heat radiated from the preheated walls of the container and from the coke. App. is described.

Retorts Low Temperature Carbonisation Ltd and Whiston A. Bristow. Brit. 431,772, July 15, 1935. Vertical or inclined retorts of narrow cross section are provided with top and bottom fittings whereby either containers enclosing the charge or the charge in a loose condition may be fed to and discharged from the retorts.

Retorts for the heat treatment of solid materials Samuel Moore Brit. 430,969, June 27, 1935. Coal, shale, peat, etc., or semisolid substances are fed by a screw conveyor into a retort between the upper and lower flights of a conveyor between the blades of which the material is propelled in sep. charges through the retort floor.

Destructive hydrogenation I. G. Farbenindustrie A.-G. (to International Hydrogenation Patents Co. Ltd) Brit. 431,435, July 8, 1935. See Fr 777,119 (C. A. 29, 3812).

Destructive hydrogenation I. G. Farbenindustrie A.-G. Brit. 431,683, July 9, 1935. In obtaining low-boiling aromatic hydrocarbons from high-boiling hydrocarbon liquids consisting of or contg. aromatic hydrocarbons and derived from mineral coal, e.g., coal-tar fractions, heavy benzene obtained by destructive hydrogenation of mineral coal, the liquids are first refined by a heat-treatment in presence of H under conditions to remove S and O compds and to hydrogenate some of the double bonds without decomposing into lower mol compds. The treatment is effected at above atm. pressure, e.g., 20 atm., and at 100-450° in presence of S-mimic catalysts. The liquid may be heated to about 200°, passed into a separator from which polymerized compds. are removed, and the remaining product led with H over a heated catalyst, e.g., at 100-300° under pressures up to 500 atm. over a hydrogenation catalyst consisting of oxides or sulfides of the 5th and 6th groups. The refined product is led with H under

100-500 atm. and at 500-700° over catalysts such as compds. of the metals of the 2nd to 8th groups.

Destructive hydrogenation. International Hydrogenation Patents Co. Ltd. Brit. 431,795, July 16, 1935. In a multi-stage process of destructive hydrogenation in which the carbonaceous materials are treated initially in the liquid phase and finally in the vapor phase, the products from the 1st stage are sep'd. in a catch-pot maintained under such conditions that no condensation takes place, i.e., at less than 20° below the temp. of the reaction vessel, and the vapors are passed, without condensation, to the vapor-phase stage. App. is described.

Destructive hydrogenation of coals, tars, mineral oils etc. I. G. Farbenind. A.-G. (Mathias Pier and Eurus Anthes, inventors). Ger. 618,315, Sept. 12, 1935 (Cl. 120.1.05). Use is made of a circulatory system around which the hydrogenating gases, and vapors produced in the process, are caused to pass without any substantial reduction in temp. Vapors are withdrawn from a point in the cycle, continuously or periodically. As an alternative, the cycle may include means whereby the vapors are modified, e.g., a catalytic treatment zone assoc. with means for varying the temp. App. is described.

Carbonizing processes Harold P. Stephenson Brit. 431,063, July 1, 1935. In the low-temp. carbonization of coal to yield a semi-coke and distillates, the powder coal is mixed with a heavy liquid hydrocarbon, e.g., tar, fuel oil and a catalyst and pre-heated under pressure before being passed by hydrostatic pressure through zones of increasing temp. A pressure of 750 lb per sq in. and a temp. of 300° is maintained in the preheater and 500-600° in the retort. App. is described.

Carbonizing coal in an externally heated intermittent carbonizing retort chamber Wilhelm Fitz and Joseph Daniels. U. S. 2,018,664, Oct. 29. Various details of app. and operation are described.

Apparatus for washing coal Simon-Carves Ltd and James Robson. Brit. 431,865, July 17, 1935. A discharge valve of a washer box is controlled by the upward and downward movement of a float which is completely submerged in the material being stratified, means independent of the discharge-valve operating mechanism being provided to prevent the float from emerging from the bed of materials.

Shaking sieve device for dewatering fine coal Fred Krupp Grusonwerk A.-G. Ger. 618,152, Sept. 2, 1935 (Cl. 1a.17).

Air filter suitable for engine intakes Howard F. Weiss (to C. F. Burgess Laboratories, Inc.). U. S. 2,019,241, Oct. 29. Intertangled, elongated wood strands are bonded together by an adhesive such as a casein or other glue which forms a discontinuous coating on the strands.

Drier for steam G. Hochstrasser & Sohn Swiss 176,728, July 1, 1935 (Cl. 107d).

Fuel gas Metallgesellschaft A.-G. (Otto Hubmann, inventor). Ger. 616,595, Aug. 3, 1935 (Cl. 12a.1.05). Addn. to 592,223 (C. A. 28, 2881). The process of Ger. 592,223 is modified by causing the gas produced to exert a hydrogenating action on the heated solid fuel before the latter is subjected to the gas-producing reaction. App. is described.

Fuel gas Metallgesellschaft A.-G. Ger. 616,580, Aug. 9, 1935 (Cl. 24e.2.05). Addn. to 592,223 (C. A. 28, 2881). Pulverulent or finely granular fuel, kept in suspension by the gasifying agent, is used in the process of Ger. 592,223. App. is described.

Purifying gases Studien-und Verwertungs-G. m. b. H. Fr. 784,357, July 22, 1935. Compds. of S are removed from gases contg. CO and H<sub>2</sub> by using alk. carbonates in amt. of at least 5% in intimate mixt. with oxides or hydrosides of Fe capable of reacting at temps. below that at which accessory reactions with CO would take place (320°).

Purifying gas such as a natural gas Thomas S. Bacon (to Lone Star Gas Co.). U. S. 2,019,468, Oct. 29. Gas contg. H<sub>2</sub>S as an impurity is brought into contact under superatm. pressure, in the presence of O, with an alkali, soln. contg. a complex of metal compds. formed by

mixing a soln. of a mixt. of Zn and Fe salts or the like with an aq. alkali soln. App. is described.

**Gas-scrubbing processes.** W. C. Holmes and Co. Ltd., Charles Cooper and Daniel M. Henshaw. Brit. 430,980, June 27, 1935. All the volatile hydrocarbons such as  $C_2H_4$  and a substantial proportion of  $CS_2$  are removed from combustible gases by scrubbing with a vol. of wash oil, unsatd. with respect to  $CaH_2$ , of the order of 50 gallons per 1000 cu. ft., and a proportion of the hydrocarbons is afterward returned to the gases. The used wash oil may be only partly stripped and returned into contact with the gases while contg. a substantial proportion of the absorbed substances, or some of the  $C_2H_4$  recovered by a complete stripping is revaporized into the scrubbed gas. App. is described.

**Gas producers, gaseous mixtures.** Metallgesellschaft A.-G. Brit. 431,030, June 28, 1935. Addn to 364,407 (C. A. 27, 2020).—In a process as described in 364,407 for obtaining a combustible gas rich in H and  $CH_4$ , and in which solid fuel is gasified under high pressure with O, or air admixed with O, and steam, air and steam are used and the CO and part of the N are removed from the resulting gas in a sep. plant. The air is compressed to 20–30 atm., but pressures up to 200 atm. may be used, the higher the pressure the lower the N content of the gas, and steam is mixed with the air (1/6), the mixt. being superheated to about 500°.  $CO_2$  and  $H_2S$  are removed by washing with  $H_2O$  under pressure and by treatment with NaOH and the CO and part of the N are removed by cooling,  $CH_4$  and other gaseous hydrocarbons together with H and N being left. The residual CO and N may be used for superheating the gasifying medium. The CO may be converted into  $CH_4$  or H catalytically prior to its sepn from the mixed gases. The sep. CO may be converted into  $CH_4$ , and (or) H which is mixed with the gas before or after it is treated in the sep. plant.

**Gas producers.** I. G. Farbenindustrie A.-G. Brit. 431,480, July 9, 1935. A gas suitable for illuminating gas is obtained by supplying gases such as waste gases from low-temp. carbonization or oil-cracking plants, fuel-distn. gases, natural gas or gases from the destructive hydrogenation of coal tars or mineral oils, to a gas producer, such as described in Brit. 214,544 (C. A. 18, 2802), in which the incandescence fine-grained fuel is kept in movement similar to that of a boiling liquid. These gases may be mixed with steam or  $CO_2$ , and the gas, rich in O for maintaining combustion, may also have an addn of steam or  $CO_2$ . The resulting gas contains at least 12 and preferably 20–30%  $CH_4$ .

**Gas producers.** Hermann Kahlbetrzer. Brit. 431,635 July 11, 1935. Fuel such as wood chips, sawdust or maize cobs is used in a producer having an outer shell enclosing an inner shell open at both ends and attached to the outer shell by a trough which receives liquid condensates from a domed top.

**Spurially-guided gas holders.** Geo. F. H. Beard, Jr. Brit. 431,086, July 1, 1935. Two carriers, which are pivoted to a carriage, each support 1 guide roller bearing on the upper surface and 1 guide roller bearing on the lower surface of the guide rail, the axle of 1 upper surface guide roller of each pair being connected by an approx. vertical link to the axle of the lower surface guide roller of the other pair.

**Sealing strip for waterless gas holders.** Friedrich Schürmann (to Stacey Bros. Gas Construction Co.). U. S. 2,019,273, Oct. 29. Structural details.

**Ammonia recovery from hot distillation gases.** Matthew J. Miller (to Smet-Solvay Engineering Corp.). U. S. 2,018,893, Oct. 29. Hot coal-distn. gases are partially cooled to obtain an aq. liquid contg. a portion of the  $NH_3$  originally present in the gases and a lesser portion of  $CO_2$  and  $H_2S$ , the liquid is subjected to a distn. treatment to remove the  $NH_3$ ,  $CO_2$  and  $H_2S$  as gases, the gases are washed with a quantity of water sufficient to remove substantially all the  $CO_2$  and  $H_2S$  but insufficient to dissolve all of the  $NH_3$ , the wash water is sep. from undissolved  $NH_3$ , heated to expel dissolved gases including  $NH_3$ , and the expelled gases are brought into contact with acid to fix the  $NH_3$ . App. is described.

**Dehydrating and distilling tar.** Stuart P. Müller (to Barrett Co.). U. S. 2,018,377, Oct. 22. Fresh hot coal-distn. gases are cooled to sep. tar from them; the cooled gases are reheated and are passed into direct contact with tar contg. water to heat the tar and vaporize the water, and resulting gases are admixed with fresh coal-distn. gases, and the admixed gases are then passed to a light-oil absorber for conjoint recovery of light oil from the coal-distn. gases and any light oil distd. from the tar during its dehydration. An arrangement of app. is described.

**Coking processes.** Société générale de fours à coke, systèmes Lécocq, Soc. anon. Brit. 431,825, July 16, 1935. In the production of semi-coke, the coke oven is heated in 2 stages, in the 1st of which the charge is heated so as rapidly to attain a temp. of about 700° in the material adjacent to the walls of the coking chamber and in the 2nd the heating is reduced so that no substantial amt. of the material exceeds 600°. The oven walls are heated by convection and radiation during the 1st stage and by radiation only in the 2nd stage. App. is described.

**Coking processes.** Hinselmann, Koksofenbaugesellschaft m. b. H. Brit. 432,390, July 25, 1935. To improve the yield of by-products, e. g.,  $CaH_2$ , from coke ovens, the distillates, during the 1st portion of the heating period, are led off by a relatively long path through the oven chamber and (or) through the hot brickwork and during the latter part of the heating period are removed by a shorter path to avoid overheating. App. is described.

**Plant for quenching glowing coke.** Buss A.-G. Swiss 175,289, July 1, 1935 (Cl. 12a).

**Coke ovens.** Clemens Delkeskamp. Brit. 431,496, July 9, 1935. In a retort bench for the distn. of fuel briquets comprising a no. of open-topped narrow retorts arranged in a ring and provided with a common discharging device, each of the retorts is substantially quadrilateral in cross section and has 2 of its sides externally heated by flues. In 431,500, July 9, 1935, divided up and added, to 431,496, the vertical retort bench is provided with a distributor element rotated continuously about the vertical axis of the bench and delivering into an annular feed shaft, which is maintained substantially full of charge material so that the briquets slide gently thereto without free fall.

**Coke ovens.** Joseph Becker (to The Koppers Co. of Delaware). Brit. 432,348, July 25, 1935. In a vertically-flued coke oven, recirculation of a portion of the combustion products is effected by connecting a down-cast flue in 1 heating wall to an up-cast flue in an adjacent wall through a duct passing beneath the intervening oven.

**Vertically-flued coke ovens of the cross-over type.** The Koppers Co. of Delaware. Brit. 432,345, July 22, 1935, 432,361, July 25, 1935 and 432,413, July 22, 1935, divided on 432,345.

## 22—PETROLEUM, LUBRICANTS, ASPHALT AND WOOD PRODUCTS

W. F. FARAGHER

**Petroleum in 1933.** R. B. Harkness. Ann. Rept. Ontario Dept. Mines 43, Pt. 5, 52–6(1935). A. H. E. Efficiency of petroleum-fractionating columns. C. W. Cannon and R. L. Huntington. Refiner & Natural Gasoline Mfr. 14, 18–20, 60–2, 424–5, 490–3(1935).—The subject is discussed under the headings of (1) capacity and

efficiency of lab.-packed column; (2) efficiency of a com. bubble tower; (3) a study of lab. packed columns; and (4) efficiency of different sections of a com. bubble-plate column.

J. L. E. Critical solution temperature of mineral oils. E. Velinger and J. D. Herrenschildt. Compt. rend. 201,

780-2(1935).—With a solvent consisting of  $\text{COMe}_2$  + 1 AmOAc the crit soln temp (T C D) of a mixt of equal parts of mineral oil and solvent decreases linearly with percentage of AmOAc. With equal amts of solvent consisting of 9COMe<sub>2</sub> + 1AmOAc, the T. C. D. of slightly refined oils and those of av. degree of refining did not vary after 25 months in a transformer, but that of a highly refined oil decreased rapidly after 10 months of use (cf Müller, C A 29, 525'5). The T. C. D. of a crude Columbia oil rose with amt (to about 9%) of  $\text{H}_2\text{SO}_4$  used in refining, and then remained approx stationary. Plotted against percentages of superrefined oils in the same solvent the T. C. D. for all percentages of 4 (Pennsylvania, Russia, Venezuela and Texas) showed very slight decreases in this order, the T. C. D. of a new oil of varying degrees of refinement showed marked increase with increased refinement. C A Silberrad

Analysis of acid sludge from mineral oil treatment. E Holzman and Stefan Suknarowski. *Ind. Eng. Chem., Anal. Ed.* 7, 378-80(1935).—A method of analysis which can be applied to all acid sludge, from both paraffin base and naphthene-base oils treated with dil. concd, or fuming  $\text{H}_2\text{SO}_4$ , in which the sepn and detn. of neutral constituents, sulfonic acids, insol parts, and free  $\text{H}_2\text{SO}_4$ , is described. The method is based on the ability of benzene to dissolve neutral constituents (after previous sludge neutralization) and the ability of AmOH to ext. the sulfonic acids, leaving the unchanged  $\text{H}_2\text{SO}_4$  in aq soln. The  $\alpha$ - and  $\gamma$ -sulfonic acids can be further sepd by formation of the Ca salts and extg with ether. Further work is necessary for detg the water content of a sludge, since the xylene method proves inadequate. J. L. E.

Inspection of oil refinery equipment. Franklin L. Newcomb. *Refiner & Natural Gasoline Mfr.* 14, 92-7, 158-62, 402-8, 449-54(1935).—A series of articles comprising comprehensive directions for the inspection of all equipment. Charts, diagrams and instructions are presented. J. L. E.

Theory of cracking [oils]. Gustavo A. Fester. *Rev. facultad quim. ind. agr. (Univ. nat. litoral, Argentina)* 3, 295-10(1934), cf C A 27, 4383. W. F. Bruce

Octane number improvement in naphtha reforming. S. D. Turner and E. J. Le Roi. *Ind. Eng. Chem.* 27, 1347-9(1935).—A correlation of plant-cracking data is effected that satisfactorily gives the octane no. obtainable in reforming naphtha in terms of the original octane of the charge and the amt. of cracking to which it is subjected. This amt. is expressed in terms of equiv. secs. at 900°F., which are derivable from or convertible to an actual time-temp. curve, on the assumption that the cracking rate doubles with a rise in temp. of 25°F. For purposes of design the method can be worked backward. J. P. Wilson, Jr.

Polymerization of ethylene under high pressures in the presence of phosphoric acid. V. N. Tsvetkov and V. N. Pines. *Ind. Eng. Chem.* 27, 1364-9(1935).—Polymerization of  $\text{C}_2\text{H}_4$  in the presence of  $\text{H}_3\text{PO}_4$  within the temp. range 250-330° and an initial  $\text{C}_2\text{H}_4$  pressure of 50-65 atm. yields a mixt. of paraffine, olefinic, naphthene and aromatic hydrocarbons. Thermal polymerization under similar conditions yields higher-boiling hydrocarbons, but no aromatic compds. The catalytic polymerization gives isobutane, the yield of which increases from 2.5% by wt. of the  $\text{C}_2\text{H}_4$ , which reacted at 250° to 18.8% at 330°. Analyses of the gases at different temps. leads to the assumption that cracking does not take place to any appreciable extent. A mechanism of  $\text{C}_2\text{H}_4$  polymerization is suggested. P. J. Wilson, Jr.

The development of specifications for lubricants. O. L. Maag. *Blast Furnace Steel Plant* 23, 696-7(1935). E. H.

Selection of lubricants for industrial purposes. F. J. See. *Oil Colour Trades J.* 88, 1173-4(1935).—A general discussion of lubricants, with some detail on steam cylinder oils, steam turbine and enclosed steam engine oils. Specifications needed for purposes of identification only, and those to insure proper functioning are noted. W. H. Boynton

Analytical recognition of fat-containing lubricating oil. A. Baader. *Allgem. Oel- u. Fett-Ztg.* 32, 381-4(1935).—The usual method of detg. fat in mineral oil, i. e., the use of 0.1 N NaOH in 1.05 benzene alc. soln., was found unsuitable. On one sample, with 1.15 benzene alc. solns of 0.1, 0.2 and 0.5 N NaOH the sapon. values were 7.49, 14.60 and 24.36, resp.; and the results with 1.05 benzene alc. solns of the above concns of NaOH were 5.50, 11.90 and 26.5. The use of the stronger concns of NaOH is recommended for sapon. fat in the presence of mineral oil. M. M. Piskur

Certain sapinic acids obtained from various sources of pine and spruce. Torsten Hasselström and Marston Taylor Bogert. *J. Am. Chem. Soc.* 57, 2118-21(1935).—The sapinic acids of *Pinus palustris*, *P. caribbea*, *P. taeda*, *P. serotina*, *P. sylvestris* and *Picea excelsa* consist chiefly of a mixt. of *d* pimaric and *l*-sapietic acids. This finding is in accord with the results of other investigators. The presence of the former acid is proved by the formation of dihydropimaric acid when the sapinic acids are catalytically reduced; the latter is indicated by the fact that, when refluxed with glacial AcOH, the acids yield Steele's abietic acid as one of the products. The stability of *d* pimaric acid and its existence in the sapinic acids explain its presence in ordinary colophony. Phys. consts. of the acids from the various sources are given. C. J. West

Polymerization of unsatd. acids [lubricating oils] (Chowdhury, et al.) 27. Tetraalkyl Pb (Fr. pat. 784,222) 10. Destructive hydrogenation of mineral oils, etc (Ger. pat. 618,315) 21. Polynuclear phenols and nitration and sulfonation products [as antioxidants and polymerization inhibitors for motor spirit] (Brit. pat. 431,945) 10. Emulsions [as lubricants] (Brit. pat. 431,642) 13

Petroleum oil distillation. Leo D. Jones (to Sharples Specialty Co.). U. S. 2,018,309, Oct. 22. Oil such as crude petroleum is heated to effect its partial vaporization, brought into contact with carrier vapor, and the heated liquid residue is also brought into contact with carrier vapor and the resulting mixed vapors are cooled to condense oil from them and the remaining vapors, contg. carrier vapor, are used in the first mentioned contacting step. App. is described.

Breaking petroleum oil and water emulsions. Harmon F. Fisher (to Petroleum Rectifying Co. of Calif.). U. S. 2,018,302, Oct. 22. Various details of app. and operation are described for continuous treatment of the emulsion with a mixt. such as one contg. iron pyrites and quartz, etc.

Vapor-phase petroleum oil cracking. Charles B. Buerger (to Gulf Refining Co.). U. S. 2,019,070, Oct. 29. Hot cracked vapors from vapor-phase cracking of material such as a gas oil or fuel oil are intimately brought into contact with charge oil so as to vaporize vaporizable constituents of the latter; liquid residuum of the charge oil is removed and discarded, and the mixed vapors are subjected to reflux condensation in a refluxing zone, uncondensed vapors are removed and condensed, and condensate is withdrawn from the refluxing zone, heated to about 315° and returned to the refluxing zone. Condensate from the refluxing zone is subjected to vapor-phase cracking for production of addnl. hot cracked vapors for intimate contact with further quantities of charge oil. App. is described.

Cracking hydrocarbon oils. Guy N. Harcourt (to Cross Development Corp.). U. S. 2,017,860, Oct. 22. Vapors from oil cracking by heat and pressure are depleted, passed through a purifying stage (such as may involve use of fuller's earth) where only those vapors are treated having approx. the b. p. range of the desired final products, other vapors being withdrawn, condensed and the condensate used for bringing the vapors being depleted into contact. App. is described.

Cracking hydrocarbon oils. Pike H. Sullivan (to Gasoline Products Co.). U. S. 2,017,874, Oct. 22. A heavy oil contg. residual constituents such as a reduced crude oil

is subjected to a mild cracking temp. under a high superatm. pressure (suitably a temp. of about 470°) under a pressure of over 200 lb. per sq. in.) and the products are fractionated to recover a clean condensate, and the latter is separately subjected to more drastic cracking conditions and the products are fractionated to sep. gases, naphtha and heavier distillate, the gases are fractionated to sep. them into a lean fraction comprising principally H and CH<sub>4</sub> and a rich fraction contg. a relatively large amt. of gaseous olefins and this fraction is commingled with the heavy hydrocarbon oil for subjection to the mild cracking temp. and high superatm. pressure to effect a polymerization of gaseous olefins to form normally liquid products. App. is described.

**Cracking hydrocarbon oils.** Harold C. Weber (to Universal Oil Products Co.). U. S. 2,018,101, Oct. 22. A pair of electrodes at least one of which is made of a catalytic metal such as Ni is immersed in an oil such as a cracked residuum and a radio frequency current is passed between the electrodes by which the catalytic metal is dispersed in finely divided form throughout the oil. If it is brought into contact with the oil and it is simultaneously subjected to cracking conditions of temp. and pressure.

**Cracking hydrocarbon oils.** Richard P. Trow (to Texas Co.). U. S. 2,018,475, Oct. 22. An oil such as a topped or gas oil is subjected to cracking in a series of cracking stages under superatm. pressure, residual liquid is withdrawn from an earlier stage and subjected to distn. in a zone of reduced pressure, a 2nd residual liquid is withdrawn in a more advanced state of cracking from a later stage; the 2nd liquid is cooled and introduced into the reduced pressure distn. zone mentioned, and a final combined condensate is withdrawn from the reduced pressure distn. zone. App. and various details of operation are described. Cl. C. A. 29, 8314.1.

**Cracking hydrocarbon oils.** Charles H. Angell (to Universal Oil Products Co.). U. S. 2,018,699, Oct. 29. An oil is subjected to cracking conditions of temp. and pressure in a primary heating zone such as a pipe coil and the heated material is passed to a reaction zone from which vaporous and liquid products pass to a sepg. zone, vapors are fractionated to sep. sufficiently converted components as vapor from insufficiently converted components as reflux condensate and the reflux condensate is subjected to conversion conditions of temp. and pressure in a secondary heating zone whence materials are passed to the sepg. zone. App. is described.

**Cracking hydrocarbon oils.** Carlton P. Dubbs (to Universal Oil Products Co.). U. S. 2,018,708, Oct. 29. A stream of oil such as a fuel oil is forced through a heating zone such as a pipe coil in which it is heated to a cracking temp. and is discharged into an enlarged reaction zone through substantially the entire length of which the material travels; vapors and unvaporized oil are withdrawn at places remote from the place of introduction of the heated material and superatm. pressure is maintained in the heating and reaction zones; vapors and unvaporized oil from the reaction zone are discharged into the lower and upper portions, resp., of a reduced pressure zone where vapors flow countercurrent to non-vaporous residue, residue is removed from the system, vapors are fractionated, and regulated portions of resulting reflux condensate are returned to the heating zone merged with the stream of charging stock being forced through it, and fractionated vapors are condensed as a final distillate. App. is described.

**Cracking hydrocarbon oils.** Walter G. Whitman (to Standard Oil Co. of Ind.). U. S. 2,018,980, Oct. 29. An app. is described comprising a means such as a pipe-still furnace for heating oil to a cracking temp. under superatm. pressure and discharging into a sepg. chamber from which a vapor conduit of restricted cross-section leads to a fractionating column with cooling coils in its upper portion. Fresh oil is passed through the cooling coils and thence to an intermediate point of the vapor conduit (various other operative and structural details also being described).

**Cracking hydrocarbon oils.** Braunkohlen-und Brikkett-

Industrie A.-G. Buhag and Woldemar Allner. Ger. 618,224, Sept. 1, 1935 (Cl. 236, 1 04). Hydrocarbon oil vapors are passed through an externally heated vertical retort packed with coal or coke which is moved through the retort in the same direction as the vapors. The conditions are adjusted so that the velocity of the vapors in the cracking zone of the retort is at least 150 times that of the coal or coke. Steam may be blown in at the base of the retort. The products are light oils, gaseous hydrocarbons and C<sub>10</sub> or high-boiling liquid residues being obtained. Various methods of procedure are indicated.

**Cracking heavy hydrocarbon oils.** Universal Oil Products Co. Ger. 618,204, Sept. 4, 1935 (Cl. 236, 1 04). See Brit. 249,001 (C. A. 21, 1003).

**Cracking and distilling hydrocarbon oils.** John C. Black (to Gasoline Products Co.). U. S. 2,018,654, Oct. 29. Oil is passed, under pressure sufficient to prevent any substantial vaporization, through a coil heated to a cracking temp., a portion of the oil is vaporized and the vapors are fractionated, a lighter fraction is condensed and withdrawn, and a heavier fraction is condensed and returned to the coil for re-cracking, crude oil contg. gasoline fractions is passed through a 2nd coil in the path of waste combustion gases from the first mentioned coil, a portion of the oil is vaporized and the vapors are fractionated in a 2nd fractionating zone, the lighter fraction is condensed and withdrawn, the heavier fraction is condensed and cooled and a portion of it is returned to the final passes of the first mentioned coil to prevent overcracking of the oil in the coil and to heat the heavier fraction to a cracking temp. App. is described.

**Distilling and cracking hydrocarbon oils.** Wm. L. Gomory (to Standard Oil Development Co.). U. S. 2,019,448, Oct. 29. Fresh charge oil is subjected to vaporization under pressure without cracking in a chamber divided into a dephlegmating zone and a vaporizing zone, reflux condensate from the dephlegmating zone is recovered and subjected to cracking conditions of temp., pressure and time in a heating and cracking zone comprising a heating coil and a conversion chamber, resulting products are discharged from the conversion chamber into the vaporizing zone, vapors are recovered, and residuum from the vaporizing zone is separately withdrawn and its lighter fractions are returned to the heating and cracking zone. App. is described.

**Coking, cracking and "reforming" hydrocarbon oils.** Charles H. Angell (to Universal Oil Products Co.). U. S. 2,017,846, Oct. 22. A charging oil such as a crude, topped or fuel oil is distd. to coke in a coking zone; resultant vapors are fractionated to form a reflux condensate heavier than gasoline and a lighter fraction contg. a substantial quantity of hydrocarbons of gasoline b. p., the reflux condensate is heated to a cracking temp. under pressure while flowing in a restricted stream as through a heated pipe coil and the lighter fraction is similarly and separately heated to a higher temp. to enhance its antiknock properties and is then mingled with the heated reflux condensate, the commingled oils are sep. into vapors and residual oil in a sepg. zone, the residual oil is withdrawn from this zone and is introduced into the coking zone in admixt. with charging oil, and the last-mentioned vapors are fractionated and condensed. App. is described.

**Cracking and hydrogenating carbonaceous materials.** I. G. Farbenindustrie A.-G. Brit. 431,519, July 8, 1935. In the thermal treatment of carbonaceous materials having an acid reaction, corrosion of the treatment vessels by the acidic materials at such places where water tends to condense, e. g., at the outlet of the reaction vessel or in the heat exchangers and condensers and pipes leading thereto, is prevented by adding at such positions As compounds, e. g., As<sub>2</sub>O<sub>3</sub>, As<sub>2</sub>O<sub>5</sub>, or As compounds, As sulfide, cyanides or org. N bases, e. g., PhNH<sub>2</sub>, pyridine, quinoline. In addn., the usual catalysts may be added. In the destructive hydrogenation of a coal contg. 0.28% Cl, 0.1% As<sub>2</sub>S<sub>3</sub>, dispersed in oil, is added to the reaction products before they enter the heat exchanger.

**Destructive hydrogenation of hydrocarbon oils.** Philip L. Young (to Standard Oil Development Co.). U. S.



120-220°F., preferably at 140-80°F. The oil and diluent are preferably chilled at a slow rate and the wax is finally sepd. in a Sharples centrifuge with use of an immiscible carrier liquid.

Removing waxes from hydrocarbon oils. Standard Oil Development Co. Brit. 431,306, July 4, 1935. See Fr. 760,999 (C. A. 29, 6043).

Separation of paraffins from oils. Standard Oil Development Co. Fr. 784,570, July 22, 1935. Paraffins are sepd. from oils, particularly hydrocarbon oils, by dig. the oil with a solvent of a type heavier than the paraffin, adding a substance which is a modifier of the paraffin and is sol. in the mixt. of solvent and oil, cooling the mixt. to solidify the paraffin and sepg. it. The solvent may be a chlorohydrocarbon such as  $\text{CCl}_4$ ,  $\text{C}_2\text{H}_5\text{Cl}$ ,  $\text{CH}_2\text{Br}_2$ ,  $\text{PhCl}$ , or a mixt. such as  $\text{C}_2\text{H}_5\text{Cl}$  70 and  $\text{C}_2\text{H}_6$  30% or dichloropropane 75, toluene 15 and xylene 10%. The modifier may be a condensation product of active derivs. of paraffin such as chloro paraffins, polymers produced from chloro paraffin and aromatic hydrocarbons, polymers produced by submitting paraffinic hydrocarbons to the action of an elec. discharge, or products obtained by condensing, with the aid of  $\text{AlCl}_3$ , distillates of high b. p. from tars which have been submitted to intense cracking.

Refining gasoline. Thomas T. Gray (to Gray Processes Corp.). U. S. 2,019,154, Oct. 29. Gasoline contg. S compds. difficult to remove in the ordinary manner is treated to convert such S compds. into easily removable compds. by subjecting the gasoline in the vapor phase to contact with fuller's earth at a temp. of about 200-450°.

Device for separating gasoline from water and sediment by filtration. Leo E. Rush (to M. L. Joyce). U. S. 2,018,901, Oct. 29. Structural details.

Strainer suitable for gasoline nozzles. Wm. H. Rice and Hancel M. Lyon. U. S. 2,019,094, Oct. 29. Structural details.

Catalyst for polymerizing unsaturated hydrocarbons as in the production of gasoline from oil-cracking gases Vladimir Ipatieff (to Universal Oil Products Co.). U. S. 2,018,065, Oct. 22. A mixt. of a phosphoric acid such as ortho- or pyrophosphoric acid and a solid adsorbent such as kieselguhr is heated to 180-300°, but not substantially higher, before the polymerization process.

Pellets for treating motor fuels with tetraethyl lead. Paul Poetschke. U. S. 2,018,570, Oct. 22. Pellets are formed with a shell of stearic acid contg. a solid core comprising  $\text{BaPb}$  and stearic acid.

Cracked motor fuel. LeRoy G. Story (to Texas Co.). U. S. 2,018,970, Oct. 29. Deterioration and gum formation are inhibited by adding about 0.01% of a compd. such as 1,2-diaminofluorene or 1,9-dihydroxyfluorene or the like.

Lubricating oils. Mathias Pier (to I. G. Farbenind. A.-G.). U. S. 2,018,571, Oct. 29. A lubricating oil substantially free from asphalt and paraffin wax and having a lower-viscosity index than desired is treated with a liquefied normally gaseous hydrocarbon such as liquefied propane, the resulting soln. is removed from pptd. constituents of high mol. wt., the liquefied hydrocarbon solvent is removed, and there is added to the resulting oil an oil-sol. synthetic high mol. wt. product richer in H than the

pptd. constituents sepd. from the oil, to increase the viscosity of the oil.

Hydrocarbon lubricating oils. Eugene Ayres and Herschel G. Smith (to Gulf Refining Co.). U. S. 2,019,037, Oct. 29. Admixed unrefined stocks of different hydrocarbons such as a heavy and light oil are treated with  $\text{AlCl}_3$  to give a synthetic product contg. a suspended  $\text{AlCl}_3$  sludge, and, for finishing, most of the sludge is settled out, the oil is decanted, the remaining sludge is decompd. with dil.  $\text{H}_2\text{SO}_4$ , and after sepg. the acid the oil is brought into contact with clay and filtered.

Lubricating oil mixture. Carleton Ellis (to Standard Oil Development Co.). U. S. 2,018,758, Oct. 29. About 0.5% or more of the reaction product of an org. acid such as stearic or oleic acid with an alkylol group of an alkylolamine such as triethanolamine is added to a mineral lubricating oil to increase the load-bearing capacity of the oil.

Lubricating and transformer oils. N. V. de Bataafische Petroleum Maatschappij. Brit. 431,609, July 11, 1935. This corresponds to Fr. 775,701 (C. A. 29, 3150).

Apparatus for testing the condition of lubricating oil by use of light beams. Clarence E. Sherman and Howard P. Boak. U. S. 2,019,024, Oct. 29. Various structural optical and operative details.

"Snuffer composition" suitable for use in transformers or for lubricating, etc. Frank M. Clark (to General Elec. Co.). U. S. 2,019,339, Oct. 29. A C-contg. compd. (such as pentachlorodiphenyl and trichlorobenzene) the vapor of which is combustible under flash-producing conditions is used together with about 10% or less of a halogenated olefin compd. such as a chlorinated ethylene to prevent silent glow combustion of vapor emanating from the compn.

Lubricants. Standard Oil Development Co. Brit. 431,434, July 8, 1935. See Fr. 776,910 (C. A. 29, 3823).

Lubricants. Eric Wahlforss, Carl L. Johnson and Geo. Lacy (to The Glidden Co.). Brit. 431,508, July 9, 1935. Lubricants for high-pressure or other purposes consist of a hydrocarbon lubricating base and 1 or more chloroderivs. of retene, mono-, di-, tri- and hexachlororetene being specified. Among examples, (1) 15 lb. of monochlororetene is blended with 85 lb. of Pennsylvania bright oil stock, and (2) dichlororetene is prepd. by (a) directly chlorinating retene and washing or distg. *in vacuo*, or (b) chlorinating retene dissolved in  $\text{CCl}_4$ , purifying by steam distn., dissolving in petroleum naphtha, filtering and evapg. the solvent; 171 g. of the product is dissolved in 2500 g. of Pennsylvania bright oil stock.

Lubricants. I. G. Farbenind. A.-G. Fr. 784,319, July 22, 1935. Stable oils are produced by chem. condensation or hydrogenation of carbonaceous materials, particularly hydrocarbons, operating in the presence of an antioxidant such as elementary S, resorcinol, pyrocatechol, pyrogallol, hydroquinone,  $\text{PhNH}_2$ , naphthylamine, aminoanthraquinone, benzylamine, acetoneitrile, benzotrile, pyridine, carbazole, thiocresol, aminothiophene, aminodiphenylsulfone and diphenylthiourea. The products obtained may be added to other oils or greases which are thereby rendered stable.

Device for removing sludge from oil tanks, etc. Benjamin Thurlay. U. S. 2,019,372, Oct. 29. Structural details.

## 23—CELLULOSE AND PAPER

CARLETON E. CURRAN

The homogeneity of native cellulose. I. Crosswise structural elements of vegetable fibers and their separation by preparative methods. A. Zakoschikov and D. Tumarkin. *Melliand Textilber.* 16, 214-10, 360-7, 499-500 (1935).—See C. A. 29, 7637.

Viscosity determinations of cellulose solutions. Technical control methods and their practical importance. D. Kruger. *Zellulose-Faser* 32, 113-20 (1935).—In this discussion, which includes the significance of viscosity measurements at different stages in the production of

rayon and cellulose derivs., the use of various types of viscometers, and the relationship between viscosity and chem. compn., mech. properties, and the "soly." of cellulose and its derivs., K. presents no new expl. data, but includes 77 references.

Louis E. Wise. Some aspects of the oxidation of cellulose. H. A. Turner. *J. Soc. Dyers Colourists* 51, 315-52 (1935).—A review with discussion.

W. H. Boynton. Nitration of cellulose with nitrogen pentoxide. René Dalmon, Jean Chédia and Louis Brissaud. *Compt. rend.*

201, 664-5(1935) —The Raman spectrum of most nitrating mixts shows the presence of  $N_2O_4$ . Accordingly the nitrating power of  $N_2O_4$  in a neutral solvent was examd. by treating cellulose (2.5 g. of dry degummed rape bleached with NaOH) with  $N_2O_4$  (45 g.) dissolved in  $CCl_4$  (105 g.) at  $13^\circ$  for 6 hrs in the dark. A 176% yield of nitrocellulose was obtained, contg. 14.14 or 13.88% N according as the product was stabilized or not. Heated at  $135^\circ$  both products turned red after 32 and 29 min., resp., but neither emitted nitrous fumes after 1 hr. at that temp.

C. A. Silberrad

Determination of the fineness of nitrocellulose by dye absorption. A. J. Phillips *Ind Eng Chem, Anal Ed* 7, 416-19(1935) —The method involves dyeing the fibers with a negatively charged dye (Congo red) and then depositing a positively charged dye (methylene blue) at and near the surface of the fibers which have been dyed red. The dyeing takes place in a borax- $NaCl-H_2BO_3$  buffer soln. of  $pH = 7.9$  at  $95^\circ$ . The dyeing tests were confirmed by analysis under the microscope and not only could the products from different beaters be identified but products from the same beater could be differentiated. Such differentiation cannot be made by the standard fineness test.

W. T. H

Are the foundations of lignin chemistry unsettled? Karl Kurschner. *Zellstoff-Faser* 32, 132-4(1935) —K. shows that Hilpert and Hellwege (cf. C. A. 29, 3704\*) have presented data insufficient to discredit the preexistence of lignin in plant tissues.

Louis E. Wise

Causes of "milky" spots on viscose rayon. Stefan Poznanski. *Przemysl Chem* 19, 42-7(1935) —The "milky" spots are caused by the formation of bubbles in the filaments as a result of evapn. of  $CS_2$  which did not succeed in diffusing through the surface skin to the acid bath. Their occurrence is increased by decreasing the quantity of  $CS_2$  used in prep., the xanthate, diminution of the viscosity of viscose, increase in concn. of free caustic in viscose, introduction of even a small quantity of sulfonated fatty oils, increase of acidity of the acid bath, in case of use of  $ZnSO_4$ , diminution of its content, diminution of the thickness of the filaments or the increase in their no. per thread.

A. C. Zachlin

Delustered rayon and its prospects. Fritz Ohl. *Spinnerei u. Weber* 53, No. 27, 10-12(1935). —A review.

Leopold Schedan

The specific gravity of coniferous woods. Reinhard Trendelenburg. *Zellstoff-Faser* 32, 129-31(1935) —The findings of previous investigators have been analyzed, and the following list includes, resp., the mean, the lowest and the highest sp. grs. observed for green wood, and in parentheses, the corresponding figures for bone-dry wood calcd. on the basis of vol. when green: spruce, 0.44 (0.38), 0.29 (0.27), 0.64 (0.55), fir, 0.42 (0.37), 0.29 (0.27), 0.00 (0.00), larch, 0.55 (0.48), 0.40 (0.36); 0.00 (0.00), Scots pine, 0.49 (0.43); 0.29 (0.27), 0.00 (0.00). With spruce, fir and larch, the greatest frequency in sp. gr. was very close to the mean sp. gr. This is not the case with fir. T. shows that marked variations in sp. gr. of spruce occur with varying site and altitude, and that in Scots pine variations are very wide even in the bole of a single tree. The relationship between width of annual rings and the sp. gr. of dry wood was also studied. In spruce, a correlation exists between sp. gr. and ring width. In Scots pine and larch, both very narrow and very wide annual rings give rise to wood of low sp. gr., indicating that there is an optimum ring width in these species.

Louis E. Wise

The influence of the preheating of wood in water on the rate of delignification by sulfite liquor. A. J. Corey and O. Maass. *Can J. Research* 13, B, 149-55(1935) —The rate of delignification of wood chips has been measured and found to conform to the monomial relation, provided that the standard method of penetration developed in this lab. is used. Pretreatment of the wood chips by heating in liquid  $H_2O$  at  $130^\circ$  decreases the rate of delignification by subsequent cooking in sulfite liquor, and the rate no longer conforms to the monomial relation. Preheating of the wood to  $130^\circ$  in the absence of  $H_2O$  does not influence the

rate of subsequent delignification. The results indicate the advantage of reducing the time of cooking, and a tentative explanation has been offered regarding the mechanism involved.

J. W. Shipley

Friction losses in pipes carrying pulp stock. W. Brecht and H. Heller. *Wochbl. Papierfabr.* 66, Tech. Teil 264-8, 312-4, 380-3, 439-43, 474-6, 529-32, 587-9, 641-4, 714 17, 747-50(1935). —An unusually thorough expl. study in which friction losses, using water alone and water kaolin suspensions, were detd. An investigation was made of the effect of the following factors on the frictional losses in the case of pulp suspensions: stock d.; rate of flow, diam. of the pipe line, roughness of the inner surface of the pipe, the av. fiber lengths; slowness of stock; temp.; type of pulp used (including bleached and unbleached sulfate, unbleached sulfate, yellow and bleached straw pulp, and white and brown mech. pulps); kaolin addn. to the pulp. Detailed descriptions and sketches of the expl. equipment used are included. B. and H.'s data are compared with those of other investigators. Sixty-five references.

Louis E. Wise

The chemist in the pulp and paper industry. M. M. Rubin. *Chemist* 12, 269-79, 288(1935).

E. H

For a (French) national paper industry. B. Navarre. *Bull. inst. pap.* 1935, 205-12 —A discussion of the possibility of reducing importations of paper and paper-making materials into France, together with an analysis of French resources and requirements of paper-making materials.

A. Papineau-Couture

Sack paper, its manufacture and properties. R. A. Krauss. *Mühle* 72, 1411-14(1935).

C. L. Brooke

Rayon fabrics (Schnell) 25. Retarding rancidity-cellulose wrappers (Morgan) 12. Soap [products used in glazing paper] (Swiss pat. 174,087) 27. Coating metal [used in the construction of a spinning pot for rayon] (Brit. pat. 431,816) 13.

Cellulose. Erich Opfermann and Gustav-Adolf Feldtmann (to I. G. Farbenind. A.-G.). U. S. 2,017,985, Oct. 22. A cellulose which can be bleached white is prepd. by the continuous addn. of alkali such as NaOH to a cellulose pulp such as one produced from partial chlorination of kraft pulp while being produced by alk. digestion with  $Ca$ , to maintain a  $pH$  between 6 and 9.

Cellulose. Henry Dreyfus. Fr. 784,333, July 22, 1935. Cellulose is extd. from lignocellulosic materials at a high temp. by a solvent mixt. contg. water and at least 2 org. liquids of different dipole moments, due to the presence in the mol. of groups of different types. Ex. samples are given of the use of mixts. contg. (1) acetone 1, water 35 and EtOH 50 parts, (2) EtOH 65, water 25 and  $CH_2Cl_2$  10 parts, (3) dioxane 50, water 30 and acetone 2) parts. Cf. C. A. 29, 7636\*.

Cellulose and half-stuff. Hofstad A.-G. Brit. 431,542, July 10, 1935. In aq. cellulose and half-stuff, ground wood, washed until it is freed from fiber fragments and fiber mucilage, or the coarse material that accumulates during the manu. or sorting of ground wood, washed free from fine fibers, fiber fragments and fiber mucilage, is subjected to chem. treatment in the usual manner. Wood rich in resin, e. g., pine, is a suitable starting material. The cellulose or half-stuff obtained may be added in suitable proportions to ground wood to make a paper suitable for newspapers. Cf. C. A. 28, 7532\*, 29, 4579\*, 6428\*.

Saccharifying cellulose. Holzhydrolyse A.-G. Ger. 618,145, Sept. 2, 1935 (Cl. 89, 1.02). The recovery of cryst. glucose and xylose from cellulose saccharification products is promoted by adjusting the proportion of mannose in the products to 5-10%, calcd. on the total sugar content. Thus, saccharification products of low mannose content, e. g., as obtained from wood of deciduous trees, may be mixed with saccharification products of high mannose content, e. g., as obtained from wood of coniferous trees, or mixts. of these varieties of wood may be saccharified together. Other methods of procedure are indicated. Solns. having a total sugar content up to

85% may be treated. Methods of effecting crystn. are described.

Preserving cellulose materials such as canvas, ropes, wood, etc. Wallace T. Conn (to the Government of the U. S., represented by the Secretary of Commerce). U. S. 2,018,659, Oct. 29. Articles such as nets, ropes, etc., formed of degummed fibers or the like contain a toxic dye such as crystal violet, a fixer for the dye comprising  $K_2Cr_2O_7$ ,  $CuSO_4$ , and  $HIOAc$  and carry a tar-like armoring. U. S. 2,018,660 relates to the use of a tannic acid and tartar emetic mordant and a  $K_2Cr_2O_7$  oxidant. U. S. 2,018,661 relates to the use of tar and an oil-sol. residue resulting from the reaction of  $\alpha$ -naphthylamine with acetaldo.

Cellulose derivatives. Samuel Wild, Frnst Hugentobler and Frich P. Gellich. Swiss 174,060, May 16, 1935 (Cl. 41). App. is described for making coned solns of cellulose derivs by circulating the solvent repeatedly over the deriv. The solns. are used for making formed articles

Aliphatic cellulose ethers R. I. du Pont de Nemours & Co. Brit. 432,277, July 21, 1935 To a crude reaction product contg. an aliphatic ether of cellulose and an org. medium a dispersing agent adapted to form an emulsion of the said crude reaction mixt. and an aq. medium is added and the product is steam distd. The alkyl cellulose is obtained as a finely divided, porous solid, which is easily purified by washing. Examples describe the prepn. of an alkyl cellulose by treatment of alkali cellulose with solid  $NaOH$ ,  $EtCl$ , which may be mixed with  $MeCl$ , or  $PrBr$ , and  $CH_4$  or  $PhMe$  as diluent, in an autoclave. The product is treated with Turkey red oil or  $Na$  oleate and  $H_2O$  and then steam distd., the alkyl cellulose remaining in suspension in the liquid.

Cellulose ester and ether compositions British Celanese Ltd. Brit. 432,271, July 23, 1935 Comps. comprise cellulose esters or ethers that are plasticized by adding hexahydrobenzoic esters of partial ethers of di- or polyhydric alcs. The comps. may be worked up into sheets, rods, tubes or blocks or molded under heat and pressure; as solns., they may be converted by wet or dry spinning processes into filaments or made into films, sheets or foils, or used as lacquers. In examples, (1) a soln. of cellulose acetate (I), the ester of ethylene glycol monomethyl ether with com. naphthemic acid, and triacetin in  $Me_2CO$  is spread on the gelatin-covered surface of a film-casting wheel, the  $Me_2CO$  is evapd., and the film stripped off, and (2) a coating compn. comprises I, di- $Me$  phthalate, the same plasticizer as in (1), diphenylolpropane- $CH_2O$  resin,  $Me_2CO$ ,  $CH_2I_2$ , etc., diacetone alc. and a pigment

Cellulose acetate. Geo. W. Miles (to Celanese Corp. of America). U. S. 2,018,028, Oct. 22. For producing cellulose acetate of high acetyl value, cellulose is treated with a restricted quantity of acetylating agent not substantially greater than that required for esterification, in the presence of a catalyst such as  $H_2SO_4$ ,  $H_3PO_4$ ,  $ZnCl_2$ , or  $NaHSO_4$ , and a substantial quantity of diluent such as  $CH_2I_2$  or  $CCl_4$ , which has no solvent action on the cellulose acetate being formed, until a cellulose acetate of an acetyl value of 63 to 70% is formed.

Cellulose ester films, lacquers, plastic compositions, etc. Deutsche Hydrierwerke A.-G. Brit. 432,404, July 25, 1935. The films, etc., contain as softening or gelatinizing agent an ester of a hydroxydicarboxylic acid with a mono- or poly-nuclear hydroaromatic alc. In examples, acetylcellulose (I) and dimethylcyclohexyl tartrate are dissolved in  $Et$  lactate and  $PhMe$  to form a lacquer, and I, didecahydro- $\beta$ -naphthyl tartrate and tricresyl phosphate are dissolved in  $EtOH$ ,  $Me_2CO$  and  $PhMe$ , the soln. being used to make films of high elasticity.

Saponifying cellulose esters. Henry Dreyfus. Brit. 432,023, July 15, 1935. The acetyl content of org. esters of cellulose is reduced by sapon. with an org. base in the presence of an alkali metal salt that is itself capable of sapon. the ester, e. g., of  $H_2CO_3$ ,  $H_2BO_3$ ,  $H_2S$ ,  $H_2SO_4$ , and  $PhOH$ . Among examples, cellulose acetate yarn is led in warp formation through a bath contg. aq.  $MeNH_2$  and  $Na_2CO_3$ , washed and dried. Substances that accelerate the reaction, e. g., metals such as  $Cu$ , as described in

Brit. 430,310 (C. A. 29, 7873\*) and 430,351 (C. A. 29, 7674\*), and swelling agents may be present.

Saponifying cellulose ester threads, etc. Henry Dreyfus. Brit. 432,027, July 10, 1935. Filaments, ribbons, etc., having a basis of cellulose acetate or other org. ester of cellulose are sapon. by treatment in a bath contg. a non-metallic base, preferably a lower aliphatic amine, e. g.,  $MeNH_2$ ,  $LiNH_2$ ,  $AmNH_2$ ,  $PrNH_2$ ,  $PtNH_2$ ,  $Me_2N$ , allylamine,  $Me_2NOH$ , ethylenediamine, 1,2,3-triaminopropane,  $\alpha,\gamma$ -diamino- $\beta$ -hydroxypropane, piperidine, etc., and a nonhydroxy swelling agent, e. g.,  $Me_2CO$ ,  $MeEtCO$ , dioxane, methylene ethylene ether, cyclohexanone, the dimethyl ether of glycol, alkali thiocyanates. To accelerate the sapon., the bath may receive an addn. of strong alkalis or of heavy metals, e. g.,  $Cu$ ,  $Ag$ ,  $Ni$  or  $Zn$  as described in Brit. 430,310-51 (C. A. 29, 7673\*)

Saponifying cellulose nitrate products Henry Dreyfus. Brit. 432,029, July 16, 1935 Artificial filaments, films, etc., having a basis of cellulose nitrate are sapon. by treatment with a liquid medium contg. an org. base, e. g., alkylamines, aromatic bases, cyclic bases, diamines and substituted diamines and piperazine. The sapon. action may be accelerated by a heavy metal, e. g.,  $Cu$ ,  $Ni$ , in the form of oxide or salt, and it may be assisted by alkalis or alk. salts, e. g., soaps. Swelling agents may be present.

Press rolls for removing water from cellulosic sheet materials, etc. Maschinenfabrik Imperial G. m. b. H. Ger. 618,431, Sept. 7, 1935 (Cl. 55d 20 01)

Apparatus for extruding a continuous web of pyroxylon composition or the like to form sheets Paul W. Crane and Reuben T. Fields (to Dupont Viscoloid Co.) U. S. 2,019,110, Oct. 29. Various details are described for the production of sheets from a compn. such as one contg. pyroxylon of 800 centipoises 40-21, camphor 10-8 and 1'10H 38-68% by extrusion through a slit-like aperture under a pressure of 30-400 lb. per sq. in.

Rayon Adrianus J. L. Moritz (to American Fknk Corp.). U. S. 2,018,505, Oct. 22. For the manuf. of rayon so that all parts of a wound package of the product will have substantially uniform dyeing properties, the spinning soln. is ejected from a spinnerette into a spinning bath and the filaments formed are tensioned by leading them over guides in advance of a collecting device, the tension being gradually increased during the formation of the package to compensate for different degrees of contraction normally occurring which would otherwise cause nonuniformity in the dyeing quality of the filaments in different parts of the package. App. is described. Cf. C. A. 29, 4532\*

Rayon Stefano Sordelli. Brit. 432,328, July 24, 1935. See Fr. 778,917 (C. A. 29, 4940\*)

Rayon Aceta G. m. b. H. Fr. 781,639, July 22, 1935. Rayon made from cellulose esters or ethers is made resistant to boiling by treating the freshly spun fibers with an aq. prepn. contg. soaps or soap components, the components forming the soap being in the cationic instead of the anionic state. Examples of preps. are (1) glue 20, oleyl acetate- $\alpha$ -pyridinium chloride 10, cacao butter 30, Japan wax 120 and water 2200 parts, and (2) glue 100, urea 50, lauryl acetate- $\alpha$ -pyridinium chloride 25, octadecyl acetate- $\alpha$ -pyridinium chloride 25,  $Me$  ester of stearic acid 400, Japan wax 100 and water 8300 parts.

Apparatus for winding freshly spun rayon. Carl Hamel Spinn- und Zwirnereischinen A.-G. and Edmund Hamel Ger. 615,592, July 8, 1935 (Cl. 20a. 6.15) Addn. to 614,441 (C. A. 29, 6077\*)

Centrifugal "bucket" for use in spinning rayon. Fredrick S. Godfrey (to General Elec. Co.). U. S. 2,010,314, Oct. 29. A bucket body is provided with circumferentially extending rows of spaced holes in its side walls and channels on the outside surface of the side walls connecting holes in the several rows and extending to the top of the body; a reinforcing sleeve is disposed about the side walls, and an acid resisting internal covering which may be formed of hard rubber is placed on the sleeve and with the channels mentioned forms outlets at the top of the body.

Paper pulp Sidney D. Wells and Gerald D. Muggleton (to Lewis L. Alsted). U. S. 2,018,037, Oct. 29. Washed

fibrous material such as wood, straw or corn stalks is crushed in a rod mill and digested and disintegrated in a cooking liquor obtained in part by forcible removal from the pulp later in the process, steam is blown into the rod mill during the digestion and disintegration, and the material is thereafter washed concurrently and finally bleached while maintained alk. so as to preclude chlorination of the lignin. An arrangement of app. is described and a bleached material may be obtained contg. not less than 20% of pentosans and not less than 10% of lignin, with the remainder substantially cellulose.

**Paper pulp from flax.** Edwin P. Jones and James M. Dempsey (to Champagne Paper Corp.). U. S. 2,018,490, Oct. 22. Mechanically decorticated flax is subjected to the action of water, and the washed fibers are cooked in a digester in the presence of an alk. chemical such as  $\text{Ca}(\text{OH})_2$ ,  $\text{Na}_2\text{CO}_3$ , or  $\text{NaOH}$  and of a sol. sulfite such as  $\text{NaSO}_3$ , and are then bleached. The resulting product is suitable for making paper.

**Apparatus for making paper pulp.** E. & M. Lamort. Fr. 784,117, July 22, 1935.

**Paper-pulp beating engines.** Walther Voith, Hermann Voith and Hanns Voith (trading as firm of J. M. Voith). Brit. 432,009, July 18, 1935.

**Hammer mills for paper pulp.** Wm. T. Doyle (to Sturtevant Mill Co.). Brit. 432,115, July 22, 1935. A stream of concd. cellulose pulp is fed to a hammer mill wherein it is subjected to swift impacting, disintegrated, hydrated and mixed with diln.  $\text{H}_2\text{O}$  which is fed as a sep. stream to the mill.

**Machines for shredding wood pulp, etc., for the manufacture of paper.** John J. Warren. Brit. 431,322, July 4, 1935.

**Slides for Fourdrinier paper making machines.** Benjamin E. Teale. Brit. 430,981, June 24, 1935.

**Flong.** Walter E. Langley. Brit. 430,850, June 26, 1935. Flong used in making molds for casting printing-plates consists of paper pulp mixed with lithopone which may be added to the pulp in the beaters.

**Apparatus for damping paper, textiles, etc.** Orion Maschinen- und Apparatebau-Gesellschaft m. b. H. Brit. 431,932, July 17, 1935. Divided on and addn. to 428,975 (C. A. 29, 7077).

**Removing printing ink from paper.** Sidney D. Wells (to Lewis L. Aisted). U. S. 2,018,938, Oct. 29. The paper is disintegrated and agitated in a soapy soln. until the ink is loosened from the paper and a foamy mass is produced contg. substantially no free liquid and having the particles of the ink pigment carried by the films of the bubbles, and the bubbles and assoc. pigment are sep'd from the fibers. An arrangement of app. is described. Cf. C. A. 29, 2744.

**Thin porous paper sheets.** Alexander V. Alm (to Dennison Mfg. Co.). U. S. 2,018,244, Oct. 22. The surfaces of the fibers of a sheet of tissue paper of open

formation are quickly parchementized to bind the fibers together with a water resistant bond without filling the interstices of the paper. An arrangement of app. is described.

**Sized papers.** Geo. A. Richter (to Brown Co.). U. S. 2,018,875, Oct. 29. A paper web is treated first with a viscose soln. and then with an acidified glue soln., thus regenerating cellulose from the viscose and producing an exposed glue surface.

**Water-repellent coatings on paper or the like.** Alfred Earle Van Wirt (to Imperial Paper and Color Corp.). Brit. 431,218, July 3, 1935. See U. S. 1,950,279 (C. A. 28, 3234). The printing step may be omitted. Washable wallpapers are produced.

**Coated paper.** International Latex Processes Ltd. Brit. 430,953, June 27, 1935. Paper is rendered resistant to  $\text{H}_2\text{O}$ , oil or grease by treating with an aq. rubber dispersion, e. g., latex, contg. added protein, e. g., casein, glue, gelatin, e. g., blood-albumin, hemoglobin, drying and then coating with a soln. of the protective material in an org. solvent, e. g., lacquers contg. nitrocellulose or cellulose acetate, resins, PhOH aldehyde or alkyl resins or chlorinated rubber. A suitable latex compn. comprises  $\text{NH}_4\text{OH}$  as stabilizer, glue,  $\text{ZnO}$ , S, accelerator, phenylanththylamine as antioxidant, casein,  $\text{NaOH}$  and PhOH as preservative. The intermediate rubber film may be vulcanized after application of the lacquer. Cf. C. A. 29, 4942.

**Apparatus for impregnating paper webs with liquids such as preserving oils.** Max Oberdorfer. U. S. 2,018,280, Oct. 22. Various structural, mech. and operative details.

**Transparent paper.** Wm. M. Driesen (to S. D. Warren Co.). U. S. 2,018,638, Oct. 22. A web of paper is formed of fibers of transparent material and a film matrix of cellulose deriv. material such as cellulose nitrate compn. with anhyd. lanolin dispersed in the material and having about the same  $n$  as the fiber substance.

**Compound paper.** Société pour l'ind. chim. à Bille. Brit. 431,958, July 18, 1935. In the manuf. of fibrous sheet material consisting of a no. of superimposed sheets having a basis of partially hydrolyzed cellulose, a primary aromatic amine- $\text{CH}_3\text{O}$  resin is added to the cellulose material before, during or after its partial hydrolysis. The product has a high resistance to  $\text{H}_2\text{O}$ .

**Ply board or ply paper.** John W. Sale (to Hummel-Ross Fibre Corp.). U. S. 2,018,382, Oct. 22. Various mfg. details are described, involving use of an app. with a traveling endless wire for continuous operation.

**Apparatus for manufacture of corrugated paper board.** Geo. W. Swift, Jr. (to George W. Swift, Jr., Inc.). U. S. 2,018,240, Oct. 22. Various mech. and operative features.

## 24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE AND C. G. STORM

**Improvements in the stability of explosives.** A. Foulon. *g Nitrocellulose* 6, 183-4(1935).—A review. E. M. S.

**Unexpected explosive effects with various shapes and positions of the charge.** Alfred Stettbacher. *Nitrocellulose* 6, 59-62, 100-8(1935).—Perforations in metal plates caused by detonation of explosive charges of various shapes and positions on or above the plate are illustrated. E. M. Symmes

**New considerations on elementary explosives production.** Alfred Stettbacher. *Nitrocellulose* 6, 79-83 (1935).—A review. E. M. Symmes

**Thermostat for stability test of blasting gelatin.** J. v. Meerscheidt-Hullessem. *Z. ges. Schiess-Sprengstoffw.* 30, 301-2(1935).—A jacketed oven maintained at 75° by a mixt. of  $\text{H}_2\text{O}$  and  $\text{MeOH}$  is provided with glass windows through which the evolution of nitrous fumes from the samples in weighing tubes may be observed. C. G. S.

**Variation of detonation spectra with nature of surround-**

ing gas. Alfred Michel-Lévy and Henri Murawow. *Compt. rend.* 201, 823-30(1935); cf. C. A. 29, 1640, 3160.—In view of the suggestion that the luminosity of an explosion depends on the nature of the gas, the spectra of explosions of the  $3\text{C}(\text{NO}_2)_4 + \text{C}_2\text{H}_2$  mixt. in A, Kr,  $\text{O}_2$ ,  $\text{CO}_2$ ,  $\text{N}_2$ , air,  $\text{H}_2$ ,  $\text{H}_2$  and  $\text{Cl}_2$  were exam'd. The amt. of continuous background varied, being considerable with A, Kr,  $\text{O}_2$ ,  $\text{CO}_2$  and  $\text{Cl}_2$ ; and the luminosity and therefore the temp. decreased approx. in the increasing order of the  $\nu$  heats of the gases. But the only bands or lines identifiable seem to have been those attributable to the explosive (especially those of  $\text{CN}$ ) or to its action on the metal supports. C. A. Silberrad

**The so-called "Congreve friction match" in Munich 100 years ago.** Max Speter. *Z. ges. Schiess-Sprengstoffw.* 30, 299-300(1935).—Historical. C. G. Storm

**Gases, explosions and fires in coal mines.** Frank Briners. *School Sci. Rev.* 17, 38-9(1935). O. R.

**Dust disasters. Consequences, causes and control of dust explosions.** David J. Price. *Food Ind.* 7, 520 (1935); cf. *C. A.* 29, 3167.—Approx. 385 dust explosions with a loss of life of 311 persons have occurred in the past 10 years. Flour, feed and starch factories are often concerned in dust explosions. It is possible to vent grain-dust explosions without structural damage. Not less than 1.25 sq. ft. of venting area per 100 cu. ft. of space is recommended. Static electricity is a prominent cause of explosions and static charges should be eliminated in mill plants. The reduction of  $O_2$  content by introducing an inert gas such as  $CO_2$  in grinding and pulverizing operations is an effective means of control. The  $O_2$  content should be reduced to approx. 12%. C. R. Fellers.

**Prevention of gas explosions by controlling oxygen concentration.** G. W. Jones and R. E. Kennedy. *Ind. Eng. Chem.* 27, 1311-6 (1935).—Tests in an explosion tube 6 ft. long X 2" diam. showed that the combustibles which occur in natural gases and in petroleum products are rendered nonexplosive when  $CO_2$  is added to the combustible mixts. so as to reduce the  $O$  content below 12.6%, if N is used as the diluent the  $O$  content should be reduced below 10%. For  $CO$  and H mixts. the  $O$  content must be reduced below 8% to eliminate explosion hazard.

C. G. Storm.  
**Explosion of mixtures of combustible gases with air, by nuclear drops of water and other nuclei and by x-rays.** Experimental conditions required for the ignition of hydrogen-air mixtures by nuclei. R. O. King and Geo. Mole. *J. Inst. Petroleum Tech.* 21, 838-45 (1935), cf. *C. A.* 28, 7017.—The view previously expressed, that self-ignition temp. rises as surface activity increases, was confirmed by exptl. results, with steel and silica tubes. The rate of steam formation in the silica tube was low

compared to that in steel tubes. The  $H_2O$  produced was measured as liquid. Self-ignition occurs only in the layer of gas adjacent to the hot surface. VI. Nuclear drop ignition temperatures of ethylene-air mixtures passing through a silica combustion tube; the relation between wall and gas temperature and the effect on ignition temperatures of the central thermocouple sheath. *Ibid.* 845-53.—Wall temps. were found to be 16-30° higher than those indicated by the central thermocouple. The sheath itself is without effect on the ignition temp. VII. Effect of a variety of nuclei, mainly mineral dusts, to ignite and explode a mixture of hydrogen and air; the exceptional efficacy of  $Ni_2O_3$  dust to ignite mixtures of air with hydrogen, ethylene or methane. *Ibid.* 853-9.—With  $Ni_2O_3$  dust the nuclear ignition temp. of  $H_2$ -air mixt. (40%  $H_2$ ) was more than 200° lower than that obtained with nuclear drops of water. In  $C_2H_4$ -air and  $C_2H_2$ -air mixts. the igniting effect of  $Ni_2O_3$  was less pronounced, but was still greater than that of water drops. C. G. Storm.

**Polynuclear phenols and nitration and sulfonation products [as explosives]** (Brit. pat. 471,015) 10.

**Explosives.** Hercules Powder Co. Ger. 618,600, Sept. 10, 1935 (Cl. 78c 17). See Brit. 299,802 (*C. A.* 23, 3576).

**Pyrotechnic compositions.** Westfälisch-Anhaltische Sprengstoff-A-Ges. m. b. H. Ger. 618,083, Aug. 31, 1935 (Cl. 78j 1.01). As agents for regulating the rate of combustion of pyrotechnic compns., particularly of charges for light-producing weapons, use is made of oleaginous materials from which oil or fat cannot be expressed at a pressure up to 5000 atm. Ground nut shells of various kinds are suitable.

## 25—DYES AND TEXTILE CHEMISTRY

L. A. OLBRY

**Dyes derived from acridic acid.** Mahadeo Prasad Gupta and Sukhshushra Dutt. *J. Indian Chem. Soc.* 12, 581-4 (1935).—From theoretical considerations (*C. A.* 22, 3891), dyes derived from acridic acid (I) should have the same color as the corresponding dyes derived from quinuclidine-1,2,3-tricarboxylic acid (II). A suspension of finely divided acridine was oxidized with 2%  $KMnO_4$  and yielded stout prisms of I, m. 128-30° (decolor.). I was condensed with  $PhOH$ ,  $m$ - and  $p$ - $HOC_6H_4OH$ ,  $m$ - $H_2NC_6H_4H_2N$ ,  $m$ - $Me_2NC_6H_4OH$ ,  $m$ - $Et_2NC_6H_4OH$ , 1,3,5-( $HO$ ) $_3C_6H_2$ , 3,5-( $HO$ ) $_2C_6H_3Me$  and  $m$ - $H_2NC_6H_4NH_2$ . The condensation takes place without condensing agent but the addn. of a trace of  $H_2SO_4$  improves the yields. The dyes resemble the corresponding phthalates but the fluorescence is slightly less and they are slightly more intense in color. They dye wool and silk beautiful and brilliant shades of orange, tan, brown, chocolate, chestnut, pink and violet. Compared with the corresponding dyes from II they are more intensely colored and far more absorptive. The extra  $CO_2H$  group in the II dyes acts as a bathochrome in reducing the intensity of color and fluorescence. Tables of the dyes derived from I, giving m. p., appearance, color in alc., color with acid or alkali, shades on wool or silk and C and H analyses, and showing the adsorption max. of the acridins and analogous dyestuffs are given. The compd. with  $m$ - $HOC_6H_4OH$  was brominated to the corresponding tetrabromo-compd., m. above 200°. C. R. Addinall.

**The selection of dyestuffs for padding.** H. C. Borg-hetty and J. Zapananick. *Am. Dyestuff Repts.* 24, 639-40 (1935).

**Lake colors and paper.** N. Holt. *Oil Color Trades J.* 83, 1219-50 (1935).—Surface-colored papers are discussed. The principal sizing agents are glue and casein and the latter required alkali which may cause the lake colors to bleed. Bleeding may also be caused by improper pptn. of the dye in the lake. W. H. Boynton.

**Light fastness of dyes on vegetable fibers.** C. M.

Whittaker. *Silk and Rayon* 9, 591 (1935); cf. *C. A.* 29, 7601<sup>1</sup>.

**Fifty years of dyeing.** Joseph Turner. *J. Soc. Dyers & Colourists* 51, 355-7 (1935).—A summary of a lecture.

**The consumption of heat in dyeing operations.** Otto Th. Koritnig. *Spinner u. Weber* 53, No. 38, 12-13 (1935).

**Chromium mordants in textile dyeing and printing.** S. L. Segson. *Can. Chem. Met.* 19, 269-9 (1935).

**Dyeing jute yarn and jute and cotton piece goods—suitable basic, acid and substantive colors; methods of producing solid shades on union goods.** James R. McIlveen. *Am. Dyestuff Repts.* 24, 600-2 (1935).

**Fast wool dyeings with particular consideration of the cloth supplied Herbert Brandenburger.** Monatschr. Textil-Ind. 50, 213-6 (1935).—Dyeing expts. were carried out with various Cr and vat dyes. Measurements were made of the strength and elasticity (dry and wet) of the Cr and vat-dyed wool. Studies were made of the effect of the addn. of fiber preservatives on the strength and elasticity of Cr-dyed textiles. The results show that the difference between Cr and vat dyeings are very slight and that the preservatives used increase both the strength and the elasticity. Detailed dyeing directions are given and the results obtained are tabulated. Leopold Scheflan.

**The printing of gold bronzes on wool textiles.** A. Molnar. Monatschr. Textil-Ind. 50, 218 (1935).—The method of printing bronzes on cotton and rayon textiles with Serikose CL extra thickeners is not feasible for wool on account of the high degree of stretch of this textile. In this case it is better to employ egg albumin as thickener. The egg albumin is beaten with a little water until it forms a foam which is allowed to shrink, then linsed oil is added and finally the bronze powder. After printing, the fixation is carried out with steam. As this mixt. is unstable

it should not be stored but must be used right after its prepn. The spraying can also be done by means of gutta-percha soln. After the print has completely dried apply the powd. bronze and calender hot. Leopold Scheflan.

The advantages of the hyposulfite glucose vat. H. Korb. *Monatsh. Textil-Ind.* 50, 246 (1935).—The mixt. contained 3.5 g indanthrene blue RS, 100 g NaOH 30%-Be and 3.5 g concd hyposulfite per l of H<sub>2</sub>O with or without glucose. Pieces of cotton were subjected to 6 dyings. The alkali contents were detd. by titration and the hyposulfite was detd. by Pyrgos' Kuptometer. The dyings without glucose yield a lighter and more turbid tone and are not as fast as those obtained with glucose. In the presence of glucose the alkali of the bath decreases rapidly while the hyposulfite contents drop only gradually. Acting as an aldehyde the glucose holds the hyposulfite loosely, thus preventing excess reduction. During the dyeing process the aldehyde groups oxidize to acid radicals which decrease the alkali concn. In the absence of glucose the action of the O of the air on the hyposulfite is much greater on account of the high temp. and the lack of protection of the hyposulfite. Conclusion: The glucose vat is carried out more readily and is more economical. Leopold Scheflan.

Yarn-dyeing equipment. II. Chas E. Mullin. *Textile Colorist* 57, 304-8, 388-91, 453-5 (1935); cf C A 29, 3157.<sup>2</sup>—A description of the various types of machines used in dyeing yarns in package form, i.e., cops, cheeses, beams, etc., with a discussion of their advantages, disadvantages, operation, etc. Chas E. Mullin.

Further studies of the effect of sunlight on the strength and color of cotton fabrics. Mary Anna Grimes. *Tex Agr. Expt. Sta., Bull.* 506, 5-42 (1935), cf C A 28, 947.<sup>2</sup>—After exposure for 500 hrs to sunlight 35 white cotton and dyed cotton fabrics showed losses in breaking strength of 18-49% in the warp and 34-65% in the filling. The mercerization of the Everfast suitings and broadcloths increased their resistance to tendering. In 8 out of 15 dyed fabrics, less breaking strength was lost than in the undyed fabrics. Certain dyes are not equally fast when used alone and in combination with other dyes. The heavier, coarser fabrics were less tendered than the thinner, finer ones. Correlation analysis of the environmental factors shows that the no. of hrs. exposure had far more effect on loss of strength than had temp. and relative humidity. Exposure to sunlight affected the color of all white and dyed fabrics, as detd. by spectrophotometric analysis. White fabrics became increasingly yellow with increased exposures. In general, guaranteed fabrics were more fast in color than those not so guaranteed. C. R. Fellers.

Progress in the textile industry. Heinrich Gölkel. *Monatsh. Textil-Ind.* 50, 229-7 (1935).—A review. Leopold Scheflan.

List of the various grades of yarns. A Hamann. *Spinner u. Weber* 53, No. 16, 2-5, No. 17, 5-7; No. 18, 5-8, No. 19, 5-7; No. 23, 6-8, No. 24, 5-7; No. 27, 4-6, No. 28, 4-7 (1935).—The compas. of a no. of yarns are described. Leopold Scheflan.

The uniformity of carded yarn. A Strang. *Spinner u. Weber* 53, No. 28, 1-4 (1935). Leopold Scheflan.

Analysis of textiles for cellulose acetate rayon, silk, regenerated-cellulose rayon, cotton and wool. Ralph T. Mease and Daniel A. Jessup. *Am. Dyestuff Repr.* 24, 613-18 (1935); *Bur. Standards, Research Paper* No. 821 (1925); cf C A 29, 7084.<sup>2</sup>—Cellulose-acetate rayon, silk, regenerated-cellulose rayon, cotton and wool are detd. by exin. of the mixed fibers with CCl<sub>4</sub>, desizing with a starch and protein hydrolyzing enzyme and with water, removal of acetate rayon with Me<sub>2</sub>CO, and silk and regenerated-cellulose rayons with solns. of Ca(SCN)<sub>2</sub> of sp. grs. of 1.2 and 1.25, resp. Cotton is detd. by removal with AlCl<sub>3</sub> and heat, to leave the wool, or by dissolving the wool in KOH soln. to leave the cotton. The accuracy of the method for each fiber is  $\pm 2\%$  of the wt. of the specimen analyzed. W. H. Boynton.

Some problems of the cotton textile industry. B. B.

Gosset. *Textile Bull.* 49, No. 11, 3-5, 34 (1935).—A survey. Leopold Scheflan.

The oiling of wool for the worsted spinning process. J. B. Speakman. *Melland Textiles* 16, 628-41 (1935).—See C A 29, 7083.<sup>2</sup> J. A. Szilard.

New methods of the finishing of blankets by means of fatty alcohol sulfonates. W. Pflumm. *Spinner u. Weber* 53, No. 38, 9-12 (1935).—Photomicrographs show wool damaged by alkalis, damages caused by the formation of Ca soap, and the appearance of a carefully washed woolen blanket. Detailed directions are given for washing and rinsing involving the use of Gardipol which was found to give very good results. Leopold Scheflan.

Sulfonated and free fatty alcohols. E. Gibson. *Silk and Rayon* 9, 589 (1935).—The uses of these compds in finishing silk and rayon are discussed. M. Harris.

Rayon fabrics. Arthur H. Schnell. *Textile Bull.* 49, No. 11, 6, 8, 10 (1935).—A review of the properties of rayon yarns and the manu. of rayon goods. L. S.

The finishing of pure rayon fabrics. Wilhelm Kegel. *Deut. Wollen-Gewerbe* 67, 1249-50 (1935).—Various formulas and sp. directions are given for sizing, treatment after bleaching and dyeing, finishing mixed fabrics from rayon and cotton, and printing and steaming of rayon fabrics. Strong alkali baths should not be employed for washing. A newly developed comp. finishing product is Ortonox K which is easily sol. in boiling water, is particularly adapted for the finishing of mixed fabrics from rayon and cotton and is employed in a concn. of 30-60 g/l. at a temp. of 30-35°. Leopold Scheflan.

The consumption of rayon in the weaving mill. Kai Dietz. *Spinner u. Weber* 53, No. 39, 6 (1935). Leopold Scheflan.

The summing of rayon and cellular wool. Fritz Ohl. *Spinner u. Weber* 53, No. 40, 7-10 (1935).—A review. Leopold Scheflan.

The utilization of waste in the jute industry. Hans Rudolph. *Spinner u. Weber* 53, No. 35, 8-10 (1935). Leopold Scheflan.

Putrefactive decomposition of Bengal silk cocoons. Sikkibhusan Dutt. *J. Indian Chem. Soc.* 12, 458-62 (1935).—The products of putrefaction of silk cocoons during the process of maceration in aq. liquids act as strong pressor substances for the heart. A preliminary examn. of cocoons obtained from the Government of Bengal showed that they contained 25.6% of sericin, 70.4% of fibrin, 2.2% H<sub>2</sub>O and about 2.0% of moist matter. The bright yellow color is probably due to their carotene content. Complete hydrolysis and subsequent estn. of the amino acids gave 28.4, 22.55, 5.75, 0.85, 0.8, 0.8 and 12.8% of glycine, alanine, serine, leucine, glutamic acid, phenylalanine and tyrosine together with traces of aspartic acid and proline. On maceration with 50 times their wt. of H<sub>2</sub>O and incubation at 37° for 1 week, the cocoons underwent extensive putrefaction and lost 35% of their wt. The liquid expressed from the fibers had a nauseating odor and on systematic extr. yielded (in the form of HCl salts) 4.2, 1.8, 5.8, 2.1 and 0.3% of NH<sub>4</sub>, NH<sub>4</sub>Me, NH<sub>4</sub>Et,  $\beta$ -HOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> and HOCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, resp. CO<sub>2</sub> was evolved freely during the incubation. Thus the putrefaction involves the simultaneous hydrolysis of the proteins into amino acids and the decarboxylation of the acids into amines. The presence of 24% of tyramine in the decoction accounts for its strong pressor action. The fibrous matter remaining consisted of practically pure bleached fibrin with the luster but only half the strength of ordinary silk. The large proportion of NH<sub>4</sub> evolved during the putrefaction must be due to the further degradation of the amines. C. R. A.

Recent developments in textile finishing. L. G. Lawrie. *Textile Bull.* 49, No. 10, 8, 34 (1935).—Reasons are given for the improvement of finishes, finishing methods and processes since the world war. Among the sulfonated textile aids are iso-Pr naphthalenesulfonic acids, sulfonated oils, alcs and long-chain compds. Commercially valuable textile aids are classified according to their effect on the finishing processes: they (1) simplify, (2) improve or (3) produce new or novel effects. Grouped

according to the effect they produce the finishing assistants are (1) detergents, (2) wetting-out agents or (3) solvents. Leopold Scheffan

The formulation of bat and fabric finishes. P. H. Fawcett. *Paint, Oil & Chem. Rev.* 97, No. 19, 12, 14, 16 (1935).—The results desired with such products, factors controlling their properties and formulation methods are discussed. J. W. Perry

Hats. T. A. Foster. *J. Soc. Dyers Colourists* 51, 351-61 (1935).—A brief review of modern hat manufacture and dyeing. W. H. Boynton

The development of the electron technic and its importance for the textile industry. Rudolf Mehlo. *Monatsschr. Textil-Ind.* 50, 217, 240 (1935).—A discussion of the applications of high-vacuum tubes and Hg vapor rectifiers. Leopold Scheffan

Working with vacuum. Teufel Z. *ges. Textil-Ind.* 38, No. 43, 10 (1935).—Applications in the textile industry are briefly discussed in connection with dyeing and drying. Leopold Scheffan

Bibliography on launderability of textiles. L. M. Gugelman. *Am. Dyestuff Repts.* 24, 593-4, 607-9 (1935).—An extensive bibliography on this subject covering the period of 1910 to 1934. W. H. Boynton

The lime soap problem in textile finishing. A. Poulon. *Allem. Oel- u. Fett Ztg.* 32, 363-5 (1935).—The use of Calgon (Na metaphosphate) is recommended to eliminate the formation of high-Ca salts when hard waters are used for textile operations. M. M. Piskur

Buckling experiments. G. Coch. *Spinner u. Weber* 53, No. 18, 9-12 (1935).—Several series of buckling experiments were conducted with and without pressure with NaOH, Nuva B, Bosit, water glass, soap, Percolloid B, Biscalon, Gardinol R and Igepon T on crude cotton. Results: Except for Percolloid B, all buckling agents tested have an effect on the white appearance of the fabric only at such a high concentration which hardly ever occurs in actual practice. Soap does not stand behind but passes all these buckling agents in this respect. A change of the wetting, foaming and dispersing properties of Nuva B, Gardinol R and Igepon T does not occur to any noticeable extent after boiling for 3 hrs. under a pressure of 1½ atms. However, admixtures to the fiber which are extruded from the crude cotton will influence these physical-chemical properties. Leopold Scheffan

Mothproofing agents for textile goods. Stötter. *Spinner u. Weber* 53, No. 36, 11-13 (1935).—A discussion of several patents on mothproofing materials and of the use of a no. of dyes for the manufacture of mothproof goods. Leopold Scheffan

Heat economy in small textile plants. J. Elbers. *Monatsschr. Textil-Ind.* 50, 210-12 (1935). L. S.

Rubbenizing fabrics (Trépan) 30. pH indicators [color change of dyed goods] (Mullin) 7. Auxochromes and resonance [in the color of dyes] (Bury) 10. Assistants [in the textile industry] for working with hard water (Ohl) 14. Electrophoresis of dyes (Waelch) 2. Industrial value of Argentine flax (Fauris) 26. App for dampening textiles (Brit. pat. 431,932) 23. Starch preps [for glazing or finishing textiles] (Brit. pat. 431,275) 28. Phenolic condensation products [as reserves in dyeing] (Ger. pat. 618,034) 29. Polynuclear phenols and nitration and sulfonation products [as dye intermediates] (Brit. pat. 431,945) 10. Hydrazinesulfonates [for azo dyes] (U. S. pat. 2,018,103) 10. Condensation products from phloroglucinol [as dye intermediates] (U. S. pat. 2,018,137) 10.

Dyes. Chemische Fabrik vorm. Sandoz. Swiss 174,541-4, Apr. 1, 1935 (Cl. 37b). Addn to 169,707 (C. A. 29, 3846). Pulling-fast dyes are obtained by treating 1-amino-2-sulfo-4-phenylaminoanthraquinone with AcCl in the presence of H<sub>2</sub>SO<sub>4</sub> (174,541). The AcCl may be replaced by 3-aminobenzaldehyde (174,542), or by 4-aminobenzaldehyde (174,543), or the anthraquinone deriv. by 1-amino-2-sulfo-4 (1'-methylphenylamino)-

1-anthraquinone (174,544). The structural formulas of the dyes are given.

Dyes. Soc. pour l'ind. chim. à Bâle. Swiss 175,242, May 1, 1935 (Cl. 37a). A new dye contg. metal is obtained by treating the Cu compd. of the dye from diazotized 5-nitro-2-aminophenol and 2-naphthol-3,6-disulfonic acid with reducing agents which, in an alk. medium, cause the NO<sub>2</sub> groups of 2 mols. to link up. The dye colors cotton and viscose in fast blue shades.

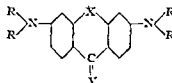
Dyes. Chemische Fabrik vorm. Sandoz. Swiss 175,893, June 1, 1935 (Cl. 37b). Addn to 169,707 (C. A. 29, 3846). A new pulling-fast dye is obtained by treating Na 1-amino-4-phenylaminoanthraquinone-2-sulfonate with CH<sub>3</sub>O in an aq. medium. The dye colors wool in fast blue shades. Its structural formula is given.

Dyes. Soc. pour l'ind. chim. à Bâle. Swiss 176,240-2, June 17, 1935 (Cl. 37d). Addns to 173,414 (C. A. 29, 5666). A S dye is obtained by heating pyrene to high temps. with S. The dye colors cotton from a hyposulfite vat in fast blackish brown shades (176,240). Similarly fluoranthene is heated with S to give a dye which colors cotton from a Na-S bath in orange-brown shades fast to Cl (176,241). Also, amonopyrene, obtained by nitrating pyrene and reducing the NO<sub>2</sub> compd. is heated with S to give a dye which colors cotton from a Na-S bath in brown shades (176,242).

Dye composition. Soc. pour l'ind. chim. à Bâle. Swiss 176,340, July 1, 1935 (Cl. 24a). The compn. consists of a dye contg. a sulfo group and capable of dyeing in AcOH, and less than 10% of a lyophilic dispersion agent contg. at least one basic N atom and at least one aliphatic or cycloaliphatic residue with at least 8 C atoms and capable of uniting with dyes contg. a sulfo group. Thus, the finely powdered dye from diazotized 1-amino-8-naphthol-3,6-disulfonic acid and 1-phenylaminoanthraquinone-8-sulfonic acid is made into a paste with alc. To this is added H<sub>3</sub>PO<sub>4</sub> and N-dihydroxypropylimidazole-HCl. The paste is dried and pulverized to give a non-sticky powder suitable for making wool dyes. Other examples are given.

Dyes. Soc. pour l'ind. chim. à Bâle. Swiss 176,533, July 1, 1935 (Cl. 37d). Addn to 173,414 (C. A. 29, 5666). A dye contg. S is obtained by treating 2-(4'-hydroxyphenylamino)anthracene with S-yielding agents, such as Na-S and S, at high temps. The new dye gives cotton from a Na-S bath olive black shades.

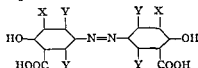
Dyes. I. G. Farbenind. A.-G. (Carl Winter and Emil Kern, inventors). Ger. 618,121, Sept. 2, 1935 (Cl. 22b). Yellow or orange dyes are obtained by the action of NH<sub>3</sub> or primary or secondary amines on compds. of the formula below, in which R is H or an alkyl, aryl or aralkyl group, and X and Y are O or S. The keto chlorides of these compds. may also be used. The reaction may be effected at atm. or raised temp. in the presence or absence of ZnCl<sub>2</sub> or like condensing agent. Water must be present if a cyclic amine is used. Examples are given. The products resemble the known dianilindimethane dyes but are relatively fast to boiling and acid.



Azo dyes. Chiford Paine (to Imperial Chemical Industries Ltd.). U. S. 2,018,764, Oct. 29. Diazotized p-nitrobenzoyl-p-phenylenediaminesulfonic acid or other diazotized amine of the general formula  $\text{YR}^1\text{XCR}^2\text{NH}_2$ , in which Y represents a nitro or monoacylamino group, R<sup>1</sup> and R<sup>2</sup> represent arylene nuclei at least one of which has substituted thereon a sulfonic acid group, and X represents a carbonylamino or sulfoamino group, is coupled with an aromatic amine such as a  $\lambda$ -substituted "J acid" and the resulting aminoazo compd. is diazotized and coupled with a N-substituted aminonaphthol-sulfonic acid having a diazotizable amino group in the N-substituent, the nitro

or monoacylamino group mentioned is converted to an amino group, and the resulting compound is tetratized and coupled with 2 equivalents of a "yellow end component" such as acylacetanilide. The resulting dyes give green shades.

**Azo dyes** Wilhelm Neelmeier and August Modersohn (to General Aniline Works) U. S. 2,018,801, Oct. 20. Dyes of the general formula



where one X stands for H or the sulfonic acid group and the other X stands for the sulfonic acid group, alkyl or halogen, in such a manner that at least one X represents the sulfonic acid group, and the Y's stand for H, alkyl or halogen, in the form of their alkali metal salts generally yellowish to brownish powders, sol. in water, and dyeing wool from an acid bath, after chroming, generally yellowish to brownish shades of good fastness are obtainable by treating with a sulfonating agent an azo dye of the general formula as above without the X substituent in one ring, where X stands for H, alkyl or halogen and the Y's stand for H, alkyl or halogen, the manufacture of these dyes being described for example in German Patent 278,613. Instead of starting with these dyes, there may be used the complex Cr compounds which are described, for example, in Fr 499,971 (C. A. 28, 650<sup>6</sup>) and which are obtainable by heating the 4-amino-1-hydroxybenzene-2-carboxylic acid or a nuclear substitution product thereof in the free form or in form of a water-sol. salt with a water-sol. Cr salt contg. the Cr in the trivalent form in water or in an organic solvent and, if desired, with the addition of a non diazotizable base. Several examples with details are given.

**Azo dyes** I. G. Farbenindustrie A.-G. Brit. 432,020, July 15, 1935. Dyes are prepared by coupling a diazotized amine of formula  $2\text{-H}_2\text{N-3-XC}_6\text{H}_4\text{SO}_3\text{H}$ , where X is H or a univalent substituent and where the  $\text{C}_6\text{H}_4$  nucleus may contain further substituents other than OH, with a naphthalene deriv. of formula  $\text{acyl-YN-1-HO-3-HO}_2\text{C}_6\text{H}_3$ , where Y is H, alkyl or aryl. The products dye animal fibers yellowish orange to bluish red shades. Examples are given.

**Azo dyes** Williams (Hounslow) Ltd., Arthur G. Green, Herbert Ackroyd and Alexander Macmaster Brit. 432,355, July 22, 1935; Fr 784,207, July 22, 1935. Dis-, tri- and tetrakis azo dyes are made from tetratized benzidine 2,2'-disulfonic acid, or the tetrazo compound of the tolidimedisulfonic acid obtained from *o*-nitrotoluene by sulfonation, alk. reduction and intramolecular change of the hydrazo compd., by (1) coupling with 2 mols of a sulfonic acid of 2,5- or 1,8-aminonaphthol with 1 mol of such an acid and 1 mol of another such aminonaphtholsulfonic acid, (2) 1 mol of a sulfonic acid of 2,5- or 1,8-aminonaphthol and 1 mol of  $\alpha$ - or  $\beta$ -naphthol or a sulfonic acid thereof, resorcinol,  $\beta$ -resorcylic acid,  $\beta$ -hydroxynaphthoic acid, salicylic acid, *o*-cresotic acid, PhOH, cresols, phenylpyrazolones or an aminophenolic or amino component, e. g., *m*-phenylene- or *m*-tolylene diamine, (3) 2 mols of an acyl deriv. of a sulfonic acid of 2,5- or 1,8-aminonaphthol contg. an acyl group derived from a fatty acid of 8 or less C atoms or from a substituted or unsubstituted aromatic acid, (4) 1 mol of an acylaminonaphtholsulfonic acid as defined in (3) and 1 mol of the 2nd components stated in (2), (5) 2 mols of a sulfonic acid of 2,5- or 1,8-aminonaphthol, 1 or both of which have been previously coupled with a diazo compd. or, alternatively, with 2 mols of the said aminonaphtholsulfonic acid and subsequently with 1 or 2 mols of the diazo compd., (6) 1 mol of a sulfonic acid of 2,5- or 1,8-aminonaphthol that is previously or subsequently coupled with a diazo compd., the 2nd diazo group of the tetrazo compd. being coupled with 1 of the 2nd components in (2), (7) in alk. soln. with either 1 or 2 mols of a sulfonic acid of 2,5- or 1,8-aminonaphtholsulfonic acid, rediazotizing the product and

coupling with 2 mols of 1 of the 2nd components of (2), or (8) with 1 or 2 mols of a middle component other than 2,5- or 1,8-aminonaphtholsulfonic acid, rediazotizing the product and coupling with 2 mols of a sulfonic acid of 2,5- or 1,8-aminonaphthol. They are suitable for leather dyeing. In examples, (1) benzidine-2,2'-disulfonic acid (I)  $\rightleftharpoons$  (alk.) H acid or J acid, (2) I or the tolidimedisulfonic acid  $\rightleftharpoons$  (alk.) acetyl H acid, (3) 1-phenyl-3-methyl-5-pyrazolone  $\rightleftharpoons$  I  $\rightleftharpoons$  (acid) H acid  $\rightleftharpoons$  (alk.)  $\beta$ -nitroaniline (II), (4) I or the tolidimedisulfonic acid  $\rightleftharpoons$  (alk.) H acid  $\rightleftharpoons$  (acid) II, (5) H acid  $\rightleftharpoons$  (acid) I  $\rightleftharpoons$  (acid) H acid  $\rightleftharpoons$  (alk.) PhNH<sub>2</sub> or  $\beta$ -naphthylamine, (6) H acid  $\rightleftharpoons$  (alk.) I  $\rightleftharpoons$   $\beta$ -xylylene  $\rightleftharpoons$  (alk.) H acid.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 174,319, June 1, 1935 (Cl. 37a). A new dye is produced by coupling *N*-nitroamine from 1-aminonaphthalene, with diazotized 1-amino-2-methoxybenzene.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 174,517-18, Apr. 1, 1935 (Cl. 37a). Addns to 163,539 (C. A. 28, 2914). A new dye is obtained by coupling diazotized 2,6-dichloro-4-nitro-1-aminobenzene with ethyl( $\omega$ -acetohydroxyethyl)aniline (174,517). The diazo component may be replaced by diazotized 2,5-dichloro-4-nitro-1-aminobenzene (174,518). The dye colors acetate silk in yellowish brown and bluish red shades, resp.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 174,519, Apr. 1, 1935 (Cl. 37a). Addn. to 169,700 (C. A. 29, 3850<sup>1</sup>). A new dye coloring acetate silk in violet-red shades, is obtained by coupling diazotized 5-nitro-2-aminophenol with 1-ethylamino-2-methoxy-5-methylbenzene.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 175,028, Apr. 16, 1935 (Cl. 37a). A new dye is prepared by coupling diazotized 4-(1'-chlorophenoxy)acetylamino-2,5-diethoxy-1-aminobenzene with the 2-methylanilide of 2,3-hydroxynaphthoic acid. The dye colors vegetable fibers in fast blue shades. The structural formula of the dye is given.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 175,030, Apr. 16, 1935 (Cl. 37a). A new dye is prepared by coupling 2-diazo-5-acetylamino-4,4'-dichloro-1,1'-diphenyl ether with the 2-methoxyanilide of 2,3-hydroxynaphthoic acid. The dye colors cotton and rayon in fast bordeaux red shades. The structural formula of the dye is given.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 175,031, Apr. 16, 1935 (Cl. 37a). A new dye is obtained by coupling 2-diazo-4-carboxylic acid 4'-methyl-1,1'-diphenyl sulfone with the anilide of 2,3-hydroxynaphthoic acid. The dye colors wool, cotton and rayon in fast orange shades. The structural formula of the dye is given.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 175,353-4, May 10, 1935 (Cl. 37a). A new dye is produced by coupling 2-diazo-diphenylene dioxide with the 1-naphthylamide of 2,3-hydroxynaphthoic acid. The dye colors cotton, wool and rayon in fast Bordeaux red shades (175,353). A second dye is obtained by coupling 2-diazo-4-chloro-1,1'-diphenyl ether with the 2-naphthylamide of 1-hydroxy-3,4-dichloro-6-carboxylic acid. The dye colors wool, cotton and rayon in fast brown shades (175,354). Examples and the structural formulas of the dyes are given.

**Azo dyes** J. R. Geigy A.-G. Swiss 175,355, June 1, 1935 (Cl. 37a). Diazotized 6-chloro-2,4-dimethylaniline is coupled with monodihydroxypropyl- $\alpha$ -naphthylamine (obtained by condensing  $\alpha$ -naphthylamine with a tech. mixt. of isomers of chlorodihydroxypropane). The dye colors acetate silk in blue shades. An example is given.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 176,922, July 16, 1935 (Cl. 37a). A new dye is formed by coupling 2-diazo-4-acetyl-4'-chloro-1,1'-diphenyl ether with 2,3-hydroxynaphthoic acid-2'-methylanilide. The dye colors cotton, wool and rayon in fast red shades. The structural formula is given.

**Azo dyes** I. G. Farbenind. A.-G. Fr 784,330, July 22, 1935. Dyes are prepared by combining diazotized amines of the formula  $\text{H}_2\text{NXSO}_2\text{NH}_2$  C(OH) C(COOH)



CH CY:CH (X is an aromatic radical which may contain

substituents and Y is H, NO<sub>2</sub>, Cl, Me or SO<sub>2</sub>H) with coupling components. Thus, *N*-(3'-amino)benzenesulfonyl-3-amino-2-hydroxybenzenecarboxylic acid → 1-(2'-chloro 5'-sulfo)phenyl-3-methyl-5-pyrazolone dyes wool in yellow shades. Other examples are given.

**Azo dyes** I. G. Farbenind. A.-G. Fr. 784,364, July 22, 1935. Dyes are prepd. by combining a diazotized alkyl ester of 1-amino-2-halobenzen-6-carboxylic acid with a coupling component contg. OH and having a coupling position next to the OH but contg. no other solubilizing groups, either in substance or on the fiber or in the presence of a substratum suitable for the prepn. of lakes. Thus, the Et ester and 1-(2',3'-hydroxynaphthylamino)-4-chloro-2,5-dimethoxybenzene give a dye which dyes fast orange shades. Several examples are given.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Fr. 784,500, July 22, 1935. Arylides of aromatic  $\alpha$ -hydroxy carboxylic acids are coupled in substance or on the fiber with 4-halo-2-diazo-5-acylamino diphenyl ethers, the 2nd phenyl group being substituted in any manner if desired. Thus a light Bordeaux red shade is obtained with 4-chloro-2-diazo-5-acylamino-4'-methoxydiphenyl ether → 2-aniside of 2,3-hydroxynaphthoic acid. Other examples are given.

**Azo dyes.** Soc. pour l'ind. chim. à Bâle. Fr. 784,514, July 22, 1935. Alkyl esters of 2-diazo-4-carboxy-1,1'-diphenyl sulfones in which the 2nd Ph group may be substituted in any manner or replaced by an alkyl group are coupled with any coupling components. Thus, 2-amino-4-carboxy-4'-methyl 1,1'-diphenyl sulfone → *o*-chloro-aniside of 2,3-hydroxynaphthoic acid is an orange dye. Other examples are given.

**Triazo dyes** E. I. du Pont de Nemours & Co. Brit. 432,122, July 22, 1935. Dyes are made in accordance with the scheme, an amine of the C<sub>6</sub>H<sub>5</sub> or C<sub>6</sub>H<sub>4</sub> series (which may be substituted by halo, alkyl, alkoxy, SO<sub>2</sub>H, COOH or NO<sub>2</sub> groups but no OH) → a *p*-coupling arylamine other than an aminopyrazolone → an aminohydroquinone dialkyl, diaralkyl or diaryl ether → an aminobenzoylaminoanthraquinone sulfonic acid. They give bright green shades on cotton when diazotized on the fiber and developed, e. g., with 1-phenyl-3-methyl-5-pyrazolone. In examples, (I) aniline-2,5-disulfonic acid → 1-naphthylamine-7-sulfonic acid (II) → aminohydroquinone dimethyl ether (III) → *p*-aminobenzoyl-J acid, and (2) aminoterephthalic acid → I → II → *p*-aminobenzoyl-1,8,4-acid.

**Indigoid dyes** Soc. pour l'ind. chim. à Bâle. Swiss 174,262-1, Apr. 1, 1935 (Cl. 37e). Addns to 163,709 (C. A. 29, 1995). 4,7-Dimethylsatin  $\alpha$ -halide is treated with a chlorinating agent in the presence of an indifferent diluent and the product is condensed with 4-chloro-1-naphthol. Thus, 4,7-dimethylsatin is converted to the  $\alpha$ -chloride by treatment with PCl<sub>5</sub> and PhCl. The  $\alpha$ -chloride is treated with SO<sub>2</sub>Cl<sub>2</sub> and the product condensed with 4-chloro-1-naphthol (174,270). Alternatively the  $\alpha$ -halide may be condensed with 1-naphthol (174,271). The dyes produce fast blue shades on cotton from a yellowish green vat.

**Indigoid dyes** Soc. pour l'ind. chim. à Bâle. Swiss 170,362-3, June 17, 1935 (Cl. 37e). Reactive  $\alpha$ -derivs. of 4-methyl-7-chlorosatin are condensed with 4-methoxy-1-naphthol to give dyes coloring cotton in fast blue shades. Thus, 4-methyl-7-chlorosatin is heated with PCl<sub>5</sub> and PhCl to give 4-methyl-7-chlorosatin  $\alpha$ -chloride. This is condensed with 4-methoxy-1-naphthol to give a dye, (176,262). Similarly, reactive  $\alpha$ -derivs. of 4,7-dimethyl-6-bromosatin are condensed with 1-naphthol to give dyes which color cotton in fast blue shades. The structural formulas of the dyes are given (176,263). Cf. C. A. 29, 1653<sup>1</sup> and preceding abstr.

**Triarylmethane dyes.** I. G. Farbenindustrie A.-G. Brit. 432,204, July 22, 1935. The dyes are obtained by condensing in the known manner in nonalk. media secondary or tertiary amines with aromatic aldehydes or with

aromatic compds. contg. 2 CH<sub>2</sub> groups, at least 2 H atoms of each CH<sub>2</sub> being substituted by halo atoms, and converting the resulting leuco bases into the corresponding triarylmethane dyes by oxidation in the presence of acids. In examples, terephthalaldehyde is condensed with dimethylaniline, diethylaniline and *N*-hydroxyethyltetrahydroquinoline in presence of ZnCl<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>, and HCl, resp., and the resulting leuco bases are oxidized by PbO<sub>2</sub> to dyes that give blue-black shades on cotton mordanted with tannic acid.

**Sulfur dyes.** Société pour l'ind. chim. à Bâle. Brit. 431,976, July 18, 1935. This corresponds to Fr. 776,145 (C. A. 29, 3539<sup>1</sup>) but the sulfuration of the SO<sub>2</sub>H and NH<sub>2</sub> derivs. and of perylene itself is excluded.

**Sulfur dyes** Soc. pour l'ind. chim. à Bâle. Swiss 174,548, June 1, 1935 (Cl. 37d). Addn. to 169,352 (C. A. 29, 606<sup>1</sup>). A black-dyeing S dye is prepd. by condensing indophenol from carbazole and *p*-nitrosophenol, with 1-acetylamino-4-aminobenzene and sulfurizing the product at temps. above 200°. Cf. C. A. 29, 767<sup>2</sup>.

**Vat dyes** Soc. pour l'ind. chim. à Bâle. Swiss 175,027, Apr. 16, 1935 (Cl. 36q). An equimol. mixt. of 1-halo-2-acylaminoanthraquinone and 1-haloanthraquinone is heated with a halogen-seizing agent and the product saponified, to give 2-amino-1,1'-biantraquinonyl.

**Vat dyes** Soc. pour l'ind. chim. à Bâle. Swiss 176,031, June 1, 1935 (Cl. 37b). The new dye 2,2'-dianthrimide 1,1'-carbazole is prepd. by the action of an acid condensing agent on 2-amino-1,1'-biantraquinonyl. It produces olive shades on cotton.

**Vat dyes.** Soc. pour l'ind. chim. à Bâle. Swiss 176,037, July 1, 1935 (Cl. 37b). Isodibenzanthrone is brominated with about 1.2 parts of Br in a PhNO<sub>2</sub> medium in the presence of at least 5% of ZnCl<sub>2</sub>. The product has a Br content of about 20% and colors cotton in violet shades.

**Vat dye** Soc. pour l'ind. chim. à Bâle. Swiss 176,027, July 16, 1935 (Cl. 37b). *Br*-1-Benzanthronyl-5-aminobenzanthrone is heated with a caustic alkali to give a dye coloring cotton from a blue vat in fast marine blue shades. The structural formula is given.

**Azo dyes** Soc. pour l'ind. chim. à Bâle. Swiss 177,260, Aug. 1, 1935 (Cl. 37a). A new dye is formed by coupling diazotized 6-cyano-2,4-dinitro-1-aminobenzene with 2-methoxy-5-methyl-1-*N*-bis(methoxyethyl)aminobenzene. The dye colors acetate silk in fast blue shades.

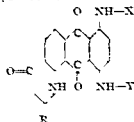
**Vat dyes** (arylaminoanthraquinones). Imperial Chemical Industries Ltd. Ger. 618,002, Aug. 30, 1935 (Cl. 22b 3 03). See I. r. 770,091 (C. A. 29, 1651<sup>1</sup>).

**Vat dyes.** I. G. Farbenind. A.-G. (Wilhelm Eckert and Ernst Fischer, inventors). Ger. 618,045, Sept. 2, 1935 (Cl. 22b 2 05). Blue vat dyes are obtained by the reaction of dihydrianthracene (I) or its substitution products with benzoquinone or its substitution products. The reaction may be effected by boiling the reagents in PhCl for several hrs. under reflux. Green dyes are obtained by halogenating the products. The starting materials specified include 3,3'-dichloro-I, m. 217-8°, 2,2'-dimethyl-I, m. 208-9°, 3,3'-dimethyl-I, m. 219°, 1,1'-dichloro-I, m. 208-9°, 3,3'-dimethyl-I, m. 239°, 1,1'-dichloro-I, m. 296°, and 1,1',8,8'-tetrachloro-I, m. 325°; methods of prep. these compds. are indicated.

**Vat dyes.** I. G. Farbenind. A.-G. Fr. 784,504, July 22, 1935. Dyes of the anthraquinonazine series are prepd. by treating  $\alpha$ -amino- $\alpha$ , $\beta$ -dianthrimides with halogenating agents. Thus, 3,3'-dibromo-4'-benzoylaminoanthraquinonedioxazine (dyes vegetable fibers in fast green-blue shades) is prepd. from 4'-benzoylamino-1-amino-2,1'-anthrimide, 4'-amino-3,3'-dibromo-*N*-methylanthraquinonedihydrazine (green-blue shades) from the anthrimide obtained from 1-methylamino-2-bromoanthraquinone and 1,4-diaminoanthraquinone, 3,3'-dibromo-4-hydroxyanthraquinonedihydrazine (blue-green shades) from the anthrimides obtained from 1-amino-2-bromoanthraquinone and 1-amino-4-hydroxyanthraquinones, also a dye from 1-methylamino-2,1'-anthrimide (by methylating 1-amino-2,1'-anthrimide).

**Arylamino anthraquinoneacridone derivatives** Fritz

Raumann (to General Aniline Works) U. S. 2,017,936, Oct. 22. Compd. of the general formula



where X and Y mean aryl radicals of the benzene series, R stands for a radical of the benzene series, forming blue to bluish gray needles, dissolves in concd.  $H_2SO_4$  with a yellow to green coloration, dyeing cotton from an alkali hypsochrome vat clear and strong blue to blue-gray shades fast to light prepd. by a process comprising reacting upon a 1,4-diaroylamino-5-aminoanthraquinone, the aryl group being of the benzene, naphthalene, biphenyl, anthracene or anthraquinone series, with an  $\alpha$ -halobenzoic acid of the benzene, naphthalene, anthracene or an anthraquinone series, which acid may be esterified by a lower aliphatic acid, in the presence of a high-boiling inert org. solvent at a temp. above about  $100^\circ$  and causing acridone ring formation of the reaction product thus obtained. A Cu catalyst and an acid-binding medium are used and a reaction temp. of about  $150-240^\circ$  is suitable. Several examples with details of procedure are given.

**p-Arylene-thiazole- and aryleneoxazole-sulfonic acids.** Max Schubert and Ernst Herderichhoff (to General Aniline Works) U. S. 2,018,513, Oct. 29. *Intermediates for the manufacture of dyes and pharmaceutical products* and which have the general formula  $arylene-X-C(SO_3H)_2$ , where

where arylene means a radical of the  $C_{12}H_8$  or  $C_{14}H_{10}$  series and X stands for S or O, are obtained by treating the corresponding 2-mercapto compds. of the arylene-thiazole and -oxazole series in an alk. medium with a quantity of an oxidizing agent corresponding to at least 3 reactive O atoms. The formed new sulfonic acids are generally colorless powders which are water sol. in contradistinction to the corresponding disulfides, which are obtainable by oxidizing the same 2-mercapto compds. in an acid medium (with nitric acid). When heated with the addn. of an acid such as HCl they split off  $SO_2$ . They form water sol. alkali metal salts which represent generally colorless crystals and yield when reduced with Na amalgam the corresponding fundamental compds. by replacing the sulfonic acid group by H in an analogous manner as with the  $\alpha$ -sulfonic acids of the  $C_{12}H_8$  series. Several examples are given, including the production of benzothiazole-2-sulfonic acid, 6-ethoxybenzothiazole-2-sulfonic acid and 6-benzoylamino-benzothiazole-2-sulfonic acid.

**Anthraquinone derivatives.** See pour l'ind. chim. à Bak. Ger. 618,001, Aug. 30, 1935 (Cl. 22) 3 022. 1-Hydroxyalkylamino-4-arylamino derivs. of an anthraquinone (I) are prepd. (1) by the action of a hydroxyalkyl amine on a 1-arylamino-I substituted in the 4-position with a reactive atom or radical, e. g., halogen or OH, or (2) by the action of an aryl amine on a similarly substituted 1-hydroxyalkylamino-I, or (3) by treating a 1-amino-4-arylamino-I with  $(CH_3)_2O$  or its homologs or alcohols, or (4) by treating a leuco-1,4-bis(arylamino)-I with a hydroxyalkyl amine, or a leuco-1,4-bis(hydroxyalkylamino)-I with an arylamine, or leuco-1,4-diamino-I with an arylamine and a hydroxyalkyl amine, and oxidizing the product in each case, during or after the reaction. The corresponding leuco compds. may also be used in methods (1) and (2), and the products similarly oxidized. Examples are given of the manifold products from (1) 1,3-bis(hydroxyethyl)amino-4-methoxy- or -4-bromo-I and  $\beta$ -phenylene diamine (II), (2) 1-amino-4-( $\beta$ -hydroxyphenylamino) I and (CH<sub>3</sub>)<sub>2</sub>O, (3) leuco-1,4-bis( $\beta$ -hydroxyethylamino)-I and II, (4) leuco-1,4-diamino-I,  $\beta$ -aminophenol, and  $NH-C_2H_4OH$ , (5) leuco-1-hydroxy-4-( $\beta$ -aminophenyl-

amino)-I and  $NH-C_2H_4OH$  (or  $NH-(CH_2)_3OH$ ), (6) leuco-1-amino-4-phenylamino-I and  $NH-C_2H_4OH$ , (7) 1-amino-4-phenylamino-I and phosgene. The products dye acetate rayon in green shades.

**Naphthal derivative.** Chemische Fabrik vorm. Sanden, Swiss 175,539, May 1, 1935 (Cl. 37g). Addn. to 159.4 b. The compd. 2-aceto-8-naphthol-3,6-disulfonic acid is obtained by heating 2,5,8,6-diazomaphtholdisulfonic acid with HI. The compd. is used as an intermediate in the prepn. of dyes and as a drug.

**Hydroxybiphenyl derivatives.** I. G. Farbenind., A.-G. (Oskar Haller and Heinrich Merschel, inventors), Ger. 618,213, Sept. 6, 1935 (Cl. 12a, 16). Arylides from 4-hydroxybiphenyl-3-carboxylic acid (I) and aromatic amines containing  $SO_3H$  or  $COOH$  group are prepd. by standard processes. Examples are given of the prepn. of the anilide, m.  $231^\circ$ , of I and of a condensation product, m.  $267^\circ$ , from 2 mols. of I and 1 mol. of benzamide. The *o*-amide, m.  $155^\circ$ , *p*-toluide, m.  $238^\circ$ , *m*-toluide, m.  $222^\circ$ , *p*-toluide, m.  $244^\circ$ , 2,4-dimethylanilide, m.  $217^\circ$ , *p*-chloroanilide, m.  $260^\circ$ , and 5-naphthalide, m.  $237^\circ$ , of I have also been prepd. The products are useful in producing dyes on the fiber.

**Diazoamino compounds.** I. G. Farbenind., A.-G. (Carl Taube and Ernst Tietze, inventors), Ger. 618,549, July 13, 1935 (Cl. 12g, 10). Addn. to 614,198 (C. 4, 29) (6749). Compds. similar to or identical with these obtainable by the process of Ger. 614,198 are obtained by coupling diazotized substituted aromatic amines with cyanamido-carboxylic acid or its salts.

**Carbazole derivatives.** See pour l'ind. chim. à Bak. Swiss 175,573-5, June 1, 1935 (Cl. 37f). Addn. to 171,365. Addn. products of aminoanthraquinone and nitrochlorobenzene are treated with a diazotizing agent at raised temps. The structural formulas of the starting compds. and the carbazole derivs. are given. The derivs. are used as dye intermediates.

**N-Nitroamines.** See pour l'ind. chim. à Bak. Swiss 177,018, June 17, 1935 (Cl. 37a). The compd. 1-benzylamino-4-N-nitroamino-2,5-dimethoxybenzene is obtained by treating the N-nitroamine of 1-amino-4-nitro-2,5-dimethoxybenzene with a reducing agent to convert the 4-nitro group to an amino group and benzylating the product. The compd. is used as a dye intermediate.

**Intermediates and dyes.** Imperial Chemical Industries Ltd. Fr. 754,229, July 22, 1935.  $\beta$ -Arylamino-arylamines of 2,3-hydroxynaphthoic acids are prepd. by causing an aromatic carboxylic halide with a  $\beta$ -amino-arylamide of a 2,3-hydroxynaphthoic acid or by condensing a monoaroyl- $\beta$ -arylenediamine on a 2,3-hydroxynaphthoic acid halide. The products are coupling components for the production of azo dyes. Examples are given of the prepn. of benzoyl- $\beta$ -aminobenzene (m.  $241^\circ$ ), 2'-chloro-4'-benzoylaminoaniline (m.  $280-17^\circ$ ), 2'-methyl-5'-chloro-4'-benzoylaminoaniline (m.  $268^\circ$ ), 2'-methyl-5'-chloro-4'-benzoylaminoaniline (m.  $253^\circ$ ) and 2'-methyl-4'-benzoylaminoaniline (m.  $230^\circ$ ), of 2,3-hydroxynaphthoic acid, and of dyes prepd. with these.

**Intermediates for dyes.** See pour l'ind. chim. à Bak. Fr. 784,543, July 22, 1935. N-Nitroamines of primary aromatic amines, nitrated in the ring, are treated with reducing agents so that the  $NO_2$  group fixed directly to an aromatic ring is transformed. The products obtained may be acetylated. Examples are given of the prepn. of the Na salts of the nitroamine of 1-amino-4-nitro-2,5-dimethoxybenzene, the mono-N-nitroamine of 1,4-diaminobenzene, 1-nitroamino-2-methyl-4-aminobenzene, 1-nitroamino 2,5-dichloro-2-aminobenzene, 1-N-nitroamino-2-amino-4-methylbenzene (I), N-nitroamine of 1-amino-4-nitro-2,5-dimethoxybenzene, mono-N-nitroamine of 1,4-diamino-2,5-diethoxybenzene, N-nitroamine of 1-amino-4-benzoylamino-2,5-diethoxybenzene, and the dinitroamine of 4,4'-diamino-3,3'-dimethylazobenzene, also 1-nitro-5-methylbenzotriazole, m.  $91.2^\circ$ , by diazotizing I.

**Dye intermediates.** See pour l'ind. chim. à Bak. Swiss 176,020, June 1, 1935 (Cl. 37a). 6-Methylphenanthrene is treated with  $H_2SO_4$  of concn. not exceeding

95% and Br, the reaction not being allowed to proceed beyond the monobromo stage. On condensing the product with 1-aminoanthraquinone, and fusing the product with alc. KOH, an olive vat dye is produced. Thus, by treatment with S gives Bs-1-Bs-1'-dibenzanthronyl sulfide, which, if fused with alc. KOH gives a violet-blue vat dye.

**Dyeing cellulose derivatives; acridine dyes.** E. I. du Pont de Nemours & Co. Brit. 432,360, July 25, 1935. See U. S. 2,005,303 (C. A. 29, 5282).

**Dyeing with vat dyes.** I. G. Farbenind. A.-G. Fr. 784,276, July 22, 1935. Basic compds resistant to alkalis, derived from tri- or quinquivalent N, quinquivalent P or quadrivalent S and contg. at least one aliphatic, cycloaliphatic or aromatic radical of at least 6 C atoms, is incorporated in the alk. dye vat. Examples are laurylamine, dimethylethylamine, oleyldiethanolamine, decylmonoethanolamine, cetyldiethanolamine, dodecylethylenediamine, condensation product of copra oil with triethylenetetramine, reaction products of polyglycerols or their halogen compds with *m*-phenylenediamine, reaction products of halo paraffins with NH<sub>3</sub> or amines, as well as their salts or derivs. See oral examples of dyeing are given.

**Coloring animal fibers with mordant dyes.** Philippe Brandt (to Durand & Huguennin S.A.) U. S. 2,018,426, Oct. 22. For producing prints or dyes on animal fibers with acid mordant dyes, fast to rubbing and of deep intensity, there is added to an otherwise usual printing paste or padding soln. at least 4% of a nonvolatile acid or salt such as oxalic acid or NH<sub>4</sub> oxalate for 3% of dye, so that the acid condition is maintained throughout steaming, and the fabric is treated with this compn. and the dye is fixed on it by steaming for about 8 min.

**Coloring organic substitution derivatives of cellulose such as cellulose acetate, etc.** Edmund Stanley, Henry C. Olpin and Geo. H. Ellis (to Celanese Corp. of America) U. S. 2,017,995, Oct. 22. The material is treated with a relatively insol. coloring compd. in the form of an aq. dispersion obtained with the aid of a sulfonated condensation product of a higher alc. such as cetyl alc. and a cyclic compd. such as naphthalene.

**Printing fabrics.** Soc. pour l'ind. chim. à Bâle. Swiss 177,231, Aug. 1, 1935 (Cl. 24c). A stable compn. for printing on cellulose or animal fibers contains a vat dye and a salt of monoalkylaminobenzene-3-sulfonic acid. In an example, a compn. consisting of tribromo-2-thionaphthene-2-indoleimido, potash thickening, Na monoethylaminobenzene-3-sulfonate and Na formaldehydesulfoxylate, is used for printing on cotton fabrics. Other examples are given.

**Stripping dyes from textile materials.** John G. Evans and Leslie G. Lawrie (to Imperial Chemical Industries Ltd.) U. S. 2,019,124, Oct. 29. Textile materials dyed with alizarin, basic or vat colors are treated with a liquor contg. a substance such as cetyltrimethylammonium bromide or other amine or amine salt having an aliphatic radical which contains not less than 10 C atoms.

**Apparatus for treating articles with dyes and other liquids.** A. E. Hawley & Co. Ltd. and Albert E. Dawson. Brit. 432,317, July 24, 1935. Work-supporting elements, to which articles or material to be treated are attached, are moved automatically 1 or more at a time from 1 end of the dye tank or vat to the other and back again in reverse order, repeatedly by endless conveyor means, to trail the articles or material through the liquid.

**Dyeing-jigs.** Philbert Deck. Brit. 432,084, July 19, 1935.

**Breaking and scutching machines for preparing flax, hemp and like bast fibers.** Hugh J. Stewart, Wm. Gardner and Matthew Russell. Brit. 432,210, July 23, 1935.

**Artificial fibers.** Rudolph H. Kagi (to Heberlein Patent Corp.) U. S. 2,019,185, Oct. 29. An artificial silk yarn such as one formed from cellulose acetate or viscose is super-twisted, the twist is allowed to set and the yarn is then untwisted to obtain a product having a lasting curliness.

**Synthetic fibers.** Heberlein & Co. A.-G. Swiss 170,899, July 16, 1935 (Cl. 10c). Wool-like fibers are formed by over-twisting artificial fibers, winding and leaving for some time. The fibers are then untwisted, having acquired a wool-like nap with a soft touch.

**Apparatus for testing the suitability for weaving of threads.** Paul Faume U. S. 2,018,971, Oct. 29. Various mech. and operative details.

**Rayon yarns.** British Celanese Ltd., Robert W. Moncrieff and Frank B. Hill. Brit. 432,280, July 24, 1935. Artificial spun yarn of staple lengths of artificial filaments are stretched beyond the elastic limit so that the staple lengths of which the yarns consist are permanently elongated. The yarns may be made from cellulose acetate or other org. deriv. of cellulose and may be stretched in the presence of softening agent. The yarns may be assembled in warp form and stretched as described in Brit. 371,461 (C. A. 27, 3089) and, after stretching, they may be slightly shrunk as described in Brit. 397,504 (C. A. 27, 4102).

**Apparatus for drying wound rayon threads.** Vereinigte Glanzstoff-Fabriken A.-G. Ger. 616,555, July 31, 1935 (Cl. 29a 608). Addn. to 613,242 (C. A. 29, 6219).

**Conditioning yarn in knitting machines by treatment with liquid.** Robert N. Apprich (to Textile Machine Works) U. S. 2,018,627, Oct. 22. Various details of app. and operation.

**Apparatus for treating threads with liquids.** Charles W. Strzalkowski and Edmund J. Wendt U. S. 2,019,201, Oct. 29. Structural, mech. and operative details.

**Fibron solutions.** Emil Hubert, Wilhelm Inon and Herbert Mahn (to I. G. Farbenind. A.-G.) U. S. 2,019,225, Oct. 29. A soln. suitable for spinning, etc., is prep'd by dissolving fibron at a temp. not exceeding 30° in an aq. soln. contg. a major amt. of a water-sol. metal thioacetate such as that of NaCNS which is itself incapable of dissolving fibron at a temp. not exceeding 30°, to which has been added a minor amt. of another thioacetate of Na, Li, Ba, Mg, Zn or Mn.

**Textile materials.** Heberlein & Co. A.-G. Fr. 784,507, July 22, 1935. Vegetable fibers are treated with an ammoniacal soln. of Cu oxide, the fibers being in the crude state or covered with their natural incrustations. A wetting agent such as a sulfo oleate may be added to the bath and the treatment may be followed by mercerization or free NaOH may be added to the bath.

**Textile materials.** Tootal Broadhurst Lee Co.-Ltd. Fr. 784,556, July 22, 1935. Textile material contg. an insol. substance, preferably a synthetic resin, is submitted to a swelling treatment, e. g., with a caustic alkali.

**Apparatus for testing the tensile strength of silk, rayon, etc.** David C. Scott (to Henry L. Scott Co.) U. S. 2,018,385, Oct. 22. Various structural, mech. and operative details.

**Rayon resembling wool.** Georges Heberlein (to Heberlein Patent Corp.) U. S. 2,019,183, Oct. 29. A viscose yarn is given a super-twist, placed on bobbins, moistened at a high temp., dried, untwisted so that a soft woolly material is produced having a lasting curliness, and the curliness is increased and the yarn is rendered substantially nonshrinkable by wetting treatments by loosely supporting the yarn in a wet steam atm. at about 3 atm. pressure for about a half hr.

**Artificial wool.** Heberlein & Co. A.-G. Ger. 618,030, Aug. 31, 1935 (Cl. 29a 609). Untwisted or normally twisted rayon threads are wound with a twist at least 4 times the normal, moistened at a high temp., e. g., by steaming, and dried. They are then retwisted to beyond the zero point in the opposite direction. Curly products which resemble wool are obtained.

**Vegetable fibers treated to resemble wool.** Ralph H. McKee and Earle H. Morse. U. S. 2,018,276, Oct. 22. Vegetable fibers such as jute fibers are subjected to oxidizing treatment at a pH between 5 and 8, and are then crumpled by subjecting them to the action of a NaOH soln. of about 10% strength.

**Cotton bands.** Chemische Fabrik G. Zimmerli A.-G. Swiss 177,226, Aug. 1, 1935 (Cl. 24f). Tightly pressed

cotton fabric is swelled with  $H_2SO_4$  and  $NaOH$ , coated with opaque paper pulp and calendered to form a band.

**Producing figured effects on textiles** Heberlein & Co. A-G Brit 432,388, July 25, 1935. Figured effects accompanied by uniform stiffening are produced by printing the fabrics with cellulose-*contg* soles mixed with pigments, fixing the printing (by drying), parchmentizing to stiffen the fabric and render the nonprinted portions transparent, washing and stretch-drying. Suitable pigments are  $BaSO_4$ ,  $Ti$  white, metal oxides, colored varnishes with body colors, they must be insol in the parchmentizing agent. In an example, a mercerized fabric is printed with a mass comprising cellulose acetate,  $Ti$  white, ethylene chlorohydrin and  $H_2O$ , dried, drawn for 8 sec through 54°Bé  $H_2SO_4$ , washed and stretch-dried.

**Imidazole derivative** Soc. pour l'ind. chim. à Bâle Swiss 175,673, May 16, 1935 (Cl 36p). The deriv. *N*-lauryl- $\mu$ -methylbenzimidazole is obtained by treating  $\mu$ -methylbenzimidazole with an ester of lauryl alc. The deriv. is used as a softening agent for viscose silk.

**Bleaching textile fibers** I. G. Farbmend A-G Fr 784,524, July 22, 1935. The chlorinated water, preferably said or supersaid, is caused to act on the material to be bleached directly after its prep. App. is described in which the bleaching and chlorination of the water form a closed system. Cf. C. A. 29, 3358.

**Apparatus for treating yarn packages with liquids such as sizing liquids, etc.** Stephen M. Fulton and Geo. C. Tyce (to Celanese Corp. of America) U. S. 2,018,017, Oct. 22, 1934. Structural and operative details.

**Mercerizing** Thomas Wm. Holt, Archibald S. Kilpatrick and John F. W. Stuart Brit 432,483, July 29, 1935. Textile fibers, e. g., cotton, wool, in lap or silver form are mercerized by treating with wetting out and mercerizing agents, mixed or otherwise, then neutralizing and finally washing. A suitable bath comprises cresylic acid, camphor oil and caustic lye of 35°Tw. App. is described.

**Mercerizing lyes** I. G. Farbmend A-G Fr 784,359, July 22, 1935. Mixts of phenols, halogenated phenols or sulfonates with amides of sulfonic acid, e. g., chloroxylenol and *p*-toluenesulfonobutylamide, are used as wetting agents in mercerizing lyes.

**Washing textiles** Ehrhart Franz Brit 432,018, July 15, 1935. Impurities are removed from raw or manuf. waste textile material by treatment with a  $H_2O$ -insol. carboxylic acid, e. g., a fatty acid, in amt. not less than half the wt. of the material, removal of the bulk of the acid, treatment with  $NH_4OH$  or alkali to convert the remaining acid into a soap and simultaneous or subsequent washing of the soap-*contg* material. In examples, (1) raw wool *contg* pitch is washed to remove sand, loose dirt and easily removable fat and is then soaked for 5-20 hrs. in twice its wt. of com. olein at normal or slightly raised temp., squeezed or centrifuged and washed in a weak alk. bath at 50°, and (2) raw cotton soiled with resin or coloring matter is soaked for 20 hrs. in twice its wt. of arachis-oleic acid, squeezed, placed in 5% potash lye, centrifuged and washed in a 2nd alk. bath.

**Bath for improving textiles, etc.** Soc. pour l'ind. chim. à Bâle Swiss 177,226, Aug. 1, 1935 (Cl 24a). Textiles, leather, paper, etc., are improved by treatment with an aq. bath *contg* dil. alkali hydroxide or mineral acid and products of the general formula  $MOC(O)AYZ$ , in which A is an org. residue, Y a  $-C(O)O-$  group, Z an aliphatic residue with at least 8 C atoms and M an alkali metal or  $NH_4$ . Thus, the  $NH_4$  salt of the product obtained by heating phthalic acid anhydride with a mixt. of hexadecyl and octadecyl alc. is dissolved in hard water and Glauber's salt added. Viscose silk, heated in the resulting bath, acquires a soft nap. Other examples are given.

**Textile lubricants** British Celanese Ltd. Brit. 431,964, July 18, 1935. A textile lubricant comprises a sub-

stantially  $H_2O$ -insol. oil or fat or higher fatty acid and a small proportion of an aromatic compd. *contg* an unsat. aliphatic substituent group, e. g., vinyl, propenyl, allyl or crotonyl. The aromatic compd. may contain other groups, e. g., OH, ether, halo or  $SO_2$  groups. Substituted  $PhOH$ , cresol, naphthol, catechol and resorcinol, e. g., or substituted phenol ethers such as anisole and phenetole may be used, and especially the propenyl, etc. Derivs. of aromatic compds. *contg* both ether and OH groups, e. g., guethol and guaiacol. Animal or vegetable oils or fats, e. g., olive, castor, coconut, neatfoot and, in general, glycerides of oleic, stearic, palmitic or other higher fatty acids, or the acids themselves may be used. Assistants, e. g., mineral lubricating oils, glycol, diethylene glycol, glycerol, etc., may be employed. The lubricant may be added to the spinning soln. or to the formed material.

**Waterproofing wool and other animal fibrous material.** Studiengesellschaft für Faserveredlung m. b. H. (formerly Deutsche Kunstseiden-Studiengesellschaft m. b. H.) Brit. 431,670, July 12, 1935. See Fr. 777,714 (C. A. 29, 41887).

**Benzimidazolium derivative** Soc. pour l'ind. chim. à Bâle Swiss 175,026, Apr. 16, 1935 (Cl 36p). The deriv.  $\mu$ -heptadecyl-*N*-benzyl-*N'*-ethylbenzimidazolium chloride is prep'd by the action of  $EtOH$  on  $\mu$ -heptadecyl-*N*-benzylbenzimidazole-HCl. The compd. is used in the textile industry. Cf. C. A. 29, 3350<sup>1,2</sup>.

**Triarylmethane derivatives** I. G. Farbmend A-G (Karl Berres and Walter Retter, inventors). Ger. 618,033, Aug. 31, 1935 (Cl 12g 20 04). Substituted triarylmethanesulfonic acids are prep'd by condensing 1 mol of a sulfonic acid of an aromatic aldehyde with 2 mols of an alkyl, cycloalkyl or aralkyl ether of a phenol or naphthol *contg* no unethersified OH group. The reaction may be effected at a temp. below 50° in  $H_2SO_4$  of 87% concn. Examples are given in which (1) *o*-sulfobenzaldehyde and *p*-chloroanisole yield 2,2'-dimethoxy-5,5'-dichlorotriphenylmethane-2'-sulfonic acid, (2) *p*-sulfobenzaldehyde and anisole yield 4,4'-dimethoxytriphenylmethane-4'-sulfonic acid, (3) *o*-sulfobenzaldehyde and *p*-chlorophenyl benzyl ether yield 2,2'-dibenzylxy-5,5'-dichlorotriphenylmethane-2'-sulfonic acid, (4) *o*-sulfobenzaldehyde and cyclohexyl phenyl ether yield 4,4'-dicyclohexyloxytriphenylmethane-2'-sulfonic acid. The products are useful as reserves in printing textiles and as mothproofing agents.

**Impregnating textile materials with rubber latex** Leon S. M. Lejeune and Jean E. C. Bongrand U. S. 2,019,420, Oct. 29. For facilitating the penetration of the latex, the  $pH$  of the latex and of the textile material are preliminarily adjusted to about the same value so that coagulation of the latex at the interfaces of the materials is avoided.

**Carpet backing** Albert W. Holmberg (to U. S. Rubber Co.) U. S. 2,018,524, Oct. 22. The backing of a carpet is coated with a mixt. of raw starch and rubber, etc.

**Upholstery padding** Joseph A. Howard Brit 431,353, July 8, 1935. For the manuf. of upholstery padding consisting of a mass of fibers held together by an adhesive, e. g., rubber, as described in Brit. 341,939 and 355,004, the fiber is brought to the form of a web by passage downwardly between extending surfaces, of which 1 is stationary. The other surface may also be stationary, or it may consist of an endless band actuated so as to impart the desired profile to the padding. After passage between the surfaces, the latex may be applied either by spraying or by passage through a bath. Several applications of latex may be made and, after each application, the web is dried or dried and vulcanized so that, by reason of the accretion of strength, the web may be passed between manual rollers after a subsequent application of latex. A coagulant for the latex, e. g.,  $Ca(NO_3)_2$ , powd.  $CaO$ , may be applied to the fibers before their formation into a web, or to the web as it leaves the die.

## 26—PAINTS, VARNISHES AND NATURAL RESINS

A. H. SABIN AND C. H. ROSE

Investigation of color permanency by the Philadelphia Paint and Varnish Production Club. Anon. *Paint Varnish Production Mgr.* 13, 22-3, 26, 28-9 (Nov., 1935); *Am. Paint J.* 19, *Convention Daily*, 16, 17, 18, 19 (Oct. 29, 1935).—An attempt to develop a practical method for detg., evaluating and recording color changes occurring in paints. Sep. gradings appear necessary for the 3 color properties: (1) hue, or dominant wave length, (2) value of brightness and (3) purity, chroma or satn., and also for the color uniformity of the surface. Color change may be due to one or more of a no. of several different causes among them (1) photochem changes caused by actinic light, (2) changes in the exposed surface of the film, (3) changes in the relative indices of the film components and (4) chem reactions resulting in changes in compn of colored pigment constituents. The permanency of org. color is tremendously impaired by the action of any other pigment. Also in *Paint, Oil & Chem Rev* 97, No. 21, 95-9 (1935). W. H. Boynton

Paint in the food factory T. Hedley Barry. *Paint Manuf.* 5, 297-300 (1935).—Wood, metal, plaster, cement and brickwork all require special attention and skilled application is necessary. W. H. Boynton

Paints and preservatives for engineering structures. C. I. Dodd. *Kansas Water Sewage Works Assoc.* 4, 18-23 (1934-5).—A discussion of the choice of and methods of application of the most suitable protective paints for wood, steel and concrete surfaces, especially those in water or sewage-treatment plants where protection from H<sub>2</sub>S, Cl<sub>2</sub>, etc., is necessary. M. G. Moore

Bibliography of articles on weathering of paints, varnishes and lacquers. L. M. Gugelman. *Am. Paint J.* 19, *Convention Daily*, 18, 20 (Nov. 2, 1935).—Contributions to the tech. literature from 1924 to 1934 inclusive are listed. W. H. Boynton

Use of metallic soaps in paint meets new problems. F. J. Licata. *Am. Paint J.* 19, *Convention Daily*, 19 (Oct. 29, 1935). W. H. Boynton

The design of flat wall paints. John C. Gehant. *Paint, Oil & Chem Rev.* 97, No. 18, 16-18 (1935).—Modern requirements and formulations are discussed. J. W. P.

Examination of antirusting paints. A. V. Blom. *Paint Manuf.* 5, 292-6 (1935).—See C. A. 29, 2001.

Measurement of body color for practical purposes. Martin Witte. *Chem. Fabrik* 1935, 418-20.—The methods of Munsell (*cf. C. A.* 17, 3837) and Ostwald (*cf. C. A.* 11, 1083; 15, 1247) are compared and discussed. J. H. Moore

Pigment particles. G. A. Campbell. *Oil Colour Trades J.* 88, 1181-3 (1935).—Pigments vary enormously in their surface properties—whether finished off by grinding or by pulverizing. Grinding problems, shape of particle, pigment and the vehicle, and grinding and oil absorption are discussed. W. H. Boynton

Colloid chemistry of pigments with special reference to zinc oxide and basic lead carbonate. F. V. Evans. *J. S. African Chem. Inst.* 18, 36-41 (1935).—A general discussion of pigment particle size and its effect on plasticity; importance of "wetting" all surfaces, etc. White lead partially saponifies the acid in oil, causing the paint to have a "creamy" flow. The combination of ZnO and white lead in a paint prevents the brittle-film tendency of ZnO and the "chalking" tendency of white lead. B. E. A.

Development of zinc pigments keeps pace with consumer progress. R. M. Neumann. *Paint, Oil & Chem Rev.* 97, No. 21, 56-7, 76 (1935).—A brief review of recent progress. J. W. Perry

Zinc pigments in relation to southern climatic conditions. S. Werthan. *Paint, Oil & Chem Rev.* 97, No. 10, 16, 18, No. 17, 11 (1935).—See C. A. 29, 7100.

Zinc white problems—economical and practical. A. Foulon. *Farbe u. Lack* 1935, 520-2.—A discussion emphasizing the nonpoisonous character of zinc white and the excellent results obtained when it is properly used in paint. J. W. Perry

Testing lithopone. W. Taut. *Farbe u. Lack* 1935, 531-2.—A discussion of various difficulties encountered in testing shade, texture, oil demand and consistency of lithopone-in-oil dispersions. J. W. Perry

Recent developments in aniline inks. Alfred McNeil. *Am. Ink Maker* 13, No. 10, 15-17 (1935). E. H.

Recent developments in the utilization of soybean oil in paint. W. L. Burlison III. *Aggr. Expt. Sta., Circ.* 438, 3-8 (1935).—A survey of recent developments indicating the increasing use of the oil in the paint and varnish industries. C. R. Fellers

A study of the industrial value of the Argentine flaxes. Roberto E. Faura. *Bol. ministerio agr. nacion* (Argentina Rep.) 36, 245-8 (1934).—The linseed oil content of 3 North American varieties of flaxseed varied between 35.12 and 38.32%, that of 13 Argentine varieties between 35.36 and 41.35%. The I no. and n of the 3 North American oils ranged from 174 to 179 and 14820 to 14840, resp., that of the Argentine varieties from 174 to 181 and 14831 to 14844. A general discussion of acidity, color, compn. and drying power is given. C. W. Whitaker

Economic and commercial factors in development of a domestic tung-oil industry. C. C. Concanon. *Am. Paint J.* 20, 18, 20, 50, 52; 54, 56 (Oct. 28, 1935). W. H. Boynton

Importance of tung oil to the varnish industry. H. A. Gardner. *Am. Paint J.* 20, 18, 48, 50 (Oct. 21, 1935).—The most likely material for replacing tung oil is the alkyl type of resin, but no oil or resin product at present exactly matches tung oil in all formulations, particularly in waterproof spar varnish. Oiticica and perilla oils may replace wood oil and linseed oil with fairly parallel results if the waterproof requirements of the varnish are not too rigid. W. H. Boynton

The tung-oil tree in Texas. P. R. Johnson and S. H. Yarnell. *Tex. Agr. Expt. Sta., Circ.* 75, 16 pp. (1935).—Soils for successful cultivation of tung-oil trees must have good drainage and at least moderate aeration. Southeastern Tex., with the exception of occasional cold injury, will grow this crop. Yields of nuts and the oil constn. of Fla. tung oil are given. C. R. Fellers

Perilla oil, center of interest, is assuming leadership based on its special features. Otto Eissenschind. *Am. Paint J.* 20, 18, 18 (Oct. 14, 1935).—Perilla oil has definite properties differing from those of linseed oil. It bodies more quickly than linseed oil and has to be watched more carefully, but is not nearly as delicate a product as China wood oil. It works well with synthetic resins but does not yield as waterproof or as hard a film as China wood oil. W. H. Boynton

Cincinnati-Dayton-Indianapolis Club presents a study of treated wood oils. V. N. Sheets. *Am. Paint J.* 19, *Convention Daily*, 9 (Oct. 29, 1935); *Paint Varnish Production Mgr.* 13, 17, 18-19, 37 (Nov. 1935); *Paint, Oil & Chem Rev.* 97, No. 23, 70, 72.—Four treated wood oils in modified phenolic resin varnishes with 20- and 33-gal. oil lengths are compared with raw wood oil in similar varnishes. In addition, treated and raw wood-oil films are compared for relative gasproofness. Conclusions: (1) treated oils are inferior to raw oils in cooked varnishes as to drying and durability; (2) treated oils have less gassing tendency at the expense of drying; (3) treated oils have lost some of the valuable characteristics of the raw oil making them more closely related to linseed and perilla oils; and (4) treated oils with progressive fume proofness have progressing increase in acid value, drying time and miscibility with progressive darkening. W. H. B.

Effect of water on drying-oil films. J. Milligan. *Oil Colour Trades J.* 88, 1329, 1331 (1935).—Drying-oil films have been found to be capable of destructive liquefaction under prolonged conditions of heat and high humidity—

the resulting product being a sticky relatively nondrying mass. It will not dry on glass at room temp. over a long period. The mode of reaction between water and dried oil films is first hydrolysis of the more liquid phase in the film and then the gradual decomposition of the solid phase. Studies of the linocyn stability have yielded information on the relative water-resisting powers of linocyn made under different conditions. Different types of driers yield different results.

W. H. Boynton  
 Lacquer solvents in commercial use. Arthur K. Doolittle. *Ind Eng Chem* 27, 1169-79 (1935).—One hundred lacquer solvents are classified according to (1) mol structure and (2) evapn rates. The properties given under (1) include mol wt, boiling range, m. p., flash p., vapor pressure, sp. gr., wt./gal., n<sub>D</sub>, expansion coeff., purity of com. product, diln ratios, soly in H<sub>2</sub>O and soly of H<sub>2</sub>O in the solvent. A review of the theory of solvent action and the pertaining literature are also given. J. A. S.

Straight run petroleum thinners assume renewed importance. E. G. Richardson. *Am Paint J* 19, *Convention Daily*, 13-14 (Oct 29, 1935). W. H. Boynton  
 Triethanolamine in the resin, lacquer, paint and insulating industry. Emil J. Fischer. *Farbe u. Lack* 1935, 519, 533-5.—A review of recent literature and patents. Thirty references.

J. W. Perry  
 Replacing benzene and benzine with alcohol. L. Kern. *Farbe u. Lack* 1935, 507-8.—The possibility of using increased amts of alc. in lacquers is illustrated by a no. of examples, and the necessity of saving benzene and benzine in Germany pointed out.

J. W. Perry  
 New materials for coatings. C. A. Klebsattel. *Paint, Oil & Chem. Rev.* 97, No. 21, 34, 36, 38 (1935).—A brief review of recent progress.

J. W. Perry  
 Changes in varnish making due to shortage of wood oil. W. Krumbhaar. *Am Paint J* 19, *Convention Daily*, 19, 20 (Oct 29, 1935). W. H. Boynton

Old and new nitrocellulose and combination lacquers. Fritz Zimmer. *Nitrocellulose* 6, 180-2 (1935).—A review. E. M. Symmes

Differences in tautening power of nitrocellulose and acetylcellulose dopes with particular consideration of the effect of moisture. R. S. Dantuma and A. M. Mees. *Farben-Ztg* 40, 744-5 (1935).—The tautness of stretched aeronautical linen decreased less on exposure to moisture if treated with nitrocellulose dope rather than with acetylcellulose dope. Addn of pigments to the dopes caused moisture to have a somewhat less detrimental effect on tautness. Applying further protective coatings, on the other hand, had little effect. Loss of tautness appeared to be assoc. with absorption of moisture by the dope film.

J. W. Perry  
 Fundamental physical properties of lac. I. Mechanical properties. Lal C. Verma. *London Shellac Research Bur. Tech. Paper* No. 3, 28 pp (1935). II. Thermal properties. *Ibid.* No. 4, 20 pp.

E. H.  
 Plasticizers in nitrocellulose lacquers. Alfred Kraus. *Paint Varnish Production Mgr.* 13, 16, 18 (Aug. 1935); 20-1, 22-3, 24, 26 (Sept. 1935).—A brief review of the various properties of the different plasticizers, examd as to their ability to gelatinize the nitrocellulose as well as their effects upon the viscosity of nitrocellulose solns. The most efficient plasticizer is the one which causes the greatest elongation with the lowest decrease in tensile strength per unit quantity added. It is concluded that the properties of mixts of plasticizers can be calcd with the sufficient accuracy from the known properties of the components. The action of ultraviolet upon the paint film is usually noticed as yellowing and as a change in film strength with a definite, yet very complex, relationship between the 2 phenomena. The degree of yellowing depends upon the nature of the plasticizer, its concn in the film, and the intensity and duration of exposure. The volatility of the plasticizers was detd and losses in wt. after 7 days' drying at 100° were tabulated. The influence of plasticizers in the cold resistance of lacquer films and that in water resistance are indicated.

W. H. Boynton  
 New England Club studies corrosive action of scrubber

water on coatings. A. F. de Ravignon, et al. *Am. Paint J.* 19, *Convention Daily*, 12-13 (Oct. 30, 1935); *Paint Varnish Production Mgr.* 13, 14-15 (Nov., 1935); *Paint, Oil & Chem. Rev.* 97, No. 23, 78-80.—Conclusions drawn after examn of a no. of types of vehicles are: (1) for tung oil and modified oil varnishes, the longer the oil length the greater is the resistance to corrosion; (2) Vinylite-type resin shows excellent corrosion-resisting properties; (3) a short coumar varnish showed better resistance to corrosion than any other clear vehicle tested; (4) a lacquer film, although not affected very much by the scrubber water, showed poor adhesion to the metal panel, peeling off in sheets; (5) several types of vehicles, having excellent corrosion-resisting properties under many other conditions, were not effective when used as clears, in preventing the corrosive action of scrubber water; and (6) of pigments ground or mixed into a common 100% phenolic vehicle and used as a primer the specially prepd. mica gave better results than all others used, red lead being slightly less efficient.

W. H. Boynton  
 Natural resins. Wladyslaw Stefanowski and S. Kraczkiewicz. *Przemysl Chem.* 19, 152-3 (1935).—Resins collected from the forests of Poland were analyzed and compared with elem.

Resin	Acid value	Sapon value	Esterification value	I value
Pine	ab 130	about 160	about 30	about 85
Fir	ab 65	about 75	10-15	160-165
Larch	65-70	115-130	50-60	about 130
Elem.	10-20	25-45	15-25	about 50
Spruce	120-125	115-120	about 5	about 160

Another table gives these values for esters collected in various parts of the country.

A. C. Zachlin  
 The technique of using Brazil Jutahucia resin. Carl Busser. *Paint, Oil & Chem. Rev.* 97, No. 20, 18 (1935).—Details are given of a method for melting or running Jutahucia, which behaves very much like kauri gum.

J. W. Perry  
 Congo copal esters, their production and use. H. Kern mer. *Farbe u. Lack* 1935, 435-6.—Production precautions and advantages are discussed.

J. W. Perry  
 Utilization of resinous products. Georges Brus. *Bull. inst. pin* 1935, 193-200.—An address discussing the directions along which the French turpentine and resin industry should be developed to find an outlet for their excess production due to loss of foreign markets and general decrease in world consumption of these products.

A. Papineau-Couture  
 Resin-manufacturing equipment. John McE. Sander-son. *Paint, Oil & Chem. Rev.* 97, No. 21, 42 (1935).—An illustrated discussion.

J. W. Perry  
 Tung-oil tree (Newell, et al.) 15 Fireproofing paints (Fr. pat. 784,649) 13. Rubber compns [used as ingredients of paints] (Brit. pat. 432,032) 30. CaSO<sub>4</sub> [for use with pigments] (U.S. pat. 2,018,955) 18. Polynuclear phenols and nitration and sulfonation products [as paints and varnishes] (Brit. pat. 431,945) 10. Cellulose ester films, lacquers, etc. (Brit. pat. 432,404) 23. Emulsions [as paints, color varnishes and printing inks] (Brit. pat. 431,642) 13.

Paints Metallgesellschaft A-G (Oskar Schöber, applicant in U.S.). Fr. 784,448, July 22, 1935. A paint for metals, etc., contains 25-60% of an Al-Si pigment in a state of such fine division that it passes, at least for the greater part, through a sieve of 10,000 mesh per sq. cm., and about 25-70% of one or more binders, such as boiled linseed oil or wood oil. A diluent about 25 and an inert filler about 25% may also be present. The Al-Si preferably contains Al about 30-68%.

Apparatus for preparing metal powders for paints. Erwin Kramer. Brit. 430,777, June 25, 1935. See Fr. 778,609 (C. A. 29, 43589).

Roller-and-breast mills for paints, etc. Anciens établissements le Clezio. Brit. 431,530, July 10, 1935.

**Pigments.** Gustave Widmer (to Société pour l'ind. chim. à Rile). Brit. 171,168, July 2, 1935. A pigment powder that is used in the manuf. of oil colors, printing colors, varnishes and inks contains a carbamide- $\text{CH}_2\text{O}$  condensation product, e. g., those from  $\text{CH}_2\text{O}$  and urea, thiourea, dicyandiamide, biuret, guanidine, guanylurea, cyanuric acid or phenylurea, which has been brought by heat treatment into an insol. and nonplastic condition, together with a pigment, e. g., ochre, amber, lampblack,  $\text{Fe}_2\text{O}_3$ , Prussian blue, ultramarine, chrome green, indigo and anthraquinone dyes, 5 dyes, insol. azo dyes or pigments that are themselves color lakes, which under the prevailing conditions of the manuf. of the powder is not converted into a lake. In examples, (1) Indanthrene blue GGS-L is ground with a varnish consisting of a soln. of a urea- $\text{CH}_2\text{O}$  condensation product contg. added thiourea, the mixt. is heated 4 hrs. at  $140^\circ$  and the product reduced to a fine powder, (2) lampblack is suspended in  $\text{H}_2\text{O}$ , the varnish of example (1) is added and dil.  $\text{HCl}$  stirred in, the curdy ppt. enclosed the lampblack and, after filtration, the product is dried at  $80^\circ$  and pulverized, and (3) a heat-treated carbamide- $\text{CH}_2\text{O}$  condensation product is finely powd. and ground with indigo.

**Emulsions for use as inks or paints.** The Twichell Process Co. Ger. 618,241, Sept. 5, 1935 (Cl. 22g 2 01). See U. S. 1,906,042 (C. A. 27, 4627).

**Printing Ink.** August J. Gautsche. U. S. 2,018,040, Oct. 22. A printing ink for lithographic use consists of an offset ink each lb. of which is mixed with about 0.5-2.0 oz. (according to the brilliancy desired) of a compound formed from a hard, resin-like phenol-condensation product 4-5, China wood oil 4, and wool grease 4 parts.

**Lacquer.** Jean Gsell. Swiss 170,071, June 17, 1935 (Cl. 54b). A lacquer for skin consists of 6 parts of collodion wool, 4 parts of shellac, 6 parts of  $\text{AcOAm}$ , 3 parts of  $\text{AcOH}$ , 1 part of spirits and 5 parts of another solvent.

**Coating compositions.** Harold A. Auden and Hanns P. Staudinger. Brit. 432,025, July 10, 1935. The compns. contain an org. ester of cellulose, particularly

cellulose triacetate (I), and 1 or more esters of pyruvic or l-valine acid, the esters used having a h. p. of at least  $150^\circ$  under normal pressure. In examples, the compns. consist of (1) I, methylcane chloride (II),  $\text{AmOH}$ , Bu valinate and resin, and (2) I, II,  $\text{EtOH}$  and Bu pyruvate. Other esters specified are those of Am, cyclohexyl and benzyl alcohols.

**Coating webs.** Wilfrid B. Park, Cyril A. Chester and Wm. Melville. Brit. 431,541, July 10, 1935. In the surface of sheet material in continuous lengths with varnishes, pigments, etc., by the flotation process, the spreading and orientation of the film on the flotation medium is controlled by regulated elec. or magnetic potential applied to the film, the flotation medium and (or) the transfer surface. App. is described.

**Pyroxylin compositions.** Wm. Saman (to Standard Oil Development Co.) U. S. 2,018,707, Oct. 29. In forming compns. such as lacquers, films, etc., pyroxylin is used with a solvent contg. an org. S-compd. of the general formula  $\text{R-S-R'}$  in which R and R' represent sep. alkyl or aryl radicals or acyl radicals of fatty acids, e. g., ethyl or methyl or propyl thioether, and various other solvents and modifiers.

**Refining rosin.** Joseph N. Bourgin (to Hercules Powder Co.) U. S. 2,018,644, Oct. 22. The rosin, in soln. in a solvent such as gasoline which is substantially insoluble with resorcinol and a B compd. such as boric acid, is admixed with resorcinol and such a B compd., and coloring substances are added together with the resorcinol and B compd. by gravity sepn. Cf. C. A. 29, 2003.

**Increasing the melting point of rosin.** Avery A. Morton (to Columbia Naval Stores Co. of Del.) U. S. 2,017,800, Oct. 22. Rosin dissolved in less than its own wt. of gasoline is treated with  $\text{H}_2\text{SO}_4$  proportioned so that for each gram of rosin treated less than 1 cc. of  $\text{H}_2\text{SO}_4$  (calcd. as conc. acid) is used, the materials are agitated together for a time sufficient to promote reaction (suitable for about 3 hrs.) while minimizing crystn. (as by agitation) and the treated rosin is recovered by distg. off the gasoline

## 27—FATS, FATTY OILS, WAXES AND SOAPS

E. SCHERER

**Melting points of solid animal fats and their mixtures.** H. Adam Koss. *Prezmyst Fatm.* 19, 139-47 (17-8 in French) (1935); cf. C. A. 29, 6083P.—Horse, pork, beef, goat and sheep fats were used individually and in combinations of 2 ranging by 10% steps in proportion from each pure fat to every other pure fat. The solidification points were detd. by the methods of Linker (modified), Zukow, and Wolfbauer, the first of which gave the lowest values but is regarded as the most reliable. The methods are critically compared. For the fats listed in the above order the values according to the F. method were found to be: 35.32°, 41.01°, 43.63°, 47.31° and 50.13°. There is no regular relationship between percentage mixts. of any 2 fats and increase of the solidifying point. M. ps. of the acids of these fats cannot be used as the only basis for identifying their identity, but the Polenske method and other chem. analytical methods should be applied. Mixts. of horse fat with beef, goat, and sheep fats, and of pork with sheep fat show minima in solidification values which may be regarded as eutectic mixts. corresponding to  $\text{C}_{18}\text{H}_{36}\text{O}_2$ ,  $\text{C}_{18}\text{H}_{34}\text{O}_2$ , or as solid solns. Mixts. of goat with sheep fats showed a max. in the solidification temps.

A. C. Zachlin

**Changes in fat during storage.** C. H. Lea. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934*, 34-8 (1935); cf. C. A. 29, 3677.—A no. of typical curves for the rates of oxidation of various natural oils and fats, detd. by the previously described method (C. A. 29, 1273), are given. The appearance of taint in the fat of chilled beef is usually to be attributed to the action of microorganisms, and the free acid content of the fat may conveniently be used as an index of spoilage. The acidity of tainted fat is almost invariably higher than normal, al-

though the actual values attained depend on the nature of the contamination. Typical data obtained from examination of chilled beef fat are given and compared with the free acidity of freshly killed beef fat and with that of fat inoculated with a suspension of spores of *Mucor*. Bleached grayish, brownish or white patches have sometimes been observed on the external fat of quarters stored in an atm. contg.  $\text{CO}_2$ , usually appearing first in regions where the covering of fat is thin, or where contact between quarters or chafing has occurred. This discoloration is apparently due to the conversion of oxyhemoglobin to methemoglobin accompanied in more advanced cases by bleaching of the yellow pigment, and seems to be accompanied by oxidation of the fat. Deterioration in flavor of the fat of chilled beef may therefore be produced by the agency of microorganisms, by the absorption of odors, or by oxidation, but the last may become significant only when the meat has been unduly exposed to light, or subjected to the long periods of storage made possible by the use of  $\text{CO}_2$ .

A. Poupillon-Couture

**Fats of sea algae.** H. Eiji Takahashi, Kiyoshi Shirahama and Shun-ichi Tase. *J. Chem. Soc. Japan* 56, 1250-7 (1935); cf. C. A. 27, 6778.—The sapon. acids of the fats of sea algae were mainly palmitic acid with lesser stearic and myristic acids. Unsatd. acids were oleic acid,  $\text{C}_{18}\text{H}_{32}\text{O}_2$ , and  $\text{C}_{18}\text{H}_{30}\text{O}_2$ .

K. Kitsu

**Antioxidants and the preservation of edible fats.** C. H. Lea. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934*, 38-43 (1935).—A study of the possibility of utilizing aliphatic amino and hydroxy compds. as antioxidants for the preservation of lard. The efficiency of the antioxidant can most conveniently be expressed in terms of a "protection" factor (P. F.),

defined as the ratio of the lengths of the induction periods in the presence and absence of antioxidant. Curves are given, leading to the following conclusions: prooxidants ( $P/F$ , 0.03)—succinic, tartaric, citric and lactic acids; inactive ( $P/F$  1.0)—mannitol, weak antioxidants ( $P/F$ , 1.0–2.0)—glycerol, glucose, sucrose, Na maleate, Na succinate, moderate antioxidants ( $P/F$ , 2.0–3.0)—Na maleate,  $\text{NH}_4$  lactate, Na tartrate, Na glycolate, Na lactate, powerful antioxidants ( $P/F$  4.5)—glycine, asparagine, Na citrate, Na malonate. The rates of oxidation of films of lard stored in contact with a series of acetate, phosphate and borate buffers indicated a maximum stability slightly to the acid side of the neutral  $\text{pH}$ , with a very rapid fall as the soln. became alk. Lard in contact with pickle also became less stable with increasing acidity, which is presumably due to a prooxidant effect of free  $\text{HNO}_3$ , the data for water, on the other hand, showed no evidence of more rapid oxidation in the acid solns. other than can be accounted for by change in the buffer system. Lard oxidizes much more rapidly in borate than in phosphate solns of approx. the same  $\text{pH}$ , except at high alkalinities when the distinction vanishes, whence it is probable that phosphate possesses antioxidant properties. Na citrate, Na malonate and glycine all exerted a protective effect at 20° and at  $\text{pH}$  values of 5.8, 6.7 and 8.2.

A Papineau-Couture  
Rate of formation of fully saturated glycerides during hydrogenation of different natural fats. T. P. Hilditch and H. Paul. *J. Soc. Chem. Ind.* 54, 336–8T (1935).—Rape, olive and cottonseed oils, 2 varieties of palm oil and pig back fat were hydrogenated progressively. The results show that the glyceride structure of a hydrogenated fat depends on the glyceride structure of the original fat, the manner in which the fat acid radicals *per se* are affected during hydrogenation, and the manner in which mixed glycerides of different configurations behave differently toward catalytic hydrogenation (i.e., the apparent reluctance with which an unsatd.  $\beta$ -acyl group is hydrogenated in comparison with an unsatd.  $\alpha$ -acyl group in the glyceride mol.). The proportions of fully satd. glycerides of the 6 fats when 50% of the original unsatd. acids have been hydrogenated are

	50 % hydrogenated fully satd. glycerides (mol)	Original Fat Total satd. acid (mol)	Palmitic acid (mol)
Rapeseed oil	20	3	2
Olive oil	24	14	12
Cottonseed oil	29	27	25
Cape Palmas palm oil	32	41	36
Belgian Congo palm oil	37	50	45
Pig back fat	41	44	30

E. Scherubel  
Detection of nickel in hydrogenated fats. M. Struszynski. *Przemysł Chem.* 19, 48–9 (1935).—From 2 to 10 g. of the fat is melted and left on the hot bath to settle out such impurities as rust, and then the clear liquid is poured off for analysis. About an equal vol. of  $\text{HCl}$ , sp. gr. 1.12, together with a small quantity of  $\text{HNO}_3$  or  $\text{KClO}_4$  is added and the mixt. is shaken well and frequently, the flask being heated on a water bath. The two layers are then allowed to settle out. When solidified the fat is punctured and the acid ext. is poured off into an evap. dish and brought to dryness. The residue is taken up with 2–3 drops concd.  $\text{HCl}$  and 1 drop  $\text{HNO}_3$ . The soln. is washed down into a small bottle with about 2 cc.  $\text{H}_2\text{O}$ . A bit of tartaric acid or  $\text{K}$  tartrate is added, then a few drops of dimethylglyoxime in alc. and finally a small excess of  $\text{NH}_4\text{OH}$ . The liq. is shaken with 1–1.5 cc. purest  $\text{CHCl}_3$  which is then collected in a pipet and placed in an evap. dish where it is dried ext. while blowing at the liquid in order to collect the residue in the smallest possible area. The min. quantity of  $\text{Ni}$  detectable by this method is  $2 \times 10^{-4}$  mg. In case the residue or the  $\text{CHCl}_3$  soln. is colored brown (presence of  $\text{Fe}^{++}$  or  $\text{Co}$ ) the residue is washed with a few drops of  $\text{H}_2\text{O}$  whereupon the red color becomes clearer. Since  $\text{Fe}^{++}$  does not

interfere with the test all  $\text{Fe}$  should be in the oxidized state.

A. C. Zachin  
Selectivity of hydrogenation. M. Zaayer. *Chem. Weekblad* 32, 333 (1935).—The selectivity of more and less active catalysts was studied by using peanut oil, at 120°, 3% catalyst (usual  $\text{Ni}$  kieselguhr either freshly made or kept for 2 months). Whereas the activity difference was marked, the more selective hydrogenation results were obtained with the most active catalyst as shown from a table and a graph of percentage of satd. acid vs. I addn. no. of fatty acids. B. J. C. v. d. H.

An innovation in the hydrogenation of oils. R. Heublyum. *Mat. grasses* 27, 10789–90, 10618–9 (1935).—Attention is drawn to Bag's highly active  $\text{Ni-Al}$  catalyst (C. A. 26, 2075) and to its advantages, one of the most important of which is the possibility of working continuously instead of in batches. A. Papineau-Couture.

Polymerization of some unsaturated fat acids. J. K. Chowdhury, A. C. Chakraborty and A. Majumder. *J. Indian Chem. Soc.* 12, 441–54 (1935).—Polymerization of oleic (I) and other unsatd. acids in the presence of  $\text{SnCl}_4$  produces highly viscous polymers which yield lubricating oils, similar to those obtained from petroleum, on decarboxylation. A known vol. of  $\text{SnCl}_4$  was added to a definite quantity of I in a new Nessler's tube provided with a mech. stirrer. The reaction was carried on at 100° for 10 hrs. and the product was extd. with petr. ether. The ext. was washed with dil.  $\text{HCl}$  and with  $\text{H}_2\text{O}$ , dried over anhyd.  $\text{Na}_2\text{SO}_4$  and evapd. Progress of polymerization was observed by noting changes in the phys. const. The amt. of  $\text{SnCl}_4$  added has a considerable influence on the degree of polymerization. Evolution of heat during the process suggests the formation of unstable intermediate compds. of I with  $\text{SnCl}_4$  which, on breaking down, activate the mol. and cause polymerization. This decomn. with evolution of  $\text{CO}_2$  indicates that the  $\text{CO}_2\text{H}$  group is involved in the reaction. It was found that stearic acid (II) was produced during the polymerization and that the polymer had a lower acid value. The Me ester of I polymerized very slowly and it is evident that the  $\text{CO}_2\text{H}$  group exercises an influence on the course of polymerization. The polymerization of linolic acid (III) took place through the conjugated system rather than the  $\text{CO}_2\text{H}$  group since the acid value remained const. though the I value fell quickly and the viscosity rose with the degree of polymerization. Mol. wt. detns. showed that several mols. had combined to form the polymerized product which was sepd. by extn. with  $\text{Et}_2\text{O}$  in place of petr. ether. In the polymerization of ricinoleic acid (IV) the I value remains const. except when 20% or more  $\text{SnCl}_4$  is added. The acid value remains const. though the  $\text{Ac}$  value falls quickly. Mol. wt. detns. show that at least 6 mols. is involved in polymerization of ester formation. Decarboxylation of the polymers from I, III and IV was effected by heating 30 g. of the products with 25 g. of  $\text{Zn}$  dust and 365° for 2.5 hrs. The acidified product was extd. with petr. ether and the degree of decarboxylation was estd. by detns. of the acid value. The products were dark green, slightly fluorescent, highly viscous hydrocarbons with the following properties. Polymers from I, II, III and their resp. decarboxylation products had  $n_D^{20}$  1.462, 1.491, 1.488, 1.513, 1.524, 1.516, I value 27.7, 45.6, 52.8, 23.0, 37.7, 44.6 and acid value 124.1, 203, 177.9, 7.6, 12.9, 11.1. The similarity of the decarboxylated polymerized products to petroleum lubricating oils seems to support the theory that petroleum hydrocarbons were formed by the polymerization and depolymerization of unsatd. fat acids followed by decarboxylation. C. R. Addinall.

Component acids and glycerides of partly hydrogenated rape oil. T. P. Hilditch and H. Paul. *J. Soc. Chem. Ind.* 54, 331–6T (1935).—Rape oil and also the Me esters prepd. from the mixed fat acids were submitted to progressive hydrogenation. In the mixed esters, hydrogenation of the linoleates proceeds selectively (as compared with that of the oleates) in the customary manner, and oleate is converted into stearate somewhat more readily than erucate into behenate. In the glycerides, the selectivity of the linoleic-oleic hydrogenation is much less



marked, while subsequently stearic and behenic glycerides appear at almost the same rate throughout. It is suggested that the differences in hydrogenation, as between the glycerides and the mixed esters, are due to the configurational positions of the unsatd radicals in combination in the glycerides. The proportions and compn of the fully satd. glycerides produced during progressive hydrogenation of rape oil have been investigated with reference to the general glyceride structure of the original oil. Neither tri- $C_{18}$  glyceride nor trierucin is present in rape oil, which conforms to the usual "even distribution" rule characteristic of the glyceride structure of seed fats. Apart from about 6% of mixed palmito-oleo-erucins, the oil investigated contained about 50% of di- $C_{18}$ -erucin and about 44% of mono- $C_{18}$ -di-erucin (the  $C_{18}$  acid being either oleic or linoleic). Consideration of the above observations on the course of hydrogenation of the 3 unsatd acid radicals leads to the belief that both  $\alpha$ - and  $\beta$ -oleo-(linoleo)-di-erucins and  $\alpha$ - and  $\beta$ -erucodiolins (linoleins) are present in the oil. E. Scherubel

The supposed occurrence of acids with uneven number of carbon atoms in vegetable oils and fats. I. Daturic acid from the seeds of *Datura stramonium*. Linn B. L. Manjunath and S. Siddappa. *J. Indian Chem. Soc.* 12, 400-4 (1935).—Extn. of 6.2 kg of specially grown and carefully collected seeds with petr ether yielded 1020 g of pale yellow oil,  $d_4^{20}$  0.9184,  $n_D^{20}$  1.4735, sapon value 187.1, I no. 122.6, acid value 5.6, Ac value 25.6, Reichert-Messl value 0.44, total fatty acids 87.7, contg 2.6% of unsapon matter. The oil was sapon and extd with Et<sub>2</sub>O. Acidification gave 855 g of fatty acids, mean mol wt. 289.1, I no. 120.7 contg 13.1% of solid acids, mean mol wt. 263.2 and liquid acids, mean mol wt 304.4 and I no. 126.5. The fatty acids were sepd into unsatd and satd. components by Twitchell's method. The unsatd acids were purified by distn of their Me esters under reduced pressure and the presence of oleic and linoleic acids was established by oxidation with  $KMnO_4$  and bromination. Crystn. of the unsapon matter from 95% alc. gave a phytosterol, m 134°, acetate, m 129°. The Me esters (111 g) of the satd acids were fractionated into 8 fractions and a residue. Fractions 1-5 (91.35 g) consisted of pure Me palmitate yielding palmitic acid (I), m. 61° on sapon. Fraction 6 contained stearic acid (II) and I, and fraction 7 yielded 6 g of pure II. Fraction 8 was pure Me stearate. The residue contained 3 g of lignoceric acid, m 76-78°;  $p$ -phenylphenacyl ester (C. A. 24, 5030) m 101-2°. No daturic acid (III) was detected at any point in the investigation. The m p. recorded for III varies from 54 to 60° and an approx. equimol. mixt. of I and II, m. 57.5 (C. A. 27, 2423), may be mistaken for III which has been stated to be the only acid with an uneven no. of C atoms known to exist in an oil. II. Acid fraction of mean molecular weight 354, from the seeds of *Butea frondosa*, (Roxb.). U. S. Krishna Rao and B. L. Manjunath. *Ibid.* 611-15.—During the examn. of the fatty oil from the seeds of *Butea frondosa* (C. A. 24, 2319) 2 fractions (one m. 74.5-5.8°, mol. wt. 353 and the other m. 77.5-9°, mol. wt. 383) were isolated. Esterification of the 1st fraction, fractionation of the Me ester, m. 54-5° and liberation of the acid from the residue gave 2 g. of lignoceric acid (I), m. 78-80°, mol. wt. 366;  $p$ -phenylphenacyl ester, m. 102-3°;  $p$ -bromophenacyl ester, m. 93-4°. Attempts to sep the 1st fraction by fractional crystn of the  $p$ -phenylphenacyl and  $p$ -bromophenacyl esters appeared to point toward the individuality of the fraction. That these derivs may not be suitable for the characterization of the higher fatty acids is apparent from a list of the mixed m. ps. of these esters of palmitic (II), stearic (III), behenic (IV) and lignoceric acids. M.-p. curves of mixts. of II, III and IV with I were detd. to find out if any evidence regarding the formation of a eutectic mixt. could be obtained. IV was prepd by the catalytic reduction in the presence of Ni of erucic acid ( $p$ -phenylphenacyl ester, m. 72.5-3.5°;  $p$ -bromophenacyl ester, m. 60.0-0.5°). Equimol proportions of IV and I form a eutectic mixt., m. 74-5°, which could not be sepd. into IV and I by the usual processes. It is probable that the

1st fraction is a eutectic equimol. mixt. of I and cerotic acid (V). The oil of *Butea frondosa* evidently contains IV and V and the presence of tricosic acid has not been established. C. R. Addinall

Oils and fats. O. E. Jones. *Oil and Soap* 12, 266-71 (1935).—An address E. Scherubel

New developments in ultraviolet irradiation of oil and fat. A. Salmony-Karsten. *Allgem. Oel-u. Fett-Zig.* 32, 358-61 (1935).—A review. Several new types of app. are described. M. M. Piskur

Oils and fats (of fresh- and salt-water fish and plants) J. A. Lovren. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 80-92 (1935); cf. C. A. 29, 948°. An extended study is being made of the fats of zooplankton and of marine and fresh-water plants. The fats of the zooplankton all show several peculiarities compared with the fats of fish, and the high degrees of unsatn. are particularly noteworthy for the fresh-water species. The characteristic differences between the fats of fresh-water and marine fish are increased proportions of  $C_{18}$  and  $C_{19}$  unsatd acids and decreased proportions of  $C_{20}$  and particularly  $C_{21}$  acids in the "fresh-water" type and the fat from the marine species of the "marine" type. With the algae, the 2 fresh-water species have fats very similar in type to those of the fresh-water zooplankton; the 1 marine alga so far examd had a most unusual type of fat with relative proportions of fatty acids: C<sub>18</sub> 0.3, C<sub>19</sub> 9.2, C<sub>20</sub> 7.3, C<sub>21</sub> 1.9, unsatd acids—C<sub>18</sub> 0.5, C<sub>19</sub> 5.0, C<sub>20</sub> 62.5, C<sub>21</sub> 33.5%. A. Papineau-Couture

Seed oils of Formosan plants. XII. Sapotaceae oil. Kinzo Kafuku and Chuta Hata. *J. Chem. Soc. Japan* 56, 1081-3 (1935). cf. C. A. 28, 5267°.—The oil of *Sideroxylon ferrugineum* Hook and Arn is a nondrying oil, it has  $d_4^{20}$  0.9082,  $n_D^{20}$  1.4650, acid no. 20.41, sapon. no. 189.13, I no. 77.63, unsaponifiable substance 0.37%. It contains 23.6% solid acids (mostly palmitic acid and small amounts of myristic and stearic acids) and 76.4% liquid acids (of which 77% is oleic acid and 23% is linoleic acid). *Palaeospermum formosanum* Hay contains 56.7% oil, solidifies at 27°, m. 57-60°, has  $d_4^{20}$  0.8855,  $n_D^{20}$  1.4520, acid no. 9.41, sapon. no. 189.75, I no. 44.96 and unsaponifiable substance 0.87%. It contains 60% solid acids (mostly stearic and less arachidic acid) and 40% liquid acids (mostly oleic). K. Kutsuta

Influence of gossypol on color of cottonseed oil. M. Podolskaya. *Fettechem. Umschau.* 42, 96-100 (1935).—See C. A. 29, 8376°

A rapid visual method for estimating the amount of lint on cottonseed. Egbert Freyer. *Oil and Soap* 12, 259-62 (1935); cf. C. A. 29, 7105°.—It was found that a difference could easily be detected visually in the case of 2 samples differing in the amt. of lint by no more than 0.5%. A graded series of mill delinted seed samples was prepd covering a range of 3.7 to 6.5% lint whose values were established by repeated detns using the acid method. The seeds were spread out in flat cigaret tins, covered with glass and mounted on a masonite board. The standards were placed in 2 horizontal rows in order that the unknown sample might always be either directly adjacent to the standard which it matched or between the 2 which it most nearly matched. The reliability of this method using 21 samples indicated no greater difference than 0.5% when compared with the acid method. The comparison may be made under 2 conditions: at close range under a strong light, and with the light subdued and the observer standing at a distance of 15 feet, when the seeds are not seen as individuals and the comparison is made by judging the relative grayness of the surfaces and the relative distribution of light and dark patches. E. Scherubel

Treatment of olive oil. R. Fusteig. *Mai. grasses* 27, 10619-20 (1935).—A brief discussion describing the production of olive oil by extn. with solvents and bringing out its advantages over pressing processes. A. P.-C.

Adulteration of peanut oil with coconut oil. H. Reubenbauer. *Przemysl. Chem.* 19, 9-10 (1935).—A 10% admixt. of coconut oil can be detected because it has a lower sp. gr.,  $n$  and I value, but a higher sapon. value, Reichert

and Meissl, and Polenske values. Polish laws prescribe labelling of mixed edible oils as "edible oils" to distinguish them from olive oil in native parlance. A C Z

**Analysis of national grape-seed oil.** Rodolfo Rouzaut. *Rev. facultad quim. ind. agr. (Univ. nac. litoral, Argentina)* 3, 102-6 (1934).—Oil from fresh grape seeds has the following characteristics:  $d_4^{20}$  0.9255,  $n_D^{20}$  1.47649,  $n_D^{25}$  1.46285,  $n_D^{30}$  1.4541, acid no. 2.084, oleic acid 1.046%, SO<sub>2</sub> 0.148%, sapon index Kottstorfer 189.57,  $\eta_{sp}$  no. (Hubl) 133.72, volatile acid (Tortelli) 77.2%, crit temp of soln (Valenta) 8.5°, (Crismer) 69.5, butyrolactometer reading at 25°, 73. It gives a neg. elaidin and Malphen tests, pos. Bellier, Heydenreich, Hauchecorne and resin (Morawski) tests. The value of this oil as a food has hitherto been largely disregarded. W. F. Bruce

**Volatile oil of Piperaceae.** Kunzo Kafuku and Ryo Kato. *J. Chem. Soc. Japan* 56, 1150-4 (1935).—Steam distn of dried leaves of *Piper betle* Linn. gives 0.62% oil. It has  $d_4^{20}$  1.024,  $n_D^{20}$  1.5035,  $n_D^{25}$  1.481, +1.00, acid no. 50.95 and ester value 145.69 (after acetylation 371.03). The oil contains chavicol and allylpyrocatechol. K. K.

**The chemical composition of the chinaberry.** R. W. Bost and Dan Fore, Jr. *J. Elisha Mitchell Sci. Soc.* 51, 134-42 (1935).—The fruits of *Melaleuca azadirach* contain a semidrying oil, glucose, other hydrocarbons, protein and a poisonous constituent of unknown compn. The oil consists chiefly of the glycerides of palmitic, oleic, linoleic and stearic acids. No linolenic, chaulmoogric or hydrocarpic acids were found. Picrotoxin is absent. A. L. Mehring

**Unsaturated acids of natural oils.** I. Highly unsaturated acids from *Onoclea* oil (Licania rigida). Wm. B. Brown and Ernest H. Farmer. *Oil Colour Trades J.* 88, 1090-1100 (1935).—See C A 29, 5294<sup>1</sup>. W. H. B.

**Finnish stickleback oil.** Toivo Karttunen. *Suomen Kemistilehti* 8A, 119 (1935).—Oil is prepd from the stickleback (*Gasterosteus aculeatus* L.) by boiling in water and finally pressing. The remaining meal has the compn raw fat 20, raw protein 50 and ash 18%. The oil prepd by skimming was light orange in color with a faint odor resembling whale-oil. Light reduced the color considerably. The following consts. were detd:  $d_4^{20}$  0.9329, acid no. 6.14, sapon no. 180.29, ester no. 180.15, Hehner's no. 91.00, hydroxyl no. 242.28, Reichert-Meissl no. 2.4, Polenske no. 3.6 and I no. 142.46. The oil yielded 47.02% of ether-insol. bromides contg. 48.25% Br. The glycerol content was 8.06% and cholesterol 24%. C. E. C.

**Gadusene, an unsaturated hydrocarbon occurring in animal and vegetable oils.** Juro Nakamura. *Sci. Papers Inst. Phys. Chem. Research (Tokyo)* 28, 16-26 (1935).—*Gadusene* (I),  $C_{21}H_{40}$ , an unsatd. hydrocarbon, has been obtained from the nonsaponifiable fraction of fish-liver oils of *Gadus macrocephalus*, *Theragra chalcogramma*, *Stereolepis ischnogi*, and further from the same fraction of rice embryo and soy oils. Catalytic hydrogenation of I gives *gadusane*,  $C_{21}H_{42}$ , b. 85-6°,  $n_D^{20}$  1.4632. I has 6 characteristic absorption bands, A (3333, 3273, 3210), B (3090, 3020), C (2865, 2785, 2721), D (2544), E (2407, total absorption). W. J. Peterson

**Tetradecenoic acid and dodecenoic acid in sperm oil.** I. Tetradecenoic acid and dodecenoic acid in sperm blubber oil. Yoshiyuki Toyama and Tomotaro Tsuchiya. *J. Chem. Soc. Japan* 56, 1030-4 (1935).—Tetradecenoic acid (I),  $C_{14}H_{28}O_2$ , and dodecenoic acid (II),  $C_{12}H_{24}O_2$ , were isolated. Consts. of I are  $d_4^{20}$  0.9046,  $n_D^{20}$  1.4571,  $n_D^{25}$  1.4552, M. R. 67.86 (theory 67.92), acid no. 248.8, I no. 107.8. Consts. of II are  $d_4^{20}$  0.9130,  $n_D^{20}$  1.4535, M. R. 58.72 (theory 58.69), acid no. 282, I no. 118.7. Examn. of decompn. products from oxidation indicated that the structure of I is  $Me(CH_2)_4CH=CH(CH_2)_4CO_2H$  and that of II  $Me(CH_2)_3CH=CH(CH_2)_3CO_2H$ . II. Dodecenoic acid in sperm head oil. Ibid. 1035-7.—Dodecenoic acid (I),  $C_{12}H_{24}O_2$ , is isolated. Oxidation of I by Huzura's method gives dihydroxydicarboxylic acid and tests on oxidation products from methylhydroxydicarboxylic acid indicated that the structure of I is exactly

the same as that obtained from the sperm blubber oil (cf. above). K. Kutsuta

**Hexadecenoic and tetradecenoic in sperm head oil.** Yoshiyuki Toyama and Tomotaro Tsuchiya. *J. Chem. Soc. Japan* 56, 1072-6 (1935).—Hexadecenoic (I) and tetradecenoic (II) were isolated from the unsaponifiable fraction of the oil. Consts. of I are  $d_4^{20}$  0.8537,  $d_4^{25}$  0.8503,  $n_D^{20}$  0.4605,  $n_D^{25}$  1.4584, I no. 98.6, M. R. 77.15 (theory 77.14); acetate of I b.p. 195.8°, has  $d_4^{20}$  0.8760,  $n_D^{20}$  1.4506. Consts. of II are:  $d_4^{20}$  0.8507,  $d_4^{25}$  0.8473,  $n_D^{20}$  1.4573,  $n_D^{25}$  1.4553, I no. 111.2, M. R. 67.98 (theory 67.91); acetate of II b.p. 165.70°, has  $d_4^{20}$  0.8729,  $n_D^{20}$  1.4477, M. R. 77.93 (theory 77.28). Studies on oxidation products of the acetates of I and II indicated that the structure of I is  $Me(CH_2)_6CH=CH(CH_2)_6CH_2OH$  and that of II  $Me(CH_2)_4CH=CH(CH_2)_4CH_2OH$ . Since these are related to zoamaric acid ( $\Delta^4$ -hexadecenoic acid) and phyteteric acid ( $\Delta^4$ -tetradecenoic acid), the names *zoamaryl alc.* for I and *phyteteryl alc.* for II are given. K. Kutsuta

**Hexadecenoic acid in sperm blubber oil.** Yoshiyuki Toyama and Goroku Akiyama. *J. Chem. Soc. Japan* 56, 1077-80 (1935).—A small amt. of  $\Delta^4$ -hexadecenoic acid was isolated from the unsaponifiable fraction. K. Kutsuta

**Application of the technic of chromatogenic adsorption to the study of the composition of the unsaponifiable residue of fish oils.** J. C. Drummond, A. Santos Ruiz and T. Thorbjarnarson. *Anales soc. españ. fis. quim.* 33, 680-91 (1935).—The Tswett chromatogenic adsorption method (Aberhalden. *Handbuch des Biochem. Arbeitsmethoden*, 6, 100 (1912)) of sepg. and detg. different constituents of the unsaponifiable fraction from various oils is reliable and simple. In cases where the classical method of fraction crystn. and distn. gave little or no success, adsorption by  $Al_2O_3$  was effective. With  $Al_2O_3$  as adsorbent, and light petroleum fractions as solvent, satd. hydrocarbons usually pass most readily through the column, while unsatd. hydrocarbons, at least those of the squalene type, pass into the filtrate. Unsatd. alcs. tend to wash down into the lower layers of the columns, but their distribution is, to some extent, detd. by the nature of other substances present. A relatively large proportion of highly unsatd. hydrocarbon, e. g., squalene, tends to bring down unsatd. alcs. lower in the column, and may even cause leakage into the filtrate. Sterols tend to be adsorbed in a relatively well-defined bed  $1/2$  of the distance down the column. Certain lipochromes of the xanthophyll type may be assoc. with sterol or may be found concd. in adjacent zones. Satd. alcs. are usually held at the top of the column. E. M. Symmes

**The highly unsaturated acids in sardine oil.** VII. The separation of highly unsaturated  $C_{22}$  acids. Yoshiyuki Toyama and Tomotaro Tsuchiya. *Bull. Chem. Soc. Japan* 10, 433-40 (1935), cf. C A 29, 8378<sup>4</sup>.—A concd. fraction of highly unsatd. acids was sepd. from sardine oil by the Na soap-acetone method. It was converted into Me esters and the latter subjected to a fractional distn. which yielded a fraction consisting of the Me esters of  $C_{22}$  acids. This fraction and the free fat acids liberated from it showed I nos. which were close to those of Me elupanodanate and elupanodonic acid, resp., but on sepg. the fat acids of this fraction by a fractional pptn. of Na soap in acetone soln. they were found to contain, in addn. to elupanodonic acid, some acids of different degrees of unsatn. After a repeated sepg. elupanodonic acid was sepd. in a much purer state than before, and a more highly unsatd. portion consisting of docosahexenoic acid,  $C_{22}H_{40}O_2$ , was also sepd. A portion less satd. than elupanodonic acid was also obtained, less satd. than elupanodonic acid,  $C_{22}H_{40}O_2$ , was not sepd. although it could not be deemed to be absent. VIII. The constitution of elupanodonic acid  $C_{22}H_{40}O_2$ . Ibid. 441-53.—The following products were found when Am elupanodanate was subjected to ozonolysis: propylaldehyde and acetaldehyde,  $CO_2$ , succinic acid, amyl H succinate and also lower acids, probably propionic and acetic. Of these compds. acetaldehyde, acetic acid and  $CO_2$  are attributable to the secondary

decompos. of the products of ozonolysis derived from the group  $\text{CH=CH}_2$ ,  $\text{CH=}$ . Accordingly elupanodonic acid was shown to contain the following groups:  $\text{CH}_2$ ,  $\text{CH}_2$ ,  $\text{CH=}$ ,  $\text{CH=CH}_2$ ,  $\text{COOH}$ ,  $\text{CH=CH}_2$ ,  $\text{CH=}$  and  $\text{CH=CH}_2$ ,  $\text{CH=}$ , of which elupanodonic acid contains 3 of the last-named groups. The dibromo deriv. of elupanodonic acid was sepd. as the chief constituent of the product obtained by adding 1 mol. Br to 1 mol elupanodonic acid. Tetrabromo and hexabromo derivs. of elupanodonic acid were sepd. similarly. In the ozonolysis of these bromo derivs., Br adds selectively first to the ethylene linking which is more distant from the carbonyl group. Ozonolysis of the dibromo deriv. of elupanodonic acid thus obtained gave a bromo compd. which on debromination and subsequent hydrogenation yielded heptonic acid. This fact coupled with the results obtained by the ozonolysis of Am elupanodonic acid indicates that elupanodonic acid has the group  $\text{CH}_2$ ,  $\text{CH}_2$ ,  $\text{CH=CH}_2$ ,  $\text{CH=}$ . Ozonolysis of the tetrabromo deriv. of elupanodonic acid gave a bromo compd. which on debromination and hydrogenation yielded capric acid and consequently elupanodonic acid was shown to contain the group  $\text{CH}_2$ ,  $\text{CH}_2$ ,  $\text{CH=CH}_2$ ,  $\text{CH=}$ ,  $\text{CH=CH}_2$ ,  $\text{CH=}$ . Ozonolysis of the hexabromo deriv. gave a bromo compd. which on debromination and hydrogenation yielded myristic acid and consequently elupanodonic acid was shown to contain the group  $\text{CH}_2$ ,  $\text{CH}_2$ ,  $\text{CH=CH}_2$ ,  $\text{CH=}$ ,  $\text{CH=CH}_2$ ,  $\text{CH=}$ ,  $\text{CH=CH}_2$ ,  $\text{CH=}$ . From the foregoing results the constitution of elupanodonic acid has been established as  $\Delta^8, \Delta^{12,14}$  docosapentenoic acid. F. Scherubel

Physical and chemical characteristics of turtle oil. Walter Lee. *Analyst* 60, 651-3 (1935).—Four lb. of refined W. African oil was mixed and filtered. The following values were obtained on analysis: M. p. 21.6-25.6°, I. p. 22.5°, titer 25.5°, d<sub>4</sub> 0.9112, n<sub>D</sub> 1.4599, sapon value 0.6%, I<sub>2</sub> value of the unsaponifiable matter 92.5, I<sub>2</sub> value of oils 64.0, acid value 2.0, percentage of insol. bromides on the free acid 5.0, m. p. of the bromides above 200°, Polenske value 0.2, Kirschner value 0.06 and acetyl value 3.6. The oil was supposedly rich in vitamins but no significant amts. of vitamin A, ergosterol or other provitamin B, or vitamin E could be detected. W. T. H.

Sulfurization of oils [and sulfur tanning]. Ahmed Abdassi. *Curr tech* 24, 236-8, 251-3, 307-7 (1935), of C. A. 29, 7672. —An abridgement of a thesis, giving A's conclusions without data or expl. details. **Conclusions.** Oils (drying, semidrying, and nondrying) dissolve 1-3% S and undergo slight darkening, increase in viscosity, and decrease in I value. Heated under pressure, the percentage of S dissolved may be increased to 10% and changes in properties are enhanced. Oxidized oils (especially cod liver) react more readily than unoxidized. When S-tanned leather is treated with oxidized cod oil, S reacts with the oil, but the product does not act as a tanning agent. H. B. Merrill

Waxes in the candle industry. L. W. Geller. *Oil and Soap* 12, 263 5 (1935).—Waxes in use for candles consist of paraffin and beeswax. Stearic acid is the only known hardening agent for paraffin. It raises the bending point, lowers the m. p. and can be used in any quantity without impairing the burning quality of the candle. The hydrogenated oils and fats serve as hardening agents for paraffin and are used chiefly for candles which are consumed in glasses. Synthetic or natural resins can be used in the form of coatings only and are used chiefly for decorative candles. No known synthetic hardening agent for candle wax can be satisfactorily substituted for stearic acid even in such a mixt. as 95% paraffin, m. 135°, and 5% stearic acid. E. Scherubel

**Determination of soap in refined oils.** Richard Durst. *Oil and Soap* 12, 271-3 (1935).—Weigh 300 g. of the oil into a 1. separatory funnel and add in 50-ml. portions 200 ml. of hot dil. 1:1 HCl. Shake and allow the 2 liquids to sep. and draw off the acid soln. in a beaker. Repeat until the 200 ml. of acid is used. Combine the 4 acid solns. and evap. to dryness. Take up the residue in distd. H<sub>2</sub>O and again evap. and take up the final residue in 50 ml. of distd. H<sub>2</sub>O and heat nearly to boiling. Add 1 ml. of 10% K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> soln. and titrate with AgNO<sub>3</sub> soln. to a brick-red color. The AgNO<sub>3</sub> soln. is standardized so that 1 ml. = 0.01 g. NaCl. From the vol. of AgNO<sub>3</sub> used, the wt. of soap in the oil is found by using the following factors: 0.05195 for Na oleate, 0.03794 for Na laurate, 0.0475 for Na palmitate. E. Scherubel

**Medicinal soaps.** P. Martell. *Allgem. Oel- u. Fett-Zig* 32, 367 8 (1935).—Tar and S-contg. soaps are recommended for skin diseases, dandruff and falling hair. The S-contg. soap is also recommended for use by workers in metal industries, especially those who work with lead compds. The addn. of HgCl<sub>2</sub> to soap has the disadvantage of producing a grayish off-colored product, however, this soap finds application in disinfection of hands before operations and also for disinfecting surgical instruments. Other antiseptic soaps mentioned contain phenols, salicylic acid, formaldehyde or peroxides. M. M. P.

#### Utilization of soybean oil in paint (Burlison) 26

**Industrial oils and fats.** Escher Wyss Maschinenfabrik A.-G. Swiss 177,269, Aug. 1, 1935 (Cl. 33a). App. is described for obtaining fats and oils from animal matter by evap. off the water *in vacuo* at temps. below 100°.

**Cashew nut shell oil extraction.** Thomas M. Rector (to Baker-Bennett-Day, Inc.). U. S. 2,018,001, Oct. 22. For extg. oil from the shells of whole nuts, the nuts are immersed in a heated bath of the shell oil, at a temp. and for a time adjusted to effect discharge of a major portion of the contained shell oil without scorching of the nut kernels. App. is described.

**Lecithin and oil mixtures from fresh soybean sludge.** August Gehrke. U. S. 2,018,781, Oct. 29. A storable mixt. is obtained by treating soybean sludge with glycerol and then sepg. undissolved solids from the aq. glycerol mixt. The product is suitable for use in foods, etc.

**Soaps.** Edward H. Taylor and Cecil W. Taylor. Brit. 432,227, July 23, 1935. Cold-process fat-resin soaps are made by treating fatty matter with just sufficient alkali for sapon., treating a mixt. of resin and fat or oil with alkali sufficient to saponify only the resin, mixing the 2 products and adding alkali to saponify the surplus fat. Perfumes, fillers, coloring matters and bleaching agents may be added.

**Soap.** Victor Wintsch. Swiss 174,087, July 1, 1935 (Cl. 38e). Alk. earth soap emulsions free from inorg. alkali salts are obtained by treating alk. earth hydroxide with at least one free fatty acid in the presence of a wetting and emulsifying agent. Thus, fused stearic acid is emulsified by an aq. soln. of a usual wetting and emulsifying agent and shaken with a hot aq. soln. of Ba(OH)<sub>2</sub>. The products are used for glazing paper.

**Washing composition.** E. Huber-Weber. Swiss 176,937, July 10, 1935 (Cl. 38e). The compn. consists of soap powder and a fatty alcohol sulfonate. In an example, the compn. contains soap powder, borax, Na perborate and fatty alc. sulfonate.

## 28—SUGAR, STARCH AND GUMS

J. K. DALP

**Saturating sugar juices with gases.** Maschinenfabrik Buckau R. Wolf A.-G. Ger. 615,144, Sept. 2, 1935 (Cl. 82c. 16). Gas is forced in through a porous ceramic or artificial stone compn.

**Sugar refining.** Holger deF. Olivarum (to Cahf. Packing Corp.). U. S. 2,018,869, Oct. 29. Molasses having a polarization of below about 60° is withdrawn from a sugar-refining system, dild. with water, treated

with yeast and invert sugar present is fermented without inversion of the sucrose, i.e., is added to the fermented molasses in the substantial absence of invert sugar, an alk earth oxide or hydroxide such as  $\text{Ca}(\text{OH})_2$  is added in an amt. sufficient to ppt org. impurities but insufficient to ppt sucrose, the ppt formed is sepd and the alc. sucrose soln is neutralized with an acid such as  $\text{H}_2\text{SO}_4$ , capable of forming substantially insol Ca and K salts, pptd salts are sepd from the substantially neutral soln, i.e., is removed from the soln and the sugar is then coned and sugar is crysdl from it. An arrangement of app. is described.

Sugar-cane mills Wm Murray. Brit. 430,532, June 29, 1935. In a mill having circumferentially grooved or corrugated rollers, the dumbturner or trash-plate is formed on its upper face with a series of transverse grooves extending the entire width of the plate and mating with the grooves in the top roller.

Roller-mills for sugar cane, etc. Fawcett, Preston and Co Ltd, John C Macgillivray and Gordon Chapman. Brit. 431,655, July 12, 1935.

Starch Maximilian Sprockhoff. Ger. 618,265, Sept. 4, 1935 (Cl. 53k 2 01). Refined potato starch is converted into a product suitable for making puddings, etc., by treating it first with  $\text{Cl}_2$  or a hypochlorite at about  $45^\circ$  and then

with a reducing agent, e.g.,  $\text{SO}_2$ , until a product is obtained which is converted completely into an easily subdivided gel when it is loaded with water.

Starch preparations Ernest E. Pendlebury. Brit. 431,275, July 4, 1935. Preps. for glazing or otherwise finishing textiles are composed of a  $\text{H}_2\text{O}$ -sol. starch and 1 or more compds of stearic, oleic or ricinoleic acid with triethanolamine, ethylene glycol, diethylene glycol or triethylene glycol. There may be added a sol. sulfonated vegetable oil,  $\text{H}_2\text{BO}_3$  or a borate, talc, and stearic, palmitic, oleic or ricinoleic alc. or esters thereof. Glycol and cetyl alc. are included in examples. The preps. may be added to "cold" or "boiled" starch.

Cold soluble starch N. V. Chemische Fabriek "Servo" and Meindert D. Rozenbroek. Brit. 430,872, June 26, 1935. Cold sol. starch, obtained by drying starch, materials contg starch or derivs of starch in thin layers, e.g., on metal plates or rollers, is moistened and gently heated, e.g., to  $50^\circ$ , to reduce its swelling capacity in  $\text{H}_2\text{O}$  and its bulk. To the treated material there may be added untreated cold sol. starch, salts, borax, soap, weak or strong alkalis, talc, china clay, gelatin, glue, milled gums or resins, dextrin, starch, rubber latex, fat, oil, wax, paraffin or fatty alcs., if desired in soln. or suspension in the  $\text{H}_2\text{O}$ .

## 29—LEATHER AND GLUE

ALLEN ROGERS

The Finnish leather industry and its products Lauri Pakkala. *Suomen Kemistilehti* 8A, 71-6 (1935)—Various kinds of leather are described. E. E. Jukkola.

Bleaching animal skins with permanganate preparatory to tanning John A. Wilson. *Hide & Leather* 90, No. 19, 17, 24-7 (1935)—Skins are bleached with  $\text{KMnO}_4$ ,  $\text{NaHSO}_4$ ,  $\text{H}_2\text{SO}_4$ , and  $\text{NaCl}$  after bating or pickling, to remove discolorations (salt stains, black hair bulbs, etc.). After bleaching, the skins are brought back to the pH value normally existing before tanning, and are tanned in the usual way. The bleached stock is now white. Comparative tests with sides showed marked improvement in shade after tanning and after coloring. Photomicrographs, analyses, phys. tests and practical examn showed no deleterious action resulted from this bleach. H. B. M.

The theory and practice of combination tanning. H. Ovrutskil. *Collegium* 1935, 178-80—See C. A. 29, 7114.

One bath chrome tanning process E. W. Merry. *Leather World* 26, 263 (1934), *J. Am. Leather Chem. Assoc.* 30, 577 (1935)—When one or more acidic groups are assoc. with the Cr atom in a manner such that the assoc. persists upon soln. and ionization, the Cr atom is said to be masked. Masking makes the Cr liquor less astringent, and is therefore useful in the early stages of tanning. Tanning in a liquor of basicity = 33% is advised. H. B. Merrill.

A theory of mineral tanning I. The flocculation and turbidity which occurs when aluminum and chromium salt solutions are made basic. A. Kuntzel and A. Königfeld. *Collegium* 1935, 257-70; cf. C. A. 29, 3189.—When alkali is added to solns. of  $\text{AlCl}_3$  and  $\text{Al}(\text{NO}_3)_3$ , a ppt. forms with every drop, on shaking, this ppt. decomposes into rod-like, double-refracting particles and rapidly or slowly disappears. Solns. of the corresponding Cr salt, at the pptn. point, give a similar cloudiness which disappears on standing. Solns. of Al salts and  $\text{Na}_2\text{CO}_3$  form carbonate complexes which are sol. but which decompose very rapidly and the soln. soon becomes cloudy as with  $\text{NaOH}$ . Sulfate solns. of Cr and Al form insol dihydroxy-tetraaquo solts, so that there is a max. basicity, which sulfates can reach without ppt., while chloride and nitrate solts can be made almost 100% basic. Cr salts are much more strongly hydrolyzed in the first step than in the second and third, Al salts are about equally hydrolyzed in all 3 steps. The reversal of pptn. is mostly caused by acids which are formed during slow hydrolysis. II. Ag-

ing phenomena in basic solutions of aluminum and chromium salts. A. Kuntzel, C. Riess and A. Königfeld. *Ibid.* 270-89.—Solns. of basic salts of Cr and Al both become more acid on standing (aging) or heating. A slow hydrolysis is responsible for this increase in acidity, and for dissolving the ppt. or cloudiness which first forms on making the solts. basic, and is caused by the formation of high mol. aggregates which are not sensitive to acid and do not enter the hydrolysis equl. The aggregates are not of uniform size, the size increases with increasing basicity, and in Al systems the size is smaller than in Cr systems of the same basicity. Al aggregates form and age much quicker than Cr aggregates. Aged Cr aggregates are much more stable to acids than Al aggregates; detn. of degree ofolation is possible only with Cr, not with Al salt solts. For the formulation of the constitution of the aggregates, theolation theory of Staudy is not as satisfactory as the O-bond theory of Jander (C. A. 26, 4765) but the 2 can be combined easily. I. D. Clarke.

The theory of mineral tanning—chromium tanning of gelatin XXVI. Bating and dyeing processes. E. Eklund and Th. Schachowsky. *Kolloid Z.* 72, 67-74 (1935), cf. C. A. 29, 3721, 3192.—Changes of light absorption of  $[\text{Cr}(\text{NH}_3)_4\text{Cl}]\text{Cl}$  in gelatin, of green  $\text{CrCl}_3$  in gelatin and of  $\text{CrCl}_3$  with addns. of  $\text{NaOH}$  and of glycooll and  $\text{NaOH}$  were investigated. The tanned end products are all similar; no glycooll like compds between gelatin and Cr occur. Tanning exps. with polyvinyl alc. support the theory that in mineral tanning attraction forces between molts. play decisive roles. Tanning with various Cr hydroxide solts prep'd. by dialysis with approx. equal basicity (20-43% basic) showed that the degree of tanning for equal amts. of Cr is decidedly dependent on the particle size—the greater the dispersion, the more intensive the tanning. No support was found for the assumption that in the tanning of gelatin with  $\text{CrCl}_3$  compds, complexes occur which in their inner sphere are especially rich in Cl ions. It seems more probable that the tanning Cr compds formed in the gelatin are of a highly basic nature.

The natural tannins Alfred Russell. *Chem. Rev.* 17, 155-66 (1935)—A review. Louise Kelley.

Determination of the plumping of skins during tanning. Fernand Pothier. *J. Intern. Soc. Leather Trades Chem.* 19, 534-43 (1935).—Turdidity coeff.,  $T$ , is defined as  $(100e)/E$ , where  $e$  = thickness of specimen under standard pressure and  $E$  = thickness under zero pressure. A

special micrometer caliper, in which contact between the specimen and the movable jaw is detected electrically, is used to measure  $E$ .  $e$  is measured by a method similar to that of Wilson and Gallun (C. A. 17, 649, 1901). Pieces of cow hide, about  $5 \times 7$  cm., were unharmed with lime, delimed with  $\text{NaHSO}_3$ , and demineralized with satd.  $\text{CO}_2$  soln.  $T$  was measured at 6 points for each piece before tanning. Pieces were tanned in strong solns. (sp. gr. = 1.030) of exts.: I chestnut, II oak, III quebracho, IV pine, V myrobalsans, VI mimosa and VII valonea.  $T$  was measured for each piece at intervals up to 30 days. For all exts. studied,  $T$  increased by 10–20% during the first 3 to 10 days, and thereafter declined slightly, the initial increase is ascribed to "hydration" under the influence of nontanning, and the subsequent decrease to dehydration by tanning.  $e$  increased rapidly at the outset, and reached a nearly const. value after about 15 days, but the initial rate of increase and the max. value attained varied widely between the different exts.  $T$  excepting for VI and VII, which showed lag periods, the final value of  $e$  increased with rate of initial increase of  $e$ .  $e_{90}$  exceeded  $e_0$  by about 120% for I and II, 100% for VI, 80% for VII, 60% for IV and V and 50% for III. Differences in  $T$  were detd. primarily by differences in  $e$ , final values for  $T$  were about 80 for I and II, 80 for VI and VII and 73 for III, IV and V. For untanned skin,  $T = 50$ . The high values of  $T$  for I and II are attributed to their high natural acidities. With 2 exceptions,  $T$  increased with decreasing initial  $p_n$  value of the ext. When the  $p_n$  value of III was lowered from 4.95 to 3.10 by adding 10%  $\text{AcOH}$ , the initial rate of increase of  $T$  was even greater than in the case of I, but  $T$  reached a max. in 6 days and thereafter declined considerably. The leather thus tanned was very brittle. Slight acidification of III, to  $p_n = 3.60$ , produced only a slight increase in  $T$ .  $T$  was measured for com. leathers, after wetting with  $\text{H}_2\text{O}$ , and values obtained increased with the firmness of the leathers as detd. by the hand bending test. Conclusion. To obtain a firm leather, employ exts. such as I and II giving high values of  $T$  by reason of their high natural acidities and favorable tannin-nontanning ratio. H. B. Merrill

Experiments on the action of vegetable-tanned leather on metallic iron. I. Effect of leather greases on iron. V. Kubelka, V. Nemec and S. Zuvavlev. *Chem. tech.* 24, 200–4 (1925).—The fact that certain leathers in contact with Fe rapidly corrode the metal with concomitant darkening and deterioration of the leather, while other leathers show no such action after years of contact, indicates that certain constituents of the leather are at fault. Iron, in thin plates, was stored with various greases for 75 days, with intermittent heating on the water bath, with and without the addn. of pulverized vegetable-tanned leather (free from mineral acids and syntans). No action occurred with satd. hydrocarbons or glycerides contg. chiefly satd. acids; slight action occurred with glycerides of unsatd. acids, and marked action with unsatd. and oxidized free acids. The action resulted in corrosion of the metal, darkening of the oil and increase in free fatty acid content. The presence of leather accelerates the action. Most of the dissolved Fe is absorbed by the leather. H. B. Merrill

Drying heavy leather in five hours. T. K. Sherwood and L. I. Carono. *Chem. & Met. Eng.* 42, 520 (1935).—The skins are coated with a paste of kieselguhr. Sol. tannin drawn to the surface by capillarity during drying is deposited on the coating instead of on the grain of the skin, and is brushed off with the kieselguhr. This permits drying at much higher temps. than can safely be employed for non-coated leather. H. B. Merrill

Comparative wear of chrome, vegetable and retanned sole leather. R. C. Bowler and W. E. Fmley. *J. Am. Leather Chem. Assoc.* 30, 672–6 (1935).—Practical wear tests were made with com. soles tanned with Cr, with vegetable tanning materials and with various combinations. Results of 996 tests show that the av. time required to wear through a Cr-tanned sole exceeds that for vegetable-tanned by about 75%. Combination tanned or retanned soles have intermediate wear resistance and

the lighter the vegetable retannage the greater the wear. In general wear resistance increases with the percentage of hide substance, falls with the percentage of  $\text{H}_2\text{O}$ -sol. matter and is unaffected by variations in grease content or percentage of insol. mineral matter. Also in *J. Research Natl. Bur. Standards* 15, 262–7 (1935) (Research Paper No. 834).

H. B. Merrill  
Causes and prevention of decay in leather. R. F. Innes. *Leather World* 26, 927 (1934); *J. Am. Leather Chem. Assoc.* 30, 577 (1935).—A résumé. Cl. C. A. 28, 1215<sup>4</sup>, 29, 3547<sup>4</sup> H. B. Merrill

Copper staining. M. P. Raffe. *Leather World* 26, 1139 (1934). *J. Am. Leather Chem. Assoc.* 30, 577 (1935); Cl. C. A. 25, 5590<sup>7</sup>.—Cu introduced into the liquors largely by corrosion by chestnut ext. is pptd. as sulfide by  $\text{Na}_2\text{S}$  carried over from the lime liquors. The presence of large amts. of  $\text{SO}_2$  in the liquors will prevent this pptn.

H. B. Merrill  
Theory of leather dyeing. Gerhard Otto. *Collegium* 1935, 371–4, Cl. C. A. 29, 4204<sup>4</sup>.—Dyeing leather with acid or substantive dyes has been shown to be a primary valence reaction between the basic groups of the hide and acid groups of the dye, but there is a great difference between different dyes and leathers. The affinity of dye for hide substance and for sumac and gambier tanned leather was measured by detg. the  $p_n$  at which the hide or leather took up the max. amt. of free dye acid. This  $p_n$  is much lower for vegetable leather than for hide substance, dyeing is better the lower this  $p_n$  "limit". The results indicate that the primary valence reaction, for vegetable but not for Cr leather, is regulated by secondary valence forces which, if active, permit disocn. of new basic groups and the absorption of more dye. In leather dyeing there is no fundamental difference between acid and substantive dyes, they should both be called anionic dyes. I. D. Clarke

Some notes on the construction and operation of an experimental rocker section. Wm. P. Doelger. *J. Am. Leather Chem. Assoc.* 30, 658–72 (1935). H. B. M.

A study of keratin. Leonor Michaelis. *J. Am. Leather Chem. Assoc.* 30, 657–68 (1935).—An address. H. B. Merrill

Swelling of collagen. A. Deforge. *Haute aux cuirs* 1935, 223–6.—A brief review. H. B. Merrill

Restraining substances in gelatin (Steigmann) 5  
Sulfurization of oils and sulfur tanning (Abbassi) 27  
Azo dyes [for leather] (Brit. pat. 432,355) 25.

Treating hides and skins. I. G. Farbenind. A.-G. (Richard Klein, Heinrich Ulrich and Paul Kording, inventors). Ger. 618,049, Aug. 31, 1935 (Cl. 29. 1). Addn. to 579,374 (C. A. 27, 4715). Dried hides and skins are softened by treatment with aq. solns. of the salts or other products obtained by reacting aliphatic hydroxyamines with  $\text{SO}_2$  or a water-sol. sulfite. Examples are given.

Soaking hides. I. G. Farbenindustrie A.-G. Brit. 470,767, June 25, 1935. Addn. to 341,851. Hides are soaked in aq. baths contg. salts or other conversion products of  $\text{H}_2\text{SO}_4$  with amines contg. 1 or more alkyl radicals having not more than 8 C atoms substituted with 1 or more OH groups. The products may be obtained by leading  $\text{SO}_2$  into solns. of the amines or by reaction of the latter with aq. sulfite solns. Suitable amines are monoethanolamine, triethanolamine, methylbutanolamine, cyclohexylethanolamine, diethylbutanolamine, hydroxyethylmethylamine, etc. H. B. Merrill

Depilatory. Oonah M. Hedberg (to Oonah Ltd.). U. S. 2,018,359, Oct. 22. A compn. suitable for application to the flesh side of hides is formed with Na sulfide, a thickening agent and a soap, etc.

Tanning compositions. Alphon O. Jaeger (to American Cyanamid & Chemical Corp.). U. S. 2,017,963, Oct. 22. A "syntan" of the sulfonic acid type such as a sulfonaphthyl methane tan is mixed with a sol. carbohydrate material such as sugar which serves to facilitate

obtainment of a permanently light-colored leather. Nat-  
ural tanning agents also are used.

Phenolic condensation products. I. G. Harbennid  
A-G (Ludwig Orthner and Arnold Doser, inventors).  
Ger. 614,034, Sept. 5, 1935 (Cl. 12g 20 04). Complex  
alkali sol substances containing phenolic OH groups are  
treated at a raised temp. with an alkylene oxide, and the  
products are sulfonated. Suitable starting materials are  
phenolic synthetic resins, various types of which are  
specified. The alkylene oxide may be replaced by a  
corresponding acid or ester, e.g., glycidic or epichloro-  
hydrin. Numerous examples are given. The products  
are sol in water and are useful as *tanning and dispersing*  
*agents and as resins in dyeing*.

Filling leather. Lester M. Whitmore (to Leas and Mc-

Vitty, Inc.). U. S. 2,014,588, Oct. 22. A vegetable  
tanning ext. such as quebracho ext., an aldehyde such as  
 $\text{CH}_3\text{O}$  and an  $\text{NH}_4$  salt such as  $(\text{NH}_4)_2\text{SO}_4$  are caused to  
react between and on the fibers and fibrils of the leather to  
deposit insol. material.

Azo dyes for leather. Francis H. Smith and Crayton K.  
Black (to L. I. du Pont de Nemours & Co.). U. S.  
2,018,234, Oct. 22. A dye which dyes leather chocolate  
brown is formed by coupling diazotized sulfanilic acid with  
resorcinol, coupling this compd. with diazotized p-tartronic  
acid, treating to convert the nitro group, ortho to the  
hydroxy group, to an amino group, then diazotizing and  
coupling with *m*-phenylenediamine sulfonic acid. Similar  
dyes may be obtained with various similar components and  
numerous examples are given.

## 30—RUBBER AND ALLIED SUBSTANCES

C. C. DAVIS

Research in the cultivation and preparation of raw rubber.  
J. Grantham. *J. Roy. Soc. Arts* 83, 1031-68  
(1935). E. H.

The acetone extraction of raw rubber. V. The influence  
of heating at 100° and of storing of the acetone extract  
on the saponification value. Hidemaro Endo. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 389-91 (1935).  
cf. C. 1 29, 6795. Heating of the acetone ext. at 100°  
gave an initial decrease in the sapon. no. of the ext. with a  
subsequent increase, while storage gave an initial increase  
with a following decrease, and a return to the original  
value. *Detn. of the sapon. no.*—After extn. evaporate  
the acetone as rapidly as possible, dry the flask on a steam bath  
for as short a time as possible (not over 15 min., and  
usually 5 min. is sufficient), cool, weigh, add 20 cc. of 0.2 *N*  
ale. KOH, heat on a water bath for 60 min. and titrate  
the excess alkali with 0.1 *N* HCl (phenolphthalein).  
Exptl. data are presented. VI. The saponification value  
of the acetone-extracted residue of raw rubber. *Ibid.*  
433-6. The acetone-extd. residue, not specially dried,  
showed a considerable increase in the sapon. value (*R*)  
after 60 min. of heating. This may have been due to the  
absorption of KOH. *R* was in general higher after 3 hrs.  
extn. than after 16 and 24 hrs. Drying of the extd. residue  
for 1 hr. in a desiccator and heating for 30 min. in the  
detn. of the sapon. value gave the best results. Part of  
the free acids and saponifiable substances decomposed  
and escaped by volatilization during acetone extn. for 16 and  
24 hrs. Karl Kammermeyer.

Rubber latex as a manufacturing material. D. F.  
Twiss. *J. Roy. Soc. Arts* 83, 1075-91 (1935).—See C. A.  
29, 4940. E. H.

The natural decomposition of the rubber in the latex  
of *Hevea* in relation to the question of the function of the  
rubber in the living plant. D. Spence. *J. Research*  
*Assoc. Brit. Rubber Manufs.* 4, 87-91 (1935). cf. C. A.  
29, 8393. Rubber in the cortical tissue of guayule has  
been decomposed by each of several strains of anaerobic  
organisms. Rubber in dialyzed and dild. *Hevea* latex was  
practically all decomposed in 1 month at 30° by 4 different  
forms of *Actinomyces* under aerobic conditions.

D. F. Cable

Natural and synthetic rubber. XV. Oxygen in rubber.  
Thomas Midgley, Jr., A. L. Henne, & F. Shepard and  
Mary W. Renoll. *J. Am. Chem. Soc.* 57, 2318-21 (1935);  
cf. C. A. 28, 4935. Natural rubber contains O, while  
synthetic rubber is O free. This O appears to be in an  
HO form, and its quantity corresponds to about 1 HO  
group for each 1000 isoprene units of the rubber mol. As  
rubber oxidizes, either by milling or exposure to O and  
sunlight, the following events occur: the H/C ratio decreases,  
the combined O increases, the active H increases and the mol. wt. (measured by cryoscopic in  $\text{C}_6\text{H}_6$ ) decreases.  
The thionyl Grignard results still lack significance.  
The O attaches to the rubber mol. in some peroxide  
linking, this linkage rearranges to an HO group, with  
liberation of O, and one of the HO groups joins with H

and are eliminated as  $\text{H}_2\text{O}$ , hence the lowering of the H/C  
ratio. The mol. is ruptured during or as the result of 1  
of the above reactions. C. J. West

Chlorinated rubber. E. Fraser Smith. *Oil Colour*  
*Trades J.* 88, 1251-3 (1935).—A review from 1859 to  
date, of the chlorination of rubber, its manuf. and the  
application and uses of chlorinated rubber. W. H. B.

Research steadily broadening rubber's automotive  
usefulness. J. D. Morton. *Automotive Ind.* 73, 506-9  
(1935).—A résumé of the increasing uses of rubber in  
automotive construction made possible by the bonding of  
rubber to metal. The old process of brass-plating steel  
and then bonding rubber to the brass has been supplanted  
by newer methods, and even Duprene can be adhered to  
practically all metals and alloys, except those  
contg. Mg, with a uniform bond which does not sep. under  
a stress of 250 lb. per sq. in. A. L. Kaye

A fatigue-cracking test for tire-tread compounds.  
Some of the laws of fatigue. F. T. Raimer and R. H.  
Gerke. *Ind. Eng. Chem., Anal. Ed.* 7, 368-73 (1935).  
A new app. and technique for evaluating the resistance of  
tire-tread vulcanizates to fatigue-cracking is described and  
illustrated in detail. The novel features of this method  
are the precision of the strain cycle, the quant. measurement  
of the degree of cracking and the initiation of the  
cracks with  $\text{O}_3$ , all of which enhance the validity of the  
method. Tests show that cracking by  $\text{O}_3$  and through  
fatigue are additive, that the rate of growth of cracks is a  
function of the max. strain, that endurance limits may  
exist, and that the addn. of antioxidants decreases the  
rate of growth and increases the limit of endurance.  
Results obtained by this method are shown to agree in a  
comparative way with shoulder-cracking of tires in service.  
The test is useful in evaluating antioxidants, accelerators,  
softeners, etc. Data obtained by this method also make  
possible the deduction of certain general laws of fatigue-  
cracking. C. C. Davis

Rubberizing fabrics by calendaring. Antoine Trépas.  
*Caoutchouc & gutta percha* 32, 17031-3 (1935). cf. C. A.  
29, 6466. A further description, illustrated with diagrams.  
C. C. Davis

Hydraulic presses for vulcanizing rubber floor coverings  
(Brit. pat. 430,979) 1. Impregnating textile materials  
with rubber latex (U. S. pat. 2,019,420) 25. Transforming  
hydrocarbons [product for vulcanizing rubber] (Brit.  
pat. 432,568) 22

Creaming latex. Rubber Research Institute of Malaya.  
Brit. 430,935, June 27, 1935. Gum extd. from the  
seeds of the locust bean is used as a creaming agent for  
latex, preferably as a 3% soln. in  $\text{H}_2\text{O}$ . The rate of  
creaming is augmented, and stability of the cream in-  
creased, by an increase of alk., of the latex prior to the  
operation, e.g., by the addn. of  $\text{NH}_3$  up to 1.5% of the  
rubber. The addn. of 0.3 part of gum to 100 parts  $\text{H}_2\text{O}$

in such a latex yields a cream of approx. 60% rubber content in 48 hrs

Rubber. Arluc R. Kemp (to Electrical Research Products, Inc.). Brit. 430,887, June 26, 1935. Partially depolymerized rubber is produced by heating crude rubber in soln in an org. solvent, e. g., decahydronaphthalene, tetrahydronaphthalene, in an autoclave at well above 100° for a sufficient time to cause the rubber to become and remain a viscous liquid after cooling, and seep the solvent.

Rubber. Ralph M. Ungar and Philip Schidrowitz. Brit. 431,869, July 17, 1935. The process of Brit. 368,902 (C. A. 27, 2338) for softening crude rubber is applied to initial crude rubber, defined as rubber 1st or freshly formed upon coagulation, gelling or pptn from latex, or upon spray drying thereof, in contrast to smoked sheet, crepe and like known forms of crude rubber. In an example, latex is coagulated by  $\text{AcOH}$  and the coagulum is washed, crumbed and drained. Further  $\text{H}_2\text{O}$  may be removed by centrifuging, pressing or air-drying. The crumbs are filled into trays which are placed on steam-heated shelves in a Passburg oven, the oven is closed and the air evacuated and the shelves are heated to heat the rubber to about 270–290°F. until it is completely dried. Air is then admitted and the heating continued for 10–30 min. The trays are then removed and stacked with an air space of about an in. between them. When the rubber has cooled to about 120°F., it is removed from the trays and may be agglomerated to condition it for shipment or use.

Rubber compositions. Alfred L. Hock and Peter Spence & Sons Ltd. Brit. 430,709, June 10, 1935. A  $\text{H}_2\text{O}$ -sol. inorg. alkali or alk. salt and an org. accelerator are incorporated in a rubber mix contg. a highly adsorbent siliceous filler. The preferred alkali is water glass and the filler is preferably pptd.  $\text{SiO}_2$  prepd. as in Brit. 294,681 (C. A. 23, 1999), 299,453 (C. A. 23, 3314) and 337,903 (C. A. 26, 4022). In an example, the accelerator is diphenylguanidine.

Rubber compositions. Pure Calcium Products Co. and Arthur H. Stevens. Brit. 432,032, July 15, 1935. Addn. to 379,942 (C. A. 27, 4130). The ingredient of rubber mixes of 379,942 that is made by grinding an earthy material, e. g., whitening, with 0–10% of an org. acid, salt or ester in a ball mill having balls of less than 2 in. in diam., occupying at least 15% of the vol. of the mill and being driven at least 25% of the crit. speed is here prepd. in a pebble, rod, tube or like mill under the same conditions of size, vol. and speed. The material may be used as an ingredient of paints, putties, etc.

Rubber compositions, etc. Edward A. Murphy, Frank T. Purkis and Douglas F. Twiss (to Dunlop Rubber Co., Ltd.). U. S. 2,018,313, Oct. 22. For producing substantially uncoagulated aq. compns. of rubber or the like from their aq. dispersions and suitable for use as adhesives in securing fibrous materials to rubber, a rubber dispersion or the like, without coagulation, is admixed with an aq. emulsion of a rubber solvent such as coal-tar naphtha in the presence of one or more suitable destabilizing agents such as acetone. Cf. C. A. 29, 3104.

Porous rubber. International Latex Process Ltd., Wilfred H. Chapman, Eric W. B. Owen and Donald W. Pounder. Brit. 431,575, July 11, 1935. Aq. dispersions of rubber or like material are converted into a froth in which the rubber is still in the reversible condition and the mass is allowed to coagulate, the pressure being reduced before, during or immediately after the coagulation. Before frothing, froth-forming agents, e. g., soap, saponin, may be added. Dispersions of coagulated, vulcanized, synthetic, waste or reclaim rubber, or natural or concd. latex, may be used. The product may subsequently be vulcanized, dried and otherwise treated.

Cellular and porous rubber. Ugo Pestalozza and Società italiana Preli. Brit. 431,410, July 8, 1935. Latex or a like dispersion is mixed with a separately prepd. foam and the dispersion is coagulated. The cellular coagulum, unless the dispersion used is pre-vulcanized, is usually subjected to vulcanization. Among examples, 1

rubber dispersion mixing comprising 55% latex preserved with 0.5% of  $\text{NH}_4\text{I}$ , 180, mineral oil 5,  $\text{ZnO}$  1,  $(\text{AcO})_2\text{Zn}$  1,  $\text{Zn}$  dimethyldithiocarbamate 0.4 and 8.2 g., having a 50% final concn. in solids, is mixed with 120 cc. of a foam obtained by introducing air into a 5% aq. soln. of glue contg. 0.5% of an alkali soap, in a whipping machine, and the resulting mixt. is poured into a mold and heated 30 min. at 60° and then 1 hr. at 100°.

Granulated rubber suitable for compounding with various materials. Royce J. Noble (to Hexatex Corp.). U. S. 2,019,055, Oct. 29. An insolubilizable hydrophilic stabilizer such as  $\text{NH}_4$  caseinate is added to rubber latex, the latex is flocculated with an agent such as  $\text{ZnCl}_2$  which insolubilizes the stabilizer, the mass of rubber flocs is sepd. from the latex water and the wet mass is dewatered until it becomes a firm but crumbly cake, and the latter is granulated.

Hard-rubber dust. International Latex Processes, Ltd. Ger. 618,181, Sept. 5, 1935 (Cl. 39b 5). See Brit. 417,031 (C. A. 29, 16824).

Coloring rubber and like materials. I. G. Farbenind. A.-G. Ger. 618,342, Sept. 6, 1935 (Cl. 39b 5). Use is made of water-insol. diazo dyes obtained by coupling tetrazotized benzidine or its alkyl, alkoxy or halogen substitution products with the Me or Et esters of 1-phenyl-5-pyrazolone-3-carboxylic acid or its derivs. substituted in the phenyl residue by a halogen atom or an alkyl or alkoxy group. Fast yellow to violet colorings are produced. Several dyes are described. Cf. C. A. 29, 84067.

Isomerizing rubber. Walter E. Lawson (to F. I. du Pont de Nemours & Co.). U. S. 2,018,678, Oct. 29. Anhyd. HF is added to a soln. of rubber dissolved in a solvent such as  $\text{C}_2\text{H}_5$ .

Plasticizing rubber. Ira Williams and Carroll C. Smith (to E. I. du Pont de Nemours & Co.). U. S. 2,018,643, Oct. 22. Unvulcanized rubber is plasticized by subjecting it, in the absence of more than 3% of S, to sufficient amts. (suitably about 0.5%) of an unsym. substituted hydrazine such as phenyl hydrazine for sufficient time (suitably about an hr. at 70°) to effect a marked increase in the capacity of the rubber to flow under a load. U. S. 2,018,644-5 also relate to plasticized rubber products and rubber cements formed with unsym. substituted hydrazines and give numerous examples.

Preserving rubber. Imperial Chemical Industries Ltd., Wm. Baird and Tom Birchall. Brit. 431,577, July 11, 1935. The aging of rubber is retarded and its resistance to flexing increased by the addn. to the mix of an *N*-alkyl- or -aralkyl-2-amino-1,3,5-xyleneol, e. g., 2-ethylamino-1,3,5-xyleneol, made by heating EtBr and 2-amino-1,3,5-xyleneol (I) at 170°, and 2-benzylamino-1,3,5-xyleneol, made similarly from benzyl chloride and I.

Rubber-preparing machines. Rupert T. Cooke and Francis Shaw & Co. Ltd. Brit. 431,012, June 28, 1935. Machines, e. g., for milling and kneading rubber, comprise 2 or more rollers having raised portions of comparatively large area, these portions and the bodies proper of the rollers being substantially concentric with the roller axes and the portions on 1 roller passing through the hollows left by the raised portions of the other roller.

Rubber threads. Hans Schuller, Emil Matzner and Armand Kalich. Brit. 430,982, June 25, 1935. Threads of rubber or similar material are made by depositing a band, from a liquid contg. rubber or the like, continuously on a moving support of the same or substantially the same width as the band being produced and feeding the latter directly and continuously into a twisting device to form the thread. The band may be dried or vulcanized before entering said device but only to such an extent as leaves it sufficiently adhesive to enable thread formation. App. is described. In 431,031, June 25, 1935, divided on 430,982, a continuously produced rubber band is twisted under tension by rotation about its longitudinal axis in a partially dried and vulcanized adhesive condition by means of a twisting device rotating continuously about the longitudinal axis of the band. The vulcanization of the threads is then completed.

Rubber threads, etc. Libbrecht Kautsch. Industrie

Gomma Torino and Alexander T. Maximoff. Brit 430,828, June 26, 1935 Rubber threads, ribbons, strips or films are made from aq dispersions of rubber, etc., vulcanized or not, by forcing the dispersion through a die into a coagulating medium, the dispersion being displaced from a closed tank by means of an auxiliary liquid which is delivered from a measuring pump. The auxiliary liquid may enter into direct contact with the latex, in which case it must be a liquid which is immiscible therewith and easily separable therefrom, e.g., Hg. Alternatively, a 2nd auxiliary liquid, which is immiscible with the dispersion, or a piston, plunger or membrane may be used. In this case, H<sub>2</sub>O may be used in the measuring pump.

Coating surfaces with liquids, etc. Dunlop Rubber Co. Ltd., Wm H. Pearce and Geo. H. Perry. Brit 431,381, July 8, 1935 For improving the gas-retaining qualities of inner tubes for tires and like hollow and distensible articles, the collapsed article, prior to vulcanization, is inserted within a chamber of an internal diam. equal to the external diam. of the unvulcanized article, the pressure between the article and the chamber is reduced so that the tube is positioned without deformation against the wall of the chamber under atm. pressure and the tube is subjected on its internal surface to a coating operation.

Means for spreading rubber dispersions on sheet materials. International Latex Processes, Ltd. (Charles Hamilton Dennison, inventor). Ger 618,180, Sept 5, 1935 (Cl. 39a 10 06). See Brit 417,559 (C. A. 29, 2394<sup>4</sup>).

Rubber products. Rubber-Latex-Poeder-Compagnie N. V. Fr 784,169, July 22, 1935 Rubber dust is mixed with a vulcanizing agent, fillers, etc., in the dry state, plastified and vulcanized.

Rubber articles having a wrinkled surface. Arthur E. Barnard (to U. S. Rubber Co.). U. S. 2,018,503, Oct 22, 1935 (C. A. 29, 4633<sup>4</sup>).

Hollow rubber articles such as inner tire tubes. Fred T. Roberts (to Frank A. Daly, as trustee). U. S. 2,019,361, Oct 29, 1935 App. and various operative details of manufacture are described.

Articles such as shoe soles, etc., from rubber dispersions. Douglas F. Twiss and Eric W. B. Owen (to Dun-

lop Rubber Co. Ltd.). U. S. 2,019,239, Oct. 29, 1935. In producing articles such as soles or matting from mixts. of aq. dispersions of rubber material, cement such as portland cement and "Ciment fondu" and a gelatin or sol alginate, the hydrophilic colloid in the mixt. is converted by an addnl. gelling reagent into an irreversible gel prior to setting of the cement and coagulating of the aq. dispersion. Various examples are given.

Rope formed of strands embedded in and surrounded by rubber composition. Andrew Robertson (to Robertson's Rope (Patents) Ltd.). U. S. 2,018,230, Oct. 22, 1935. Various structural and mfg. details are described.

Trimming devices for tire-building machines. National Rubber Machinery Co. Brit 431,217, July 3, 1935.

Extrusion apparatus for molding and vulcanizing coverings on cables. Archie Reed Kemp (to Electrical Research Products, Inc.). Brit. 432,110, July 22, 1935.

Heavy cables are covered with thick layers of very soft rubber compn. by extrusion and the coating is vulcanized while the cable is maintained in a vertical position to prevent decentration. A typical coating compn. is crepe rubber 100, S 1, ZnO 1, stearic acid 0.5, diphenylguanidine 0.5, aldehyde-amine condensation product 3, petrolatum base wax of high m. p. 2 and phenyl- $\alpha$ -naphthylamine 2 parts.

Vulcanizing. Alexander Johnston and The North British Rubber Co. Ltd. Brit 430,911, June 24, 1935.

In vulcanizing rubber goods or treating other articles under heat and gas pressure, the heat is obtained by compressing the gas, which travels in a closed circuit, above the working pressure and allowing it to expand against that pressure. App. is described.

Vulcanized rubber solution. Walter Alexander. U. S. 2,019,207, Oct. 29, 1935. A rubber soln. suitable for use in varnishes, etc., is prepd. by depolymerizing rubber at 200-300° until a syrupy consistency is attained, then dissolving and vulcanizing the depolymerized rubber in a solvent (such as xylene) for the depolymerized rubber and the resulting rubber product formed by the vulcanization.

Machine and method for vulcanizing rubber belting and like articles. Boston Woven Hose and Rubber Co. Brit. 430,885, June 26, 1935.



# CHEMICAL ABSTRACTS

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## 1—APPARATUS, PLANT EQUIPMENT AND UNIT OPERATIONS

W. L. BADGER

An efficient fume hood Geo W Muhleman *J. Chem. Education* 12, 591(1935) E. H.

German lumber as building material in the chemical industry. Romstetter *Chem. Fabrik* 1935, 446-50 — Various uses of wood, some of its advantages over metals for certain purposes, and methods of protection are discussed. J. H. Moore

Theory and calculation of the two-phase process in a rectifying column. J. P. Kahlrov. *Khimistrol* 7, 402-10 (1935).—A discussion with math treatment. C. B.

A flexible-spiral manometer for measurement of low pressures. Louis Henry. *Bull. soc. chim. Belg* 44, 311-14 (1935).—Movement of the manometer pointer tip is detected by an amplified photoelectric current provided with compensation for variations in intensity of the incident light source. This method nullifies the effect of vibration of the pointer, and permits a sensitivity of  $2 \mu$  of Hg difference in pressure. H. A. Beatty

Application of the methane cycle in the liquefaction of air. M. Z. Ruzman and Yu. M. Ryabinin. *Khimistrol* 7, 461-81(1935).—An auxiliary methane cycle for the reduction of energy consumption in the liquefaction of air by the Linde method is treated mathematically. The calculations include the simple methane cycle, methane cycle with ammonia cooling and methane cycle with high pressure and preliminary cooling. Chas. Blanc

Theory and calculation of the diffusion burner and its application in industry. A. K. Shakhia. *Khimistrol* 7, 420-6(1935).—A discussion of literature with 10 references. Chas. Blanc

An x-ray powder camera for specimens at various known temperatures. W. H. Barnes and W. F. Hampton. *Can. J. Research* 13A, 73-81(1935).—An x-ray camera designed specifically for pin-hole photographs of frozen gelatin gels at temps. between  $0^\circ$  and  $-60^\circ\text{C}$ . is described. The novel feature of the camera is the method of mounting the specimen on the surface of a copper block, the temp. of which depends on a controllable circulation of acetone at a controllable temp. The adaptability of the camera to modifications required by specific conditions of specimen or temp. other than those for which it was primarily designed is pointed out. It is shown that the camera appears to be suitable for the study of a great variety of materials over a considerable temp. range both below and above  $0^\circ$ , whenever a single crystal spectrograph, or an ionization spectrometer, is not required. Also in *Rev. Sci. Instruments* 6, 342-4(1935). J. W. S.

Neon lamp as a counter. II. Potential and delay in discharge, processes of extinction and rekindling. Giorgio Valle. *Nuovo cimento* 12, 426-40(1935); cf. C. A. 29, 2399.—The critical time is of the order of  $10^{-3}$  sec. Beyond this the counter is not effective. J. B. Austin

Mercury-vapor pumps with somewhat elevated back pressures. Louis Henry. *Bull. soc. chim. Belg* 44, 307-10(1935).—A simple Hg-vapor ejector is described, which operated at a vapor pressure of 250 mm. against a back pressure of 15 mm. produces a vacuum of 0.001 mm. H. A. Beatty

Diffusion coeffs. in alc-water mixts (Mouquon, Cathcart) 2.

Bubbler plate column suitable for fractional distilla-

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tions John H West U. S. 2,020,761, Nov. 12. Structural and operative details.

Means for transforming an intermittent distillation installation into a continuous one. Koehler, Bosshardt & Cie. Fr. 784,699, July 22, 1935

Apparatus for distilling and rectifying alcoholic liquids, etc. Akt.-Ges. vorm. Skodawerke und Eugen Gregor. Austrian 142,882, Sept. 25, 1935 (Cl. 6c)

Rectification apparatus. Oesterreichische Ags-Werke A.-G. Austrian 142,786, Sept. 10, 1935 (Cl. 6c)

Crystallizing salts. Soc. générale d'évaporation (Procédés Frache & Bouillon). Fr. 784,753, July 24, 1935

App. for circulating the soln. through a crystallizer and a heat exchanger

Mixing devices. American Machine & Foundry Co. Brit. 432,849, Aug. 2, 1935. A whisk or stirrer is described.

Gas-pressure regulator. Edward Zahn (to Zahn & Nagel Co.) U. S. 2,020,492, Nov. 12. Various structural, mech. and operative details.

Photoelectric color comparator. Clarence G. Stone and Abraham Edelman. U. S. 2,020,281, Nov. 5. Various structural, optical and operative details.

Oil-temperature regulator suitable for use with aircraft engines. Roland Chilton (to Reed Propeller Co.). U. S. 2,021,129, Nov. 19. Various structural and operative details.

Stream-lined hydrometer bulb. Geo. Ruehfel U. S. 2,020,968, Nov. 12. Structural details.

Manometer. Erich Bandoly (to Taylor Instrument Cos.). U. S. 2,021,250, Nov. 19. Structural details.

Thermocouple pyrometer. Charles H. A. F. L. Ross U. S. 2,019,695, Nov. 5

Thermometer with a glass tube. Herman Neuwirth U. S. 2,021,687, Nov. 19. Structural details.

Tubular thermometer. Harry D. Bolton (to Taylor Instrument Cos.). U. S. 2,021,283, Nov. 19. Structural details.

Annealing furnace. Wilhelm H. Engelbertz. Ger. 616,787, Aug. 5, 1935 (Cl. 18c. 10.01).

Filters. Wm. A. Hoveman and Frederick C. Hoveman. Brit. 431,586, July 8, 1935. To facilitate efficient cleansing by reverse upward flow, filter beds are divided into small areas by partitions, each of which supports a trough.

Filters. Joseph A. Pickard. Brit. 432,102, July 22, 1935. Filter cells are formed with walls of paper or textile fabric sufficiently stiff or rigid to form a self-supporting structure without internal supporting means, the walls being spaced apart only by the inherent roughness of the material or by inequalities produced by suitable treatment of the material.

Filters. Frank W. Young. Brit. 432,852, Aug. 2, 1935

A filter comprises a rotary drum mounted in a tank and having independent filtering compartments on its periphery, each compartment being provided with a port communicating with the interior of the drum and opened and closed successively by a gravity-operated valve, and the 2 ends of the drum being closed except for a passage through 1 end establishing communication between the interior and exterior of the drum.

Rotary-drum filter. Frank W. Young. Fr. 785,671, Aug. 16, 1935.

Filtering apparatus (with a rotary filtering drum) suitable for use with various liquids. Kinya Nagao. U. S.

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2,021,081, Nov 12 Various structural, mech. and operative details

Gas filters Louis Raichlen, André Gottraux and Jean Laroche Fr 784,773, July 24, 1935 Finely powd. charcoal, activated carbon, lampblack or animal black is incorporated in paper or paper pulp during its manuf. for use in filtering air, e. g., in gas masks

Gas filter (plate type). Midwest Steel & Supply Co Inc Ger 619,183, Sept 25, 1935 (Cl 12e 2 01) This corresponds to Brit 322,684 (C A 24, 2644)

Air filter Lullius L S Nelson U. S. 2,019,867, Nov 5 Structural features

Air filter Charles A Winslow (to Catherine B Winslow) U. S. 2,021,215, Nov 19. Structural details

Air filters St George's Engineers Ltd, and Frederick W Wilson Brit 432,760, Aug 1, 1935.

Air filter including cotton fiber. Robert S Curley (to Saco-Lowell Shops) U. S. 2,021,853, Nov. 19. Structural features

Air filtering and -cleaning device containing oil Leonard A Sondburg U. S. 2,021,524, Nov. 19 Structural details

Air-filtering and -heating apparatus, carried as a unit in a casing below a railway or other vehicle J. Stone & Co Ltd and John F B. Vidal Brit. 432,117, July 22, 1935

Dust filters Società italiana Pitelli Brit 433,186, Aug 9, 1935 A filter for removing aerosols, etc., from the atm. comprises an intimate mixt. of fibers, e. g., of wool, cotton, kapok, rayon or asbestos, with an inert powder, e. g., talc, cork, ebonite or resin, 1 of the 2 constituents being fusible by heat and the constituents being bonded by such fusion In 433,190, Aug 9, 1935, a filter is formed of an intimate mixt. of fibers with an inert powder, e. g., wool and resin, spun into a yarn and used as such or as a fabric woven wholly or partly therefrom The mixt. may be produced by twisting the fiber in a liquid suspension of resin and ebonite, or the materials may be bonded by slight fusion of 1 of them.

Apparatus for removing dust from gases Joseph C E V Le Gras de Marillac Fr 785,917, Aug 22, 1935

Filtering materials Società italiana Predelli Fr 784,857, July 27, 1935 A material such as that of Fr 744,530 (C A 27, 4325) is heated till either the fibrous material or the powder begins to melt and is kept at that temp. until an intimate union of the constituents takes place Settling out of the powder is thereby prevented

Apparatus for cleaning sand filters Norman G Elliot Brit 432,742, Aug 1, 1935

Discharging filter cake from filter elements of a continuous rotary suction filter Horace B Whitmore. U. S. 2,022,069, Nov. 26 The filter cake is washed from the filter element by a projected stream opposite in direction to the direction of rotation of the filter elements so that the discharge cake will pass over filter areas still under suction Various app details are described

Sifting apparatus and dust bins Rance H Stevens Brit 432,890, Aug 6, 1935

Pneumatic separating Blaw-Knox Co Brit 433,147, Aug 9, 1935 In cleaning segg. or classifying granular material by the winnowing action of an air current, the material is poured in a hollow freely-falling stream and the air is blown downwardly into the hollow space and passes out through the material transversely

Centrifugal separator of solids or liquids from gases Maschinenfabrik Friedrich Haas Ges m b H Fr. 784,911, July 29, 1935

Magnetic separators Herbert H Thompson and Alfred F Davies Brit 432,431, July 26, 1935

Gravity separation apparatus The Dorr Co., Inc. Brit 433,051, Aug 8, 1935 A rectangular settling tank is provided with scrapers which, as they move from 1 end to the other in either direction, push the deposited matter laterally into a longitudinal channel or zone from which it is discharged by another scraper.

Gravity separation apparatus Alexandria Water Co Ltd Brit 433,372, Aug. 6, 1935 A settling basin is of less depth than its length or diam. and the upper portion

of its walls is vertical The liquid under treatment enters from conditioning tanks through a chamber and an inlet-opening extending upward from the lower end of the vertical part of the wall and controlled by adjustable gates and arranged so that the liquid enters so as to give a rotary motion to the liquid in the basin.

Apparatus for separating materials such as grease and waste water by gravity. Maurice Loeb U. S. 2,022,118, Nov. 26 Structural and operative details

Apparatus for separating powders of different sizes by the action of air currents Paul S Roller. U. S. 2,019,507, Nov 5 Structural and operative details.

Apparatus for separating dust and other light material from heavier matter, e. g., from abrasive after its use in sand-blast apparatus St George's Engineers Ltd. and Frederick W Wilson Brit 432,222, July 23, 1935

Apparatus for treating gases in the presence of contact agents Soc. anon française pour la fabrication des essences & pétroles (Saffep). (Harrison, applicant in U. S.) Fr. 785,698, Aug. 16, 1935 The vessel, containing the contact material and inlet and outlet tubes for the gas in the material, is so shaped on its inner walls that the tubes near the walls are surrounded by approx the same amt of contact material as the rest of the tubes.

Apparatus for washing air and other gases. Kurt Reichert. Ger 611,483, Sept. 30, 1935 (Cl 36d. 1.13)

Apparatus (with supersaturated liquid pools) for cooling gases as in oxidation of ammonia Stanley L Handforth (to E. I. du Pont de Nemours & Co.) U. S. 2,019,533, Nov 5 Structural and operative details

Liquefying gases. The General Electric Co Ltd and Robert E. Keelan Brit. 433,232, Aug. 12, 1935 In obtaining gases, e. g., O, from the atm., an engine is used in which compressed gases are expanded, the power of the engine driving an induction generator which is electrically connected with a -c power supply

Apparatus (with Venturi tube eductors) for mixing liquids and gases as in precipitating zinc sulfide, etc Arne J. Myhren and Byron Marquis (to New Jersey Zinc Co.) U. S. 2,020,850, Nov 12 Various structural and operative details

Balanced valve for controlling the rate of flow of liquids through conduits Hans Ernst (to Cincinnati Milling Machine Co.). U. S. 2,020,773, Nov. 12 Numerous structural, mech and operative details

Pressure-actuated meters for measuring the flow of liquids through conduits full of liquid. Electroflo Meters Co. Ltd. and Friedrich V A E Engel Brit. 432,416, July 25, 1935

Storage and delivery apparatus for highly volatile liquids such as liquefied propane and butane. Henry N Wade U. S. 2,021,394, Nov 19 Various structural, mech and operative details

Sedimentation apparatus suitable for sludge separations and liquid clarifications Harlowe Hardinge (to Hardinge Co.). U. S. 2,021,304, Nov. 19 Various structural, mech and operative details

Solidifying liquid materials such as milk, fruit juices, soap or resins, in finely subdivided form Robert R Bottoms (to Girdler Corp.) U. S. 2,020,719, Nov 12 A liquid material to be solidified and a liquefied refrigerant gas such as liquefied  $\text{C}_2\text{H}_2$  are introduced into a chamber (of a described app) in such relative proportions that the first mentioned material is completely solidified and the refrigerant is completely gasified

Spraying liquids in air to effect its purification Frederick P. Bingham (to R. C. Mahon Co.). U. S. 2,021,251, Nov. 19 Various details of app. and operation are described, suitable for cleaning air for paint spray booths

Rotating-drum drier. Büttner-Werke A-G Ger 616,823, Aug 6, 1935 (Cl 82a 19 04)

Apparatus for drying crystals obtained from solutions Ateliers Neyret Beylier Fr 785,729, Aug 17, 1935

Vertical apparatus for drying descending comminuted materials by hot gases Albert Hantley (to Mid-Co Products Co.). U. S. 2,020,504, Nov 12 Various structural and operative details

Centrifugal apparatus suitable for drying small coated

metal articles. Irving H. Peck. U. S. 2,020,950, Nov. 12 Various structural and operative details

Multiple-effect evaporator. Hugh K. Moore (to Brown Co.). U. S. 2,020,038, Nov. 5. Centrifugal pumping of liquor at its b. p. from one effect to another is facilitated by injecting into the centrifugal chamber a small stream of cold liquor of higher concn. Various structural and operative details are described

Evaporating apparatus operated with superheated steam. Irma Rud. O. Meyer. Ger. 619,072, Sept. 21, 1935 (Cl. 12a, 2). The app. is useful for the recovery of salts of low-water content from solns., particularly for the recovery of anhyd. or approx. anhyd.  $MgSO_4$  from solns. of crude kieserite

Multi-stage evaporating apparatus operated with superheated steam. Hermann Klingler. Ger. 619,073, Sept. 21, 1935 (Cl. 12a, 4)

Apparatus for evaporating liquids and solutions. Paul Knecht. Fr. 784,847, July 25, 1935

Rotary evaporation or distillation apparatus. Edwin M. F. Guignard. Ger. 619,098, Sept. 27, 1935 (Cl. 12a, 2)

Method of joining tubes to plates in evaporating apparatus of the type in which heating fluid circulates through concentric tubes mounted in an evaporating chamber. Apparatus et evaporateurs. Kestner. Brit. 432,934, Aug. 6, 1935

Gas-heated regenerative furnace. Ofenbau-Ges. m. b. H. Ger. 619,391, Sept. 30, 1935 (Cl. 24c, 6)

Rotating drum furnaces. Metallgesellschaft A.-G. (Carl P. Debusch, inventor). Ger. 616,680, Aug. 28, 1935 (Cl. 40a, 550). Details of the gas- and liquid-tight joint between the rotating and stationary parts are given

Regulation of air and liquid fuel supply to furnaces. Barton H. Noland. U. S. 2,020,955, Nov. 12 Various details of app. and operation

Wall for high-temperature furnaces such as open-hearth furnaces. Arthur S. Nichols (to Illinois Clay Products Co.). U. S. 2,021,742, Nov. 19 Various structural details

Relief valve for use at high temperatures as in high-pressure steam lines. Perry H. Gentzel (to Crosby Steam Gage & Valve Co.). U. S. 2,021,773, Nov. 19 Various structural, mech. and operative details

Heat-exchanger. Maschinenfabrik Hans Summon. Austrian 142,817, Sept. 25, 1935 (Cl. 17c). Addn. to 117,563 (C. A. 24, 3407)

Tubular heat-exchangers, e. g., for refrigerant condensers or evaporators. The Liverpool Refrigeration Co. Ltd. and James Allan. Brit. 432,230, July 23, 1935

Straight-tube heat-exchangers, e. g., for gas condensers and coolers. Joseph Bishop, Wm. A. Leaver and Newton, Chambers & Co. Ltd. Brit. 432,080, July 19, 1935

Circulatory heat-exchange apparatus for effecting exothermic or endothermic reactions between fluids in flow. Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. Ger. 619,295, Sept. 27, 1935 (Cl. 12g, 1.01). Details are given of the use of the app. in the manuf. of acetone from  $C_2H_5$  and steam and from  $EtOH$  and steam

Tube bundle heat-exchange apparatus. James B. Forbes (to Alco Products Inc.). U. S. 2,021,856, Nov. 19 Structural features

Tube and fin heat-exchange apparatus suitable for use with heating gases. Charles E. Lucke (to Babcock & Wilcox Co.). U. S. 2,021,117, Nov. 12 Structural details

Tube, fin and chamber heat-transfer apparatus suitable for use with liquids and gases. Clive M. Alexander. U. S. 2,022,173, Nov. 26 Structural details

Tube and header heat-exchange apparatus suitable for use with steam and water, etc. Thomas H. Ireland. U. S. 2,021,009, Nov. 12 Structural details

Metallic plate heat-exchange apparatus. Delos P. Heath. U. S. 2,021,995, Nov. 26. Structural details of a device suitable for use as an evaporator in a refrigerating system

1 Gilled tubes for heat-exchangers. Charles A. Hubert. Brit. 432,936, Aug. 6, 1935 Addn. to 337,236 (C. A. 25, 1713)

Thermoelement. G. Siebert G. m. b. H. Ger. 616,023, Aug. 1, 1935 (Cl. 42, 8.01). The element consists of Pt, Rh or a Pt-Rh alloy conig. 5-15% Rh.

Apparatus for heating and filtering suction air for starting direct-injection engines. Soc. anon. Adolphe Saurer. Brit. 432,150, July 22, 1935

2 Impact pulverizers. International Pulverizing Corp. Brit. 432,101, July 22, 1935 Fluid under pressure is injected into a pulverizing chamber to rotate and pulverize a mass of material therein, the fine material being classified by the action of centrifugal force opposing the force exerted on the material by the pressure fluid as it escapes toward a coaxially disposed outlet

Jet-impact pulverizers. Paul Anger. Brit. 433,034, Aug. 7, 1935 In a pulverizer in which material is fed to the bottom of a funnel-shaped vessel where it is blown by an air jet through a mixing tube onto an impact member, the discharge of the pulverized material is principally at the sides of the pulverizer

Combined roller mill and impact pulverizer for seeds, bones, etc. Cathrina Gudman Jørgensen (trading as P. Jørgensen's Maskinfabrik ved C. Jørgensen). Brit. 433,001, Aug. 7, 1935

4 Ball or tube mills. Ernest Newell & Co. Ltd., Geo. L. Woodhouse and Arthur H. Moss. Brit. 432,999, Aug. 7, 1935 In a tube mill wherein the material is discharged through peripheral openings in the end of the drum which are surrounded by a stationary casing communicating with a discharge shoot, a chimney or shaft is provided on the casing to draw air through a hollow trunnion and through the discharge openings to exhaust from the mill the vapor generated by the grinding

Roller and breast mills. Franz Bernhard Lehmann (trading as (firm of) J. M. Lehmann). Brit. 432,708, July 31, 1935

Disk mills. Frederick J. E. China and Premier Colloid Mills Ltd. Brit. 432,570, July 30, 1935 In a disk mill in which the distance between working faces of the rotor and stator decreases from the point of entry of the material toward their periphery, the contours of the working faces of the rotor and stator are identical

6 Gyrotory crushers. Jean Meaux. Brit. 432,848, Aug. 2, 1935

Gyrotory crushers. Société ateliers Neyret Beylier. Brit. 432,954, Aug. 6, 1935

Air-conditioning apparatus. Geo. W. Langford. U. S. 2,019,575, Nov. 5 Structural and operative details

Air-conditioning apparatus. Clarke Redfield. U. S. 2,019,583, Nov. 5 Structural and operative details

7 Air-conditioning apparatus. Frank P. Walsh. U. S. 2,021,437, Nov. 19. Various structural details

Air-conditioning apparatus. Don A. Sargent. U. S. 2,021,521, Nov. 19. Various structural and operative details

Air-conditioning apparatus. Frank A. Whiteley. U. S. 2,021,583, Nov. 19. Various structural and operative details

8 Air-conditioning apparatus. Clark T. Morse and Edward L. Hogan (to American Blower Corp.). U. S. 2,022,133, Nov. 26 Structural and operative details

Air-conditioning apparatus utilizing ice. Samuel M. Anderson (to B. F. Sturtevant Co.). U. S. 2,020,003, Nov. 5 Various structural and operative details

Control device suitable for regulating the flow of fluids in air-conditioning apparatus, etc. Wm. G. Hillen and Frederic W. Bailey (to Carrier Engineering Corp.). U. S. 2,021,727, Nov. 19 Structural, mech. and operative details of a device which has thermostatic controls

Air-cooling device of the air-expansion type. Ernest A. von Seggern. U. S. 2,020,923, Nov. 12 Various structural and operative details

Vacuum apparatus and method for preserving perishables, drying, tanning, impregnating lumber, etc. Harry Hotcock. Brit. 432,532, July 22, 1935

Purifying and sterilizing liquids. Katadyn A.-G. Brit.

431,872, July 17, 1935 This corresponds to Fr 760,287 (C A 29, 3929)

Rotary-drum apparatus for treating materials with hot gases Johan M Pehrson and Ragnar V Pehrson. U. S. 2,020,960, Nov 12 Structural and operative details

Apparatus suitable for taking samples of pulverized materials from conveying air currents Thorleif Thorsten U. S. 2,020,529, Nov 12 Various structural and operative details

Apparatus for shipping pulverulent or granular materials such as flour, malt, cement, wheat or coal Graham C Woodruff (to L C L Corp.) U. S. 2,020,628, Nov. 12 Various structural features

Apparatus for applying coating or filling material to fabrics British Celanese Ltd., Albert Mellor and Ralph J Mann Brit 432,429, July 26, 1935

Apparatus for coating cores such as metal tubes with cement plaster by spiral windings, etc Agostino Rocca U. S. 2,022,009, Nov 26 Mech features

Machine for coating bodies with metal, particularly for coating the interior surfaces of hollow cylindrical bodies Heinrich Schlupmann Brit 432,209, July 22, 1935

Apparatus (with rotary pockets) suitable for the discharge of materials from vertical calcining retorts Edward P Gillette (to Gillette Research Corp.) U. S. 2,019,963, Nov 5 Mech and operative details

Nozzle for the shockless expansion of hot liquids such as water Henri B Reithinger (to Office national des recherches scientifiques et industrielles et des inventions and Camille Husson) U. S. 2,019,694, Nov 5 Structural details

Apparatus for carbonating water Wm C Buttner (to Bastian-Blessing Co.) U. S. 2,019,479, Nov 5 Various structural and operative details

Apparatus for removing solid deposits from the mouth of conduits such as vapor outlets of oil-conversion apparatus Howard Dummig (to Gasoline Products Co.) U. S. 2,020,241, Nov 5 Mech details

Apparatus (with a storage tank and compressor) for control apparatus using compressed air Willy Ochel (to A. Borsig Maschinenbau, A-G) U. S. 2,019,723, Nov 5 Structural, mech and operative details

Gas-storage cylinders Joseph H L Trevorrow and The Chesterfield Tube Co Ltd Brit 432,718, Aug 1, 1935

Solid carbon dioxide. Charles L Jones (to Adico Development Corp.) U. S. 2,020,189, Nov. 5. Materials such as water 0.01-0.15% and diethylene glycolmonoethyl ether 0.01-0.04% are added to improve the physical properties of solid CO<sub>2</sub>. Normal octane, glycerol, ethyl-

ene glycol, triethanolamine or clean low-cold-test lubricating oil also may be used, as may also EtOH, etc.

Solid carbon dioxide. Guido Mauri (to Mauri Refrigeration Patents Ltd.). U. S. 2,021,073, Nov 12 For obtaining a dense block of solid CO<sub>2</sub>, the CO<sub>2</sub> is absorbed in an absorption medium such as a soda soln and the CO<sub>2</sub> is driven off by heat and the sensible heat of the driven-off CO<sub>2</sub> and the sensible and latent heat of the jointly vaporized absorption medium are used as the "prime mover" of a refrigerating system, the driven-off CO<sub>2</sub> is compressed to slightly above its triple-point pressure, and the compressed CO<sub>2</sub> is cooled by the refrigerating system to a solidifying temp. App is described

Container suitable for holding solid or "semi-solid" carbon dioxide Harry B. Rudd (to Rudd Patents Corp.) U. S. 2,020,329, Nov. 12. Various structural details of a self-sealing container.

Apparatus for use of solid carbon dioxide as a refrigerating medium. Duncan McNeil (to Chas R McCormick Lumber Co.). U. S. 2,019,759, Nov. 5 Structural and operative details

Portable shipping container suitable for holding "dry ice" or various gases under high pressure. Percy C Avery (to Avery Products Co.). U. S. 2,021,622, Nov 19 Various structural and mech details

Electrically controlled valve suitable for refrigerating systems Charles C Hansen (to Refrigerating Specialties Co.). U. S. 2,020,833, Nov 12 Mech, elec and operative details

Endless belt, etc., in apparatus suitable for freezing ice cubes, etc. Ralph H Chilton (to General Motors Corp.) U. S. 2,021,047, Nov 12 Various structural, mech and operative details.

Refrigerating apparatus suitable for use with ammonia, etc. Gordon Varney U. S. 2,019,551, Nov. 5 Various structural and operative details

Separating components of solutions such as dichloro methane and oil in refrigerating systems Mahlon W Kenney (to General Household Utilities Co.). U. S. 2,021,691, Nov. 19. A porous device such as a wick-like material is placed on an inclined wall having a discharge at its lower end and extends over the upper end of the wall and dips into the soln and takes it up by capillary attraction, pressure and temp. conditions being such as to effect selective evapn of one component of the soln such as CH<sub>2</sub>Cl<sub>2</sub> and leave another, such as oil App. is described. Cf. C. A. 29, 64771.

Acetylene generator Rudolf Brendler, Ger. 616,553, July 31, 1935 (Cl 25b 25). Details of construction and arrangement are given

## 2-GENERAL AND PHYSICAL CHEMISTRY

FREDERICK L. BROVINE

Thomas Andrews R. Plank *Z. Elektrochem* 41, 804-6(1935)—Obituary G G

Carl Dussberg, 1861-1935. Alfred Stock *Ber.* 68A, 111-48(1935)—Obituary with portrait. G G

The life and works of Hippolyte Copaux Henri Perrot *Bull soc chim* [5], 2, 1777-85(1935)—Obituary with portrait G G

The life and works of Maurice Hannot André Kling *Bull soc chim* [5], 2, 1753-76(1935)—Obituary with portrait and list of publications G G

Jakob Meisenheimer, 1876-1934 W. H. Mills *J Chem Soc* 1935, 1355-9—Obituary G G

In memory of Stanislaw Tollaczko Emil Votoček *Collection Czechoslov Chem Communications* 7, 359-63 (1935)—Obituary with portrait and bibliography. E. H.

The teaching of physical science. J. A. Lauwerys *School Sci Rev.* 17, 161-70(1935). O. Reimnuth

The present state of the problem of electrolytic solutions Charles A Kraus *J Chem Education* 12, 567-71(1937). F H

The synthesis of ammonia E. B. R. Prudeaux. *School Sci. Rev.* 17, 191-202(1935) O. Reimnuth

Velocity-of-reaction experiments (avoiding the use of a thermostat). H. C. Palmer. *School Sci. Rev.* 17, 238 (1935). O. Reimnuth

Remarks on the publications "Theory of Flotation," Philipp Siedler. *Z. physik. Chem.* A174, 73-6(1935)—A discussion of papers by Wark (C. A. 29, 77039) and Ostwald (C. A. 29, 77031). R. H. Baechler

The theory and practice of adsorption investigations A. W. Wellings *School Sci. Rev.* 17, 181-90(1935) O R

Magnetochemical investigations of organic substances III. The deeply colored dimeric ketene of W. Langenbeck. Eugen Müller. *Ber.* 68B, 1883-5(1935)—The exptl method previously described (C. A. 29, 5320<sup>4</sup>, 6116<sup>4</sup>) indicates that neither Langenbeck's formula (C. A. 22, 2740) nor Wittig's formula (C. A. 23, 134) is adequate for L's dimeric ketene More exptl data are necessary for establishing its formula L. E. Steiner

When and by whom was alcohol first prepared from ethylene? B. Herstein *Chemistry & Industry* 1935, 881-4—Berthelot has often been incorrectly credited with

this discovery. Hennell made the discovery and presented partial proof, but the extension of this reaction to olefins generally belongs to Berthelot. J. S. Hicks

Boron content of sea water of the North Atlantic coast Norris W. Rakestraw and Henry E. Mahneke. *Ind. Eng. Chem., Anal. Ed.* 7, 425 (1935).—Moberg and Harding (C. A. 27, 4142) detd. the B content of samples drawn for the most part from the Pacific Ocean and found an av. of 4.50 mg. B per kg. of water. Some 75 samples taken in the Atlantic, analyzed by the same method, contained 4.75 mg. per kg. of water. The av. ratio of B/Cl was 0.0024.

The organic metabolism of sea water with special reference to the ultimate food cycle in the sea. Ancl Keys, E. H. Christensen and August Krogh. *J. Marine Biol. Assoc. United Kingdom* 20, 181-96 (1935).—Sea water, sterilized by filtration, loses little O when stored in the dark. Metabolic activities of sea water stop when there is still a reserve of  $\text{NH}_4$ ,  $\text{O}_2$  and org. matter. Phosphate may at times limit bacterial activity in sea water but this activity is independent of  $\text{NH}_4$ . C. M. McCay

The rate of liberation of phosphate in sea water by the breakdown of plankton organisms L. H. N. Cooper. *J. Marine Biol. Assoc. United Kingdom* 20, 197-200 (1935).—The phosphate content of sea water was detd. after addn. of animal and plant plankton. The former decomposes rapidly and sets free some of the org. P of the water as well as that in the plankton. Phytoplankton sets free only part of its P. C. M. McCay

Magneto-optics C. G. Darwin. *Proc. Roy. Soc. (London)* A151, 512-39 (1935).—Most discussions of magneto-optics lack generality because they are based on particular at. hypotheses and hence must be reexamined, if not revised, whenever changes are made in the atom models used. To avoid this difficulty, D uses a process, originally developed by Born, but now generalized to include the consideration of the absorption of light. All magneto-optic effects are described in terms of the "refractive tensor" and involve no appeal to the fundamental nature of the material. After a study of the properties of the "refractive tensor," various special problems are considered, such as what constitutes the effective static magnetic field for the purposes of optics, the development of formulas for the refractive effects of a general magnetized substance contg. both free and bound electrons, changes introduced by quantum theory, etc. The expl. data of the Kerr effect are applied to test the theory at various stages but, because of the uncertainties in the expl. values, these checks are qual. rather than quant. W. W. S. Stiller

Chemical elements and the unity of matter. I. F. A. Paneth. *Scientia* 58, 219-28 (1935).—A philosophical discussion. L. E. Gilson

Periodic classification of the rare earths. Herman Yagoda. *J. Am. Chem. Soc.* 57, 2329-30 (1935).—General discussion. A study of the radioactivity of thulium preps. is desirable. C. J. West

Preliminary study of the fractionation of isotopic isomers by distillation. D. F. Stedman. *Can. J. Research* 13B, 114-21 (1935).—Slight sepn. of some isotopic isomers were achieved by equil. rectification. With Cl the total sepn. amounted to 0.045 at. wt. units; 28.6% of the  $\text{O}^{18}$  was also removed from normal O by the fractionation of water, and in a short run with liquid O the normal concn. of  $\text{O}^{18}$  was raised from 0.2 to 0.25%. The last-mentioned sepn. can be carried considerably further with present equipment.  $\text{CH}_3\text{D}$  was synthesized. Its b. p. appears to be 0.5° lower than that of  $\text{CH}_4$ . The vapor pressures of a 56.8% soln. of  $\text{D}_2\text{O}$  were measured, and it is suggested that the published values of the vapor pressure of  $\text{D}_2\text{O}$  at temps. lower than 40° may be slightly too high.

J. W. Shipley  
The critical increment of ionic reactions. Influence of dielectric constant and ionic strength. W. J. Szwabry and J. C. Warner. *J. Am. Chem. Soc.* 57, 1883-6 (1935).—On the assumption that ionic reaction rates are functions only of temp., dielec. const. (D) and ionic strength ( $\mu$ ), equations are derived to predict the effect of D and  $\mu$  upon the crit. increment. The predictions agree satisfactorily

with observed effects in the reaction between  $\text{NH}_4^+$  and  $\text{CNO}^-$  ions to form urea in water soln. at 30° to 70° and in MeOH-H<sub>2</sub>O mixts. at D = 63.5 and D = 55.0.

L. W. Elder  
The mechanism of ionic reactions. The heat of ionic substitution reactions. R. A. Ogg, Jr. *Trans. Faraday Soc.* 31, 1385-92 (1935); cf. C. A. 29, 3901<sup>1</sup>.—The study of exchange reactions between alkyl halides and halogen ions was extended to the reaction of Me halides with the following neg. ions:  $\text{SO}_4$ ,  $\text{SO}_3$ ,  $\text{OH}$ ,  $\text{HS}$ ,  $\text{CN}$ ,  $\text{C}_2\text{H}_3\text{O}$ ,  $\text{C}_2\text{H}_5\text{O}$ ,  $\text{CH}_3\text{COO}$ . The theorem suggested previously, i. e., if a compd. is substituted by a series of neg. ions, whose repulsive forces are approx. const., arranged as to increasing heats of reactions, the energies of activation decrease in that order, is confirmed experimentally. Activation energies do not vary as widely as the heats of reaction, indicating some variation of repulsion. This affects identical substitution. The racemization of methylphenylcarbamol by alc. KOH, claimed by Ogg, Polanyi and Werner, C. A. 28, 6429<sup>2</sup>, was due to oxidation, for no reaction occurred over long periods in the absence of air. This indicates an activation energy greater than 40 Cal. This result confirms the theory. R. E. DeHight

Magnetic double refraction and light scattering in fused nitrates V. N. Thatté. *Proc. Indian Acad. Sci.* 2A, 244-8 (1935); cf. C. A. 28, 6635<sup>1</sup>.—The depolarizing factor, intensity of light scattering, and magnetic double refraction of fused  $\text{Zn}(\text{NO}_3)_2$  and  $\text{Cd}(\text{NO}_3)_2$  were measured at several temps. The ease with which these fused salts can be maintained in a supercooled condition extended the temp. range from 140° to 30°. The magnetic and optical anisotropies of the  $\text{NO}_3$  ion calcd. from these data are practically identical with those found for it in soln. and in crystals. W. W. Stiller

Influence of a magnetic field on the coefficient of viscosity of liquids. H. S. Venkataramiah. *Current Sci.* 4, 156 (1935).—In view of a recent publication by Raha and Chatterjee (cf. C. A. 29, 7143<sup>2</sup>) attention is called to expts. made in 1932 by V. on the rate of flow of  $\text{C}_6\text{H}_6$  through a viscometer, with and without a transverse magnetic field. The effect noted was less than 0.5% and the results at the time were considered indicative.

W. W. Stiller  
Experiments at very low temperatures obtained by the magnetic method. II. New superconductors. N. Kirtu and F. Simon. *Proc. Roy. Soc. (London)* A151, 610-23 (1935); cf. C. A. 29, 3888<sup>1</sup>.—Methods of cooling materials by means of a paramagnetic salt are discussed. The method adopted was to press Fe NH<sub>4</sub> alum and the material to be cooled into a pill. The existence of supercond. was indicated by magnetic measurements at low temps. Cd, Zr, and Hf became superconductive, the transition points on the abs. scale being 0.54°, 0.70° and 0.35°, resp. Au, Cu, Bi, Mg and Ge did not become superconductive down to 0.05°.

J. B. Austin  
The validity of Becker's relation for the initial permeability of stretched nickel wire. Gertrud Scharif. *Z. Physik* 97, 73-82 (1935); cf. Becker, C. A. 24, 5191<sup>1</sup>.—The relation between the initial susceptibility of stretched Ni wire and the temp. is investigated, for temps. up to the immediate neighborhood of the Curie point. The results obtained confirm Becker's magneto-elastic theory.

B. Swirles  
The negative Matteucci effect. E. Englert. *Z. Physik* 97, 82-93 (1935); cf. Becker and Kersten, C. A. 24, 241<sup>1</sup>.—Becker's theory of magnetization is extended to twisted Ni wires and the neg. Matteucci effect is evaluated theoretically for Ni and (92% Fe + 8% Ni) and also for a Ni tube. The temp. dependence of the neg. Matteucci effect and the magnetization of a twisted Ni wire are investigated expl. Approx. quant. agreement between theory and expt. is obtained.

B. Swirles  
Phenomenon of negative hysteresis in nickel. S. Sharan. *Current Sci.* 4, 157 (1935).—Specimens of Ni wire were first carried through a few complete magnetization cycles and then left in a weak field to produce a small pos. magnetization while alternating fields were applied. Longitudinal alternating fields produced effects similar

to those in Fe. Circular alternating fields, produced by passing an a. c. through the specimen itself, brought out the phenomenon of neg. hysteresis, similar to effects observed in studies of the magneto resistance change in Ni. S. calls attention to the fact that in both cases the Ni is carrying current so that the effect may be assocd. with movement of the conduction electrons. W. W. S.

**Magnetochemical investigations. XVII. The magnetic behavior of chalcogenides of bivalent chromium.** Haakon Haraldsen and Elisabeth Kowalski. *Z. anorg. allgem. Chem.* 224, 329-30 (1935); cf. C. A. 29, 7719<sup>1</sup>.—The magnetic susceptibilities of CrS, CrSe and CrTe were measured over a wide range of temp. and field strength. CrS and CrSe are paramagnetic at all temps. studied (90-629°K.). CrTe is ferromagnetic at lower temp. The Curie point is 70°. The results confirm the belief that at binding between the metal atoms exists in a whole series of compds. of the transition elements.

**C. E. P. Jeffreys**  
**Measurement of the gyromagnetic effect in pyrrhotite.** Frederik Coetzer. *Helv. Phys. Acta* 8, 522-64 (1935).—The math. theory of the gyromagnetic effect, the inherent difficulties of the various methods of studying it, and the magnetic properties of pyrrhotite are discussed. Several samples, both cryst. and powd., were investigated by the resonance method of Einstein and de Haas, which is treated at great length. The mean of 30 detns gives  $g = 0.63$ . If this be taken to be  $1/2$ , it corresponds to the case of a spinning electron revolving in a  $p$ -orbit, the spin moment and the orbit moment being oppositely directed. According to Hund's calcn., this gives a magnetic moment of 0.57 Bohr or 2.8 Weiss magnetons. This corresponds approx. to results obtained by Weiss by extrapolation from exptl. results. W. W. Stiller

**Diamagnetism of the trivalent bismuth ion S. S. Bhatnagar and Bhim San Bahl.** *Current Sci.* 4, 153-4 (1935).—From measurements made on 10 of its compds., values for the diamagnetic susceptibility of the trivalent Bi ion are calcd. The extreme values for  $\chi_A \times 10^4$  are 28.45 and 43.45, with 41.24 as the mean value. The susceptibility is also calcd. by Slater's theory to be 43.8. It is pointed out that Angus's modification of Slater's method would give still better agreement between theoretical and exptl. results. W. W. Stiller

**Diamagnetism of copper.** S. Ramachandra Rao. *Proc. Indian Acad. Sci.* 2A, 249-59 (1935); cf. C. A. 29, 7719<sup>1</sup>.—Colloidal Cu was prepd. by an elec. dispersion method in  $C_6H_6$  or  $PROH$ , and the diamagnetic susceptibility measured by the Curie method. For Cu *en masse*  $\chi = -0.080 \times 10^{-4}$  and the larger particles give approx. this value. As the particle size diminishes, the abs. value of  $\chi$  increases gradually at first, but for particles less than 0.8  $\mu$  in diam. the increase is quite rapid. These results are correlated with those of Honda and Shimizu on the effects of cold working, indicating that the lattice const. for the surface layers is greater than that for the interior of the metal. The thickness of this layer is extd. as 200 Å, its diamagnetic susceptibility as  $-0.200 \times 10^{-4}$ , and its  $d$  as 4.64 Å. Thomson's (cf. C. A. 29, 3593<sup>1</sup>) expts. on electron diffraction by electrolytic Cu are interpreted as confirming this theory of a surface layer with larger lattice const. W. W. Stiller

**Influence of the formation of hydrates on the diamagnetism of chemical compounds.** P. S. Varadachari. *Proc. Indian Acad. Sci.* 2A, 161-75 (1935).—The magnetic susceptibilities of  $H_2SO_4 \cdot H_2O$  mixts. were studied at various concns. Above 86% of the acid, the susceptibilities are greater than those given by the additive law, while below 86% the values are lower than the additive values. Max. deviations were observed at concns. corresponding to  $2H_2SO_4 \cdot H_2O$ ,  $H_2SO_4 \cdot H_2O$ ,  $H_2SO_4 \cdot 3H_2O$ ,  $H_2SO_4 \cdot 6H_2O$  and  $H_2SO_4 \cdot 18H_2O$ . These hydrates are also indicated by the other phys. properties of the mixt. The deviations are attributed to increased deformation of the anion in the acid in one case and decreased deformation in the other. Acetic acid-water mixts. were investigated at various concns. The results show that the additive law is obeyed even at equimol. concns. of the components, although a

compd. is formed at this compn. The decahydrate of  $Na_2SO_4$  was studied both in the solid state by the Curie method and in soln. by the modified Quincke method used for  $H_2SO_4$  water mixts. When the hydrate was heated to more than 33° (at which temp. the water of crystn. breaks away and the crystal becomes anhyd.) no change of magnetic susceptibility was noted. This suggests that the binding of the water mols. to the  $Na_2SO_4$  is very loose in contradistinction to the case of the hydrates of  $H_2SO_4$ . The theoretical basis for the increase of susceptibility on hydration suggested by Cabrera and Fahlenbrach (C. A. 29, 14<sup>1</sup>) to explain their results in aq. solns. of KI is critically examd. and evidence is advanced to show that no such changes are likely to occur. H. G.

**Dipole moments of hydrazine and its derivatives.** H. Ulich, H. Pensker and L. P. Audrieth. *Ber.* 68B, 1677-82 (1935); cf. C. A. 29, 1791<sup>1</sup>.—Dipole moments (in Debye units) calcd. from measurements of the dielectric constants of hydrazine derivs. in dil.  $CH_2Cl_2$  solns. are: methylhydrazine, 1.68 = 0.14,  $p$ -tolylhydrazine, 2.04,  $m$ -tolylhydrazine, 1.64,  $o$ -tolylhydrazine, 1.54,  $p$ -nitrophenylhydrazine, 2.49,  $p$ -nitrophenylhydrazine, 7.2 = 0.5, and 2,4-dinitrophenylhydrazine, 5.8 = 0.6. These moments and those previously reported (C. A. 27, 1791<sup>1</sup>) are inconsistent with free rotation on the N-N axis in hydrazine and with a trans position. They can be explained almost quantitatively on the basis of Penney and Southerland's model for hydrazine, which allows "inner" and "outer" positions of the substituent group. Discrepancies between the moments observed and calcd. for  $p$ -nitrophenylhydrazine can be explained by inner-salt formation. L. E. Steiner

**Paramagnetism of metallic gadolinium above its Curie point.** Felix Trombe. *Compt. rend.* 201, 652-3 (1935); cf. C. A. 29, 5370<sup>1</sup>.—The sp. susceptibility,  $\chi$ , of Gd was detd. from 16° to 360°. The  $1/\chi \cdot T$  (temp.) curve is curved below 90°, a straight line above. At 27.4°  $\chi = 4707 \times 10^{-4}$ . The paramagnetic and ferromagnetic Curie points are at 29.5° and 16°, resp., the paramagnetic moment by Langevin's formula is 23.28 Weiss magnetons, the theoretical value for the  $Gd^{3+}$  ion being 29.20.

**C. A. Silberrad**  
**Refinement of the Heisenberg theory of ferromagnetism.** 6. 1sm, applicable to simple cubic crystals. Charles H. Fay. *Proc. Natl. Acad. Sci.* 21, 537-42 (1935).—In the "simple" form of the Heisenberg theory of ferromagnetism, the energy levels of the spin multiplets of the microcrystal for a given spin quantum no.  $s'$  are replaced by the av. energy for  $s'$ . The assumption of a Gaussian distribution of energies for  $s'$ , as used by H. as a possibly better approximation, leads to the introduction of energies lower than that of the state of max.  $s'$  for very small spins, with the result that the crystal becomes paramagnetic at very low temps. instead of approaching its true satn. magnetization. The case of simple cubic crystals in which each microcrystal contains 8 atoms is treated. This makes it possible to treat half the interaction between atoms rigorously, and to the remainder an av. energy approximation, similar to that of the "simple" theory, is applied. The results are in close agreement with those of the simple theory but differ widely from those based on a Gaussian distribution. It is concluded, therefore, that except at low temps. the simple approximation is probably valid as the other assumptions of the Heisenberg theory. The treatment is highly math. throughout. W. W. Stiller

**The measurement of dielectric constants and the conductivities of electrolytes at ultra-high frequencies.** A. A. Tatarinov. *J. Exptl. Theoret. Phys.* (U. S. S. R.) 5, 533-9 (1935).—Theoretical. F. H. Rathmann

**Motion of an electron in an electric and a magnetic field with allowance for the space charge.** S. Ya. Braude. *J. Exptl. Theoret. Phys.* (U. S. S. R.) 5, 621-6 (1935).—A plane condenser system was used and the trajectory, potential distribution and charge  $d$ , were detd. The electron  $d$  is a max. at  $e/mHt = 2k$  and a min. at  $e/mHt = (2k + 1)/\omega$ , where  $k$  takes on values of 0, 1, 2, ...,  $t$  time,  $e$  and  $m$  are elec. charge, mass and magnetic field, resp. F. H. Rathmann

Dependence of the dielectric properties of Rochelle salt crystals on mechanical stresses. René David. *Helv. Phys. Acta* 8, 431-84 (1935).—Rectangular parallelepipeds, about  $20 \times 20 \times 9$  mm., were cut from Rochelle salt crystals with the 2 largest faces perpendicular to the *a*-axis and the others parallel to it and at  $45^\circ$  to the *b*- and *c*-axes. Electrodes of Al foil, with guard rings, were cemented to the larger faces. A Braun tube oscillograph with suitable amplifying system was used to study the dielec. behavior at various field strengths, without mech. stress, when the crystal was subjected to pressure perpendicular to the *a*-axis from 2 or 4 faces, and when pressure was applied in all directions. The results in general are analogous to those obtained in similar investigations of the magnetic behavior of ferromagnetics, viz. dielec. hysteresis and satn are observed. Up to 50 v./cm. hysteresis is absent and the polarization is proportional to the field and reversible, the dielec. const. (parallel to the *a*-axis) having a const. value of approx. 500. Above 50 v./cm. the dielec. const. increases very rapidly, reaching 20,000 at about 100 v./cm., when satn is reached. For higher fields it decreases with increasing field. When the crystal is subjected to mech. pressure, the hysteresis loop becomes unsymmetrical although the reversible part at small field strengths persists. From analogy with ferromagnetic theory, the dielec. const. of the reversible polarization is ascribed to "wall displacements" of spontaneously polarized domains. This spontaneous polarization in turn is detd. by the distortion of the domains. The results, given in detail, both graphically and in tabular form, are discussed and are correlated quantitatively with the piezoelec. properties of the cryst. Sixty references. W. W. Stiller.

Measurement of the variation of the dielectric constant of water with extent of adsorption. G. H. Argue and O. Maass. *Can. J. Research* 13B, 160-66 (1935).—An expl. technic was devised for measuring the dielec. consts. of cellulose materials contg. various amts. of adsorbed  $H_2O$ . From measurements made with standard cellulose the dielec. const. of the adsorbed  $H_2O$  was calcd. over the concn. range 0 to 18% of  $H_2O$ . The dielec. const. of the  $H_2O$  initially adsorbed is less than one quarter of that of liquid  $H_2O$ , but it increases with the amt. of  $H_2O$  subsequently adsorbed, until the dielec. const. approximates that of liquid  $H_2O$  as the  $H_2O$  content of the fiber approaches the satn point. These results agree with the hypothesis concerning the nature of the system cellulose-water. J. W. Shipley.

Friction dispersion of polar solutions by short electric waves. Wilhelm Müller. *Ann. Physik* 24, 99-112 (1935).—Dielec. consts. of 10% solns. of  $PhNO_2$  in paraffin oil and in a mineral oil of high viscosity, and of the pure solvents were measured at a wave length of 60.0 cm in the temp. range  $2^\circ$  to  $40^\circ$ . The dielec. consts. of the pure solvents and of  $PhNO_2$  in paraffin oil were independent of temp.; that of the soln. in the high-viscosity oil increased with temp., indicating that the applied frequency lay within the region of friction dispersion for this soln. The relaxation time of the  $PhNO_2$  mol. in the high-viscosity oil and the coeff.  $\alpha$  (ratio of apparent to actual viscosity) of the solvent were calcd. from the results.  $\alpha$  increases rapidly with temp. Measurements at longer wave lengths (500 m.) appear to confirm the results of Luthi (C. A. 27, 3371) that solns. of  $PhNO_2$  in a high-viscosity solvent have a second dispersion in addn. to the dispersion region at short wave lengths. The change of dielec. const. in the long wave dispersion region is smaller than in the principal region of dispersion at short wave lengths. A. S. S.

Electric breakdown in gases and in solid insulators. A. v. Hippel. *Ergebnisse exakt. Naturw.* 14, 79-129 (1935).—A review. C. D. West.

Relations between chemical constitution and dielectric properties. P. Debye. *Bull. soc. chim. Belg.* 44, 167-75 (1935).—A review.  $H_2O$ ,  $H_2S$ ,  $SO_2$ ,  $CO_2$ ,  $CS_2$ , as well as hydroquinone, *o*-, *m*- and *p*-chlorobenzene and the halogen acids are discussed. R. E. DeRicht.

Studies in the physical chemistry of betaines and related substances. I. Studies of dielectric constants and apparent molal volume. John T. Edsall and Jeffries Wy-

man, Jr. *J. Am. Chem. Soc.* 57, 1064-75 (1935).—Dielec.-const. increments and apparent molal vols. were detd. for various betaines, and for *N*-dimethylanthranilic acid (I) and its Me ester in a variety of solvents ranging from  $H_2O$  to  $C_4H_{10}$ . For the rigid betaines, approx. dipole moments were calcd. on the basis of known interatomic distances. These were compared with the values calcd. from the observed dielec. increments. In solvents of low dielec. const., the dielec. increments (and probably the moments) of all betaines studied fall below their values in  $H_2O$ . This effect appears to be an actual deformation of the mols. rather than an assocn. I appears to exist largely as an amphoteric ion, in  $H_2O$  and alc. as well as  $C_4H_{10}$ , in which its dipole moment is about 3 times greater than that of its Me ester. The electrostriction of the solvent produced by betaines in  $H_2O$  is less than that produced by similar  $NH_2$  acids but that due to the betaines is much greater in solvents of low dielec. const. than in  $H_2O$ . The magnitude of the effect is in good accord with theory.

H. W. Leahy.

Dipole moments of ethyl and isomyl borates and triphenyl phosphates. I. G. Cowley and J. R. Partington. *Nature* 136, 643 (1935).—In  $C_4H_{10}$  as solvent, in an all-glass cell, with Pt plates, the following values were obtained:

	$B$	$p$	$d^{25}$	$n_D^{25}$	$P_{25}^\infty$
$Et_2B_2O_4$	110	2/756 mm.	0.8635	1.3741	50.5 cc.
$iso-Am_2B_2O_4$	256	1/761 mm.	0.8514	1.4156	94.3 cc.
$Ph_3PO_4$	40	8 (m p)			262.5 cc.
		$P_E$			
		38.63 cc.		0.75D	
		80.20 cc.		0.81D	
		87.41 (calcd.)		2.70D	

Gregg M. Evans.

An elementary derivation of the law of the space-charge current. P. Selényi. *Z. Physik* 97, 395-7 (1935).—The laws giving the space-charge current in a high vacuum and in a dense gas can be obtained, apart from a numerical factor, from elementary considerations. B. Swirles.

The variation of the viscosity of gases with temperature over a large temperature range. A. B. Van Cleave and O. Maass. *Can. J. Research* 13B, 140-8 (1935). cf. C. A. 26, 3416.—The coeffs. of viscosity of  $NH_3$ , propylene,  $C_4H_8$  and  $Me_2O$  over the temp. range  $23^\circ$  to  $-80^\circ$  were measured. The present data are compared with those of other authors for temps. above  $0^\circ$ . It is stated that the authors' results are correct to 0.2% and have a relative accuracy of 0.1%. It is claimed that they are the most accurate data for the viscosity of gases at low temps. to date. The validity of a number of viscosity-temp. relations was tested with the present data and those previously published. In general, it is found that the equations of Sutherland and Jones hold at high temps. but fail at low temps. for substances such as  $CO_2$ ,  $SO_2$ ,  $NH_3$ ,  $Me_2O$  and propylene, which have viscosity-temp. curves that are convex to the temp. axis below room temp. An empirical equation is suggested which adequately represents the variation of viscosity with temp. for these five gases over the temp. range  $23^\circ$  to  $-80^\circ$ . However, this relation fails at high temps. for all gases, and even at low temps. for substances such as  $H_2$ , air and  $C_4H_{10}$ . The viscosity-temp. curves for  $CO_2$ ,  $SO_2$ ,  $NH_3$ ,  $Me_2O$  and propylene each show a definite inversion or inflection point. Below this inversion temp. the viscosity curves are convex to the temp. axis; above it they are concave to the temp. axis. In general, it seems that this inversion temp. bears a direct relation to the polarity of the mol. and to the crit. temp. J. W. Shipley.

The effect of dissociation on flame temperatures. L. Reingold. *Chaleur & Ind.* 16, 463-72 (1935); cf. C. A. 29, 6810<sup>1</sup>.—The reciprocal influence of the simultaneous dissocs. of  $H_2O$  and  $CO_2$  is formulated for combustion with insufficient, theoretical and excess air.

H. A. Beatty.

A simple modification of Victor Meyer's method for finding molecular weights of substances of high boiling points. H. C. Palmer. *School Sci. Rev.* 17, 138 (1935).

O. Reimnuth.

The experimental proof of the theory of the new equation of state Vitold Jacyna *Z. Physik* 96, 119-20 (1935)—The new equation of state is applied to the experimental data of Hausen, Roebuck and Osterberg on air and A, also to the data of Kapitza on He. Proof of the calculation of the inversion of the Joule-Thomson effect is briefly shown. Air and A are similar to He as previously reported. Tables are given showing the inversion point of the Joule-Thomson effects in both air and A. W. George Parks.

A kinetic interpretation of the "inner effect" V. Njegovan *Z. Physik* 97, 390-4 (1935)—An interpretation of the "inner effect" on the kinetic theory of an ideal gas. B. Swirles.

Expansion of alcohol and water Curt Luckow *Pharm. Zentralhalle* 76, 641-2 (1935)—The phys. data reported are essentially identical with those in Landolt-Bornstein. W. O. E.

Surface tension and solvation H. G. Trieschmann *Z. Physik Chem.* B29, 328-34 (1935)—Surface tension was measured at 22° by the max.-bubble method for  $H_2O$ ,  $EtOH$ ,  $C_2H_5$ , hexane, cyclohexane,  $BuOH$  and hexyl alc. and also for binary mixt. with hexane, cyclohexane and  $C_2H_5$  for solvent. From the results, certain conclusions could be drawn with regard to the surface activity and degree of solvation. G. M. Murphy.

The refractive dispersion of eugenol and isoeugenol G. Thompson *Pharm. J.* 134, 290 (1935); cf. Pickard and Hunter, *C. A.* 17, 1949—The Sellmeier equation (1871) can be resolved into an expression (1) for  $n$  at different wave lengths, believed to be equal to another expression (2) for the various wave lengths in which  $\lambda_0 = 2820 \text{ \AA}$  and  $2610 \text{ \AA}$ , resp. for eugenol and isoeugenol. To test the equality of (1) and (2),  $n$  was detd. for eugenol and isoeugenol at 9 different wave lengths (6708 to 4678  $\text{\AA}$ ) and the results were inserted in (1). Calculation of the corresponding values in (2) gave results fairly equal for isoeugenol, but deviating for eugenol. S. W.

Influence of the proximity of a solid wall on the consistency of viscous and plastic materials. IV. R. K. Schofield and G. W. Scott Blair, *J. Phys. Chem.* 39, 973-81 (1935)—The rate of flow of an aqueous paste of  $BaSO_4$  through tubes differing considerably both in radius and length was measured under a series of pressure heads. For tubes of the same radius, and under the same pressure gradient, the rate of flow is independent of the length of the tube. From this it is concluded that, under the conditions of these expts., this material shows no progressive breakdown with time under shear, as suggested by Ambrose and Loomis (*C. A.* 27, 5231) for bentonite. For different radii, however, curves for  $V/R^4$  against  $PR/2L$  were obtained, which, as previously recorded, do not coincide as they should if at every point in the tube the velocity gradient depends only on the shearing stress. The hypothesis previously advanced that the proximity of the wall of the tube causes a sheath of material to shear more easily than does the bulk of the material appears, therefore, to be the only one so far advanced that accounts for the facts. The case of  $BaSO_4$  is particularly interesting because the particles are roughly cubic in form and the thickness of the modified layer is many times the av. particle diam. Harold Gershinowitz.

The parachor V. Desreux *Bull. soc. chim. Belg* 44, 249-87 (1935)—The parachor,  $P$ , cannot be taken equal to the mol. vol. at the temp. where  $\gamma = 1$ , because at that temp.  $d$ , in the formula  $P = M/\gamma^{1/3}(D - d)$ , cannot be neglected. Hence a comparison of values of  $P$  at equal values of  $\gamma$  is possible only at low temps. Comparison at unequal values of  $\gamma$  may be uncertain, since the exponent in the relation  $\gamma = K(D - d)^3$  is not strictly const. but for a large no. of compds over a 15-20° range is found to vary from 3.1 to 4.8, the mean value being 3.8. The following values of  $d$ ,  $\gamma$  (dynes/cm<sup>2</sup>) and  $P$  (accurate to 0.2%), resp., were detd. at 20°:  $BuF$  0.7763, 17.72, 201.5,  $sec-BuF$  (at 15°) 0.7700, 16.93, 201.0,  $AmF$  0.7905, 20.06, 241.4,  $C_2H_5F$  0.8004, 21.83, 281.2,  $sec-C_2H_5F$  0.7914, 20.39, 279.7;  $C_2H_6$  0.8071, 23.20, 231.2,  $CH_3FCO_2Et$  1.0912, 23.35, 226.2,  $CHF_3$ ,  $CO_2Et$  1.1765, 24.60, 234.8,  $CHF_2CH_2Ac$  1.2075, 27.13, 234.5,

$CF_3CO_2Et$  1.1905, 16.76, 241.7,  $CH_2ClCO_2Et$  1.1504, 31.70, 252.7;  $CHCl_2CO_2Et$  1.2827, 31.34, 289.54,  $CCl_3CO_2Et$  1.3830, 30.87, 326.1;  $CCl_4F$  (at 15°) 1.4995, 19.09, 192.2,  $CHBr_3$  2.8905, 41.91, 222.5,  $CBu_3F$  2.7648, 31.68, 232.4;  $CH_2BrCH_2F$  1.8169, 27.02, 182.1;  $CHBr_2CH_2F$  2.3203, 31.60, 228.7;  $CH_2FCH_2I$  2.2259, 31.08, 203.7,  $CHClFCH_2Cl$  1.5393, 29.95, 230.1;  $CH_2ClCH_2Cl$  1.4405, 33.57, 222.9,  $PhF$  1.0252, 27.71, 215.0,  $m-C_6H_4F$  1.1572, 25.93, 222.4,  $p-C_6H_4F$  1.17006, 27.05, 222.35,  $m-C_6H_4I$ ,  $FMe$  0.9974, 27.47, 233.7,  $PhCF_3$  1.1880, 23.39, 270.2,  $PhCCl_3$  1.3723, 34.03, 353.6, trifluoromethylcyclohexane 1.0818, 22.44, 305.9. Consideration of all available data shows that atomic  $P$ 's are not strictly const. but may exhibit marked constitutive effects. Recalculation of the atomic  $P$  for the normal primary aliphatic series gives  $CH_3$ , 39.9,  $C_2H_5$ , 15.8,  $F$ , 26.1,  $Cl$ , 55.0,  $Br$ , 68.5,  $I$ , 90.0. Replacement of  $H$  by halogen in mixed halides, esters and aromatic compds gives  $P$ 's that may differ considerably from those calcd. from the foregoing values. No correlation is shown between the parachor and other functions of mol. vol. and it appears that these are not similar functions of the same phys. property. H. A. Beatty.

Aluminum alkyl oxides and their parachors. Robert A. Robinson and Douglas A. Peck, *J. Phys. Chem.* 39, 1125-33 (1935)—The following compds were prepd and their surface tensions and  $d$ 's were detd.: Al acetylacetonate, Al Et acetoacetate, Al diethyl malonate, Cr acetyl acetate and Al ethoxide, dipropoxide, isopropoxide, butoxide, isobutoxide and *sec*-butoxide. *Sn* ethoxide also was studied. The Al alkoxides do not have a simple structure and are unsuitable for parachor detn. Details of mol. wt. indicate a fourfold polymerization. If single linkage formulations are assumed for the other Al compds, the atomic parachor of Al is 39.5. The parachor deficiency found with the Al alkoxides is larger than that of *Sn* ethoxide. E. J. Rosenbaum.

Linear thermal expansion of sodium tungstate between 20° and 600° J. B. Austin and R. H. H. Pierce, Jr. *J. Chem. Physics* 3, 683-6 (1935)—The linear thermal expansion of anhydrous  $Na_2WO_4$  was detd. in the range 20-600° with an interferometer (*C. A.* 27, 2077). The results are  $\Delta l/l = 1 + 53.194(t - 20)10^{-6} + 41.5(t - 20)^2 10^{-9}$ ,  $\alpha_m = 17.73 \times 10^{-6} + 13.83(t - 20)10^{-9}$ ,  $\alpha_t = 17.73 \times 10^{-6} + 13.83(t - 20)10^{-9}$ ,  $\alpha_s = 17.73 \times 10^{-6} + 27.66(t - 20)10^{-9}$ . The salt is trimorphous at atm. pressure. Modification III is stable up to 585°, where it changes to II with an increase in vol. of 17.4%. Form II is stable over a very short temp. interval, and changes to  $Na_2WO_4$  I with a decrease in vol. of 0.12%. G. M. P.

Temperature dependence of the shearing strength of glass rods Karl Mengeloch, *Z. Physik* 97, 48-63 (1935)—Cylindrical glass rods were examd, the cross section being maintained const. The temp. dependence of the shear strength was measured between -100° and 545°. A relation is found between the values of the breaking strengths and the size of the mirror-like portion of the torn surface. The theory is discussed. S. T.

The change in brittleness during the formation of crystals with increasing temperature G. Tammann and W. Müller, *Z. anorg. allgem. Chem.* 224, 194-212 (1935)—The action of crystals on deformation is discussed at length. The effect on rock salt is particularly stressed. The method of prep. of flawless brittle crystals is described. The dependence of etched figures on temp. and the effect of edge angles is noted. Fractures in the different planes of the crystal give characteristically different figures. Pressure by means of a needle point on a surface gives sectors of a cube face and pressure figures when viewed by crossed nicols are observed. Effects of temp. and pressure are described also for galena, fluorapatite, calcite, ice, quartz and diamond. Some expts. were made with certain plastic easily melted org. compds to which pressure was applied. They seem to fall into 3 classes, namely, (1) those forming thin layers with rounded edges, (2) those having irregular boundaries with the interior transparent and (3) those falling into an entirely opaque powder. Raymond H. Lambert.

Thickness of the amorphous layer on polished metals



H. G. Hopkins. *Trans. Faraday Soc.* 31, 1095-1101 (1935); cf. *C. A.* 29, 3593.—Au was selected as a metal with which to work because interference with oxide films would be minimized. Electron diffraction patterns were made after successive removal of portions of the Beilby layer by sputtering. The specimen, 1 cm. square, was ground smooth on 00 and 000 emery with  $\text{CaH}_2$ . The metal was ground finally with 0000 emery paper and it was then polished on clean chamois leather. Four min. rubbing on 0000 emery, 2 min. in each of 2 directions at right angles, with  $\text{CaH}_2$  as a lubricant, and a subsequent 15-min. polish on chamois was sufficient to induce the polish layer. Electron diffraction patterns alternating with sputtering showed the thickness of the Beilby layer to be about 30 Å. There is a gradual increase in crystal size below the polished surface and at 100 Å. below the surface the crystals are still extremely small, about 5 to 6 atoms in the edge.

H. A. Smith. Molecular layers of fatty substances on metals. J. J. Trillat and H. Motz. *Trans. Faraday Soc.* 31, 1127-35 (1935).—On almost all electron diffraction photographs of metallic and org. surfaces there appear reticular distances among which are 4.20, 3.80, 3.0, 2.50, 2.35, 2.20, 2.02, 1.87, 1.60, 1.76 Å. as calcd. from diffraction rings. Spot diagrams correspond to a rectangular lattice with sides of 4.80 and 7.30 Å. On a carefully prepd. Au film such diffraction effects are eliminated. If such a film is impregnated with paraffin wax rings appear that are identical with those obtained from metallic films as well as from films of cellulose or polyoxymethylene. Similar expts. were made with octadecane, stearic acid, tristearin, ceryl alc., beeswax, cecic acid and oleic acid. With the exception of the last two acids all of these compds. gave reticular distances that are included in the above list. It is concluded that all films and surfaces unless special precautions are taken show diagrams corresponding to those of long-chain aliphatic compds. These diagrams gradually disappear and are missing altogether if a single region is irradiated with electrons for several min. The orthorhombic form of paraffin always appears after irradiation of the monoclinic form.

H. A. Smith. Crystallization of thin metal films. E. N. da C. Andrade. *Trans. Faraday Soc.* 31, 1137-50 (1935).—The polarizing microscope was used for examg. sputtered films of Ag and Au 50 atoms thick on glass. The microscopic method possesses 2 advantages over the electron diffraction method, viz (1) the microscope can delineate the distribution in space of the units which the electron beam detects, (2) the microscope gives certainly the crystal size. The Ag and Au films were heated at 250-280° and 400°, resp. After an hr. small spherulitic crystals about 1  $\mu$  across were found. Continued heating increased the no. of these spherulitic centers but not their size. On further heating Ag films at temps. up to 350° the spherulites change to the usual crystal form of Ag with the (111) faces parallel to the glass surface. The crystals grow reluctantly in a direction perpendicular to this face. In building up these crystals the surrounding film is decreased in thickness. The temp. at which spherulites form is inversely proportional to the thickness of the films. Quartz glass, glass, diamond and mica substrates yield the same results. The behavior of Au films is similar but changes occur at about 100° higher. When films are contaminated with Hg the same sort of initial spherulitic aggregation occurs at room temp. Small Au particles (1-2  $\mu$ ) grown by slow reduction in silicic acid gel first presented this optically anisotropic form of aggregation. It appears that when Au and Ag atoms are brought together by slow diffusion the first stage in crystn. is the spherulitic aggregation. The discussion of the paper brings out some doubt as to the validity, (1) of calling the initial particles spherulites and (2) of assuming the sputtered film to be monocryst. H. A. S.

Optical research on evaporated metal layers. L. S. Ornstein. *Trans. Faraday Soc.* 31, 1158-66 (1935).—The importance of measurements of optical transmissivity and reflectivity for the study of metallic films and surfaces is emphasized. A new theorem is presented which simplifies the exptl. technic needed. If a surface of opaque ma-

terial is illuminated homogeneously from all sides, the ratio of the intensity reflected within a solid angle,  $d\Omega$ , in a direction  $A$  to the intensity incident within an equal solid angle (is equal) to the total reflectivity for direction  $A$ . This makes necessary only one measurement of the reflected intensity. The method may be used for the study of the transition points in metals. H. A. Smith.

Validity of Drude's optical method of investigating transparent films on metals. Leif Tronstad. *Trans. Faraday Soc.* 31, 1151-8 (1935).—The optical method of Drude for examn. of thin transparent films and of the assumptions on which the method is based are reviewed. The validity and applicability of the method for a wide range of surface problems are discussed. The limitations are not yet thoroughly investigated, but considering the disadvantages of other methods for the examn. of thin surface films, the optical method deserves wider attention by investigators of surface problems. H. A. Smith.

The oxidation of metals. II. Copper, brass, aluminum brass, aluminum bronze, magnesium and some magnesium alloys. G. D. Preston and L. L. Burcumshaw. *Phil. Mag.* 20, 706-20 (1935), cf. *C. A.* 29, 2813.—The structure of thin oxide layers was studied by electron diffraction. The oxide film formed on pure Cu in air or O at temps. up to 183° consists of  $\text{Cu}_2\text{O}$  crystals with a (111) plane parallel to the polished surface. Repeated oxidation and reduction produced a surface that consisted of many very small crystals oriented at random. No evidence of  $\text{CuO}$  was found. With brasses of various composition it was found that small amts. of As had no effect on the results. Al inhibits the formation of  $\text{Cu}_2\text{O}$  on the surface at lower temps., probably by the formation of a film of  $\text{Al}_2\text{O}_3$ . At 400° all the samples showed lines resembling those of ZnO. ZnO and  $\text{Cu}_2\text{O}$  existed as separate phases. Pure Mg and alloys of Mg with Mn, Zn, Cd, Cu and Al were studied. In all cases, even when the Mg content was very small, the surface of the metal oxidized at 400° consisted of cubic MgO. At lower temps. the spectra could not be interpreted. The nature of the orientation of thin films is discussed. E. J. Rosenbaum.

Inner adsorption in salt crystals. Nature of the inclusion of zinc sulfide in copper sulfide. D. Balarev. *Z. anal. Chem.* 102, 408-11 (1935).—It is shown by expts. that the copptn. of ZnS by CuS is a typical case of inner adsorption and not one of postpptn. as Kolthoff and Pearson assumed (*C. A.* 26, 1831). W. T. H.

Grain changes in polymorphic conversion. G. Tamman and W. Boehme. *Z. anorg. allgem. Chem.* 223, 365-8 (1935).—Discussion of the change in grain size and number when a substance undergoes a change from one crystal form to another. The number of grains per unit area increased from 4 to 70 during the conversion of  $\text{KNO}_3$  at 120°; from 2 to 36 for  $\text{NH}_4\text{NO}_3$  at 87°; and from 10 to 175 for AgI at 145°. The number decreased from 7 to 4 for  $\text{AgNO}_3$  at 160° and from 2 to 1 for  $\text{TiNO}_2$  at 72.8°.

Arthur A. Vernon.

Inner potentials of crystals and the electron diffraction. V. E. Lashkarev. *Trans. Faraday Soc.* 31, 1081-95 (1935).—The difficulties encountered in the detn. of inner potentials by electron diffraction methods are discussed at length. Most of the present difficulties with fine structure and "prohibited" maxima may be eliminated mathematically by further development of wave mechanics along the lines set down by Bethe (*C. A.* 23, 332) and Morse (*C. A.* 24, 5604). H. A. Smith.

The mechanism of ionic motion in solid electrolytes. W. Schottky. *Z. physik. Chem.* B29, 335-55 (1935).—The theory of elec. cond. of solids of Bost (cf. *C. A.* 27, 4457) is discussed. The method used there for estg. the absolute value of the concn. of empty spaces in the crystal lattice is not exact enough. A few qual. conclusions can be drawn. The alk. earth oxide lattices are also assumed to conduct ionically by a mechanism of empty spaces. The variation in the anodic and cathodic cond. in alkali halides is due not to different nos. but to different mobility of the two kinds of empty spaces. If the vapor pressure of the metallic component is raised, at first a decrease and then, with further increase in the vapor pressure, an increase in

the total value of the electrolytic cond. takes place. Liectronic cond. of alk. earth halides now can be calcd. semi-empirically and an approach to the fundamental theory seems possible.

G. M. Murphy.  
Internal and superficial conductivity of cuprous oxide. Leon Dubar. *Compt. rend.* 201, 883-5(1935), cf. *C. A.* 29, 4986<sup>2</sup>.—Comparison of the cond. of a rod and plate of Cu O before and after drying over  $P_2O_5$  shows this to depend on both the internal ( $\sigma$ ) and superficial ( $\omega$ ) cond., the latter alone being affected by adsorbed moisture. In similar fashion  $\omega$  for a dried sample was reduced to  $1/10$  of its initial value after slight attack with dil.  $H_2PO_4$  followed by washing and drying.

C. A. Silberrad.  
The temperature dependence of the electro-optical Kerr effect of nitrobenzene at the transition point. W. Herzog. *Z. Physik* 97, 233-41(1935).—No discontinuity in the Kerr const. could be detected as the temp. passed through the Wollke-Mazur transition point (*C. A.* 26, 2758).

B. Swirles.  
Displacement of the Curie point due to tension. E. Englert. *Z. Physik* 97, 94-6(1935).—A displacement of the Curie point was found for a wire under tension by Ray-Chaudhuri (*C. A.* 26, 3154), this was in contradiction to the expts. of Adams and Green (*C. A.* 25, 5806). In the present expts. no effect was detected, it can certainly be no more than  $1/10$  that found by Ray-Chaudhuri.

B. Swirles.  
The atomic factor of zinc. Cecilia Mossin Kotin and Jose Losada. *Anales soc. espan. fis. quim.* 33, 597-601(1935).—The scattering factor of Zn is det'd., the first obtained from an element belonging to the hexagonal system. The photometric method was used to measure the intensities of the reflections, according to which the curve of relative values is drawn. Intensity of the (101) reflection of Zn is compared to intensity of the (200) reflection of NaCl, and for  $\sin^2 \theta/\lambda = 0.234$ ,  $f_{\text{Zn}} = 31.0$ , which permitted the curve of the exptl. values to be drawn. Comparison of the exptl. with the theoretical curve shows a greater slope for the first, and 2 undulations not found in the latter.

E. M. Symmes.  
Fine structure of x-ray absorption edges in close-packed cubic and close-packed hexagonal crystal lattices. D. Coster. *Physica* 2, 605-10(1935).—The fine structure of x-ray absorption edges was compared for Ca and Ti of cubic and hexagonal close packing, resp. By reducing the distances of the max and min absorption for Ti by a factor  $(2/91/3/97)^{1/2}$ , corresponding to the distances of neighboring atoms in the 2 structures, the Ca and Ti curves are found identical. Likewise curves for  $\alpha$ - and  $\beta$ -brass are identical after reduction to a common distance between atoms. Thus the positions of the edges in the spectrum are found the same, however, there still are typical differences in the form of the max and min due to the difference in lattice.

B. J. C. van der Hoeven.  
The law of the points of fusion and the lattice bonds. Robert Forrer. *Ann. phys.* (11), 4, 202-09(1935).—Continuing previous work on the Curie point, F. shows that in ps. of ordinary metals obey the same law as that detg. the Curie points, namely:  $T = F/\sqrt{N}$ , where  $F$  is a const. close to  $300^\circ$  and  $N$  is the no. of "contacts" attributed to the atom. These contacts are interpreted as representing contacts between the external electron orbits of neighboring atoms, these orbits forming an "electronic lattice."

Morris Muskat.  
Lattice constant of galena determined with a new x-ray spectrometer. Evald von Zeipel. *Archiv Mat. Astron. Fysik* 25A, No. 8, 18 pp (1935).—By precision methods developed in Siegbahn's laboratory the  $Ag K\alpha$  line was measured in the first four orders of reflection from the cube face of a selected galena crystal. Results are  $2d_{100} = 5923.34 \text{ \AA}$  at  $18^\circ$ , and  $d/N = 8.0 \times 10^{-4}$  (in  $\text{\AA}$ ,  $d = 1 - n$ ). The latter agrees well with the theoretical value  $7.3 \times 10^{-4}$  based on the measured  $d = 7.82$ . The half-width of the first-order line,  $5.6^\circ$ , showed the crystal used to be of good quality.

C. D. West.  
Differences in lattice constants. G. Wassermann. *Metallwirtschaft* 14, 813-15(1935).—A review of recent literature. Thirteen references. C. E. Macfarlane

1. Crystal structure of silver azide. Marc Bassière. *Compt. rend.* 201, 735-7(1935).— $AgN_3$  is orthorhombic with  $a$  5.58,  $b$  5.93,  $c$  6.04  $\text{\AA}$ ,  $d$  4.81, 4 mols in the unit cell, space group  $I_{2h}^{21}$ . The structure closely resembles those of  $KN_3$  and  $RbN_3$  (Hendricks, *et al.*, *C. A.* 20, 318; Günther, *et al.*, *C. A.* 24, 2930), being ionic and consisting of alternate planes parallel to (100) of Ag and  $N_3$  ions, the latter being linear with  $N-N = 1.18 \text{ \AA}$ .

C. A. Silberrad.  
The crystal structures of rubidium and ammonium fluoroborates. J. L. Hoard and Virginia Blair. *J. Am. Chem. Soc.* 57, 1983-8(1935).—X-ray data show orthorhombic units of structure  $RbBF_4$ ,  $a_0 = 9.07$ ,  $b_0 = 5.60$ ,  $c_0 = 7.23 \text{ \AA}$ , and for  $NH_4BF_4$ ,  $a_0 = 9.06$ ,  $b_0 = 5.64$  and  $c_0 = 7.23 \text{ \AA}$ . In the latter there is some evidence for the existence of H bonds between N and F.

L. W. Elder.  
The crystal structure of hexagonal silver iodide. Lindsay Helmholz. *J. Chem. Physics* 3, 740-7(1935).—From oscillation data, a structure is assigned to hexagonal AgI at room temp. in which the Ag atoms are distributed at random among 4 positions tetrahedrally surrounding the ideal position in the wurtzite structure. Previous work is discussed, and evidence favoring this structure is presented. At liquid-air temps. the structure approximates closely that of wurtzite with the ideal parameter value of  $1/8$ . Phys. characteristics of AgI are discussed in relation to the proposed structure.

G. M. P.  
A study of the molecular structure of diiodoethane. Iodine bond resonance and the molecular structure of diiodoethylene. Molecular packing in their crystal lattices. Harold P. Klug. *J. Chem. Physics* 3, 747-53(1935), cf. *C. A.* 29, 7731<sup>1</sup>.—From data obtained by an x-ray study of the crystal structure of  $(CH_2I)_2$ , and *trans*- $CH_3CHI$  the positions of the I atoms in the crystal cells were located. I band resonance is discussed and a factor of 0.92 of the length of the longer bond proposed for evaluating the interatomic distance for any resonating bond. On the basis of the concepts of tetrahedral C bonds, bond resonance, Pauling's normal covalent bond radii, etc., reasonable mol. models for these compds. were predicted. Comparison of these with the x-ray data showed that the I-I distances agree excellently with certain I-I distances within the crystal cells, thus locating the mols. within the cells. These models together with Mack's concept of the atomic domain radius of atoms in crystals permitted a consideration of the packing of the mols. in the crystal lattice, thus locating the atomic coordinates of the C and H atoms in the cell. The domain radii were: I = 1.97  $\text{\AA}$ , H = 1.23  $\text{\AA}$ . The resulting mol. packing explains and agrees excellently with observed phys. properties of the two crystals.

G. M. P.  
The dynamic theory of the diamond lattice. III. The diamond-graphite transformation. N. S. Nagendra Nath. *Proc. Indian Acad. Sci.* 2A, 143-52(1935); cf. *C. A.* 29, 5713<sup>1</sup>.—The transformation is explained by a general application of dynamics to crystal lattices. At a certain temp. the diamond lattice attains its max. energy and then passes over to that of graphite. Calcul. of this temp. from energy relationships agrees with exptl. detns. The theory also agrees with the calcd. energies of Lasareff, cf. *C. A.* 29, 6133<sup>1</sup>.

R. E. DeRight.  
Formation of new nuclei in crystallization. I. Dependence of the time of incubation on the deformation and the heating conditions. M. O. Kornfeld. *J. Exptl. Theoret. Phys.* (U. S. S. R.) 5, 556-62(1935).—K. discusses the concepts: speed of formation of centers, recryst. wave and tendency toward recryst. At wires tempered at  $410^\circ$  were deformed by tension, and allowed to recrystallize at  $300^\circ$ . For samples deformed 4.5, 5.0 and 7.0%, the speeds of nuclei formation are not linear functions of time or degree of deformation but increase more rapidly than linearly with time and deformation. The linear speed of the growth of nuclei is greater for smaller deformation and falls rapidly and linearly with time.

F. H. Rathmann.  
The adsorption of methane by coal. J. Bartlett Sutton and Earl C. H. Davies. *J. Am. Chem. Soc.* 57, 1785-7(1935).—The adsorption of  $CH_4$  by coal was measured at

10 94°, 17.77° and 24 60° at pressures up to about 1 atm. The results agree with the Freundlich equation. The heat of adsorption calcd. from the adsorption data is about 5000 cal per mol of  $\text{CH}_4$ . P. H. Emmett

Densities of adsorbed gases. I. Carbon dioxide on charcoal. Thos De Vries *J. Am. Chem. Soc.* 57, 1771-4 (1935).—By using  $\text{He}$  as an inert gas the  $d$  of adsorbed  $\text{CO}_2$  on C detd. at 30° was found to lie in the range 0.85 to 0.91. From the exptl. results together with an  $r$ - $\rho$  curve calcd. according to Polanyi's theory it was shown that  $\text{He}$  did not penetrate the  $\text{CO}_2$  layer when its  $d$  was more than 0.09 g. per ml. P. H. Emmett

The sorption of dimethyl ether on alumina. J. Edwards and O. Maass *Can. J. Research* 13B, 133-9 (1935), cf. C. A. 28, 18°, 29, 38941.—The sorption of  $\text{Me}_2\text{O}$  on alumina was investigated at pressures from 0.5 to 52 atm., the crit. pressure, over the temp. range 25-135°. The results are comparable to those for the propylene,  $\text{Al}_2\text{O}_3$  system. No discontinuity in the sorption accompanies the transition of sorbate from vapor to gas at the crit. temp., this differs from the previous results for the liquid-to-gas change. The initial stages of the sorption involve the formation of a unimol layer followed, with increasing pressure, by a multimol layer of increasing depth. It is unlikely that condensation to liquid occurs in the pores except at high relative pressures. The increase in crit. temp. of such a liquid must be exceedingly great to account for the continuous form of the isobars up to 135°. J. W. Shipley

Effect of temperature on selective adsorption by silica gel from binary mixtures. M. R. Aswathanarayana Rao *J. Indian Chem. Soc.* 12, 371-5 (1935).—The variation of selective adsorption with temp. was measured,  $\text{SiO}_2$  gel was used with  $\text{C}_2\text{H}_6$ - $\text{C}_2\text{H}_4$ ,  $\text{EtOH}$ - $\text{C}_2\text{H}_6$  and  $\text{C}_2\text{H}_5\text{N}$ - $\text{H}_2\text{O}$  mixts. over an entire range of concns. It was found that the selectivity (apparent adsorption) decreases with a rise in temp. By plotting selectivity against the equl concn. by the selectively adsorbed component, U- and S-shaped curves were obtained. The equation  $S = C_1(K'x + (b/T) - m')$  where  $m'$  is the wt. of the mixt. adsorbed by 1 g. of the gel was derived for a U-shaped curve, which indicates that selectivity decreases with rise in temp. The equation applies to dil. solns. only and can be used for S-shaped curves if these are considered as being made up of 2 U-shaped curves each representing the selectivity of one of the components. When  $C_1$  is const., the variation of  $m'$  with temp. is comparatively small, and for small values of  $C_1$  a straight line should be obtained when  $\log S$  is plotted against  $1/T$ , a prediction verified by the exptl. findings. C. R. Addinall

Surface potential differences of unimolecular films of fatty acids. Yohei Yamaguchi and Saburo Mizuno *Bull. Chem. Soc. Japan* 10, 453-64 (1935).—See C. A. 29, 5718°. G. G.

Unimolecular films of molecules which lie flat on the surface of water. I. Surface pressures and potentials of films of long molecules. Polymers of  $\omega$ -hydroxydecanoic acid. Wm. D. Harkins, Everett F. Carman and Herman E. Ries, Jr. *J. Chem. Physics* 3, 692-8 (1935).—Film pressures and surface potentials were measured for a series of linear polymers of  $\omega$ -hydroxydecanoic acid, with mol. wts. of 780-25,200. The mols. are oriented parallel to the surface. An improved form of the Harkins and Fischer app. (C. A. 29, 9549) was used. The chief modification in the procedure was the use of two radioactive Po electrodes over the film. The pressure-area relations show that the area per mol. in the condensed films is nearly proportional to the mol. wt. and that the compressibility of the films is large. The film  $d$  is lowest for the polymers of smallest mol. wt. The films are not very sensitive to changes in  $\rho$ , but on changing from an acid to an alk. substrate the films in the expanded state give higher mol. areas at given pressures, and the pressures for film collapse are considerably higher. The vertical cross section of the mols. lies between 19.4 and 19.9 sq. Å, nearly the same as the horizontal cross section of vertically oriented mols. such as stearic acid. Differences between films with perpendicular and with parallel orientation are tabulated

1 The surface potentials of the polymer films rise to 400-50 mv., although the surface  $d$ . of the polar groups is only  $1/2$  to  $1/3$  that of stearic acid type films, which give comparable potentials. The contribution of each polar group is, therefore, considerably greater than that of the dipole of the vertically oriented stearic acid mol. The surface potential of a condensed film increased about 13% less rapidly than the surface  $d$ . of the dipoles. G. M. P.

2 Colloidal chemical study of systems of three liquid components. I. The colloidal behavior of critical mixtures of three liquids. Naoyasu Sata and Osamu Kimura *Bull. Chem. Soc. Japan* 10, 409-20 (1935).—Systems consisting of conductivity  $\text{H}_2\text{O}$  as one component (A),  $\text{C}_2\text{H}_6$ ,  $\text{C}_2\text{H}_5\text{Me}$ ,  $\text{C}_2\text{H}_5\text{Et}$ ,  $\text{C}_2\text{H}_5\text{Cl}$ ,  $\text{C}_2\text{H}_5\text{NO}_2$ ,  $\text{C}_2\text{H}_5\text{NH}_2$ ,  $\alpha$ -xylene,  $m$ -xylene,  $p$ -xylene, cyclohexane,  $n$ -pentane,  $n$ -hexane,  $\text{CCl}_4$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ , chloroethylene and  $\text{Et}_2\text{O}$  for component B and  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{PrOH}$ ,  $i$ - $\text{PrOH}$  and  $\text{Me}_2\text{CO}$  for component C, were studied. The presence of a colloiddally dispersed phase was shown by a bluish opalescence in the Tyndall cone in the systems contg. the cyclic compds.,  $\text{CCl}_4$ ,  $\text{CHCl}_3$  and  $\text{CS}_2$  with the  $\alpha$  cs., but those contg. pentane, hexane,  $\text{Et}_2\text{O}$  and  $\text{Me}_2\text{CO}$  did not show this effect, this indicates some relation between chem. structure and the formation of emulsions. The most stable emulsions were obtained with the 2 systems contg.  $\text{C}_2\text{H}_6$  with  $\text{MeOH}$  and  $\text{EtOH}$ . The opalescent range for the system  $\text{C}_2\text{H}_6$ - $\text{EtOH}$ - $\text{H}_2\text{O}$  is shown on a triangular diagram by a space between 2 branches of the curve, called an island. The temp. range in which the emulsion is stable was also detd. for this system, and the resp. increases in vol. of the  $\text{H}_2\text{O}$  and  $\text{C}_2\text{H}_6$  layers were measured, this showed that  $\text{H}_2\text{O}$  has the greater attraction for the  $\text{EtOH}$ . The bluish opalescence is due to a dispersed phase consisting principally of  $\text{C}_2\text{H}_6$  in a dispersion medium of  $\text{EtOH}$  and  $\text{H}_2\text{O}$ . E. R. Rushton

3 Measurement of colloid-osmotic pressure by Krogh and Nakazawa's method. Hiroshi Nagaoka *J. Biochem. (Japan)* 22, 351-66 (1935).—Equilibration of the colloid-osmotic pressure requires at least 24 hrs. Also the exptl. error of the detn. decreases as the reading at equl. is taken. The pressure is proportional to the abs. temp., which confirms the earlier findings of Imai. The colloid-osmotic pressure decreases more rapidly than the concn., the relation between the values expressed by the general formula  $P = aTC/(1 - bC)$ , where  $P$ ,  $C$  and  $T$  are the pressure, colloid concn. and abs. temp., resp., while  $a$  and  $b$  are consts., which are detd. empirically. S. Morgulis

4 Studies in electroendosmosis. VII. Some measurements with nonaqueous liquids and high voltages. Harold P. Dakin, Fred Fairbrother and Alfred E. Stubbs *J. Chem. Soc.* 1935, 1229-33; cf. C. A. 29, 46491.—By use of an improved app. with photographic recording, the rate of electroendosmosis of 9 alcs. and ethers through a Jena Geräte glass diaphragm is found to increase linearly with the potential at low voltages and more than linearly at high voltages up to 6000 v. per cm. The flow rate becomes const. within 0.002 sec. after the application of potential. H. A. Beatty

5 The electrical conductance of colloidal solutions at high frequencies. Howard J. Curtis and Hugo Fricke *Phys. Rev.* 48, 775 (1935); cf. C. A. 29, 57191.—Measurements of elec. cond. were made on colloidal solns. at frequencies up to 16,000 kc. per sec. At higher frequencies there is a rise in cond. assoc. with a small decrease in dielec. const. Measurements were made on powder glass and kaolin suspended in  $\text{KCl}$ , mineral oil in  $\text{Na}$  oleate, and colloidal solns. of  $\text{Al}_2\text{O}_3$ ,  $\text{V}_2\text{O}_5$ , starch and gelatin (at its isoelec. point). The concn. of the  $\text{KCl}$  was varied to give sp. conductances of  $10^{-1}$  to  $10^{-4}$  mho/cm. with no appreciable change in the cond.-frequency curves. The aged  $\text{V}_2\text{O}_5$  soln. showed a marked increase in the high-frequency conductances. Most of the frequency-cond. curves show a sharp change in their rate of increase between 500 and 2000 kc. per sec. This increase in cond. of suspensions and colloidal solns. is probably due to an effect of the same type as the Debye-Falkenhagen effect in strong electrolytes (C. A. 22, 2705), the static forces

between charges on the colloidal particles and those in the adjacent portion of the continuous phase produce, in an external field of low frequency, an interference with the displacement of the ions of the continuous phase, which disappears at high frequencies. Soils show similar increased cond. of high-frequency radio waves (Strutt, *Elek. Nach. Ted.* 7, 387 (1930). Feldman, *Proc. Inst. Rad. Eng.* 21, 764 (1933). Smith-Rose, *J. Inst. Elect. Eng. London* 75, 221 (1934)).

Highly polymerized compounds CXVII. The classification of colloids H. Staudinger *Ber.* 68B, 1682-91 (1935), cf. C. A. 29, 7135<sup>2</sup>—Colloids may be classified either as globular colloids (spherocolloids) or as fiber colloids (linear-colloids). The former form only slightly viscous solns which follow Einstein's formula (cf. following abstr.) up to relatively high concns (5%), and deviate only slightly up to 10%. The viscosity is independent of the sphere size, for a given concn. The solns. obey the Hagen-Poiseuille law. To this group belong most lyophobic colloids or suspensions. The fiber colloids form solns that do not obey Einstein's formula, the viscosity increases rapidly with concn and with length of fiber (size of particle). Highly viscous solns form gels. These solns do not obey the Hagen-Poiseuille law. Lyophilic colloids (emulsoids) belong to this group. Fiber colloids have much greater surface than globular colloids of the same mass and differ in all properties assoc. with large surface. Most inorg. colloids are globular; the silicic acids are linear. Org. colloids are classified as macromol., held together by covalent bonds, and as micelles, which consist of simple mols. held together by van der Waals' forces. Both macromols. and micelles may be either spherocolloids or linear-colloids CXVIII. Viscosity investigations on organic spherocolloids and linear-colloids. H. Staudinger and E. Husemann *Ibid.* 1691-7.—The Einstein viscosity formula,  $\eta_r = 1 + 0.025 \frac{c}{c_0}$ , where  $\eta_r$  is the relative viscosity of the soln,  $c$  the concn and  $c_0$  the d. of the solute, holds for S solns as well as for true solns. of sugars and polyalcs. with approx. spherical mols. The emulsification of a monomeric styrol by Na oleate results in a polymeric styrol latex (similar to rubber latex) in which the polystyrol particles are spherical and obey Einstein's formula. The polystyrol may be pptd. from the latex and then redissolved by  $\text{CaH}_2$  in the form of fibers. The viscosity of the fiber solns. does not follow Einstein's formula, the sp. viscosity of a 1% soln of the fiber being 1000 times greater than that for the latex. Similar differences are obtained between the viscosities of emulsions of vinyl acetate and vinyl butyrate, and those of the corresponding  $\text{Me}_2\text{CO}$  solns. L. E. S.

The structural viscosity of solutions of high polymers J. Coumou. *Chem. Weekblad* 32, 426-9 (1935)—Liquids have structural viscosity if the rate of flow in capillaries is not proportional to the pressure and the viscosity decreases with pressure. The Hess viscometer was used to det. the effect. Structural viscosity is found in gelatin solns (0.7%) In 4% starch solns it is found provided the soln has not been heated more than 2 hrs. at 122°; after 30 min. at 150° there is no longer any structural viscosity. Com. dextrin shows slight structural viscosity; concd. sucrose or glucose solns. do not. Of plant materials carobin soln (0.5%), tragacanth soln. (0.5%) and husked mangel show the effect, gum arabic (20%), cashew gum (30%) do not; pectin soln. (4%) and lichen soln (5% in dil. alk.) do slightly. No structural viscosity is found in solns. of phenol-formaldehyde resins, but polystyrene (tetralin soln. 2%) does show it if the polymer is pptd. at low temp (100°) in the absence of  $\text{O}_2$  and  $\text{H}_2\text{O}$ . The conclusion is that structural viscosity is found only in solns. contg. long stretched particles of a min. size. Solns. with structural viscosity have considerably higher viscosity values, a 0.5% carobin soln. has a viscosity equal to that of a 20% gum arabic soln.

B. J. C. van der Hoeven  
Viscosity of solutions of substances of high molecular weight, especially cellulose J. Marchlewska *Przemys. Chem.* 19, 160-9 (1935)—A review with more than 85 references.

Distances between colloidal particles in iridescent strata of some ferric oxide sols. Wilfried Heller. *Compt. rend.* 201, 831-3 (1935); cf. C. A. 24, 2029; 29, 5330<sup>2</sup>.—The distance,  $d$ , between the layers of tactoids which produce by interference the iridescent colors in sols of  $\text{Fe}_2\text{O}_3$  prep. under certain conditions by hydrolysis of solns of  $\text{FeCl}_3$  is given by the equation  $d = (\lambda \lambda - 2d_1 \sqrt{n_1^2 - \cos^2 \alpha}) / 2\sqrt{n_1^2 - \cos^2 \alpha}$ , where  $d_1$  is the thickness of the tactoid assumed const.,  $\lambda$  wave length of light,  $\alpha$  order of color,  $\alpha$  inclination of incident beam,  $n_1$  and  $n_2$  are refractive indexes of the tactoid and liquid, resp. Such distances were detd. for solns of  $\text{FeCl}_3$  of (initial) concn. (c) 0.0408-0.0628 M, after periods (t) of 50-1095 days. For t const.  $d$  is a max. (2000-3000 A.) for approx.  $c = 0.05$  M, for c const.  $d$  increases with time, but with some indication of a max. between 190 and 248 days and subsequent decline. The changes are consistent with slow increase in charge of the particles resulting from increase in concn. of H ions through slow continuing hydrolysis. C. A. Silberrad.

The properties of silver halides peptized in gelatin by supersonic waves H. W. Dangers *Z. Physik* 97, 31-45 (1935).—The photographic behavior of peptized emulsions that are mechanically produced by high-frequency sound waves only, is examd. The properties of the emulsions are affected by heat treatment. The effects of varying binding materials upon the grain and on the character of the emulsions are studied. Under suitable conditions the sensitivity and the color sensitivity can be made very high. The importance of the surface state of the Ag halide grains before the addn. of the gelatin is discussed in relation to opening. S. Tolansky.

The chemical and physical characteristics of water of crystallization I. Hydrates containing up to three molecules of water. Luigi Passerini. *Gazz. chim. ital.* 65, 502-11 (1935)—The present paper begins a systematic investigation of the infrared absorption of the  $\text{H}_2\text{O}$  of crystn. in inorg. and org. compds. The infrared absorption spectra between approx. 1.30 and 2  $\mu$  of 8 compds. were detd. with an app. already described (cf. C. A. 27, 4801) and with a technic which is described in detail. The following data give the compds. examd. and the wave lengths of the maxima of their 2 bands (in  $\mu$ ): water vapor (I), 1.35, 1.83 (cf. Hettner, C. A. 13, 1559); Sclator and Phelps, C. A. 19, 3218; liquid water (II) at 18°, 1.443, 1.930 (cf. Collins, C. A. 17, 684; Dreisch, C. A. 19, 779); ice (III), 1.474, 1.976 (cf. Saunders, *Johns Hopkins Univ. Circ.* 18, No. 180, 58 (1899); Bode, C. A. 2, 274), ( $\text{CO}_2\text{NH}_2$ ) $\cdot$  $\text{H}_2\text{O}$  (IV), 1.385, 1.828,  $\text{HO}_2\text{CCH}_2\text{C}(\text{CO}_2\text{H})(\text{OH})\text{CH}_2\text{CO}_2\text{H}$  (V), 1.380, 1.832, ( $\text{CO}_2\text{H}_2$ ) $\cdot$  $\text{H}_2\text{O}$  (VI), 1.468, 1.967;  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  (VII), 1.475, 1.973,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (VIII), 1.426, 1.904;  $\text{K}_2\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$  (IX), 1.412, 1.881;  $\text{AcONa} \cdot 3\text{H}_2\text{O}$  (X), 1.470, 1.968, ( $\text{AcO}$ ) $\cdot$  $\text{Pb} \cdot 3\text{H}_2\text{O}$  (XI), 1.474, 1.970. For IV and V the bands correspond to those of water in the vapor state; for VI, VII, X and XI they are almost the same as those of ice; and for VIII and IX they occupy intermediate positions. The coincidence in the positions suggests that in the 1st group the  $\text{H}_2\text{O}$  of crystn. has a mol. structure analogous to that of water vapor, i. e., simple  $\text{H}_2\text{O}$  mols., in the 2nd group to that of ice, i. e., dimer ( $\text{H}_2\text{O})_2$ , or trimer ( $\text{H}_2\text{O})_3$  mols., and in the 3rd group to a mixt. of the different types of mols. To test this theory, a mixt. of compds., the absorption bands of which differ approx. the same as those of I and III were examd., viz.,  $\text{CaCl}_2$  and hexane, and a 1:1 mixt. of them, in the range of 1-2  $\mu$ .  $\text{CaCl}_2$  showed bands with maxima at 1.175, 1.419 and 1.704  $\mu$  (cf. Leconte, *Le spectre infrarouge*, 1928, 215, C. A. 22, 4383), hexane at 1.228, 1.439 and 1.778 (cf. Ellis, *Phys. Rev.* 23, 48 (1924)). The mixt. showed bands at 1.198, 1.430 and 1.741, compared with theoretical values of 1.200, 1.429 and 1.739  $\mu$ , resp. These results support the theory that the  $\text{H}_2\text{O}$  of crystn. in compds. such as VIII and IX is composed of different kinds of mols. A similar theory of nonhomogeneous  $\text{H}_2\text{O}$  of crystn. in hydrated salts has been advanced by several investigators to explain the varying behavior of the several mols. of  $\text{H}_2\text{O}$  of crystn. during dehydration. II. Hydrates containing more than

three molecules of water. *Ibid.* 511-17—The absorption bands between approx. 1.30 and  $2\ \mu$  of 16 more hydrated salts were measured. The following data give the compds. examd., and the wave lengths (in  $\mu$ ) of the maxima of the 2 bands:  $\text{NaNH}_2\text{SO}_4 \cdot 4\text{H}_2\text{O}$ , 1.451, 1.941;  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , 1.465, 1.900;  $\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$ , 1.464, 1.938;  $\text{Na}_2\text{SO}_4 \cdot 5\text{H}_2\text{O}$ , 1.464, 1.938;  $\text{Na}_2\text{SO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  (I), 1.470, 1.971;  $\text{ZnSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  (II), 1.471, 1.973;  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , 1.459, 1.955;  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ , 1.460, 1.958;  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ , 1.459, 1.957;  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ , 1.453, 1.943;  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ , 1.462, 1.964;  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ , 1.466, 1.965;  $\text{Al}(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ , 1.443, 1.930;  $\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$  (III), 1.473, 1.970;  $\text{K}_2\text{SO}_4 \cdot \text{Cr}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$  (IV), 1.471, 1.973; In the previous work (cf. above), some of the hydrates (I, II, III, IV and V) give evidence of contg.  $\text{H}_2\text{O}$  of crystn. in the same form as ice; in the other salts, the  $\text{H}_2\text{O}$  of crystn. is probably a mixt. of mols. like those in ice and in water vapor.

C. C. Davis

The chemical and physical characteristics of the water contained in colloidal substances. I. An investigation of some organic colloids. Luigi Passerini *Gazz. chim. ital.* 65, 518-28(1935), cf. preceding abstr.—The infrared absorption spectra between approx. 0.8 and  $2\ \mu$  of 6 hydrophilic colloids (3 gels and 3 sols) were examd. by the same method as before. The following data give the substances examd. and the wave lengths (in  $\mu$ ) of the maxima of the bands: water (at  $18^\circ$ ), 1.203, 1.443, 1.930, agar-agar gel (3%), 1.250, 1.472, 1.973, starch (30% sol) —, 1.443, 1.930,  $\text{adcm}$  (40% sol) —, 1.448, 1.937,  $\text{idcm}$  (50% sol) —, 1.460, 1.950,  $\text{idcm}$  (60% sol) —, 1.465, 1.957, egg albumin (natural) (I) 1.210, 1.454, 1.940,  $\text{idcm}$  (10% sol) —, 1.443, 1.930,  $\text{idcm}$  (20% sol) —, 1.443, 1.930,  $\text{idcm}$  (30% sol) —, 1.443, 1.930,  $\text{idcm}$  (40% sol) (II) —, 1.450, 1.937;  $\text{idcm}$  (60% sol) (III) —, 1.455, 1.942, dextrin (30% sol) —, 1.443, 1.930,  $\text{idcm}$  (40% sol) —, 1.443, 1.930; gum arabic (30% sol) —, 1.443, 1.930,  $\text{idcm}$  (40% sol) —, 1.443, 1.930,  $\text{idcm}$  (60% sol) (IV) —, 1.460, 1.945. The spectra of all the substances contain the individual bands of the water present. In the gels the bands are displaced considerably, viz., up to approx. 0.05  $\mu$ , toward greater wave lengths with respect to the bands of pure water. The greater the concn. of a given substance the greater is this displacement, and at a given concn. the displacements vary among the individual substances. There is a certain parallelism between the extent of this displacement and certain phys. properties of the colloid, e. g., its rigidity, but no explanation can be offered for this parallelism. In the sols the bands are displaced noticeably (0.01-0.02  $\mu$ ) toward greater wave lengths only for sols with relatively high viscosities, viz., I, II, III and IV. In an analogous way to the  $\text{H}_2\text{O}$  of crystn. in hydrated inorg. salts (cf. preceding abstr.), this behavior of colloids may depend on the fact that the mol. compn. of their water differs from the mol. compn. of pure water. The direction of the displacements indicates that there is an increase in the no. of mols. of the type of those in ice, i. e. ( $\text{H}_2\text{O}$ )<sub>2</sub> or ( $\text{H}_2\text{O}$ )<sub>3</sub>. In gels this increase is greater than in sols. The hypothesis that a colloidal substance forms, with the dispersing medium, labile compds. similar to solvates must be discarded, both because in the present work there was no difference in the intensity of the bands of the colloids and that of pure water, and because with changes in the concn. of the colloids there would have been no displacements such as those observed. II. An investigation of some inorganic colloids. *Ibid.* 529-33—The same technique was used as before. The range was approx. 1.30 to 2.00  $\mu$ . The following data give the substance (its percentage of water in parentheses) and the wave lengths (in  $\mu$ ) of the maxima of the absorption bands:  $\text{H}_2\text{SiO}_3 \cdot \text{nH}_2\text{O}$  gel (I) 46; 62; 89; 1.443, 1.930;  $\text{Sn}(\text{OH})_4 \cdot \text{nH}_2\text{O}$  gel (2; 66; 83); 1.443, 1.930;  $\text{Al}(\text{OH})_3 \cdot \text{nH}_2\text{O}$  gel (41; 55; 70; 82); 1.443, 1.930;  $\text{Ti}(\text{OH})_4 \cdot \text{nH}_2\text{O}$  gel (56; 81); 1.443, 1.930;  $\text{ZnHAsO}_4 \cdot \text{nH}_2\text{O}$  gel (48; 76); 1.443, 1.930;  $\text{MnHAsO}_4 \cdot \text{nH}_2\text{O}$  gel (54; 78); 1.443, 1.930;  $\text{FePO}_4 \cdot \text{nH}_2\text{O}$  gel (47; 54; 76); 1.443, 1.930;  $\text{NiH}_2\text{P}_2\text{O}_7 \cdot \text{nH}_2\text{O}$  gel (56; 61; 78); 1.443, 1.930; I after aging (15 S), 1.466, 1.955; *Alr* opal

(II) (13 S), 1.457, 1.946; Casellette opal (III) (8 O), 1.457, 1.946; Slesia opal (IV) (6 O), 1.457, 1.946; pure water (at  $18^\circ$ ) (V) 1.443, 1.930. The water in all the colloids except I, II, III and IV gave the same bands as did that in V, indicating that the water is absorbed physically by a phenomenon analogous to capillarity. In I, II, III and IV the water is united by firmer bonds, in which cases the displacements depend upon a change in the mol. compn. of the water. Inorg. colloids can be divided in 2 groups. (1) substances of the type of hydroxides, phosphates and arsenates, in which the water is not intimately bound but only absorbed, and which may be called *pseudo-colloids*, and (2) substances of the type of opal, in which the water is at least in part united chemically with the rest of the mol., and which are *true colloids*. The great differences in the infrared absorption by org. and inorg. colloids correspond to differences in the chem. behavior of the 2 groups with water, which lead to their classification as hydrophilic and hydrophobic colloids.

C. C. Davis

An investigation of the constitution of zeolites. Luigi Passerini *Gazz. chim. ital.* 65, 534-42(1935), cf. preceding abstracts.—The expts. are of importance in throwing light on the long-standing controversy of the state of water in zeolites and the intimate constitution of the latter. The infrared absorption spectra, between approx. 1.30 and 2.00  $\mu$ , of 6 zeolites, including analcite (I) from Flinders, natrolite (II) from Langesund, heulandite (III) from Teigarhorn, stilbite (IV) from Teigarhorn, cabasite (V) from Paterson and from Idar, scolecite (VI) from Teigarhorn, and an opal were examd. The following data give the wave lengths (in  $\mu$ ) of the maxima of the bands: water (at  $18^\circ$ ) 1.443, 1.930, I, 1.443, 1.930, II, 1.443, 1.930, III, 1.443, 1.930, IV, 1.443, 1.930, V, 1.443, 1.930, VI, 1.455, 1.913, opal, 1.457, 1.916. These results, in conjunction with those obtained with inorg. colloids and hydrated salts (see preceding abstracts), controvert the idea that the water in the zeolites examd. is in the form of  $\text{H}_2\text{O}$  of crystn. On I, II, III, IV and V it is in a different form from that in hydrophilic inorg. colloids, for in I, II, III, IV and V the water is simply absorbed physically. In VI on the contrary it is at least in part in the same form as that in inorg. colloids such as opal, and probably in VI that part of the water which causes a displacement of the bands is united to the 3  $\text{SiO}_2$  groups. The results in general indicate that the characteristic state of water in zeolites is in a simple state of absorption brought about by phys. forces alone.

C. C. Davis

Extension of the theory of complex coacervation to ionic disperse systems. H. R. Kruyt and H. G. Bungenberg de Jong. *Proc. Acad. Sci. Amsterdam* 38, 714-21(1935).—Unmixing in supersatd. solns. of electrolytes is a frequently occurring phenomenon which in many cases cannot be demonstrated on account of the exclusion of expts. at low temp. because of the nature of the solvent,  $\text{H}_2\text{O}$ . Coacervates have been demonstrated with luteo and hexal cations reacting with  $\text{KC}_2\text{H}_3\text{O}_2\text{SO}_4$  and  $\text{K}_2\text{Co}(\text{CN})_6$ . When drops of the following pairs of salts were allowed to run together under a cover glass upon a microscope slide, thus attaining all degrees of mixing, droplets due to unmixing could be observed,  $\text{Cd}(\text{NO}_3)_2 \cdot \text{Na}_2\text{C}_2\text{H}_3\text{O}_4$ ,  $\text{Cd}(\text{NO}_3)_2 \cdot \text{BzONa}$ ,  $\text{Pb}(\text{NO}_3)_2 \cdot (\text{CH}_3\text{CO}_2\text{Na})_2$ . With proper mixing proportions coacervates were shown with  $\text{BaCl}_2$  and  $(\text{NH}_4)_2\text{MoO}_4$  and with  $\text{SnCl}_4$  and  $(\text{NH}_4)_2\text{MoO}_4$ .

C. E. P. Jeffreys

Phosphatide autocomplex coacervates as ionic systems and their relation to the protoplasmic membrane. H. G. Bungenberg de Jong and J. Bonner. *Proc. Acad. Sci. Amsterdam* 38, 797-806(1935); cf. C. A. 29, 4651.—The phosphatide autocomplex coacervates are considered as fluid, ionic, autocomplex systems. The interactions of the phosphatide ions are detd. by the electrostatic attraction due to oppositely charged ionic groups, repulsion due to hydration, and attraction due to lyophilic groups. The behavior of the coacervates toward surfaces, such as C and glass, similar to that of paraffin, as well as theoretical considerations lead to the conclusion that phosphatide ions at the surface of the coacervates are oriented with

polar groups inside and hydrocarbon chains outside. It is assumed that when 2 such oriented films meet, a highly stable double film is formed. This assumption explains the peculiar behavior of vacuoles in the phosphatide auto-complex. It is suggested that the special properties of protoplasmic membrane depend upon one or more double layers of phosphatide ions similar to the double films of these coacervates. C. E. P. Jeffreys

The iodine-starch reaction. G. van Isteron, Jr., and J. C. M. Proc. Acad. Sci. Amsterdam 38, 700-8 (1935).—The I starch soln is considered to contain crystallites made up of mols of the structure  $(R)(R)O(M)(I)_n$ , where M represents a univalent metal or H atom. The addn of KI and  $I_2$  to starch soln raises the point of gelatinization up to an increase of  $5.9^\circ$  at a concn of 0.05% KI, with 0.014%  $I_2$ . This concn of KI or stronger ones alone had no effect. 1 xpts with I ions from a long series of acids and salts indicate the specific effect of this ion as required by the theory. Also, multivalent cations exert a greater effect than univalent on the change in gelatinization point. Neither  $Cl^-$  nor  $Br^-$  with or without KCl affects this property.  $Br^-$  in the presence of KBr produces a gelatinous ppt. C. E. P. Jeffreys

Interaction of amino acids and salts. I. Zinc chloride. Norman R. Joseph. *J. Biol. Chem.* 111, 479-87 (1935).—Potentiometric detns of activities in systems of amino acids and strong electrolytes in soln. possess the advantage over f-p and soly methods of permitting temp. and concn to be varied. Cells of the type  $Ag|AgCl||MCl||HgM_2||MCl||$  amino acid  $|AgCl|Ag$ , similar in design to those of Harned and Akerlof (*C. A.* 21, 1051), were studied by H. and A.'s exptl. procedure. From the data thus obtained for mixts of  $ZnCl_2$  (I) and glycine (II), alanine,  $\alpha$ -aminobutyric acid,  $d$ -valine and  $d$ -leucine activity coeffs ( $\gamma$ ) were calcd. Amino acids of the glycine leucine series decrease  $\gamma$  of I, hence I decreases  $\gamma$  for these acids. The effect of I on each amino acid appears to vary relatively little with concn of the latter and the relation between  $\gamma$  m f and amino acid concn is substantially linear. The results thus obtained were correlated with f-p data in the case of II and the interaction of I and II as detd is characteristic of that observed by the f-p method for salts of the same valence type. By means of the ionic force theory the results were compared with those of soly studies in media of low dielec. const. II. Sodium chloride and thallous chloride. *Ibid.* 483-90.

—By a similar method the interaction of amino acids with TlCl was studied. The value for  $\log(\gamma/\gamma_0)$  ( $\gamma_0$  = activity coeff. of the isomolal salt soln) at 0.4 M amino acid concn decreases from about 0.06 for glycine to 0.03, 0.02 and 0.01 for amino acids contg., resp., 2, 3 and 4 CH<sub>3</sub> groups. This value in dil solns of  $ZnCl_2$  is about 3 times as great as the corresponding value for TlCl. Measurements with NaCl were complicated by decompn. of NaHg by the amino acids. At  $1.4^\circ$  reversible conditions were approached with low concns. and at  $25^\circ$  with salt concns. higher than 1 M.  $\log(\gamma/\gamma_0)$  at high salt concns increases linearly with the concn. of the amino acid and the proportionality factor is independent of the concn. of the electrolyte. Salting-out coeffs. as detd. by this method compare favorably with those obtained by soly detns. The results are interpreted as indicating not only electrostatic attraction between ions and amphoteric ions, but also repulsive forces which are relatively greater, the greater the hydrocarbon chain of the amino acid and the dielec. const. of the solvent. R. C. Linderfeld

Studies in the physical chemistry of amino acids, peptides and related substances. V. Influence of amino acids, urea and alcohol upon the velocity constants of chemical reactions. Danella Straup and Edwin J. Cohn. *J. Am. Chem. Soc.* 57, 1704-1800 (1935).—The rates of reaction of  $S_2O_8^{2-}$  with  $CH_3Br$ ,  $CO$  ions and uncharged alkyl iodide mols were studied. The rates were increased by 1 OH and to a small extent by  $(NH_4)_2CO$ , and decreased by ions and amino acids. The rate of reaction with ions is increased by salts, amino acids and  $(NH_4)_2CO$  but slightly affected by  $FeOH$ . The effect of amino acids and  $(NH_4)_2CO$  varies with the concn., salts increase the log

of the velocity const. in proportion to the sq. root of the concn. Kirkwood's equation extended for reaction rates describes satisfactorily the observed results. H. W. L.

Physicochemical studies of the colloidal state of cholesterol, cholesterol esters and lecithin. X. Reducing power of cholesterol sols. Igor Remezev and Olga Zepalova. *J. Biochem. (Japan)* 22, 71-83 (1935).—Cholesterol sols have strong reducing power, a 5% sol corresponding to about 63 mg. and a 10% sol to 108 mg. pure glucose. Pptn. of proteins by alk.  $ZnSO_4$  causes a loss of 50-60% of this reduction by cholesterol. Addn. of cholesterol sol to glucose likewise increases the total reduction materially. An injection of 5% cholesterol sol into the portal vein in rabbits results in a marked rise in the reducing power of blood from various vessels, which sometimes represents a doubling of the value previous to the injection. Histological examn. of the liver fails to reveal even a trace of the cholesterol but there is a tremendous increase in the glycogen content. S. Morgulis

The concentration of heavy isotopes in cellulose. Kenzo Okabe and Toshizo Titani. *Bull. Chem. Soc. Japan* 10, 465-6 (1935), cf. *C. A.* 29, 6139<sup>1</sup>.—Several substances contg. cellulose were dried in air at  $100^\circ$ , then burned in a current of air and the  $H_2O$  formed was collected. Measurements of the sp. gr. of the  $H_2O$ , by means of a quartz float, were expressed in  $\gamma$  units ( $\gamma = 10^{-4}$ ) as follows: filter paper +0.3, cotton +5.7, cedar wood +4.5, bamboo +4.4, peeling of bamboo sprouts +4.4, contents of sprouts +0.6 and sap pressed out of the sprouts +1.0, compared with a standard cond.  $H_2O$ . The sp. gr. increases with the purity of the cellulose, and is not greater in the growing parts of the plant. Cf. *C. A.* 23, 3637<sup>2</sup>, 7151<sup>3</sup>. E. R. Rushton

Solubility of several compounds of the mannose series in alcohols. I. red W. Upson, Edwin A. Gluevog and Walter D. Albert. *J. Phys. Chem.* 39, 1079-91 (1935).—Soly. data were detd. for the following carbohydrate derivs. in 9 different alcs.: l-rhamnose,  $\alpha$ -d-mannose,  $\beta$ -d-mannose, d-mannonic- $\gamma$ -lactone, d-mannonic- $\delta$ -lactone,  $\alpha$ -methyl-d-mannoside and d-mannitol. The concn. in terms of mols of solute per 100 mols of solvent and the soln temp. are given for each detn. II. W. Leahy

Solubility of some strong, highly soluble electrolytes in methyl alcohol and hydrogen peroxide-water mixtures at  $25^\circ$ . Gosta Akerlof and Harlow E. Turck. *J. Am. Chem. Soc.* 57, 1746-50 (1935); cf. *C. A.* 21, 1051. —Soly. measurements at  $25^\circ$  were made for NaCl, KCl,  $NaNO_3$ ,  $KNO_3$ , KBr, KI,  $K_2SO_4$ ,  $NH_4Cl$  and  $Pb(NO_3)_2$  as solutes in  $H_2O$ -MeOH mixts. and for NaCl, KCl,  $NaNO_3$ ,  $KNO_3$ ,  $K_2SO_4$ ,  $KClO_4$  and NaI as solutes in  $H_2O$ - $H_2O_2$  mixts. The data for  $H_2O$ -MeOH mixts. are compared with corresponding data for mixts. of  $H_2O$  with other org. solvents, in most cases the soly. varies with the dielec. polarization of the solvent. A tentative explanation of the soly. changes in the  $H_2O$ - $H_2O_2$  mixts. is based on the changes in the dielec. const. of the medium and Huckel's theory for concd. solns. of strong electrolytes. J. W. Shipley

Mutual solubility of heavy water and organic liquids in systems with negative saturation curve. Jean Timmermans and Gustave Poppe. *Compt. rend.* 201, 608-10 (1935), cf. *C. A.* 29, 7759<sup>4</sup>.—While addn. of 1.07%  $KCl$  to a 31.1% soln. of a picoline in  $H_2O$  produces sepn., the upper and lower crit. soln. temps. ( $t_{uc}$ ,  $t_{lc}$ ) being  $128.5^\circ$  and  $87.5^\circ$ , the corresponding figures for the same addn. to a 33.2% soln. of the same in  $D_2O$  are  $112.5^\circ$  and  $92.0^\circ$ , complete miscibility occurring at  $102.5^\circ$  under 90 kg./sq. cm. Similarly 1.85%  $KCl$  added to a 40.8% soln. of *tert*-BuOH in  $H_2O$  causes sepn.,  $t_{uc}$  being  $137.5^\circ$  and  $t_{lc}$   $71^\circ$ , while 1.55%  $KCl$  does the same in a 37.2% soln. in  $D_2O$ ,  $t_{uc}$  being  $135.5^\circ$ ,  $t_{lc}$   $72^\circ$ . Thus more  $KCl$  is required to produce sepn. in  $H_2O$  than in  $D_2O$ , the latter having a larger domain of immiscibility. C. A. Silberrad

Equilibrium distribution of acetic acid between isopropyl ether and water. Andrew A. Smith and Joseph C. Igin. *J. Phys. Chem.* 39, 1149-53 (1935).—The distribution at  $20^\circ$  of AcOH between purified iso-Pr<sub>2</sub>O and distd.  $H_2O$  and between technical iso-Pr<sub>2</sub>O and tap water was detd. by titration of each layer with standard alc. NaOH after

they had stood 48 and 170 hrs. with frequent vigorous shaking. The results agreed closely. In order to approach the equil. from both sides the AcOH was dissolved initially in the  $H_2O$  in some cases, in the ether in others. The apparent distribution coeff.  $D = C_a/C_w$  slowly increases from 0.185 to 0.242 as the equil. concn. in  $H_2O$  increases from 0.0732 to 2.7800; i. e., the distribution law is only approx. obeyed, a result expected because AcOH is assoc. in ether and disassoc. in  $H_2O$ . The results with tap  $H_2O$  and technical ether are slightly higher, probably because of the presence of about 3% iso-PrOH in the ether. The equation  $C_a/C_w^{1.08} = 0.148$  may be used for approx. calcn. of the distribution ratio, but the equation  $D_{apparent} = C_a/C_w = 0.027 C_w + 0.178$  (based on the slope 0.027 and intercept 0.178 of the plot of  $C_a/C_w$  against  $C_w$ ) is recommended and the calc'd values are probably more accurate than the individual values. From the equation  $C_a/C_w = 2 K_1 K_2 C_w + K_2$  (cf. *C. A.* 28, 953<sup>3</sup>) the distribution const. for single mols. of AcOH between iso-PrOH and  $H_2O$  is  $K_2 = 0.178$ , and the assocn. const. of AcOH in iso-PrOH is  $K_1 = 0.43$  (concns. in mols. per l.).

Janet E. Austin

Alteration of chemical equilibria by adsorption at interfaces illustrated by color changes of dyes. A. J. Mee *School Sci. Rev.* 17, 298-301 (1935).—Directions are given for demonstration or student expts.

O. Reimnuth

Apparent volumes and apparent compressibilities of solutes in solution. II. Concentrated solutions of lithium chloride and bromide. Arthur F. Scott and G. L. Bridger *J. Phys. Chem.* 39, 1031-9 (1935).—New measurements of the ds. and compressibility coeffs. of conc'd solns of LiCl and LiBr are reported. On the basis of these and previous measurements, the irregular properties of the conc'd solns of these electrolytes are discussed. A graphic method for calcg. the factor  $f = (d\rho/dc)/(d\rho/dp)$  is described.

Harold Grishnowitz

Compressions and specific volumes of aqueous solutions of resorcinol and methanol at 25° and the behavior of water in these solutions. R. E. Gibson *J. Am. Chem. Soc.* 57, 1551-7 (1935), cf. *C. A.* 28, 1245<sup>7</sup>, 3292<sup>7</sup>, 3643<sup>7</sup>, 29, 4995<sup>7</sup>.—The sp. vols. and compressions of 6 solns of resorcinol and 15 solns. of MeOH in  $H_2O$  were measured at 25° and the corresponding apparent and partial quantities computed. The apparent compression of resorcinol varies slightly with concn. and an equation linear in the sq. root of its concn. expresses this variation within the exptl. error. The apparent vols. of resorcinol and of MeOH and the compressions of MeOH in aq. solns. are not linear functions of the sq. roots of their concns. The curves of the apparent compressions and apparent vols. of  $H_2O$  in MeOH solns against the sq. root of the concn.  $H_2O$  are sigmoid but a linear function fairly represents the data over the whole concn. range. MeOH and resorcinol apparently promote the assocn. of  $H_2O$ , thus differing from most other types of solutes. The sp. compressions of all solns. of MeOH up to 15% are the same as the sp. compressions for pure  $H_2O$ . The values for the compression of MeOH up to 1000 bars do not agree with those cited in the literature.

J. W. Shipley

The solute as liquid. Wilder D. Bancroft. *Science* 82, 388-9 (1935).—All liquid solns. are mixts of liquids, regardless of whether any or all of the pure components are solids, liquids, vapors or gases at the temp. of the expt. The ions in a fused salt or in a soln. are present as liquids and may exert a pptg. or a solvent action. In low concn. a dissolved liquid or a suspended particle will behave in some respects as a gas. This postulate reconciles the conclusions of van't Hoff on true solns. and of Einstein on solns with the fact that true solns. are mixts of liquids, and with the fact that the dispersed phase in a sol is rarely a gas. While it is customary and profitable in phys. chemistry to treat the solute thermodynamically as a gas under certain circumstances, it is actually a liquid and is a gas only in a metaphysical sense. The osmotic pressure may be that of a gas in a vol. into which the liquid solute could not possibly be compressed. In dealing with the effect of one salt on the soly. of another salt, it is not safe to ignore the direct or indirect solvent or pptg. effect of the third

ion or the undissoc. salt on the soly. of the second salt. G. M. P.

Pyrosulfuric acid as a solvent. C. R. de Robles and C. Moles. *Anales soc. españ. fís. quim.* 33, 643-54 (1935).—The cryoscopic const., detd. by the aid of sulfonal, veronal, trional and PhNO<sub>2</sub>, is 111.5, in good agreement with the Auerbach value 105 (*C. A.* 21, 1746). Abs.  $H_2SO_4$  is highly polymerized. The mol. magnitude near the f. p. is about 300. Its solns in  $H_2O$  probably react as  $H_2SO_4 + H_2O \rightarrow 2H_2SO_3$ . The mol. wt. of  $SO_3$ , at first near that of  $(SO_3)_2$ , falls rapidly; this explains the abnormal f. p. of fuming  $H_2SO_4$ . E. M. Symmes

The application of equations for the chemical potentials to equilibria between solid solution and liquid solution. George Scatchard and Walter J. Hamer. *J. Am. Chem. Soc.* 57, 1800-11 (1935).—Calcs. are made for the systems Ag-Pd and Au-Pt. These and the earlier results of others are discussed.

F. D. Rossini

The application of equations for the chemical potentials to partially miscible solutions. George Scatchard and Walter J. Hamer. *J. Am. Chem. Soc.* 57, 1805-9 (1935).—Equations for the free energy of mixing are calc'd. from the mutual solubilities of partially miscible substances; the vapor compns and pressures computed therefrom are in accord with exptl. data.

F. D. Rossini

Velocity of sound in solutions. Horace M. Trent. *Proc. Indiana Acad. Sci.* 44, 192-5 (1934).—An app. is described for detg. the change in velocity of sound through  $H_2O$  while a salt is dissolving. Data are obtained for  $Na_2PO_4$ ,  $SnCl_4$ ,  $NaOAc$ ,  $Na_2CO_3$ ,  $CuSO_4$ ,  $ZnSO_4$ ,  $NaCl$ ,  $CaCl_2$ , and powdered quartz as solutes. In general the velocity rose from its initial value. After a certain value was reached, the velocity remained const. A change of 2.3% was observed between the velocity in pure  $H_2O$  and that in a 10% NaCl soln.

W. J. Peterson

Diffusion coefficients in alcohol-water mixtures. H. Mouquin and W. H. Cathcart *J. Am. Chem. Soc.* 57, 1791-4 (1935), cf. Ostwald and Quast, *C. A.* 24, 3939. An improved app. is described, consisting of a rotating totally enclosed cell with fritted-glass diaphragm and provision for stirring by means of glass balls. Diffusion coeffs. ( $D$ ) are reported for 0.1% crystal violet solns. in solvents covering the range 100% EtOH to 100%  $H_2O$ . A discontinuity in the curve relating  $D$  to the percentage of EtOH in the solvent at 65 wt. % EtOH is correlated with other irregularities in phys. properties of solns. in 65% EtOH.

L. W. Elder

The rate of diffusion of deuterium hydride in water. W. J. C. Orr and J. A. V. Butler. *J. Chem. Soc.* 1935, 1273-7; cf. *C. A.* 29, 909<sup>4</sup>.—The diffusion of  $H_2O$  contg. about 3% DOH through sintered-glass membranes was studied at different temps. The diffusion coeff. of DOH increases from  $1.46 \times 10^{-4}$  at 0° to  $4.75 \times 10^{-4}$  at 45°. From the results the rate of self-diffusion in  $H_2O$  was estimated. The diffusion was also measured in  $N H_2SO_4$  and in  $N Na_2SO_4$  soln. at 13.5° and 13.85°, resp.; it differs only slightly from that in pure  $H_2O$ . H. S. van K.

The vapor pressure of phosphoric acid solutions. I. A. Kablukov and K. I. Zagorodkin *Z. anorg. allgem. Chem.* 224, 315-21 (1935).—See *C. A.* 29, 1308<sup>7</sup>.

G. G.

Viscosity of dilute solutions of nonelectrolytes. Balaghadra Prasad. *J. Indian Chem. Soc.* 12, 499-503 (1935).—The  $m$  and  $k$  consts. in the straight-line equations connecting the viscosity and vapor pressure of dil. solns. and  $H_2O$  differ by 0.8 and 1.3%, resp., for 1% sucrose solns. On the assumption that in still more dil. solns. the difference would become negligible, the 2 equations, one for the soln. and the other for the solvent, could be combined to give an equation which, on substitution and expansion, would assume the form  $\eta/\eta_0 = 1 + aC$ , where  $a = 0.00652$  if  $C$  is measured in g. mols. per l. To test this equation and, consequently, the assumption made, the viscosity of aq. solns. of various concns. ranging from 0.002 to 0.027 g. mols. per l., of sucrose (I) and fructose (II) were measured at 30°, 35° and 40°, and of glucose (III) at 35° and 40°. The variation of  $a$  with temp. was found to be within exptl. error and its value was fairly const. for

a given solute. The values of  $\alpha$  for solns. of I, II and III are 0.78, 0.42 and 0.44 and consequently depend on the nature of the solute, conclusively proving the invalidity of the assumption on which the equation was based.

C. R. Addinall

Cryoscopic investigation of anomalies in the behavior of ethylene chloride. Hugo Huettig, Jr., and Charles P. Smyth. *J. Am. Chem. Soc.* 57, 1923-6 (1935).—F. p. were measured on solns. of  $C_2H_4Cl_2$  in  $CaH_2$  and in  $(C_2H_5)_2O$  to investigate the possibility of intermol compd formation. Equil diagrams constructed from the f. p. data gave no indication of compd formation in the  $C_2H_4Cl_2$ - $CaH_2$  system but the formation of an unstable compd  $C_2H_4Cl_2 \cdot 3(C_2H_5)_2O$  was indicated in the  $C_2H_4Cl_2$ -( $C_2H_5$ )<sub>2</sub>O system.

J. W. Shipley

Cryoscopic determination of total hydration of ions of sodium nitrate. F. Bournon and O. Hun. *Compt. rend.* 201, 660-2 (1935).—By the method of C. A. 29, 7759<sup>4</sup> the hydration of the ions in M and 0.5 M NaNO<sub>3</sub> corresponds, resp., to NaNO<sub>3</sub> 9.3H<sub>2</sub>O and NaNO<sub>3</sub> 11.1H<sub>2</sub>O. If the hydration of the NO<sub>3</sub> ions is regarded as nil (cf. Manchot, et al., C. A. 19, 916) the above amts of H<sub>2</sub>O would represent the hydration of the Na ions.

C. A. S.

Mobility of the hydrogen ion constituent in aqueous mixtures of hydrogen chloride and calcium chloride at 25°. L. G. Longworth. *J. Am. Chem. Soc.* 57, 1638-1700 (1935), cf. C. A. 26, 4325.—The transference nos. of the H<sup>+</sup> in aq. mixts of HCl and CaCl<sub>2</sub> at a const. total concn of 0.1 N were measured at 25°. The conductivities of the same mixts were also detd. and from the combined data obtained the mobility of H<sup>+</sup> was calcd. The decrease in the mobility of the H<sup>+</sup> in passing from a soln. of pure HCl to one of pure CaCl<sub>2</sub> was compared with the value computed from the interionic attraction theory of electrolytic cond. and with a similar effect observed in mixts of HCl and KCl solns.

J. W. Shipley

The transference number and valence of silver in liquid silver amalgam. Karl Schwarz. *Monatsh.* 66, 218-21 (1935).—Electrolysis was performed in the same way as with Au amalgam (C. A. 27, 5007) with a const. current of 2 amps. for periods of 20 to 88 days. The Ag is carried to the cathode. The av. value for the transference no. divided by the concn ( $n/z$ ) is  $4.0 \times 10^{-4}$ . To det. the at. wt. of the Ag, an approx. detn. of the sp. gr. of the 0.025% amalgam was made, and gave 1.00040 compared with Hg as 1. From this,  $V_{Ag} = 6.67$  cc. Diffusion consts. were calcd. for valences of 1-3, and from the analogy to Au, the value  $1.0 \times 10^{-4}$  cm. per sec., corresponding to a valence of 2, is the most probable.

E. R. Rushton

The predominant role of association in the dissociation of simple straight-chain sulfonic acids in water. I. Conductivity. J. W. McBain and Margaret D. Betz. *J. Am. Chem. Soc.* 57, 1905-9 (1935).—In dil. solns the straight-chain sulfonic acids are simply partially dissociated electrolytes. At about 0.05 N the cond. goes through a min. and then with increasing concn. rises above the min. by 18, 36 and 45% for undecyl, lauryl and myristyl sulfonic acids, resp. This behavior is explained by an assocn. of like ions to form ionic micelles, where equiv. cond. in concd. solns. is as large as that of the OH ion. The assocn. masks the effects of interionic attraction. II. Freezing point. *Ibid.* 1909-12.—The osmotic coeffs. of undecyl and lauryl sulfonic acids pass through a min. in dil. soln., rising again with concn. by about 25%. The graphs of  $\rho/\sqrt{m}$  against  $\sqrt{m}$  (C. A. 24, 3417) exhibit a highly characteristic form, which departs strikingly from that of strong electrolytes or that of any simple weak electrolyte or nonelectrolyte. The interpretation of the data is that, whereas in dil. solns. they are simple moderately weak electrolytes, with increased concn. mols. and ions assoc. into neutral and ionic micelles, resp., but that ionic micelles, owing to the wide spacing of their charges, have ionic strength similar to that of univalent electrolytes. III. Electromotive force. *Ibid.* 1912-16.—Existing e. m. f. data for HCl with a 3.5 N KCl bridge were compared with values computed from diffusion potentials and the activity coeffs. of HCl on the assumption that  $f_{H^+} =$

$f_{Cl^-} = f_{HCl}$ . Discrepancies were noted which became large, rising to 10 mv. for 1 N HCl. The addn. of HCl to these colloidal electrolyte solns. of sulfonic acids produced more effect upon e. m. f. than in water, showing that the ionic micelles cannot be regarded as equiv. to multivalent ions in which the charges are coned in a point. In very dil. soln. the sulfonic acids behave as weak electrolytes and the degree of dissocn. rapidly falls, but in more concd. soln. remains almost const. or tends to rise again. IV. Comparison of results, and so-called "Hammarsten effects". James W. McBain. *Ibid.* 1916-20.—The H-ion concn. deduced from cond., f. p. and e. m. f. for solns. of simple sulfonic acids above 0.1 N are compared. Cond. and e. m. f. (single electrode) agree in showing that the dissocn. of the colloidal electrolyte is about 50%. If the data for thymonucleic acid and for undecyl, lauryl, myristyl and cetyl sulfonic acids are valid, it is shown that the activity coeffs. of single ions may be measured. For example, in more concd. solns. of HCl, the activity coeff. of the H ion appears to be about half that of the Cl ion and  $f_{H^+} < f_{Cl^-}$  where  $f_{HCl} = f_{H^+} f_{Cl^-}$ . A new explanation of the genuine Hammarsten effect is given, based upon steric hindrance to close packing.

E. R. Smith

The conductance of nonaqueous solutions. I. Sodium triphenylboron and disodium tri- $\alpha$ -naphthylboron in diethyl ether. Henry E. Bent and Maurice Dorfman. *J. Am. Chem. Soc.* 57, 1924-7 (1935).—The sp. resistances of Na triphenylboron and of disodium tri- $\alpha$ -naphthylboron at dilns. from  $10^{-4}$  to  $10^{-7}$  mols. per l. in Et<sub>2</sub>O were measured at 0° and 25° by both a c. and d-c methods. The bearing of these results on the structure of disodium tri- $\alpha$ -naphthylboron is discussed.

L. W. Elder

Structure of electrolytic solutions. H. Falkenhagen. *Ergebnisse exakt. Naturw.* 14, 130-200 (1935).—A review of the Milner-Debye theory of strong electrolytes. This accounts quantitatively for thermodynamically reversible and irreversible processes in very dil. solns. Investigation of concd. solns. and of those in which the solvent has a lower dielec. const. than water, is only just beginning from this standpoint.

C. D. West

Verification of Brønsted's principle. Seiji Kaneko. *J. Chem. Soc. Japan*, 56, 1035-6 (1935).—The Brønsted principle (cf. C. A. 16, 2057) was derived from the theory of a strong electrolyte.

T. Katsural

The dissociation constant of cresol red in sea water. Philip H. Mitchell and Ivon R. Taylor. *J. control. inters. exploration mer.* 10, 169-72 (1935).—Values for  $pK_a$ , the logarithm of the reciprocal of the indicator dissocn. const., were calcd. for  $\alpha$ -cresol red in sea water, from the equation  $pH = \log(\text{depth alk. standard}/\text{depth acid standard}) + pK_a$ . The  $pK_a$  was detd. by the glass electrode and the ratio of the two indicated colors by a Hastings bicolormeter. At 30°  $pK_a$  varied with chlorinity between 7.761 at 20.68 g. per kg. and 8.058 at 3.38 g. per kg.; at 20°  $pK_a$  was 7.879 at a chlorinity of 19.92 g. per kg. and 8.102 at 4.07 g. per kg.

Rex J. Robinson

The second dissociation constant of carbonic acid. Y. Kauko and V. Mantere. *Suomen Kemistilehti* 8B, 34 (1935) (in German).—A preliminary report. Measurements were made in soln. with a concn. less than  $10^{-4}$  from which extrapolation to infinite diln. gave value for  $k_2$  of  $5.0 \times 10^{-11}$  at 18°.

E. E. Jukkola

Titration curves and dissociation constants of L-ascorbic acid (vitamin C) and diethyl dihydroxymaleate. W. D. Kumler and T. C. Daniels. *J. Am. Chem. Soc.* 57, 1929-30 (1935).—The titration curves of L-ascorbic acid (I) and diethyl dihydroxymaleate (II) in alc.-H<sub>2</sub>O soln. and of I in H<sub>2</sub>O soln. were detd. The  $pK_a$ -values of these compds were measured and I was found 1000 times as strong as II. The apparent inability of the enolized  $\alpha$ -hydroxy- $\beta$ -ketonic ester type of compd. to account for the acid properties of I suggests that some other grouping is responsible for its large first dissocn. const.

H. W. L.

The activity coefficient of bicarbonate ions. Yrjö Kauko and J. Carlberg. *Z. Elektrochem.* 41, 721-4 (1935).—Kauko (C. A. 28, 3337<sup>7</sup>) worked out a method for detg. the concn. of CO<sub>3</sub> by measuring the change in  $pH$  of a bicarbonate soln. through which the air is passed,



and calcg. the partial pressure of  $\text{CO}_2$  from a formula, in which one of the terms is the activity coeff. of the bicarbonate ion. This coeff. is calcd. from the  $p_n$  obtained by the authors (C. A. 29, 5721) and found to agree with the value found by E. Güntelberg and E. Schödt (C. A. 23, 1335). The soly. of  $\text{CO}_2$  did not vary with the rate at which it was passed through the soln., as reported by McInnes and Belcher (C. A. 28, 239). It is necessary to work very carefully to keep the temp. const. The heat of the reaction  $\text{CO}_2 + \text{H}_2\text{O} \rightleftharpoons \text{H}^+ + \text{HCO}_3^-$  was calcd. thermodynamically and found to agree with the values detd. calorimetrically.

The "salting-in" of hydrogen peroxide by electrolytes M. H. Gorin. *J. Am. Chem. Soc.* 57, 1976-8 (1935); cf. Livingston, C. A. 23, 753.—The activity coeff. of  $\text{H}_2\text{O}_2$  detd. by measuring its distribution between  $\text{H}_2\text{O}$  or salt soln. and isomyl alc., is less than unity in solns. of  $\text{KCl}$ ,  $\text{NaCl}$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{KNO}_3$ ,  $\text{NaNO}_3$ ,  $\text{NH}_4\text{NO}_3$  and  $\text{NH}_4\text{Cl}$ . The phenomenon is attributed to the different dipole moment of  $\text{H}_2\text{O}_2$  relative to  $\text{H}_2\text{O}$  in salt solns. The order of salting-in is  $\text{K}^+ > \text{NH}_4^+ > \text{Na}^+ > \text{H}^+$  and  $\text{SO}_4^{2-} > \text{Cl}^- > \text{NO}_3^- > \text{ClO}_4^-$ . The relation of this series to the salting-out series for other nonelectrolytes is discussed on the basis of the relative configurations of  $\text{H}_2\text{O}_2$  and  $\text{H}_2\text{O}$ .

L. W. Ilder

Activity coefficients of bismuth chloride in hydrochloric acid solutions. Thos. De Vries and E. M. Hattox. *Proc. Indiana Acad. Sci.* 44, 138-43 (1934).—Cells of the type  $\text{Bi}|\text{Hg}$ , (2 phase),  $\text{BiCl}_3(\text{m})$ ,  $\text{HCl}(\text{m})|\text{Hg}_2\text{Cl}_2$ ,  $\text{Hg}$  were studied at 25° at three different  $\text{HCl}$  concns and widely different  $\text{BiCl}_3$  concns. The results show that practically all the  $\text{Bi}$  is in the soln. as the complex ion  $\text{BiCl}_4^-$ , with a disocn. const. of the order of magnitude  $2.88 \times 10^{-4}$ . The molal electrode potentials for the cells  $\text{Bi}(s)$ ,  $\text{Bi}^{+++}$  and  $\text{Bi}(s)$ ,  $\text{BiCl}_4^-$ ,  $\text{Cl}$  are 0.277 and 0.1678. The mean activity coeff. for  $\text{BiCl}_3$  in  $\text{HCl}$  solns. with an

ionic strength  $\mu$  is  $\log \gamma = \frac{-1.512\sqrt{\mu}}{1 + 0.0231\sqrt{\mu}}$ . This assumes that the activity coeff. is the same for  $\text{BiCl}_3$  as for  $\text{Cl}^-$ .

W. J. Peterson

Activity of chlorine ions in solutions of some complex chlorides of cobalt and chromium. Marcel Chatrelet and I. rancos Kertesz. *Compt. rend.* 201, 817-19 (1935).—The potential of the  $\text{AgCl}$  electrode in the cell  $\text{Ag}|\text{AgCl}, \text{X}$ , gelose  $|\text{Hg}_2\text{Cl}_2$ , satd.  $|\text{Hg}$  was detd. for  $\text{X} = \text{KCl}$ ,  $\text{BaCl}_2$ ,  $[\text{Co}(\text{NH}_3)_4]\text{Cl}_2$  (I),  $[\text{Co}(\text{NH}_3)_5]\text{Cl}_2$  (II), and  $[\text{Cr}(\text{NH}_3)_6]\text{Cl}_3$  (III). The results for I and II closely resemble those with  $\text{KCl}$ , those with III those with  $\text{BaCl}_2$ , implying that I and II apparently have only one ionizable  $\text{Cl}$ , while III, especially in solns. of concn. less than 0.0005  $M$ , has two.

C. A. Silberrad

Standard electrode potential of lithium in methyl alcohol. Angus Macfarlane and Harold Hartley. *Phil. Mag.* 20, 611-16 (1935); cf. C. A. 23, 3127.—The e. m. f. of the cell  $\text{Li}$  amalgam (0.33%)  $|\text{LiCl}$  in  $\text{MeOH}|\text{AgCl}$   $\text{Ag}$  is 2.296 v for a 0.1  $M$   $\text{LiCl}$  soln. and 2.321 v for a 0.05  $M$  soln. From these data in conjunction with others previously found, the standard electrode potential of  $\text{Li}$  in  $\text{MeOH}$  is  $-3.095$  v. The failure of the Born equation to reproduce the values of the partial molal free energy of transfer from infinitely dil. solns. is discussed.

E. J. Rosenbaum

First ionization constant of carbonic acid, 0° to 38°, from conductance measurements. Theodore Sheldovskoy and D. A. MacInnes. *J. Am. Chem. Soc.* 57, 1705-10 (1935).—To check the results of MacInnes and Belcher (C. A. 28, 239), who detd. the first ionization const. of  $\text{H}_2\text{CO}_3$  working with concn. cells this const. was detd. from cond. measurements at 0°, 15°, 25° and 38°. The relative conductances of satd.  $\text{CO}_2$  solns. and of 0.001  $N$  solns. of  $\text{KHCO}_3$ ,  $\text{KCl}$  and  $\text{HCl}$  were detd. The conductivities of  $\text{H}_2\text{CO}_3$  and  $\text{KHCO}_3$  were measured at 25°. The thermodynamic disocn. const. of  $\text{H}_2\text{CO}_3$  as a monobasic acid was calcd. from the data and the ionization const.  $K_1$  found. The values of  $K_1$  are  $4.21 \times 10^{-7}$  at 25° and  $1.82 \times 10^{-7}$  at 38°, corresponding closely to the values of  $4.5 \times 10^{-7}$  and  $4.0 \times 10^{-7}$  obtained at these temps. by MacInnes and Belcher. The heat  $\Delta H$  of the reaction  $\text{H}_2\text{O} + \text{CO}_2 =$

$\text{H}^+ + \text{HCO}_3^-$  was calcd. for each of the temps.

J. W. Shipley

Ionization constant of acetic acid in methyl alcohol-water mixtures from 0° to 40°. Herbert S. Harned and Norris D. Embree. *J. Am. Chem. Soc.* 57, 1669-70 (1935); cf. C. A. 28, 6611.—The e. m. f. of the cells  $\text{H}_2|\text{HOAc}(\text{m})$ ,  $\text{NaAc}(\text{m})$ ,  $\text{NaCl}(\text{m})|\text{AgCl}|\text{Ag}$  in 10 and 20% by weight  $\text{MeOH}-\text{H}_2\text{O}$  mixts. were measured at 0°, 10°, 20°, 25°, 30° and 40° in cells without liquid junctions. From these and the molal electrode potentials of  $\text{Ag}|\text{AgCl}$  electrode in these mixts. the ionization const. was computed. Apparently  $\log K$  varies linearly with  $1/D$ ,  $D = \text{dielec. const.}$

J. W. Shipley

Some thermodynamic properties of univalent halide mixtures in aqueous solution. Herbert S. Harned. *J. Am. Chem. Soc.* 57, 1865-73 (1935).—Measurements of the e. m. f. of the cells,  $\text{H}_2|\text{HCl}$  (0.01  $M$ ),  $\text{NaCl}$  (m)  $|\text{AgCl}|\text{Ag}$ , with  $m$  varying from 0 to 3, and at temps. from 0° to 60°, are reported. Calcs. were made for other halide mixts. from previous data, and the results are discussed in relation to the original and extended theory of sp. ionic interaction.

F. D. Rossini

The thermodynamics of ionized water in sodium chloride solutions. Herbert S. Harned and George E. Mannweiler. *J. Am. Chem. Soc.* 57, 1873-6 (1935).—Data are reported on the e. m. f. of the cells,  $\text{H}_2|\text{NaOH}$  (0.01  $M$ ),  $\text{NaCl}$  (m)  $|\text{AgCl}|\text{Ag}$  and  $\text{H}_2|\text{HCl}$  (0.01  $M$ ),  $\text{NaCl}$  (m)  $|\text{AgCl}|\text{Ag}$  at temps. from 0° to 60°, and with  $m$  varying from 0 to 3. From these data were calcd. the ionization const. of water, the ion activity coeff. product of water in  $\text{NaCl}$  solns., the heat of ionization of water, and the partial molal heat of ionization of water in  $\text{NaCl}$  solns. The logarithm of the ionization in alkali halide solns.,  $\log m_{\text{H}_2\text{O}}$ , at a given concn. varies linearly with the sum of the reciprocals of the ionic radii.

F. D. Rossini

Hydrogen-ion concentration ( $p_n$ ) determination with two-color indicators by a dilution method. J. McCrae. *J. S. African Chem. Inst.* 18, 62-6 (1935).—The accuracy of results from the double-wedge and the colorimeter methods of detg.  $p_n$  is due to the continuous changing of the comparison colors. Results comparable in accuracy with the double-wedge method are achieved by maintaining the concn. of an indicator in one condition (acid or alk.) const., while reducing by diln. the concn. of an indicator in the other condition (alk. or acid).

B. E. Anderson

Glass electrodes. Philip L. Varney. *Science* 82, 396-7 (1935).—The construction of glass electrodes is described in detail.

G. M. P.

Potential oscillations of iron in nitric acid. H. M. Karschulin. *Z. Elektrochem.* 41, 664-7 (1935); cf. C. A. 29, 4244.—When Armo Fe is placed in a 30 or 40%  $\text{HNO}_3-\text{H}_2\text{O}$  soln. a thin red-brown layer is formed in the soln. at the metallic surface. The film is thinner in the less concd. soln. In the less concd. acid a fine gray etched surface is formed on the Fe while with the higher acid concn. the surface is etched with a bright metallic luster. The metal goes periodically into soln. with the periodic formation of gas. The absorption spectrum of this brown film of liquid is identical with that of  $\text{Fe}(\text{NO})_2(\text{NO})_2$ . The periodic soln. of Cu in  $\text{HNO}_3-\text{HCl}$  mixts. behaves similarly in the formation of such a complex.

H. A. S.

Equilibrium between propyl alcohol, propyl ether and water at 190°. N. G. Gajendragad and S. K. K. Jaitkar. *J. Indian Chem. Soc.* 12, 486-93 (1935).— $\text{H}_2\text{O}$  is of little use industrially in India on account of its low b. p. and consequently studies of the equil. of the prepn. of  $\text{Pr}_2\text{O}$  (I) by the dehydration of  $\text{PrOH}$  (II) have tech. as well as theoretical importance. Dehydration of II in the vapor phase in the presence of fresh activated K alum was carried out in the app. previously described (C. A. 27, 654). The products were analyzed by sepg. the I with satd.  $\text{NaCl}$  soln. and removing II by the addn. of 60%  $\text{H}_2\text{SO}_4$ . The opt. temp. for the reaction is 190°, at which temp. the equil. corresponds to 84% conversion. I is more stable than II to the action of the catalyst and conse-

quently I cannot be the intermediate product in the dehydration of II to propylene. C. R. Addinall

**Hydrolysis of cupric sulfate solutions** Ossias Binder *Compt. rend.* 201, 893-4 (1935); cf. C. A. 28, 4997<sup>1</sup>.—A series of aq. solns. of  $\text{CuSO}_4$  of varying concn. was boiled for 1 hr., and a second series, all of the same concn., for periods of 0.25-12 hr. In all the  $pH$  decreased, the more so as concn. was greater. Examd. by the method of residues the solid phases were  $4\text{CuO} \cdot \text{SO}_3 \cdot 4\text{H}_2\text{O}$  when the concn. did not exceed 0.06 M, and  $3\text{CuO} \cdot \text{SO}_3 \cdot 2\text{H}_2\text{O}$  from more concd. solns. Both are insol and show characteristic x ray spectra. There was no trace of Fowles' salts (C. A. 20, 3401). C. A. Silberrad

**The dissolving of silver in cyanide solution** A. Simon and H. Deckert *Z. Elektrochem.* 41, 737-8 (1935).—The presence of 82.5% of the theoretical amt. of  $\text{H}_2\text{O}_2$  as an intermediate product in the soln. when air was passed in showed that the reactions are analogous to those for Au. The addn. of  $\text{H}_2\text{O}_2$  increased the rate of soln., a max. being obtained at 0.3%  $\text{H}_2\text{O}_2$  in 1% CN soln. At higher concns. of  $\text{H}_2\text{O}_2$  and lower concns. of CN, the Ag was colored yellowish brown to brownish black by oxidation products which interfere with soln. Fe, Cu and Mn salts and  $\text{K}_2\text{SO}_4$  decrease the rate of soln. in the presence of  $\text{H}_2\text{O}_2$ .  $\text{MnSO}_4$  forms a dirty green adherent deposit which stops soln. almost completely. Data on the effect of  $\text{FeSO}_4$  are given. Its effect may be due either to the formation of complex CN ions or to the catalytic decompn. of  $\text{H}_2\text{O}_2$ . The velocity of the air-stream has a decided effect, in proportion to the concn. of CN, on the rate of soln., which also varies with  $pH$ . Ag is insol in aq. HCN, which retards soln. in the presence of air. Alkalis and  $\text{Ce}(\text{OH})_3$  also decrease the rate of soln. E. R. Rushton

**Thermal decomposition of  $\text{CrO}_3 \cdot 3\text{NH}_3$**  Wm. F. Ehret and Arthur Greenstone *J. Am. Chem. Soc.* 57, 2330-1 (1935).— $\text{CrO}_3 \cdot 3\text{NH}_3$  (I) loses practically no  $\text{NH}_3$  upon standing for 4 months over concd.  $\text{H}_2\text{SO}_4$  in *vacuo* at room temp.; at 1 mm or less and at 120° there results the compd.  $\text{CrO}_3 \cdot \text{NH}_3$  (II), at 1 cm a mixt. of the 2 compds results. The presence of small amts of impurities has a marked effect on the rate of decompn. of I, which may be explosive. Radiation is essential for starting the reaction. II detonates near 200°, leaving  $\text{Cr}_2\text{O}_3$ , d. is 2.073 at 23°. C. J. West

**Considerations of the mechanism of chemical reactions** C. N. Hinshelwood *J. Chem. Soc.* 1935, 1111-15, cf. C. A. 28, 1254<sup>1</sup>.—Various uncoordinated results of chem. kinetics are discussed in an endeavor to bring them into relation. The efficiency of activating collisions, the catalysis of many homogeneous unimol. reactions by I vapor, and the occurrence of bimol. reactions in soln. are discussed particularly. P. H. Emmett

**Kinetics of the reaction between permanganate and manganese ions** Milton J. Polissar *J. Phys. Chem.* 39, 1057-66 (1935).—In acid soln. the reaction between  $\text{MnO}_4^-$  and  $\text{Mn}^{++}$  to give  $\text{MnO}_2$  is autocatalytic and has an incubation period.  $\text{MnO}_2$  prepd. in neutral soln. is a more effective catalyst than that prepd. in acid soln. The reaction rate is increased by a decrease in the acidity. Sulfate and fluoride ions form complexes with the manganese ion. In a mixt. of  $\text{MnO}_4^-$ ,  $\text{Mn}^{++}$  and oxalic acid, the oxalic acid can form a manganic oxalate complex or it can be oxidized. The amt. oxidized depends on the ratio  $[\text{Mn}^{++}]/[\text{H}_2\text{C}_2\text{O}_4]$ . E. J. Rosenbaum

**Rates of reaction of sodium atoms with hydrogen and deuterium chlorides** C. E. H. Bawn and A. G. Evans *Trans. Faraday Soc.* 31, 1392-1400 (1935).—The rates of these reactions were studied by the diffusion flame method. For  $\text{Na} + \text{HCl} \rightarrow \text{NaCl} + \text{H}$  the rate is 0.412  $\text{cm}^3 \text{mol}^{-1} \text{sec}^{-1}$ , while for  $\text{Na} + \text{DCl} \rightarrow \text{NaCl} + \text{D}$  the rate is 0.344  $\text{cm}^3 \text{mol}^{-1} \text{sec}^{-1}$ . The activation energies calcd. from the rates differ by 300 cal. This is approx. equal to the difference between the zero point energy differences in the initial and crit. transition states of HCl and DCl. E. J. Rosenbaum

**Mechanism of the chain reaction in the oxy-hydrogen mixture** Marcel Prettre *Compt. rend.* 201, 728-30 (1935); cf. C. A. 29, 3900<sup>1</sup>.—By detg. the velocity of the

reaction  $2\text{H}_2 + \text{O}_2 = 2\text{H}_2\text{O}$  during the time in which the proportion combined increases from 10 to 20% at various pressures the equation  $-dp/dt = k e^{E_{\text{act}}/RT}$  is deduced. The figure thus derived, 95,000 = 6000, for the energy of activation agrees fairly well with that (80,000) deduced from Haber's representation of the mechanism of the reaction (cf. C. A. 23, 772). C. A. Silberrad

**Kinetics of oxidation of metallic wires** Gabriel Valeus *Compt. rend.* 201, 602-4 (1935); cf. C. A. 29, 7843<sup>1</sup>.—The rate of oxidation of metallic wires is investigated mathematically. For cases where the oxide forms a protecting layer, the rate of diffusion of O across the layer is the chief detg. factor. The results agree with expt. for Ni wires of varying diam. C. A. Silberrad

**The factors determining the velocity of reactions in solution** The formation of quaternary ammonium salts in benzene solution. C. A. Winkler and C. N. Hinshelwood *J. Chem. Soc.* 1935, 1147-51.—The rates of 12 reactions of alkyl halides with tertiary amines and with pyridine and quinoline in  $\text{C}_6\text{H}_6$  solns. were detd. at a series of temps. In the general equation for a bimol. reaction,  $\text{rate} = P Z e^{-E/RT}$ , it is found that P is of the order of  $10^{-4}$  to  $10^{-5}$  for the 12 reactions, E varies from 8790 cal. for  $\text{Me}_2\text{N}$  and  $\text{MeI}$  to 18,000 cal. for pyridine and  $\text{iso-PrI}$ . In general the rate of reaction seems to depend primarily upon the value of E though marked decreases in the values of P occur on passing from Et halides to Pr halides, from  $\text{Me}_2\text{N}$  to  $\text{Et}_3\text{N}$ , from pyridine to quinoline and from iodides to bromides. P. H. Emmett

**The homogeneous unimolecular decomposition of gaseous alkyl nitrates. IV. The decomposition of isopropyl nitrate.** L. W. R. Steacie and G. T. Shaw. *Proc. Roy. Soc. (London)* A151, 685-93 (1935); cf. C. A. 29, 4999<sup>1</sup>.—The thermal decompn. of  $\text{iso-PrONO}$  is a homogeneous first-order reaction in the range 170-210°. The mechanism of the decompn. is analogous to those of the other alkyl nitrates:  $\text{MeCHONO} = \text{NO} + 1/2 \text{Me}_2\text{CO} + 1/2 \text{MeCHOH}$ . The rate of reaction:  $K = 1.25 \times 10^{14} e^{-21400/RT} \text{ sec}^{-1}$ . For  $\text{PrONO}$ ,  $K = 2.75 \times 10^{14} e^{-21400/RT} \text{ sec}^{-1}$ . The ratio  $K_{\text{iso}}/K_{\text{Pr}}$  is 1.09. The decompn. activation energies are:  $n$ -, 37,560,  $\text{iso}$ -, 37,000 cal/g. mol. C. M. P.

**The thermal decomposition of triethylamine** F. A. sin Taylor and Edwin E. Juterbock *J. Phys. Chem.* 39, 1103-10 (1935).—The thermal decompn. of  $\text{Et}_3\text{N}$  over a pressure range of 15-450 mm. at temps. from 450° to 500° is homogeneous with an energy of activation in its early stages of 50,000 cal. Analysis of intermediate products suggests that the mechanism involves the formation of  $\text{Et}_2\text{NNEt}$  and  $\text{C}_2\text{H}_5$  with subsequent decompns. H. W. Leahy

**The thermal decomposition of nitromethane** H. Austin Taylor and Vladimir V. Vesselsky *J. Phys. Chem.* 39, 1095-1101 (1935).—The main reaction appears to be a homogeneous unimol. splitting of  $\text{MeNO}_2$  into  $\text{MeNO}$  and O, with an energy of activation of approx. 61,000 cal. The complexity of the subsequent reaction does not permit a further analysis of the observed kinetic data. H. W. Leahy

**Photolytic and thermal decomposition products of azo methane** Thermal reaction rates in quartz, 260° to 290°. Lawrence J. Heidt and Geo. S. Forbes *J. Am. Chem. Soc.* 57, 2331 (1935).—Pressure readings at room temp., -78° and -183° at various stages of decompn. of azo methane (I), show that upon complete decompn. the pressure is approx. double the initial pressure of I and the ratio of the pressure increase at room temp. due to the presence of "permanent" gases formed by the reaction diminishes from a value of at least 2, in the initial stages, to 1 at complete decompn. To explain these facts it is assumed that the products include, with N, largely  $\text{CH}_4$ . These observations hold for photochem. decompn. at 30° and for thermal decompn. at 300°. The thermal reaction rates in quartz fit the unimol. formula, like Ramsperger's, but the rate consts. are roughly half as great as his. The 10° coeff. from the dark reaction rates at 260°, 270°, 280° and 290° is 2.9 and apparently affected but little by the presence of Hg vapor. C. J. West

**Kinetics and mechanism of thermal conversions of unsaturated hydrocarbons. I. Thermal polymerization of 1,3-butadiene at atmospheric pressure.** V. G. Moor, N. V. Strigaleva and L. V. Sulyayeva. *J. Gen. Chem. (U. S. S. R.)* 5, 818-29(1935).—The kinetics and mechanism of the thermal polymerization of 1,3-butadiene were studied at atm. pressure and a temp. interval of 400° to 700°. The reaction was performed in a Cu coil heated in a Pb bath and in a vertical porcelain tube heated electrically. The primary thermal reaction is bimol of the general formula:  $2C_4H_6 \rightarrow C_8H_{12}$ . The rate is expressible by the formula  $\log_{10} K = (b/400/T) + 7.32 \pm 0.04$ . The energy of activation is  $E = 28,000$  cal/mol. The temp. coeff. is 1.24. The 2 methods of thermal polymerization produced substantially equal results. C. B.

**The kinetics of the reaction between carbonyl sulfide and water.** H. W. Thompson, C. F. Kearton and S. A. Lamb. *J. Chem. Soc.* 1935, 1033-7.—The kinetics of the reaction between COS and H<sub>2</sub>O occurring in aq. soln. in alc. and in the gas phase were exam'd. In the latter 2 cases, the bimol. reaction observed is some powers of 10 slower than would be calc'd from theory by use of the observed energy of activation. In aq. soln. the measured rate is slightly greater than the calc'd rate, OH ion apparently being an effective catalyst. P. H. I.

**The kinetics of absorption of oxygen by catechol.** M. A. Joslyn and G. E. K. Branch. *J. Am. Chem. Soc.* 57, 1779-85(1935).—The rate of O<sub>2</sub> absorption by catechol solns. ranging from  $p_H$  6.5 to 10 is  $k[cat]^{1/2}[O_2]^{1/2}/[C]/[C + A]$ , where  $cat$  is the concn. of the univalent catechol ion,  $C$  the concn. of total catechol and  $A$  is a const. The effects of  $p_H$  outside of this range and of added KCl are also reported. The heat of activation from 20° to 35° is higher in phosphate buffer than in NaOH soln. because of the difference in heats of neutralization of phosphate and OH anions by catechol. L. W. L.

**Kinetics of solid-phase reactions of certain carbonates with mullite, silica and alumina.** Gordon R. Pole and Nelson W. Taylor. *J. Am. Ceram. Soc.* 18, 327-37(1935).—Rates of reactions between sized solids were det'd at several temps. by detg. the CO<sub>2</sub> evolved as a function of time. While the abs. rate is a function of grain size and other factors, the temp. coeff. gives a const. characteristic of the chem. system involved. The reaction product is found to lessen the period of reaction in accordance with the relation  $dy/dt = k/y$ , where  $y$  = thickness of the layer of reaction product. Under comparable conditions, Na<sub>2</sub>CO<sub>3</sub> attacks mullite most easily, with less attack on quartz, and least on corundum. The activity of CaCO<sub>3</sub> on mullite, quartz and corundum is less marked. The results have direct bearing upon the mechanism of glass melting and the corrosion of refractories. In prep. mullite by heating mixts. of cristobalite and Al<sub>2</sub>O<sub>3</sub> plus a little CaO as flux, batches that lie in the compn. triangle mullite-anorthite and SiO<sub>2</sub> give best results. C. H. Reer.

**The determination of the absolute temperature of the freezing point of water,  $\psi_0$ .** Witold Jacynza. *Z. Physik* 97, 107-12(1935).—By use of the exact form of the Joule-Thomson expression for  $\psi_0$ , it is deduced from exptl. data of Holborn, Otto, Heuse, Roebuck, Osterberg and others that  $273.17 < \psi_0 < 273.25$ . B. Swirles.

**The change, with time, of the vapor pressure of mixtures of the hydrogen isotopes at 20.38° absolute.** K. Steiner. *Physik. Z.* 36, 659-60(1935).—Mixts. contg. 2, 4, 13.9, 24.6, 47.8 and 94.3 atoms % of D, resp., showed lowerings in vapor pressure, in 10 hrs., of 5.6, 13.3, 18.6, 23.3 and 50 mm. Hg. The observed relation between the change in vapor pressure and the atoms % of D is approx. parallel with the HD content of the resp. samples. F. D. R.

**Difference in vapor pressures of ortho- and para-deuterium.** F. G. Brickwedde, R. B. Scott and H. S. Taylor. *J. Chem. Phys.* 3, 653-60(1935); cf. C. A. 28, 6005.—The differences between the vapor pressures,  $\Delta P(e - n)$ , of the 20.4°K. equl. mixt. and the normal mixt. of the 20.4°K.  $\Delta P(e - n)$  for D<sub>2</sub> is small as compared with  $\Delta P(e - n)$  for H<sub>2</sub>, but  $[\Delta P(\text{ortho-para})/P(n)]$  is approx. equal for D<sub>2</sub> and H<sub>2</sub> at the same temp. The uncatalyzed

change with time of the vapor pressure of liquid normal D<sub>2</sub> is less than 1 mm. Hg. in 200 hrs.; that of liquid normal H<sub>2</sub> increases 1 mm. in 4 hrs. The difference in rates is attributed to the difference in magnetic moments. If Wigner's theory of ortho-para conversion by paramagnetic molts. in the gaseous phase is extended to the liquid phase, a ratio of  $1/1000$  is obtained for the ratio of the rates of change of the vapor pressures of liquid normal D<sub>2</sub> and H<sub>2</sub>. By use of purer D<sub>2</sub> than that previously used, the b. p. was det'd to be 23.50°K. and the triple point 18.71°K. and 123.7 mm. Hg. Differences between the latent heats of vaporization of the ortho- and para-forms of solid and liquid D<sub>2</sub> are calc'd, and are found to be smaller than that for liquid H<sub>2</sub>. There is free rotation in condensed states of D<sub>2</sub> just as with H<sub>2</sub>. G. M. P.

**Vapor pressures of solid krypton.** W. H. Keessom, J. Mazur and J. J. Meihuizen. *Physica* 2, 669-72(1935).—Vapor pressures of solid Kr were det'd at temps. from 78 to 116° abs. by the Cath. app. (C. A. 13, 2477). The triple point is at 115.94° abs.  $\pm 0.03$ ,  $p = 54.9$  cm.  $\pm 0.15$ . The values are in fair agreement with a 5-term theoretical equation in which the heat of vaporization at 0° abs. is taken as 2633 cal. per mole, the at. heat of the solid is calc'd from a Debye equation with  $\theta = 55$ . Values for  $p$  in cm. are at 75°  $\pm 0.101$  and at successive 5° points up to 115° abs. 0.311, 0.834, 2.005, 4.294, 8.92, 16.94, 30.2 and 50.7 cm. B. J. C. van der Hoeven.

**Comments on "vapor pressure measurements of high-boiling metals" by Baur and Brunner.** Joseph Fischer. *Helv. Chim. Acta* 18, 1028-9(1935), cf. C. A. 29, 973.—The app. of von Wartenburg as used by B and B is unsuitable for b. p. measurement. Reply: Emil Baur and Roland Brunner. *Ibid.* 18, 1030. L. W. Elder.

**The interpretation of the dissociation pressures of the palladium-hydrogen system.** Max H. Hey. *J. Chem. Soc.* 1935, 1254-8. cf. C. A. 29, 7718.—A kinetic equation was derived for expressing the behavior of compds. of a volatile and a nonvolatile component that can undergo partial or complete dissociation without the formation of a new solid phase. This equation was applied to the Pd-H<sub>2</sub> system on the assumption that only one phase is present throughout. From the available data, which do not strictly represent equilibria, the conclusion is drawn that the limiting compn. is PdH<sub>2</sub>, which has possibly the same structure as NaCl. H. S. v. K.

**Piezometric researches. I. Effect of high pressures on the temperatures of fusion and transformation of organic compounds.** Louis Deffet. *Bull. soc. chim. Belg.* 44, 97-139(1935); cf. C. A. 29, 3221.— $\delta$  for the temps. of fusion and transition, resp., are: cetyl alc., 1.070, 1.058; C<sub>7</sub>-H<sub>16</sub>, 1.067, 1.065; methylcyclopentanol, 1.058, 1.071; acetophenone, 1.071, 1.071. Values of  $\delta$  for the m. p. of the stable and unstable forms, resp., are: cetyl iodide, 1.060, 1.055;  $\alpha$ -nitrotoluene, 1.075, 1.073;  $\alpha$ -toluidine, 1.071, 1.068; salol, 1.082, 1.082; Mel, 1.067, 1.066. Complete data of values for other compds. are tabulated. The equation  $\delta = 1 + \frac{\Delta V}{V}$ ,  $\Delta V$  = changes

in vol. with pressure,  $V$  = the heat of fusion, is shown to apply. An attempt is made to relate  $\delta$  with the chem. structure. R. E. DeRicht.

**The dimorphism of trinitroresorcinol, bromophenylhydrazine and benzaldehyde p-nitrophenylhydrazones.** R. Fischer and A. Köfler. *Atkrochimie* 19, 35(1935).—As a result of microsublimation tests, 2 kinds of crystals are found with each of the 3 substances mentioned. With trinitroresorcinol the stable crystals are monoclinic and  $m.$  at 177°, the unstable crystals are trigonal and  $m.$  at 165.5°. The stable modification of bromophenylhydrazine forms monoclinic crystals  $m.$  165°, the unstable crystals are also monoclinic but  $m.$  at about 159°. With benzaldehyde p-nitrophenylhydrazones, both species of crystals appear to be monoclinic. The  $m. p.$  of the more stable form is 261-2° and of the less stable form 234-6°. W. T. H.

**Application of the "thaw-melting point" method to binary inorganic systems.** A. Benrath, P. Hartung and

M. Widen *J. prakt. Chem.* 143, 238-244 (1935), cf. Rheinboldt, *C. A.* 20, 693.—The results are given in 20 diagrams. Binary melts of  $M(NO_3) \cdot 6H_2O$ , where  $M = Co, Ni, Mn, Mg$ , all give a single solid soln, while systems of  $Zn(NO_3)_2 \cdot 6H_2O$  with the preceding show 2 solid solns. The systems  $M(NO_3) \cdot 6H_2O - M(NO_3)_2 \cdot 4H_2O$ ,  $M = Co$  or  $Zn$ , are of the single solid soln type. Systems of  $NH_4NO_3$  or  $AgNO_3$  with these nitrate hexahydrates are of the eutectic type. The m.p.s of  $M(NO_3) \cdot 6H_2O$ ,  $M = Ni, Mg, Co, Zn, Mn$ , are 209°, 193°, 191°, 163° and 153°, resp. Binary systems of these compounds with one another are all of the single solid soln type. The systems  $M(NO_3) \cdot 6H_2O - M(ClO_4)_2 \cdot 6H_2O$  are of the eutectic type. The reciprocal salt pair  $Mg(ClO_4)_2 \cdot 6H_2O - Ni(ClO_4)_2 \cdot 6H_2O$  is discussed. Michael Fleischer

Solubility studies in the systems benzene-*p*-nitrotoluene and benzene-*o*-nitrotoluene. H. D. Crookford and E. C. Powell, Jr. *J. Elisha Mitchell Sci. Soc.* 51, 143-6 (1935).—Temp.-compn. data give no evidence of compd. formation in these systems. The eutectic temp. for the *p*-nitrotoluene system is -7.2°, for the *o*-system -10.3°. The latent heats of fusion were calcd. for each component as follows: *o*-nitrotoluene 2905, *p*-nitrotoluene 4100 and benzene 2475 for the *o*-system and 2730 cal. for the *p*-system. A. L. Mehning

Heterogeneous equilibrium in two-component systems with thymol as one component. Konstanty Hrynakowski and Marja Smyt. *Arch. Pharm.* 273, 418-27 (1935).—Among other data reported for the systems thymol-urea, -acetanilide, -phenol, -salicylic acid, -salol, -antipyrine and -camphor are the eutectic values, resp. 43.0°, 10.5°, 6.7°, 46.2° and 15.6° (none obtained with antipyrine and camphor), the corresponding contents of thymol were 93.5, 15.0, 48.2, 96.2 and 37.5%. W. O. E.

The system calcium oxide-sulfur dioxide-water. I. Determination of vapor pressures and conductivities. G. W. Gurd, P. E. Gishler and O. Maass. *Cow J. Research* 13B, 599-17 (1935), cf. 27, 5231, Beuschlein and Conrad, *C. A.* 28, 7824.—The system  $CaO-SO_2-H_2O$  is being investigated to det. the nature of existing equilibria, and the way in which these vary with concn. and temp. A technique is described by means of which vapor pressures and conductivities were measured over the temp. range 25° to 150° and over the concn. range 0 to 2.5%  $CaO$  and 0 to 6%  $SO_2$ . The data are systematized in tabular form. J. W. Shipley

The system  $NH_4Cl-NH_4NO_3-H_2O$  at 0.4°, 25° and 50°. C. F. Prutton, J. C. Brosheer and S. H. Maron. *J. Am. Chem. Soc.* 57, 1656-7 (1935).—Sol. relationships in the system  $NH_4Cl-NH_4NO_3-H_2O$  were detd. at 0.4°, 25° and 50°. The results indicated no complex salt, solid soln or hydrate formation. J. W. Shipley

The ternary system stannous oxide-sulfur trioxide-water. Henry G. Denham and William E. King. *J. Chem. Soc.* 1935, 1251-3.—By Schreinemaker's residue method the system  $SnO-SO_3-H_2O$  was studied at 25° and 50°.  $SnSO_3$ , when dissolved in  $H_2SO_4$  at 25° and 50° does not form a hydrated normal sulfate, the only other stable phase being  $SnSO_3 \cdot SnO$ . In ternary solns the ternary phase stable at 25° is  $SnSO_3 \cdot 2SnO \cdot 4H_2O$  and the one stable at 50° is  $SnSO_3 \cdot 2SnO \cdot 2H_2O$ . H. S. v. K.

The ternary system isomyl alcohol-propyl alcohol-water. James Coul and Henry B. Hope. *J. Phys. Chem.* 39, 907-71 (1935).—The solubilities,  $d_s$  and  $w_s$  were detd. for the system iso-AMOH-*n*-POH- $H_2O$  at 25°. The lines were detd. for 9 pairs of solns. H. G.

Studies of equilibrium and surface processes in the system water-formic acid-phenol. V. I. Nestorova, N. N. Petin and K. V. Topchieva. *J. Gen. Chem. (U. S. S. R.)* 5, 848-61 (1935), cf. *C. A.* 26, 3168.—Study of the changes in the vol. surface concns. of liquid two-phase systems with 3 components and of the electrochem. properties of these solns was begun with the system  $H_2O-HCO_2H-PhOH$  by the method previously described. In the presence of sufficient  $HCO_2H$  in the mixt.  $H_2O$  and  $PhOH$  are miscible at room temp. The conductometric method of titration is most suitable for the detn. of the concn. of  $HCO_2H$  in the presence of  $PhOH$  and  $H_2O$ ,

when  $PhOH$  is not present in excess. Detd. by this method the coeff. of distribution of  $HCO_2H$  between  $PhOH$  and  $H_2O$  begins to increase with rising concn. of the acid and on reaching the value 1.732 (at the concn. of  $HCO_2H$  in the mixt. of  $10.76 \times 10^{-4}$  g./mol. per l.) drops to the mixing point. The agreement in the results of detn. of  $HCO_2H$  in the contiguous layers by the conductometric method and the "vol. method" of Kablukov indicates that the latter procedure can be used in the study of the equl. of 2-phase systems with 3 components. The "vol. method" of detn. of the changes of the concns. of  $H_2O$  in the top and bottom layers disclosed that at the crit. point of concns. these tend sharply to equalize, while the coeff. of distribution of  $H_2O$  between the contiguous layers decreases rapidly nearing 1 asymptotically. With the increasing concn. of  $HCO_2H$  the sp. elec. cond. of the lower layer ( $PhOH$ ) invariably increases up to the crit. point, while that of the upper layer ( $H_2O$ ) at first sharply increases and then drops, whereby the position of the max. is observed at the same partial concn. of  $HCO_2H$  as in its aq. solns in absence of  $PhOH$ . The position of this max. also nearly corresponds with that of the coeff. of distribution of  $HCO_2H$  ( $C_1/C_2$ ). The consts. of disson of  $HCO_2H$  for the bottom  $PhOH$  layer, called by Ostwald's law of diln., show a definite trend toward an increase on approaching the crit. point, while those for the aq. solns of  $HCO_2H$  and for the top  $H_2O-PhOH$  layer remain const. All 3 consts. differ in magnitude from each other about 100 times. Computed on the basis of elec. cond., the coeff. of distribution of undissoc. mols. of  $HCO_2H$  shows the same course in relation to its total concn. as the coeff. of distribution of the entire acid. The surface tensions of the contiguous layers at the boundary with air are equal and are independent of the total concn. of  $HCO_2H$ , remaining practically equal to the surface tension of the two-component system of  $H_2O$  and  $PhOH$ . With increase of the total concn. of  $HCO_2H$  in the system, the magnitude of surface tension at the boundary of liquid phases increases nearly rectilinearly to a crit. point. This increase is parallel to the increase of the coeff. of distribution of  $PhOH$ ; it is almost proportional to the increase of the total concn. of  $HCO_2H$  and is not in any simple relation to the coeffs. of distribution and partial concns. of  $HCO_2H$  and  $H_2O$ . Chas. Blane

Inner adsorption in salt crystals. II. Reason for inhomogeneity in a precipitate and the requirements for forming an homogeneous precipitate as possible. D. Balarev. *Z. anorg. Chem.* 102, 241-62 (1935), cf. *C. A.* 29, 2815, 3570, 3573.—Theories concerning the formation and aging of ppts. are explained which suggest that a chemist can never hope to obtain a perfectly pure ppt., conforming absolutely to the laws of definite and multiple proportions. Ppts. as they begin to form are colloids and as such are inclined to adsorb dissolved ions or mols. present in the soln. and this adsorption is, to some extent at least, selective. The purity of a ppt. depends upon such factors as the size and habit of the "growth conglomerate," the rate of pptn. and the time it is allowed to remain in contact with the mother liquor, the lattice structure of the solid changes that may take place (such as hydrolysis) on heating the crystal, etc. A few expts. are described for illustration. W. T. H.

Catalysis of hydrogenation. II. Technique of hydrogenation. A. S. Ginzberg. *J. Gen. Chem. (U. S. S. R.)* 5, 795-8 (1935), cf. Ginzberg and Ivanov, *C. A.* 25, 4173.—With the use of the previously described catalysts prepd. by the addn. of Pd or Pt chloride to powd. Ni, Al, etc., it is possible to measure exactly the amt. of H consumed in the hydrogenation of a definite quantity of an unsatd. compd., and, therefore, to det. its degree of unsatd. state, or the "H no." (cf. Grün and Holden, *C. A.* 18, 912, Fokin, *C. A.* 3, 1009). In the preliminary discussion, the app. is illustrated and described. Chas. Blane

The energetics of catalysis. V. The temperature coefficient of hydrogenation processes. Edward B. Masted and Charles H. Moon. *J. Chem. Soc.* 1935, 1190-2, cf. *C. A.* 29, 4659.—The low-temp. pos. temp. coeff. of the catalytic hydrogenation of crotonic and maleic acids

over Pt changes to a neg. temp. coeff. between 60° and 80°. The true energy of activation appears to be about 25,000 cal. per mol. for both acids. P. H. Emmett

The adsorption of hydrogen by iron synthetic-ammonia catalysts. P. H. Emmett and R. W. Harkness. *J. Am. Chem. Soc.* 57, 1631-5 (1935); cf. *C. A.* 28, 1213; 1583. —Exptl. evidence is presented to show that the 2 types of activated adsorption of H<sub>2</sub> that occur on Fe synthetic-NH<sub>3</sub> catalysts are in part at least adsorptions on the surface of the catalysts rather than any type of soln. within the metal. The rate of the lower temp. (type A) activated adsorption agrees closely with that calcd. by assuming that all H<sub>2</sub> molts. are adsorbed that strike the catalyst with the observed energy of activation, 10,400 cal.

P. H. Emmett  
The catalytic interconversion of ortho-para hydrogen over iron, platinum and nickel catalysts. P. H. Emmett and R. W. Harkness. *J. Am. Chem. Soc.* 57, 1621-31 (1935); cf. *C. A.* 27, 4730. —The ortho-para interconversion of H<sub>2</sub> over Fe synthetic-NH<sub>3</sub> catalyst was studied as a function of the temp., pressure, time of contact and presence of various poisons. Both types of activated adsorption of H<sub>2</sub> on Fe are poisons for the conversion of ortho to para H<sub>2</sub> at liquid-air temp. The low-temp. ortho-para conversion over Ni and Pt is also inhibited by the activated adsorption of H<sub>2</sub>. P. H. Emmett

Chemical reactivity and catalysis of solids in transformations. J. Arvid Hedvall. *Stenok Kem Tids.* 47, 161-88 (1935) (in German). —A review covering 42 papers in which the following topics are discussed: X-ray radiation; metastable crystal forms with incomplete lattice structure; lattice structure with varying polarization; microstructure; crystallographic transformation and magnetic states. A. R. Rose

Catalytic decomposition of hydrogen peroxide in the presence of mixed manganous and cupric salts. M. Bobelsky and L. Bobelsky-Chajkin. *Compt. rend.* 201, 604-6 (1935). —Although Mn ions (e. g., MnSO<sub>4</sub> aq.) exert no catalytic action on the decompn. of H<sub>2</sub>O<sub>2</sub>, and Cu ions (e. g., CuSO<sub>4</sub> aq.) very little, a mixt. of the 2 is very active. With 0.05 N MnSO<sub>4</sub>, the velocity const., *K*, is a max. with the addn. of 0.04 N CuSO<sub>4</sub>, and with const. concn. (0.04 N CuSO<sub>4</sub>) *K* increases with the concn. of MnSO<sub>4</sub> to at least 0.15 N MnSO<sub>4</sub>. Other salts (except chlorides, whose action is peculiar, cf. *C. A.* 28, 6065), generally retard or stop the reaction, but those of Zn and Cd accelerate it. C. A. Silberrad

The effect of ferromagnetic transformation on the catalytic power of nickel in the reaction  $2CO = CO_2 + C$ . J. Arvid Hedvall and Folke Sandford. *Z. physik. Chem.* B29, 455-63 (1935); cf. *C. A.* 29, 1314. —Four kinds of Ni catalysts were prepd. and carefully analyzed for impurities. The Curie point was detd. for each. The reaction  $2CO = CO_2 + C$ , catalyzed with this Ni, was then studied and, as with other reactions, the rate increased sharply at the Curie point. The effect of impurities on the reaction was also studied as well as the condition of formation of Ni carbide and its effect on the reaction. G. M. Murphy

The mechanism of carbon oxidation in the presence of certain metallic oxides. Don Martin and J. E. Day. *Proc. Indiana Acad. Sci.* 44, 118-20 (1931). —Since the oxidation temp. of C, with Ag<sub>2</sub>O as a secondary substance, is the same in O<sub>2</sub> as in N<sub>2</sub>, Ag<sub>2</sub>O must be a stoichiometric O<sub>2</sub> carrier. With Fe<sub>2</sub>O<sub>3</sub> and CuO as secondary substances, the oxidation temp. is lower in an atm. of O<sub>2</sub> than in N<sub>2</sub>; therefore these oxides are adsorption O<sub>2</sub> carriers.

W. J. Peterson  
Oxidation of sugars by air in the presence of ceric hydroxide sol and cerous hydroxide gels. J. C. Chosh and P. C. Rakshit. *J. Indian Chem. Soc.* 12, 357-70 (1935). —Heterogeneous oxidation processes in the presence of catalysts were studied by use of a modification of the Barcroft-Warburg app. for respiration. Mixts. contg. 5 cc. of (I) and 5 cc. of dil. alkali were introduced into the cell and the pressure drop due to absorption of O was noted from time to time. The *p*<sub>H</sub> remained const. for 5 hrs., the av. duration of the expt. For *p*<sub>H</sub> up to 9.6 no oxidation

was observed during 5 hrs. in the absence of the sol. Oxidation takes place at the surface of the colloid particles which adsorb the sugar molts. almost instantaneously in quantities independent of the concn. of the sugar solns. Even at very low concns. of alkali, sugars undergo oxidation by air in the presence of Ce(OH)<sub>3</sub>. Oxidations were performed at *p*<sub>H</sub> 7.05, 7.0 and 8.6 with phosphate buffers and a suspension of Mg(OH)<sub>2</sub>. Levulose is slightly oxidized by air in phosphate buffers even in the absence of catalysts. The brown ppt. obtained during the oxidation in the presence of Ce(OH)<sub>3</sub> was proved to be ceric hydroperoxide [C. A. 25, 3764] and not Ce(OH)<sub>3</sub>. In the presence of visible radiations, the rate of absorption of O is somewhat increased. H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>H at *p*<sub>H</sub> as low as 7.9 is stable to air in the presence of Ce(OH)<sub>3</sub> at 32°. The oxidation of glucose is, however, strongly retarded in the presence of H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>H buffers. In the above expts. Ce(OH)<sub>3</sub> not only serves as a detector for H<sub>2</sub>O<sub>2</sub> produced during the course of anaerobic oxidation, but its particles provide an active surface for the dehydrogenation of the sugars preliminary to the combination of the liberated H<sub>2</sub> with O<sub>2</sub> to form H<sub>2</sub>O<sub>2</sub>. Equations representing the postulated chem. reactions are given. The initial catalytic surface of Ce(OH)<sub>3</sub> is rapidly converted into ceric hydroperoxide by preliminary dehydrogenation of the substrate sugars. After the 1st hr. the relative surface areas attain const. values and O<sub>2</sub> is absorbed at a steady rate which is the difference between the rate of absorption of O<sub>2</sub> to form H<sub>2</sub>O<sub>2</sub> and the rate of decompn. of ceric hydroperoxide. H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>H is a powerful inhibitor of the preliminary dehydrogenation and the subsequent formation of peroxide. The slightly catalytic action of illumination on aerobic oxidation is ascribed to the acceleration of the rate of reaction in the breakdown of 4Ce(OH)<sub>3</sub>·OOH<sub>2</sub> to 4Ce(OH)<sub>3</sub>, 2H<sub>2</sub>O and 3O<sub>2</sub>. Such a reaction would exert a larger relative ratio of surface of Ce(OH)<sub>3</sub> to that of H<sub>2</sub>Ce(OH)<sub>3</sub>. C. R. Addison

Active oxides. XCII. Reciprocal actions between solid substances. Gustav F. Härtig, Theodor Meyer, Herbert Klitel and Susanne Cassier. *Z. anorg. allgem. Chem.* 224, 225-62 (1935). —Active states of the systems CuO·Fe<sub>2</sub>O<sub>3</sub> and CuO·Cr<sub>2</sub>O<sub>3</sub> during the transition from the mixt. to cryst. compd. were characterized by their catalytic effect on N<sub>2</sub>O decompn. The changes in magnetic properties during aging of the systems: Fe<sub>2</sub>O<sub>3</sub>/Cr<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> were studied and the changes in hygroscopicity of the systems: MgO/Fe<sub>2</sub>O<sub>3</sub>, BeO/Fe<sub>2</sub>O<sub>3</sub>, ZnO/Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>/Fe<sub>2</sub>O<sub>3</sub>, ZnO/Cr<sub>2</sub>O<sub>3</sub> and CuO/Al<sub>2</sub>O<sub>3</sub> were detd. These data furnish evidence for certain ranges of existence of active intermediate states. C. E. P. Jeffreys

Conditions of formation of manganese trichloride and of its catalytic decomposition. M. Bobelsky and R. Cohen. *Compt. rend.* 201, 662-4 (1935). —A Mn<sup>++</sup> salt is oxidized to MnCl<sub>3</sub> in HCl aq. soln. of any concn. by HMnO<sub>4</sub>, H<sub>2</sub>CrO<sub>4</sub> or HClO<sub>4</sub>; in HCl aq. soln. of concn. above 6 N by H<sub>2</sub>O<sub>2</sub>, HNO<sub>3</sub>, HNO<sub>2</sub> and H<sub>2</sub>S<sub>2</sub>O<sub>8</sub>; other oxidizers have no effect. The MnCl<sub>3</sub> soln. is more stable the greater the concn. of HCl. Its decompn. is very slow at room temp., but is rapid even at 0° in the presence of (in decreasing order of activity, and more rapidly save in presence of Ag, as the concn. of HCl is greater) Ag, Pt<sup>IV</sup>, Pb and Cu<sup>II</sup> ions. Co, Ni, Fe<sup>III</sup>, Mo<sup>VI</sup>, V<sup>V</sup>, Ti<sup>III</sup>, Hg<sup>II</sup>, Au<sup>III</sup> and U<sup>VI</sup> have no catalytic effect. C. A. S.

A generalized thermodynamic notation. H. A. C. McKay. *J. Chem. Physics* 3, 715-16 (1935). —A generalized notation is described which leads to a method of writing down from one generalized equation all the members of a family of thermodynamic equations (cf. Koenig, *C. A.* 29, 1316). In particular, the signs of the various terms are very easily manipulated. A method is given for obtaining the generalized equation when any one member of a family is known. The method is easily extended to problems of capillarity, magnetization, etc.

G. M. P.  
Thermodynamics of stationary systems. I. The thermoelement. B. Bruins. *Proc. Roy. Soc. (London)* A151, 640-61 (1935). —The quant. application of the first and

second laws of thermodynamics to stationary systems requires the introduction of 2 arbitrary assumptions: (1) the max work to be expected from a reversible process remains unaltered by the presence of a simultaneous irreversible process, and (2) there exists a const. ratio between the Joule effect and the "polarization" effect. The first assumption leads to the 2 Kelvin relations connecting the Seebeck, Peltier and Thomson coeffs; and the second leads to a new relation connecting the Seebeck coeff with the thermal and elec. conductivities of the material. It is concluded that the whole problem of the thermoelement reduces to a knowledge of the thermal and elec. conductivities. II The diffusion element. *Ibid* A151, 651-65 — Utilizing the 2 arbitrary assumptions already introduced for the thermoelement (see above), B. develops the quant. thermodynamic relations for the diffusion element, and obtains equations relating the Helmholtz coeff with the Peltier coeff, with the coeff. of the "homogeneous" effect, and with the coeffs. of diffusion and elec. cond. The whole problem of the diffusion element reduces to a knowledge of diffusion and elec. cond.; the only difference between the case of the thermoelement and the diffusion element follows from the fact that there exist more than one energy carrier in electrolytic systems.

F. D. Rossini

The thermal conductivity of quartz at low temperatures W. J. de Haas and Th. Biermasz *Physica* 2, 673-82 (1935) — The thermal cond. of a quartz cylindrical rod, axis coinciding with the crystal axis, 5 cm. long, 0.308 cm. diam. was measured between Cd wire resistance thermometers. The ends were clamped in Cu caps, electrically heated. The app. was submerged in a cryostat and evacuated, with cooling by liquid O or H. For the lowest temp. (liquid He) a 3 X 0.13-cm. rod was used, and a Pb wire thermometer. From the final results between  $T = 2.77$  and 80.9° abs. is seen that the resistance increases sharply below 10° abs., the min. lying at about this temp. No proportionality of sp. resistance  $W$  with  $T$  or  $T^2$  is found. A few values of  $W$  are:  $1.12 \times 10^{-4}$  at 2.77° abs., 0.483 at 3.87°, 0.285 at 4.74°, 0.07 at 14.94°, 0.123 at 18.84°, 0.199 at 23.60°, 0.339 at 29.6°, 1.47 at 74.6°, 1.75 at 88.9°. Inhomogeneities can increase the resistance to the double value in the liquid-II range, which is particularly sensitive. After heat treatment, 8 hrs. at 340°, 5 hrs. at 570° and slow cooling (recombination of ions) the resistance values around 20° abs. increase up to 10%. The increase of thermal resistance of pure metals at very low temps. has evidently a character similar to that for insulators (de Haas and Bremmer, *C. A.* 26, 1490, 4518, 4992).

B. J. C. van der Hoeven

Thermochemical data for titanium oxides Nobuyuki Nasu *Kinzoku-no-Kenkyu* 12, 411-18 (1935), cf. *C. A.* 29, 5720 — By use of the exptl. values obtained in the previous study (N. Nasu, *C. A.* 29, 7773)  $n$  calcd.  $\Delta F^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$  of the following reactions: (a)  $2TiO_2 + H_2 = Ti_2O_3 + H_2O$ ,  $\Delta H^\circ = 4095 + 0.874 T + 0.003856 T^2$ ,  $\Delta F^\circ = 4095 - 2.0125 T \log T - 0.003856 T^2 + 10.1 T$ ,  $\Delta H_{1000}^\circ = 4700$  cal.,  $\Delta F_{1000}^\circ = 5280$  cal.;  $\Delta S_{1000}^\circ = 1.95$  cal./degree. (b)  $Ti_2O_3 + 0.5 O_2 = 2TiO_2$ ,  $\Delta H^\circ = -61,520 - 1.814 T - 0.005506 T^2 + 0.00000074 T^3$ ,  $\Delta F^\circ = -61,520 + 4.1760 T \log T + 0.005506 T^2 + 0.00000037 T^3$ ,  $\Delta H_{1000}^\circ = -62,530$  cal.,  $\Delta F_{1000}^\circ = -59,770$  cal.,  $\Delta S_{1000}^\circ = -9.26$  cal./degree. (c)  $2Ti + 3/2 O_2 = Ti_2O_3$ ,  $\Delta H_{1000}^\circ = -388,070$  cal. (d)  $2TiO_2 + CO = Ti_2O_3 + CO_2$ ,  $\Delta F^\circ = -6005 + 2.1552 T \log T - 0.008306 T^2 + 0.00000008 T^3 + 10.64 T$ ,  $\Delta F_{1000}^\circ = -6220$  cal. The lattice energy of  $Ti_2O_3$  was calcd. from the Born-Haber thermochem. cycle to be  $U = 3559.51$  cal., and was compared with the theoretical value calcd. from Kapustinsky's new equation of the lattice energy. The discrepancy between the exptl. and the theoretical values is 2.6%. This is attributed to the uncertainty which will be caused by applying effective ion radius of  $Sc^{+++}$  for that of  $Ti^{+++}$  to the crystal.

Tomo-o Sato

Thermochemistry H. C. Palmer *School Sci. Rev.* 17, 298 (1935) — Cheap thermos flasks are recommended for use in instructional experiments.

O. Reinmuth

Specific heat, entropy and free energy of sulfur (S)

vapor at temperatures between 100° and 5000°K. 1 Godnev and A. Sverdlin. *Z. Physik* 97, 124-30 (1935) cf. *C. A.* 29, 6131. — The sp. heat at const. vol., entropy and free energy at const. pressure are calcd. from spectroscopic data for  $S_2$  vapor in the perfect gas state. The values of the free energy agree with those obtained by Montgomery and Kassel (*C. A.* 28, 5324).

B. S.

The theory of solid bodies at high temperatures, with special regard to the temperature dependence of  $C_p$  Gerhard Damköhler. *Ann. Physik* 24, 1-30 (1935) — The Grüneisen-Debye theory of solid bodies is extended on the assumption of a temp. dependence of the vibration frequency. The existing data on  $C_p, 1/V_0 (\partial V/\partial T)$ , and  $1/V_0 (\partial V/\partial P) T$ , are utilized to compute values of  $C_p$  over a large range of temp. for NaCl, KCl, KBr, Ag, Cu and Pb. With the exception of Cu and Pb, a decrease in  $C_p$  occurs with increasing temp., at temps. above about 200° to 300°. For a linear chain, an expression is derived for the Helmholtz free energy as a function of the temp. and the length of chain, though the method employed is practicable only for the temp. region in which the anharmonic part of the potential energy is equal to or less than  $kT$ . The Gibbs thermodynamic potential of a linear chain as a function of the temp. and pressure is derived. The Morse and Born-Mayer potential-energy curves are discussed in relation to D's theory.

F. D. Rossini

A note on the specific heat of liquid deuterium oxide R. S. Brown, W. H. Barnes and O. Maass. *Can. J. Research* 13B, 167-6 (1935) — Preliminary detns. of the sp. heat of liquid  $D_2O$  from 4° to 65° are described. The sp. heats over the temp. ranges 4° to 26°, 26° to 45°, and 26° to 65°, are 1.018, 1.003 and 1.008, resp., while the av. sp. heat between 4° and 65° is 1.01 cal. per g. per deg.

J. W. Shipley

The heat capacity of potassium chloride from 2.3° to 17° abs. W. H. Keesom and C. W. Clark. *Physica* 1, 698-706 (1935) — Variability of Debye  $\theta$  has previously been found for metals (Ag), it is now studied for a non-conductor (cf. Blackman, *C. A.* 29, 3903). A cylindrical Cu calorimeter was used, 103 X 34 mm., suspended in a vacuum vessel, equipped with phosphor bronze and constant resistance thermometers and an elec. heater. The calorimeter contained 1.03 mols. KCl; He served as heat conductor. In agreement with Blackman's theory there is a gradual rise of  $\theta$  down to 6° abs., then a rapid rise, max. at 4.5°, followed by a drop in  $\theta$ . The drop may be due to desorption of He from the crystal surfaces. As compared with the Ag heat-capacity curve, the drop in  $\theta$  with  $T$  is not as rapid, owing to electron heat. If a term  $1.526 \times 10^{-4} T$  is subtracted from the Ag values they approach the KCl values satisfactorily.

B. J. C. van der Hoeven

The heat of vaporization of water and the specific volume of the saturated vapor to 202 kg./sq. cm. (365°) M. Jakob and W. Ritz. *Physik. Z.* 36, 651-9 (1935) — Data were obtained at 270°, 330°, 350°, 360° and 365° (56, 131, 169, 190 and 202 kg./sq. cm.). Utilizing these and their own previous calorimetric data, together with the data of L. B. Smith and Keyes (*C. A.* 28, 7095) on the sp. vol. of the liquid, J. and F. calc. values for the heat of vaporization and the sp. vol. of the vapor from 0° to 372°. Comparison is made with other existing data.

F. D. Rossini

Some physical properties of mixtures of water and glycol G. Romstatt. *Industrie chimique* 22, 648-51 (1935) — Data are given showing the rise in temp. produced on mixing various proportions of  $H_2O$  and glycol at 15°, the vols. occupied by the resulting solns., the wts. of glycol per l. and the contraction in vol. of the solns. Curves are given showing the  $d$  at 0°, 15°, 30° and 45° of aq. solns. contg. 0-100% glycol, the  $n$  at 15°, 30° and 45° of similar solns., and their initial  $b$  ps. The various detns. were made on solns. contg. 5, 10, 15, 20, 25, 35, 50, 60, 75 and 85% glycol. A Papineau-Couture dissociation energy of carbon monoxide and the heat of sublimation of carbon H. Lessheim and R. Samuel. *Nature* 136, 608 (1935); cf. Goldfinger and Lasareff, *C. A.* 29, 5730. — From spectroscopic data the disson energy

of CO is calculated to be 10.45 kcal. and the heat of sublimation of C ( $\beta$ -graphite) 155.7 kg.-cal./mol. E. O. W. Detm. of U, and the U content of sea water (Hirnberger, Karlik) 7.

### 5—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

W. ALBERT NOYES, JR.

Theory of elementary particles. II. Electromagnetic whorls and elementary particles. N. S. Japolsky. *Phil. Mag.* 20, 641-700 (1935). cf. C. A. 29, 7783.

E. J. Rosenbaum

Electronic structures of molecules. XIV. Linear triatomic molecules, especially carbon dioxide. Robert S. Mulliken. *J. Chem. Physics* 3, 720-39 (1935). cf. C. A. 29, 7787. Electron configurations are given for  $\text{CO}_2$ ,  $\text{BO}_2$ ,  $\text{N}_2\text{O}$ ,  $\text{C}_2\text{O}$ ,  $\text{NO}_2$ ,  $\text{N}_2$ ,  $\text{NCO}$ ,  $\text{NCS}$ ,  $\text{BeF}_2$ ,  $\text{HgCl}_2$ ,  $\text{ClCN}$ ,  $\text{CIBO}$ ,  $\text{NO}_2$  and the  $\text{HeI}_2$  type of mol. All the electron configurations are formally analogous, they are formally identical in isoelectronic molecules. The relations of electron configurations to valence conceptions are discussed. This method gives a simple description of intermediate cases (double-bonded  $\text{A}=\text{B}=\text{C}$ , single-bonded  $\text{A}-\text{B}-\text{C}$ , homopolar  $\text{A}=\text{B}-\text{C}$ , heteropolar  $\text{A}^+-\text{B}^+-\text{C}^-$ ,  $\text{A}-\text{B}=\text{C}$ ,  $\text{A}^+=\text{B}=\text{C}^-$ , etc.). The min. ionization potentials of  $\text{CO}_2$ ,  $\text{CS}_2$  and  $\text{HgCl}_2$  correspond to the removal of a nearly nonbonding  $\pi$  electron of the O, S or Cl atom. The ultraviolet emission bands of  $\text{CO}_2$  probably represent transitions among 4 electron states of  $\text{CO}_2^+$  which correspond to the 4 lowest ionization potentials of  $\text{CO}_2$ : 13.72, 17.09, 18.00 and 21.5 eV. If this interpretation is correct, the  $\text{CO}_2^+$  mol. is linear in these states. The min. potential of 13.72 eV disagrees with the value based on electron impact. Linear  $\text{NO}_2^+$  is composed of a stable system of closed shells, the final electron added to make  $\text{NO}_2$  would have to go into a fairly high energy antibonding orbital. Evidently the actual  $\text{NO}_2$  avoids this by being triangular. Ultraviolet absorption spectra of  $\text{CO}_2$ ,  $\text{CS}_2$  and  $\text{COS}$  are also considered.

G. M. P.

Relation between the internuclear distances and force constants of molecules and its application to polyatomic molecules. Richard M. Badger. *J. Chem. Physics* 3, 710-14 (1935); cf. C. A. 29, 2908. Clark, C. A. 23, 7143; Allen and Longair, C. A. 29, 4671. The relation between internuclear distances and force constants is found to persist in polyat. mol. The internuclear distances of  $\text{CCl}_4$ ,  $\text{SiCl}_4$ ,  $\text{SnCl}_4$ ,  $\text{SF}_6$ ,  $\text{SeF}_6$  and  $\text{TeF}_6$  were predicted from vibrational data alone, and were found to agree well with exptl. values.

G. M. P.

A new conception of the chemical elements and the forces between bearing metal and lubricant. Heinrich Herbst. *Chem.-Ztg.* 59, 777-8 (1935). D. F. Brown

Rotatory power of quartz for rays perpendicular to the optic axis and its dispersion between 2537 and 5780 Å. Georges Bruhat and Louis Weil. *Compt. rend.* 201, 887-9 (1935); cf. *Ibid.* 719. A repetition of the authors' previous experiments, and their extension to the region 2537-5780 Å. by means of their quartz compensator gave substantially the same result for  $\epsilon$  and  $\rho$ , while  $\epsilon/\rho$  (the ratio of the rotatory power of rays  $\perp$  to the axis to that of rays  $\parallel$  thereto) averages  $-0.54$  instead of  $-0.52$ . Thus for this interval at least the rotatory dispersion of quartz is the same for rays  $\perp$  and  $\parallel$  to the axis.

C. A. Silberrad

Electron diffraction by gases. I. R. Maxwell, S. B. Hendricks and V. M. Mosley. *J. Chem. Physics* 3, 699-709 (1935). The method of electron diffraction was used to det. the C—O—C valence angle in  $p,p'$ -( $\text{C}_6\text{H}_4$ ) $_2\text{O}$ ; it is  $118^\circ \pm 3^\circ$ . By this method it was detd. that  $\text{P}_4$  and  $\text{As}_4$  have regular tetrahedral structures, with at. sepns of 2.21 and 2.44 Å, resp. These at. distances are approx. the same as those found by crystal structure analysis. This shows that these distances do not change greatly when the bond angle decreases from  $100^\circ$  to  $10^\circ$ . The app. is described.

G. M. P.

The theory of image errors of the electron microscope. Walter Glaser. *Z. Physik* 97, 177-201 (1935).—The 3

distortion errors are fully discussed. A necessary and sufficient condition for their vanishing is given for a general rotation-symmetrical optical medium.

B. Swirles

Use of electron lenses for  $\beta$ -rays. Otto Klemperer. *Phil. Mag.* 20, 545-61 (1935).

E. J. Rosenbaum

Ion-optical images with electrical lenses. Jürgen Koch and Wilhelm Walcher. *Z. Physik* 97, 131-7 (1935).—Images of apertures are formed by the use of an electron system and a stream of K ions. The ion images are compared with electron images. Magnetic deviation experiments show that the images are formed by heavy positively charged particles. An image of a surface emitting ions can be obtained.

S. Tolansky

A new interference phenomenon in the passage of fast electrons through crystals. F. Kirchner and H. Lassen. *Ann. Physik* 24, 113-23 (1935).—A new type of crystal lattice interference has been found in the investigation of thin Ag layers. The interference points form a cross. This phenomenon is ascribed to the irradiation of many small Ag crystals, which are spread on the surface, oriented exactly parallel.

Harold Gershinowitz

The influence of the space-charge on the paths of the electrons in the magnetron. Hans Awender, Alfred Thoma and David M. Tombs. *Z. Physik* 97, 202-10 (1935).—A solution is given of the equations of motion of electrons in an electrostatic field and under their mutual electrostatic interaction. It is shown that the electrons then move in straight lines. The influence of a magnetic field is found by a perturbation method.

B. Swirles

The mechanism of high frequency discharge. H. Beck. *Z. Physik* 97, 335-75 (1935).—The spectrum of a high-frequency discharge in Hg vapor is compared with that of the pos. column. No difference of intensity for corresponding lines could be detected. Also absorption measurements gave the same no. of excited states in high-frequency discharge as in the pos. column; if a Maxwell distribution is assumed for the velocities in both cases the electron temps. are found to be the same for a large pressure region. Conclusion. The mechanism of high-frequency discharge can be interpreted similarly to that of the pos. column.

B. Swirles

The disappearance of hydrogen in the presence of positive ions. C. H. Kunsman and R. A. Nelson. *J. Chem. Physics* 3, 754 (1935).—Evidence is presented to show that the authors' previous conclusions (C. A. 23, 3402; 26, 4246; 29, 521) are valid, although they are not in agreement with those of Schechter (C. A. 29, 5732).

G. M. P.

Striations in the positive column in the hydrogen glow discharge. Hans H. Paul. *Z. Physik* 97, 330-54 (1935).—The mechanism of striations is studied on the basis that these are equipotential surfaces. The conditions are varied. The cross section gradient is important. Current voltage characteristics are found by use of 2 probes, one of which is movable in the tube axis. In the middle of the striations 2 velocity groups of electrons occur. A theory is proposed and is supported by the data. The varying forms of striations are due to a varying "ion mantle effect".

S. T.

Products and processes of ionization in methyl chloride as determined by a mass spectrometer. S. H. Bauer and T. R. Hogness. *J. Chem. Physics* 3, 687-92 (1935).—The ions  $\text{MeCl}^+$ ,  $\text{Me}^+$ ,  $\text{CCl}^+$ ,  $\text{CH}_2^+$ ,  $\text{Cl}^+$ ,  $\text{HCl}^+$ ,  $\text{CH}^+$ ,  $\text{C}^+$  and  $\text{H}^+$  were found to be produced in  $\text{CH}_3\text{Cl}$  as primary products (they are listed in order of decreasing intensity). The appearance potentials for  $\text{MeCl}^+$ ,  $\text{Me}^+$  and  $\text{Cl}^+$  are 11.0, 14.7 and 26 v., resp.

G. M. P.

Sputtering of metals by the impact of slow ions and measurement of the threshold value of the sputtering. Heinrich Lüder. *Z. Physik* 97, 158-70 (1935).—Argon ions are drawn perpendicularly from a glow cathode and

directed onto a thin metal wire acting as a sputtering electrode. The amt sputtered is detd. by measuring the wire resistance. The velocity of the ions is measured and the sputtering carried to an impact velocity of 40 v. The probable threshold values using  $A^+$  are Ni = 7 v, Cu = 12 v, Fe = 14 v, W = 24 v. The sputtering produced by alkali atoms is measured, the wires being heated to prevent alkali condensation. The sensitivity is improved by thinning the sputtering wires by evapn. Threshold values are,  $K^+$  on W = 36 v,  $Ca^+$  on W = 15 v,  $Li^+$  on W = 80 v,  $Cs^+$  on Cu = 15 v. Calcd values are compared with the measurements and the various theories discussed. S Tolansky

Comparison of the decrease in intensity of the primary cosmic radiation in different materials. J. Clay *Physica* 2, 645-9 (1935).—Generally the decrease in ionization is found greater for equiv. layers of materials (Pb, Fe, water, air) if the d is lower. Absolute intensity of cosmic rays. J. Clay *Ibid* 650-1. B. J. C. van der Hoeven

Absorption of the asymmetric component of cosmic radiation. V. M. Dukelskii and N. S. Ivanova. *J. Exptl. Theoret. Phys. (U. S. S. R.)* 5, 512-19 (1935).—The azimuthal asymmetry of the cosmic radiation was measured by a set of 3 Geiger-Müller coincidence counters on Mount Alagüez in Soviet Armenia, 35° 12' N latitude, 3250 m altitude. With zenith angle 45° the west-east azimuthal ratio was 1.08 ± 0.015. A 10-cm lead absorber between the counters reduced this to 1.03 ± 0.014. F. H. Rathmann

The frequency and magnitude of the showers produced in lead by penetrating radiation. H. Geiger and O. Zeiler. *Z. Physik* 97, 300-11 (1935); cf H. Geiger and E. Funke, *C. A.* 29, 3907. —With different geometrical arrangements of counters the threefold and fourfold coincidences due to showers produced in lead are counted. It is calcd. from the results that on an av. 0.6 shower per hr and per sq. cm. are emitted from a sheet of lead 1.5 cm. thick. By use of the theory of Geiger and Füller that each shower consists of one very energetic electron pair (C-rays) and their continuous radiation (D-rays), it is calcd. that with each C-electron approx. 200 D-rays are emitted. The "recoil" effect observed by Füller is explained on this theory. The mean absorption coeff. of the D-rays is approx. 1 cm<sup>-1</sup> Pb, corresponding to about 6 × 10<sup>4</sup> e. v. B. Swirles

Theory of radioactive fluctuations. Arthur E. Ruark and Lee Devol. *Phys. Rev.* 48, 772 (1935).—Math. G. M. P.

Nuclear theory. Kugao Nakabayashi. *Z. Physik* 97, 211-20 (1935).—The mass defects and nuclear radii are calcd. by the Thomas-Fermi method, exchange forces of Majorana's type (C. A. 27, 5000), between neutron and proton, and Coulomb forces between the protons are assumed; the particle density is assumed const. An expression is obtained for the binding energy, and the outstanding discrepancies with expt. are discussed. B. Swirles

$\beta$ -Radioactivity of neutrons. Lloyd Motz and Julian Schwinger. *Phys. Rev.* 48, 704-5 (1935).—Math. G. M. P.

Velocity of slow neutrons by mechanical velocity selector. J. R. Dunning, G. B. Pegram, G. A. Fink, D. P. Mitchell and E. Segré. *Phys. Rev.* 48, 704 (1935). G. M. P.

Absorption and detection of slow neutrons. D. P. Mitchell, J. R. Dunning, E. Segré and G. B. Pegram. *Phys. Rev.* 48, 774-5 (1935).—The transmission of slow neutrons is plotted against the mass per sq. cm. of the absorber, with the absorber-detector pairs B-B, B-Li, Li-B, Li-Li, Cd-B and Cd-Li. The B and Li absorption curves deviate from the exponential curve, because of the nonhomogeneity of the neutron beam and the dependence of the absorption upon the velocity distribution of the neutrons. G. M. P.

Excitation of  $\gamma$  rays by slow neutrons. Seshi Kikuchi, Kōji Fushimi and Hiroo Aoki. *Proc. Imp. Acad. (Tokyo)* 11, 253-5, *Proc. Phys.-Math. Soc. Japan* 17, 369-77

(1935).—Hg, Au, Cd, Ni, Fe, Cu, Pb and Al were studied to det. if  $\gamma$ -rays were emitted from them by slow neutrons, all but Pb and Al were shown to emit  $\gamma$ -rays at least for Pb and Al the radiation was much weaker. The cross section of the interaction was estd. at 5 (10<sup>-24</sup>) sq. cm. for Cu, Fe and Ni, somewhat larger for Au and very much greater for Cd. No combination of protons with neutrons to form deuterons was detected. The app. is described and some discussion is given. W. E. V.

The  $\gamma$ -rays emitted in the capture of slow neutrons. F. Rasetti. *Z. Physik* 97, 64-9 (1935).—The absorption of slow neutrons by  $\alpha$  nuclei is in general accompanied by the emission of radiation. A rough measurement of the energy of these  $\gamma$ -rays has been made by the method of the coincidences of the secondary Compton electrons. For the elements investigated, Cl, Co, V, Ag, Cd, Ir, Hg, the energies were found to be between 4 × 10<sup>4</sup> and 7 × 10<sup>4</sup> e. v. B. Swirles

The number of  $\alpha$ -particles emitted by uranium. Raimund Schedt. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse Abt. IIa*, 144, 191-211 (1935).—A total count of 53,088  $\alpha$ -particles was obtained from 2 preparations of U<sub>3</sub>O<sub>8</sub>. The app. procedure and calcs. are described in detail. The no. of  $\alpha$ -particles emitted per sec. by 1 g. of U is 12.64 ± 0.70%. Accordingly, the decay const. of U<sub>1</sub>,  $\lambda_{U1}$  = 1.5671 × 10<sup>-19</sup> years<sup>-1</sup> = 0.70%, and the period of half decay,  $T_c$  = 4.422 × 10<sup>9</sup> years = 0.70%. G. M. P.

Counting the  $\alpha$ -particles emitted by uranium. Friedrich Hecht. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse Abt. IIa*, 144, 213-15 (1935).—A micro method was used for detg. U which had been used for measurements of radioactivity. The U<sub>3</sub>O<sub>8</sub> was dissolved in HNO<sub>3</sub> and taken to fumes with HCl. The vol. was made up to 6-8 cc. and H<sub>2</sub>S passed in. Afterward, all Fe was removed by the hydroxylamine-HCl method (C. A. 27, 684; 28, 6390). The filtrate was taken to fumes with aqua regia, and the U pptd. with 8-hydroxyquinoline. This compd. contains 33.86% U. All operations except the last filtration were made in porcelain dishes and filters; it was made in Pt. G. M. P.

Mass spectrum of the positive radiation from radium C'. (Supplement. the spectrum of natural H-rays.) Lambrecht Wissgott. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse Abt. IIa*, 144, 227-41 (1935).—The construction and operation of a mass spectrograph are described, and the theory of its operation is given. A long camera was constructed for the detection of the undeviated radiation. By use of simultaneous elec. and magnetic deflection, it was found that the long-range radiation of Ra C' has a specific charge  $e/m$  =  $1/4$ . It is, therefore, an  $\alpha$ -ray. By comparing the spectrum of the normal radiation of Ra C' ( $R_{w1}$  = 6.96 cm.) with that of the long range radiation, the 9-cm. group was found to have a ratio of 25/10<sup>4</sup>, in good agreement with Rutherford's results. The mass spectrum of natural H-radiation was recorded, and used as a basis for the mass spectrographic registration of  $\alpha$  fragments. G. M. P.

Calculation of the velocity of  $\alpha$ -particles from their range, and its relation to the number of ion pairs produced. Stefan Meyer. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse Abt. IIa*, 144, 317-30 (1935).—The calcn. of the velocity  $v$  (in 10<sup>8</sup> cm./sec.) of  $\alpha$ -particles of av. range  $\bar{R}$  (in cm. of air at 15° and 760 mm.) is possible from the formula  $\bar{R} + v = A + Bv^2 + Cv^3$  ( $r = 0.26508$ ,  $A = 2.1850$ ,  $B = -2.5393$ ,  $C = 1.7335$ ). Further,  $(v - v_0) = aR$  (the initial velocity  $v_0 = 0.143$ ,  $a = 0.6648$ ,  $n = 1 + 1.15185v - 0.07441v^2$ ), or better,  $v = aR$ , where  $R$  is the sum of the point value of the ionization curve  $R_0$  and the addn.  $r$ ,  $R_0 + r = \bar{R} - 0.2$ ,  $a = 1.0977$  and  $n = 1.67851 + 1.16468v - 0.23333v^2$ . Geiger's relation  $v^2 = aR$  is valid for the range 3-11 cm. The initial energy of  $\alpha$  particles is of the order of 5 × 10<sup>4</sup> e. v. The equation for the relation between the range and the total ionization  $k = k_2 R^{1/2}$  is only a crude relationship for small values of  $R$ . Not only is the formula  $v^2 = aR$  inaccurate, but the relation of the no. of ion pairs



to the energy of the  $\alpha$ -particles increases with increasing velocities. G. M. P.

The  $\gamma$ -radiation of uranium X. E. Stahel and D. J. Comou. *Physica* 2, 707-18 (1935).—In a high-pressure CO<sub>2</sub> ionization chamber (17.8 atm.) and with Geiger Müller tubes the hard  $\gamma$ -radiation of U X<sub>2</sub> was studied. The app. has been described (C. A. 27, 5632). Absorption curves show an av. absorption coeff. of  $\mu_{\text{Fe}} = 0.83$  per cm.,  $\mu_{\text{Pb}} = 0.47$  per cm., in fair agreement with Hahn and Meitner (C. A. 17, 3645). Their second radiation with  $\mu_{\text{Pb}} = 2.3$  per cm. could not be found and is thought to be of secondary nature. B. J. C. van der Hoeven.

The nuclear  $\gamma$ -radiation of beryllium. Friedrich Koch and Fritz Rieder. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse Abt. IIa*, 144, 331-7 (1935).—The  $\gamma$ -radiation from  $\alpha$ -radiated Be was studied in a magnetic field with a Wilson chamber. In the formation of electron pairs there occurred preponderantly, if not always, equal distribution of the available energy. The numerical distribution of positrons and of photoelectrons of various energies indicates  $\gamma$ -lines at 5.6, 4.2, 2.6, 2.1 and  $1.4 \times 10^6$  e. v., with possible lines at 10.2, 5.6 and  $1.7 \times 10^6$  e. v. G. M. P.

Branching ratio of the actinium family of radioactive elements. Marcus Francis and Tcheng Da-Tchang. *Phil. Mag.* 20, 623-32 (1935).—The branching ratio was detd. for 2 pitchblende samples. Copptn. with both ZrF<sub>4</sub>O<sub>2</sub> and Ta<sub>2</sub>O<sub>5</sub> was used. Concordant results were obtained whose mean is  $4.05 \pm 0.17$ . E. J. R.

The disintegration of lithium by swift protons. K. Jakovlev. *J. Expil. Theoret. Phys. (U. S. S. R.)* 5, 595-8 (1935).—The formation of 8.4-cm  $\alpha$ -particles from Li bombarded by high-velocity protons began at 27 kv. velocity and increased rapidly up to 118 kv. The results agree well with those of Oliphant and Rutherford (C. A. 27, 4729). F. H. Rathmann.

Nuclear evolution of iron, cobalt and nickel. H. J. Walke. *Phil. Mag.* 20, 738-9 (1935), cf. C. A. 28, 6822.—Equations are written for the possible formation of Ni and Co from  $^{56}\text{Fe}$ . E. J. Rosenbaum.

Ercitation of secondary  $\gamma$ -rays by means of neutrons I. General. Effects in paraffin. R. Fleischmann. *Z. Physik* 97, 242-64 (1935).—Neutrons are slowed down with paraffin and are then able to produce nuclear  $\gamma$ -rays from H, Fe, Cu, Cd and Pb. The absorption of the H  $\gamma$ -rays is measured in paraffin, Al and Pb. The interaction of the neutrons with paraffin is discussed. The  $\gamma$ -rays arise from the fusion of slow neutrons with H<sup>1</sup> forming H<sup>2</sup>. The rays possess a quantum energy of  $1.5 \times 10^6$  e. v. The mass of the neutron is calcd. as  $1.0083$ . II. Measurements on iron, copper, cadmium and lead. *Ibid.* 263-76.—By suitable arrangements of absorbing layers neutrons are slowed down and measurements made on the absorption of the excited  $\gamma$  rays. The absorptions for various thicknesses of paraffin, Fe, Cu, Cd and Pb are measured for slow neutrons. The absorption coeffs. of the excited radiation are measured in paraffin, Al, Fe, Cu and Pb with various sources. The quantum energies of the excited radiations vary between  $0.45$  and  $2.6 \times 10^6$  e. v. S. Tolansky.

Protons from the disintegration of lithium by deuterons L. A. Delassus, W. A. Fowler and C. C. Lauritsen. *Phys. Rev.* 48, 848 (1935); cf. C. A. 29, 5733.—Li, bombarded by 700-kv. deuterons, gave proton groups with ranges of  $31.7 \pm 0.5$  and  $23 \pm 1$  cm., and  $\alpha$ -particle groups with ranges of  $13.8 \pm 0.7$  and  $8.9 \pm 1.0$  cm. The masses of Li<sup>6</sup> and Li<sup>7</sup> are calcd. to be  $8.0185$  and  $8.0072$ , resp. The protons are assoc. with the formation of radio-Li<sup>8</sup>. G. M. P.

Synthesis of radioelements by deuterons accelerated by means of an impulse generator. Frédéric Joliot, André Lazard and Pierre Savel. *Compt. rend.* 201, 829-8 (1935).—A generator capable of producing impulses of voltage  $3 \times 10^6$  v. (cf. Schup and Sollma, *Rev. gen. Elec.* 37, 799 and 38, 241 (1935)), combined with an acceleration tube similar to that of Brach and Lange (C. A. 25, 5545), is described. This method of accelerating the ions by impulses is more powerful than the continuous

method and does not require the extremely low pressures in the tube. It was used to produce accelerated deuterons with which C and B were bombarded, producing radio-carbon, and amts. of radionitrogen much greater than obtainable by bombarding B with  $\alpha$ -particles from natural radioelements:  $^{12}\text{C} + \text{H}^2 = ^{13}\text{N} + \text{H}^1$  (cf. Cockcroft, *et al.*, C. A. 28, 2900). C. A. Silberrad.

The radioactive climate and the radioactive waters of Badgastein, and their bioclimatic and balneological significance. Walter Kosmath and Otto Gerke. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse Abt. IIa*, 144, 339-55 (1935).—A survey of the radioactivity of the soil, water and air was made at several stations in and near Badgastein, during April, 1935. G. M. P.

Radiometric examination of a rhyolite flow. Edmond Rothé and A. Hée. *Compt. rend.* 201, 892-3 (1935); cf. C. A. 29, 6771.—Portions of a rhyolite flow in a quarry near Weischnbrunn (Alsace) show increased radioactivity (up to double) as compared with the average shown by rocks in that neighborhood. C. A. Silberrad.

Experiments on heavy hydrogen V. The elementary reactions of light and heavy hydrogen. Thermal conversion of ortho-deuteronium and the interaction of hydrogen and deuterium. Adalbert Farkas and Ladislav Farkas. *Proc. Roy. Soc. (London)* A152, 124-51 (1935), cf. C. A. 29, 414.—The rate of recombination of ortho-D and the interaction of H and D were measured between  $550^\circ$  and  $1000^\circ\text{K}$ . and at pressures between 3 and 74 mm. Hg. The mechanisms are (I) D + ortho-D<sub>2</sub> → para-D<sub>2</sub> + D, (IIa) D + H<sub>2</sub> → HD + D, (IIb) H + D<sub>2</sub> → HD + D. The rate for I is 2.4 times slower than the corresponding reaction with para-H. The difference is due mainly to differences in the frequency of collisions and the concn. of D atoms. The energy of activation is 0.5 kcal. greater than for the corresponding H reaction. For II, the difference in rate is due to the energy of activation, which is 1.7 kcal. greater for IIb than for IIa. The activated complexes HHH, DHH, HDD and DDD have zero-point energies similar to those of H<sub>2</sub>, HD and D<sub>2</sub>. From a consideration of the possible vibrational frequencies in the complexes, an estimate can be made of their zero point energies and an energy-level scheme constructed. The conclusions are consistent with the exptl. results. VI. Ratio of the magnetic moment of the proton to the magnetic moment of the deuteron. *ibid.* 152-7.—The conversion of ortho-D to normal D catalyzed by O was studied between  $83^\circ$  and  $203^\circ\text{K}$ . The rate const. obtained was compared with that for the para-H conversion. From the results, the ratio of the magnetic moment of the proton to that of the deuteron can be calcd. (cf. C. A. 29, 6826) as  $5.96 \pm 0.11$ . G. M. Murphy.

The principles of a new method for the measurement of the intensity of x-rays. V. I. Vekker, A. V. Ribergal and M. P. Ivanov. *J. Expil. Theoret. Phys. (U. S. S. R.)* 5, 490-5 (1935).—A Geiger-Müller counter is modified by combination with a thyatron relay and used to measure the intensity of x-rays at the rate of ten to a thousand quanta per second. Data are given on the time of discharge in the ordinary counter and in the modified system with the discharge chamber walls supplied by d. c. F. H. Rathmann.

Relation between density of blackening and x-ray intensity for characteristic copper radiation and Agfa-Lau film. F. D. Miles. *Trans. Faraday Soc.* 31, 1452-60 (1935).—The d. of blackening is found to be proportional to the x-ray intensity up to a d. of 1.0, with an error of less than 4% at the highest d. There is no three-fold effect. E. J. Rosenbaum.

The L absorption edges of protoactinium (91). V. Dolzjel and J. Marek. *Z. Physik* 97, 70-2 (1935).—The wave lengths of the  $L_{\text{III}}$  and  $L_{\text{II}}$  edges are given with an accuracy of  $\pm 0.1 \text{ \AA}$ . U. and of  $L_{\text{I}}$  edge to within  $\pm 0.2 \text{ \AA}$ . U. The  $L_{\text{I}}$  edge is much more diffuse than that of the others. The values of the positions of the further  $L_{\text{a}}$  levels are calcd. from the measured  $L$  values. S. T.

Scattering of x-rays varies discontinuously with the angle of scattering. Jean Laval. *Compt. rend.* 201, 889-91 (1935).—The scattering by a cryst. solid of mono-

chromatic x-rays outside the limits of selective reflection is examined. If scattering power,  $A$ , is defined as  $dI/dm \sin \theta$ , where  $dI$  is the mass concerned of the crystal,  $s$ , the cross section and  $I$  the intensity of the beam, of x-rays, which by scattering gives a beam of intensity  $dI$ , solid angle  $d\omega$ , in the direction making the angle  $\theta$  with that of the incident beam, the curve  $A \sim \sin \theta^2$  consists of a series of discontinuous portions sep. by "scattering edges," e.g., 6 for Al with  $K\alpha$  of Cu ( $\lambda = 1.955 \times 10^8$ ). In the same substance  $\sim \sin \theta^2$  (the "absolute abscissa" of an edge) is const. For different substances it is a linear function of the at. no.,  $\sim \sin \theta^2 = aZ + b$ . These laws are verified for Al, Cr, Ni, Zn, Ag, Sb, Pt and Au. C. A. Silberrad

The width of the  $K\alpha$  lines of gaseous krypton and of elements in chemical combination. Ernst Wilhelm Z. Physik 97, 312-20 (1935).—By use of cathode-ray excitation, and analyzing by means of a double-crystal spectrometer the breadths of the  $K\alpha$  lines of Kr, Rb (RbCl<sub>2</sub>), Br (KBr), and Cu (Cu, CuO, CuF) are measured. The values for Kr lie upon the same curve as the elements of at. no. 26-47, the state of aggregation having no observable effect upon the width. The values found for Rb, Br and Cu lie above the curve and show an effect due to the chemical combination. S. Tolansky

The intensity of x ray lines of gold. Leo Fuchler. Physica 2, 597-605 (1935).—See C. A. 29, 5015

Selective x ray diffraction from artificially stratified metal films deposited by evaporation. Jesse W. M. DuMoind and J. Paul Youtz. Phys. Rev. 43, 703 (1935).—A method is described by which 100 alternate layers (each) of Cu and Au were deposited on glass. The thickness of the deposit was 10,000 Å. Because of the diffusion of the atoms in the solid state, the intensity of the Mo K diffraction maxima decreases exponentially, with a "half-life" of 2 or 3 days. G. M. P.

An x ray method for the study of "bound water" in hydrophilic colloids at low temperatures. W. H. Barnes and W. F. Hampton. Can. J. Research 13B, 215-27 (1935), cf. C. A. 26, 4226.—A new method is described for the study of hydrophilic colloids by the application of x-ray methods of analysis to the frozen gels. The possibilities of the method and its limitations are shown by a qual study of the amt and variation with temp. of the so-called "bound" H<sub>2</sub>O in gelatin gels over the temp. -3° to -50°. J. W. Shipley

Kinetic interpretation of the coloring of glass by hard x-rays. Robert Livingston and Carl E. Nurnberger. J. Phys. Chem. 39, 1011-19 (1935).—Some expts have been performed on the rate of coloring of Pyrex glass by irradiation with hard x-rays, and on the distribution of relative absorptions, of the colored glass, as a function of the wave length for visible light. A simple hypothesis of the rate of coloring has been suggested. An equation has been derived from this hypothesis and it has been shown that the exptl measurements are in agreement with this equation. A discussion of the analogous effects of radiations from Ra is included. Harold Gershinowitz

The electrical and magnetic effect on the helium lines with rectangular crossed fields. Walter Steubing and Wilhelm Redepenning. Ann. Physik 24, 161-82 (1935).—The expts previously described (C. A. 29, 4201) have been extended. The at. theoretical difficulties which stood in the way of the investigation are discussed and it is explained why He, rather than H, was investigated first. Harold Gershinowitz

The vacuum arc spectra of rubidium and lithium. S. Datta and Parimal Chandra Bose. Z. Physik 97, 321-9 (1935).—The source used gives sharp lines. For Rb the satellites for the third and fourth members of the diffuse series are found, giving the splitting of the D levels. The structure of the red Li line 6708 Å is examined at various pressures. The existence of Li in the sunspots is questioned. S. Tolansky

Perturbation of higher cesium terms through paraffin hydrocarbons and measurements on the 1s-3p doublet of the principal series of potassium. Chr. Fuchthauer

and H. J. Reimers. Z. Physik 97, 1-7 (1935).—The displacements in the Cs lines 1s-7p to 1s-30p which are produced by methane, ethane and propane are measured. All the lines are strongly displaced to the red, the max values being 16.45, 20.99, 25.79 cm.<sup>-1</sup> for methane, ethane and propane, resp. In each case the displacements increase up the series to a limit value. Displacements increase with heavier mol. The effective cross section to slow electrons found for CH<sub>4</sub> is 73 sq. cm./cc. and for C<sub>2</sub>H<sub>6</sub> 120 sq. cm./cc. A table of the effective cross sections is given for He, Ne, Ar, Kr, Xe, H<sub>2</sub>, CH<sub>4</sub> and C<sub>2</sub>H<sub>6</sub>. Measurements are made of the half width and displacement of the 1s-3p doublet of K when mixed with various gases. The displacements and half width do not move parallel. S. Tolansky

The strengthening, due to drying, of the mercury lines in a mercury-hydrogen discharge. H. Beck. Z. Physik 97, 332-9 (1935).—The strengthening of the lines observed in H<sub>2</sub> dried with P<sub>2</sub>O<sub>5</sub> by Güntherschulze (C. A. 27, 4732) and others is confirmed. The spectroscopic results do not agree with those of Schmitzer (C. A. 23, 6629); the discrepancies are traced to lack of uniformity in his exptl conditions. It appears probable that the protons are responsible for the strengthening of the lines. B. Swick

A photographic method of measuring the intensities of spectral lines. Erwin W. Müller. Z. Physik 97, 97-106 (1935).—A photographic method is described enabling relative intensities to be measured. A high accuracy can be obtained without the use of a photometer. A double line method enables intensity variations of lines, and also the comparison of neighboring lines, to be made. The absorption of light filters can easily be measured without complex instruments. The relative intensities of heterochromatic lines can be determined with the help of a continuous source. S. Tolansky

Hydrogen in the upper atmosphere. Joseph Kaplan. Nature 136, 549-50 (1935).—The afterglow of N<sub>2</sub> is quenched by minute amts of H<sub>2</sub>. This and spectroscopic evidence indicate that H<sub>2</sub> (as well as H<sub>2</sub>O) is absent, or present in very low concn, at the atm. level at which the aurora occurs. G. M. P.

Excitation of spectral lines of the hydrogen molecule by electron impact. A. A. Krut'kov and L. S. Ornsen. Physica 2, 611-22 (1935).—A narrow electron beam, passed through 2 grids for acceleration and capture of ions, is used for excitation of H<sub>2</sub> in a Faraday cage. The excitation functions of the singlet and triplet systems of H<sub>2</sub> were detd. (4634, 4617, 4581 and 4340) at pressures from 0.045 to 0.0123 mm. The disson of the gas is detd from the change in intensity with pressure. The change in intensity of atom lines differs from that of mol lines at 30 e.v., indicating that excited atoms are formed from disson of excited mols as well as by direct atom excitation. Several characteristic curves are given. D. J. C. S. d. H.

Ultraviolet fluorescence spectra of iodine vapor, Mc Lennan bands. I. Duchinsky and Peter Pringsheim. Physica 2, 633-44 (1935); cf. C. A. 29, 7194.—The diffuse Mc Lennan bands (C. A. 7, 5704; 9, 406) were studied at various pressures, temps and excitation conditions (Cd, Hg, Zn lamps). Typical differences were obtained and their origin is discussed. The interpretation is considered as yet incomplete. B. J. C. S. d. H.

Emission spectrum of selenium oxide. Léon Bloch, Eugene Bloch and Choong Shim-Paw. Compt. rend. 201, 824-5 (1935), cf. C. A. 30, 25.—The emission spectrum of SeO extends from 3800 to 2880 Å, any portion of greater  $\lambda$  being masked by that of Se due to decomposition. It consists of some 30 bands, mostly double-headed, all degraded toward the red, and expressed by the formula  $\lambda = 3337.5 + [53.4(v' + 1/2) - 8.7(v' + 1/2)^2]$  (408  $v'$  and 5 + 48  $v'' + 1/2$ ), whence the energy of disson of the normal mol is 5.31 e.v. (C. A. 29, 4201). C. A. Silberrad

The fine structure of the C bands of calcium hydride. B. Grundström. Z. Physik 97, 171-6 (1935).—Details are given for the rotation doublets of the CaD C bands.

The appearance of the doublets stands in close relation to the perturbations described by Watson (*C. A.* 20, 2082<sup>2</sup>). The perturbations are discussed. S. Tolansky

A method of measuring light absorption at low light intensity and short exposure. Manifest v. Ardenne and P. W. H. H. *Z. Physik, Chem.* A174, 115 21 (1915).—An arrangement is described for measuring a light intensity of  $7 \times 10^{-10}$  cal/sec. with an accuracy of 1% and an exposure time of 1 sec. A photocell was used whose beam produces a large (greater than  $10^6$  Ohm) connection resistance reduction in voltage which is measured by an amplifying instrument. By a periodic interruption of the beam of light an a-c. amplifier may be used. R. H. Baechler

Emission spectrum of the flame of bromine burning in hydrogen and mechanism of the reaction. I. K. Klotz, *Proc. Imp. Acad. (Tokyo)* 11, 262 (1915).—Four strong bands, degraded to the red, are found between 6756 and 6400 Å. from a Br flame burning in H<sub>2</sub>. The band heads are tabulated. The band heads are given by  $\nu = 16841.2 \pm 1.0181 \mu^{-1} - 1.59 \mu^{-2} - 0.0087 \mu^{-3}$ ;  $(1022.73 \mu - 1.15 \mu^2) \text{ cm}^{-1}$  for  $\nu = 5, 6, 7, 8$ ; 21;  $\nu = 1, 2, 3, 4$ . The spectrum is due to excited Br<sub>2</sub> molecules. The mechanism is discussed. W. I. V.

Photography of the third harmonic of hydrogen chloride. Alden P. Graves and Charles W. Edwards. *Phys. Rev.* 48, 840 (1915).—The third harmonic of HCl at 10161 Å. is being investigated. The band origin is at 10222 Å. G. M. P.

Two new bands of CO<sub>2</sub> in the photographic infrared. G. Herzberg and H. Verleyer. *Phys. Rev.* 48, 700 (1915).—Two new CO<sub>2</sub> bands were photographed, with heads at 1.2311  $\mu$  and 1.2178  $\mu$ . The path length was 4 m., the pressure, 1.10 atm. These lines give values of 8309 and 8290  $\text{cm}^{-1}$ , agreeing well with the values 8291 and 8188  $\text{cm}^{-1}$ , predicted for the pair of bands  $\nu_2$  (1, 2<sub>g</sub>) by Adel and Demulson (*C. A.* 27, 3698<sup>2</sup>). G. M. P.

Infrared absorption of cyanides and thiocyanates. Walter Gordy and Dudley Williams. *J. Chem. Physics* 3, 181-7 (1915).—An study of HCN, NaCN, KCN, Ba(CN)<sub>2</sub>, KAg(CN)<sub>2</sub>, NaCl(CN)<sub>2</sub>, KCl(CN)<sub>2</sub>, KCN<sub>3</sub>, NH<sub>4</sub>CN<sub>3</sub>, KCNO, MeCN, Ph(CN)<sub>2</sub>CH and MeCN<sub>3</sub> were studied in the region 1.2-7.5  $\mu$ . An absorption band which occurred in all the compounds in the region 1.28-1.90  $\mu$  was attributed to changes in the vibrational energy of the bound CN group. An additional band at 1.1  $\mu$  appeared only in highly ionized cyanide salts, and was attributed to free CN ions. A double band appeared near 7  $\mu$  in the KCNO salt; it was not characteristic of the cyanide or thiocyanate salts. *Hydrolytic and solvation effects were observed in some of the salts.* G. M. P.

Absorption spectra of iodine solutions and the influence of the solvent. O. J. Barker. *Trans. Faraday Soc.* 31, 1172-8 (1915).—Molar extinction coeffs. were determined in the range 450-600 m $\mu$  for dil. solutions of I in CCl<sub>4</sub>, CHCl<sub>3</sub>, CH<sub>2</sub>Cl<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>, C<sub>6</sub>H<sub>5</sub>Cl, CH<sub>3</sub>Cl, CH<sub>3</sub>Br, cis- and trans C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub>. For CCl<sub>4</sub> solutions, the wave length of max. absorption ( $\lambda_{\text{max}}$ ) is  $1818 \pm 3 \text{ m}\mu$  and the molar extinction coeff. is about 600. For trans-C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub>,  $\lambda_{\text{max}}$  is about the same but for all the other solvents it is displaced toward the ultraviolet, indicating an increasing attraction of the solvent molecules for I<sub>2</sub>. The amt. of the displacement increases with the dipole moment of the solvent molecule. The effects on the absorption spectrum of various types of interaction with solvent molecules are discussed. L. J. Rosenbaum

Infrared absorption spectrum of methyl deuteride. Nathan Glasberg and P. V. Barker. *J. Chem. Physics* 3, 1084-7 (1915); cf. *C. A.* 28, 4110<sup>2</sup>.—The 6 fundamental frequencies of MeD<sub>3</sub> were observed and the fine structure of the bands was resolved. The frequencies,  $\nu_1$  1160.3,  $\nu_2$  1300.8,  $\nu_3$  1377.1,  $\nu_4$  2205.2,  $\nu_5$  2984.0,  $\nu_6$  3011.0. The moments of inertia,  $I_A$  7.760  $\times 10^{-40}$  g. cm.<sup>2</sup>;  $I_B$  8.298  $\times 10^{-40}$  g. cm.<sup>2</sup>;  $I_C$  1.003  $\times 10^{-39}$  g. cm.<sup>2</sup>;  $I_{AB}$  1.785  $\times 10^{-39}$  g. cm.<sup>2</sup>. From the 4 previously known frequencies of CH<sub>4</sub> and each of the frequencies of this mol., the values of the potential constants are computed. Three of the sets are consistent and agree well with the values found by Dennison and Johnston

(*C. A.* 20, 1297<sup>2</sup>), but the other 3 sets have neg. or imaginary values. The frequencies from which these are calculated are the ones which take part in a resonance interaction which was not included in the theory. From the observed spacings of the zero branch of the perpendicular bands, extreme ranges of the rotation-vibration interaction factors are calculated. An overtone was observed  $2\nu_2$  at 2221. Three frequencies of CH<sub>3</sub>D<sub>2</sub> were observed at 1010.0, 1090.5 and 1215.5  $\text{cm}^{-1}$ . G. M. P.

Infrared spectrum of heavy water. I. V. Barker and W. W. Sleator. *J. Chem. Physics* 3, 660-3 (1915).—Samples of water vapor containing 90 and 40% of D<sub>2</sub> were examined in the infrared, and the absorption bands  $\nu_2$  and  $\nu_3$  for D<sub>2</sub>O and  $\nu_2$  and  $\nu_3$  for H<sub>2</sub>O found at 1170, 2761, 1401 and 2720  $\text{cm}^{-1}$ , resp. Of the 6 fundamental frequencies for the 3 varieties of water, only  $\nu_{1,2,3}$  has not been observed. It is apparently completely masked by  $\nu_{1,2}$ . Calculated and experimental values of these frequencies are in good agreement. A fair degree of resolution is obtained in the bands  $\nu_2$ . The fine structure observed agrees approx. with that predicted, with Meck's molecular dimensions (*C. A.* 27, 3698<sup>2</sup>). The magnitude of the interactions is so great, however, that precise details of these constants must await a more complete study of the mechanism. G. M. P.

The light absorption of porphyrins. H. A. Stern and Hans Wendelein. *Z. Physik, Chem.* A174, 81 103 (1915); cf. *C. A.* 29, 1114<sup>2</sup>.—The absorption spectra of several porphyrins were studied in dioxane. The effect of various substituted groups on the position of the maximal bands is discussed. The absorption spectra of several porphyrins and their chloro derivatives were measured; these compounds being special cases of isomeric compounds of the "dehydrochlorophyll" type show similar spectra. Various substitutions in dihydrochlorophyll are discussed and some uroporphyrins compared. R. H. Baechler

Constitution of water in solutions of nonelectrolytes. I. Acetone. C. S. Silva Rao. *Phil. Mag.* 20, 687-94 (1915); cf. *C. A.* 29, 7190<sup>2</sup>.—The effect of acetone on the structure of the hydrogen bond of water was studied. With increasing concentration the Raman band gets sharper but the frequency of maximum intensity does not shift. The lower frequency branch of the band shifts to higher frequencies and the graph of intensity vs. frequency for this branch changes from convex to concave. J. R. Robinson

Intensity and polarization of the Raman scattering in liquids. A. Veeralindra Rao. *Z. Physik* 97, 164-7 (1915).—By employing a narrow slit intensity measurements are made for the Q branches of the various Raman vibration lines of CCl<sub>4</sub>, CCl<sub>2</sub>, CHCl<sub>3</sub>, A-Cl<sub>3</sub>. The total intensity of the depolarized Raman lines, which are quite noticeable in all these liquids, introduces a contribution to the depolarization of the total scattered radiation. This is especially noticeable in cases like CCl<sub>4</sub> which are characterized by small depolarization factors. S. T.

Raman spectra of 1-methyl-1-ethyl-1-propanol,  $\alpha$ ,  $\alpha$ -dimethylbenzyl alcohol and the corresponding ethylene hydrocarbons. Jean Savat. *Compt. rend.* 201, 847-8 (1915).—The Raman spectra of MeEtCOH (I), MeC(CH<sub>3</sub>)<sub>2</sub>COH (II), MeC(CH<sub>3</sub>)<sub>2</sub>CHMe (III), and PhC(CH<sub>3</sub>)<sub>2</sub>COH (IV) have been determined and compared with that of MeEtCOH (V) (cf. Nevel, *et al.*, *C. A.* 28, 6613<sup>2</sup>). The results make previous allocations of the lines to certain groups of atoms doubtful, but examination of other compounds is necessary before definite corrections are proposed, save that the line near 1645, characteristic of the double bond, varies with the nature of the substituting groups (cf. Prevost, *C. A.* 28, 3720<sup>2</sup>). C. A. Silvestro

The absorption spectrum of carbon "hydro-sulfide" and its constitution. R. Padmanabhaiah. *Current Sci.* 4, 95-6 (1915).—Although the formation of crystalline compounds between  $\alpha,\beta$ -unsaturated ketones and H<sub>2</sub>S has been known since 1870, the constitution of these "hydro-sulfides" is still in controversy. In the case of chrysone "hydro-sulfide", which consists of 1 mol. of H<sub>2</sub>S and 2 mols. of the ketone, 3 different structures have been proposed. A quantitative measurement of the ultraviolet absorp.

tion spectrum of the compd. reveals a band at 2900 A. which furnishes direct evidence for the existence of free CO groups in the mol., thus favoring one of the proposed structures for the constitution of carvone "hydrosulfide" as the most probable. W. F. Meggers

Investigation of the polarization of Raman scattering. Fritz Heidebrech. *Z. Physik* 97, 277-99(1935).—The polarization of the Raman spectra of dichloroethane, hexachloroethane, cyclohexane and  $\text{Ac}_2\text{O}$  is examd., by the method of circular polarization. The observed values can be well correlated with the mol. structures. The relation between the linear and circular polarization of the scattered radiation from benzene,  $\text{CCl}_4$ , EtOH, toluene and cyclohexane is quantitatively examd. The normal circularly polarized Raman lines agree better with the results of other workers (who use unpolarized primary radiation), than the inverse circularly polarized lines. The broadening of the Rayleigh lines, usually attributed to unresolved rotation Raman structure, is examd. Pure rotation produces inverse circular polarization, the degree of broadening increasing with the anisotropy. Other types of broadening produce depolarization. Only toluene, benzene and  $\text{AcOH}$  give inverse circular polarization, while EtOH, water and cyclohexane show normal circular polarization, i. e., broadening due to other causes than Raman rotation exists in these mol. Rotation branches in  $\text{NH}_3$  are observed. Normal circular polarization is observed in  $\text{CS}_2$ . S. Tolansky

Raman spectrum and constitution of fuming sulfonic acid. N. Gopala Pai. *Phil. Mag.* 20, 616-23(1935).—In 21% fuming  $\text{H}_2\text{SO}_4$  all the lines characteristic of  $\text{H}_2\text{SO}_4$  are present, although weakened, no  $\text{SO}_3$  lines are found and addnl. lines are found which are attributed to the  $\text{H}_2\text{S}_2\text{O}_7$  mol. At greater concns. the lines of  $\text{SO}_3$  and also those of  $\text{S}_2\text{O}_8$  appear and become more intense with increasing concn. The more intense  $\text{H}_2\text{SO}_4$  lines persist even in 80% solns. E. J. Rosenbaum

Relation between ultraviolet absorption and Raman spectra of pyridine. Victor Henri and Pierre Ancenot. *Compt. rend.* 201, 895-6(1935); cf. C. A. 29, 3235<sup>7</sup>.—The absorption spectrum of  $\text{C}_5\text{H}_5\text{N}$  has been examd. at various temps. and pressures up to 231° and 910 mm. There are 5 fundamental frequencies (600, 857, 903, 1031 and 1159  $\text{cm}^{-1}$ ) corresponding with those deduced from the Raman spectrum, and practically identical with the fundamental frequencies of  $\text{C}_5\text{H}_6$  (because of the nearness of the at. wts. of N and CH). The 5 series forming the  $\text{C}_5\text{H}_5\text{N}$  spectrum are given. Light of  $\lambda$  greater than 2750 A. has no photochem. action on  $\text{C}_5\text{H}_5\text{N}$  vapor. C. A. Silberrad

The Raman effect of organic molecules. (The vibration spectrum of acrylonitrile and ethylene oxide.) B. Timm and R. Mecke. *Z. Physik* 97, 221-4(1935); cf. C. A. 29, 4265<sup>7</sup>.—The following Raman lines are found:  $\text{C}_3\text{H}_3\text{CN}$ , 241(2), 384(1), 1289(1), 1612(2), 2338(5), 3076(3), 3115(3). For  $\text{C}_2\text{H}_2\text{O}$ , 807(0), 873(1), 1128(1), 1268(5), 2924(1), 2970(1), 3008(4), 3047(1). The valency bonds and vibration frequencies are discussed. S. Tolansky

Reaction rate of acetic anhydride and water. E. K. Plyler and E. S. Barr. *J. Chem. Physics* 3, 679-82(1935).—The infrared absorptions of  $\text{AcOH}$  and  $\text{Ac}_2\text{O}$  were measured in the range 1-6.5  $\mu$ . Several bands below 5  $\mu$  which were similar for the 2 compds. were attributed to CH bonds. An intense band was found in  $\text{Ac}_2\text{O}$  at 5.45  $\mu$  and in  $\text{AcOH}$  at 5.75  $\mu$ . By measuring the intensities of these bands, the reaction velocity of  $\text{Ac}_2\text{O}$  with  $\text{H}_2\text{O}$  was detd. The reaction was approx. unimol. for any concn. The reaction const. decreased as time increased its value differed at different concns. The reaction const. was detd. at 25°, 50° and 75°. Values of the reaction const. and of other const. are tabulated. G. M. P.

The Raman spectrum of arsenic trichloride. Don M. Yost and Thomas F. Anderson. *J. Chem. Physics* 3, 754(1935).—The modes of vibration of  $\text{AsCl}_3$  given by Brodskil and Sack (C. A. 29, 6504<sup>7</sup>) are reassigned, on the basis of polarization expts. (Cabannes and Rousset, C. A. 26, 2117). Conclusion. Central forces alone are in-

adequate for the treatment of the  $\text{AsCl}_3$  type mol.

G. M. P.

Raman spectra of oxalic acid. James H. Hibben. *J. Chem. Physics* 3, 675-9(1935).—Raman spectra of anhyd. oxalic acid, its aq. and alc. solns., and of  $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$  are given, and are compared with the results of Rao (C. A. 29, 5350<sup>4</sup>). Spectra of the aq. soln. and of  $(\text{COOH})_2$  show that the 2  $\text{COOH}$  groups do not behave identically: in alc. soln. both groups behave alike. The magnitude of the force consts. in the CO groups is consistent with the strength of this acid. CO oscillations are either very weak or absent in  $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$ . Chichibabin's tautomeric form (C. A. 23, 1114) is excluded. G. M. P.

Use of Raman effect to distinguish the cis-trans isomerism of the methylcyclohexanols. Jean Tabuteau. *Compt. rend.* 201, 897-8(1935).—The Raman spectra of cis- and trans-o-, m- and p-methylcyclohexanols and the corresponding acetates have been compared. The differences between the spectra of the cis- and trans-isomers are most marked for frequencies between 800 and 900  $\text{cm}^{-1}$ , also in the spectra of the alc. and corresponding acetate the percentage of identical lines is greater for the cis- than for the trans-isomer, the difference decreasing in the order o, m, p. C. A. Silberrad

Polarized fluorescence of organic compounds. S. M. Mitra. *Z. Physik* 97, 138-53(1935); cf. C. A. 29, 2087<sup>4</sup>.—Measurements are made of the polarization of the fluorescence radiation from succinylfluorescein and succinilousin in soln. in glycerol, castor oil, sugar soln., collodion-ether, glycerol-water and gelatin. The dependence of the polarization on wave length is measured. The effects of temp. viscosity and concn. are examd. The exptl. results are discussed. S. Tolansky

Inorganic photosyntheses. Gennaro Calcagni. *Gen. chim. ital.* 65, 558-66(1935).—A study was made of the possibility of obtaining  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{SeO}_4$ ,  $\text{H}_2\text{PO}_4$ ,  $\text{H}_2\text{AsO}_4$ ,  $\text{H}_2\text{SbO}_4$ ,  $\text{H}_2\text{BO}_3$  and  $\text{SiH}(\text{OH})_3$  by the action of light on suspensions of S, Se, yellow P, As, Sb, amorphous and cryst. B. and amorphous and cryst. Si in water. The subject has been studied very little. Each purified powder element was kept in highly purified water at atm. pressure for 7-11 months, after which the filtered solns. were analyzed. With S,  $\text{H}_2\text{SO}_4$  but no  $\text{SO}_3$  was formed, whereas S in boiling water for many hrs., even with a current of O gave neither  $\text{H}_2\text{SO}_4$  nor  $\text{SO}_3$ . Accordingly the formation of  $\text{H}_2\text{SO}_4$  from S and water is a strictly photochem. reaction. The residual S was in the same stable form. With Se only  $\text{H}_2\text{SeO}_4$  was in soln., while the residue was in part red Se. Therefore Se probably forms  $\text{H}_2\text{SeO}_4$ , and the latter decomposes into red Se, until an equal is reached. With P, there were formed  $\text{H}_2\text{PO}_4$ , less  $\text{H}_3\text{PO}_4$ , and a doubtful trace of  $\text{H}_4\text{P}_2\text{O}_7$ . The residue was red P. Probably therefore the chief reaction is the transformation of yellow to red P. Red P then reacts thus:  $\text{P}_4 + 6\text{H}_2\text{O} \rightarrow 3\text{H}_2\text{PO}_4 + \text{PH}_3$ . These products in turn react thus:  $2\text{H}_2\text{PO}_4 + \text{H}_2\text{PO}_4 + \text{PH}_3$ ,  $\text{PH}_3 + \text{O} \rightarrow \text{HPO}_3 + \text{H}_2$  and  $4\text{H}_2\text{PO}_4 + 3\text{H}_2\text{PO}_4 + \text{PH}_3$ , while  $\text{HPO}_3$  combines with water and is oxidized to  $\text{H}_2\text{PO}_4$ . Perhaps, however,  $\text{H}_2\text{PO}_4$  is first formed, and then gives  $\text{H}_2\text{PO}_4$  and  $\text{H}_3\text{PO}_4$ . With As, the soln. contained  $\text{H}_2\text{AsO}_4$  and a trace of  $\text{H}_3\text{AsO}_4$ . Likewise with Sb,  $\text{H}_2\text{SbO}_4$  and a trace of  $\text{H}_3\text{SbO}_4$  were formed. It is probable therefore that arsenolite, senarmonite, arsenites, arsenates, antimonites and antimonates in nature are formed by oxidation of As and Sb. With B. the soln. contained only  $\text{H}_2\text{BO}_3$ , less being formed with cryst. B. than with amorphous B. With Si, there was recovered from the soln. the  $\text{SiH}(\text{OH})_3$  of Mendeleev (cf. The Principles of Chemistry, 1903). Less was obtained with cryst. Si than with amorphous Si. C. C. Davis

The action of radon on polymethylcyclopentene and cyclopentene. G. B. Hessig. *J. Phys. Chem.* 39, 1067-73(1935); cf. C. A. 26, 4535. —Pure cyclopentene (I) was prepd. from dicyclopentene by depolymerization at 185-215° and from cyclopentene (II) by hydrogenation in alc. soln. in an Adams app. with a Pt oxide catalyst and was distd., washed with  $\text{H}_2\text{O}$ , concd.  $\text{H}_2\text{SO}_4$ , dil. NaOH (in the second case also alk.  $\text{KMnO}_4$ ), dried over

CaCl<sub>2</sub>, then bright Na, distd, under its own vapor pressure at 0°, and condensed at -80°,  $b_{\text{Na}} = 48.8^\circ$ , vapor pressure (cf. C. A. 29, 1771) 107.3 mm. at 0°, 4.4 mm. at -52°. II was prep'd. by heating 110 g. of cyclopentanone (from cyclopentanone reduced at 150° by H<sub>2</sub> at 1600 lb. pressure in the presence of a Raney Ni catalyst, yield 91%) with 600 g. hydrated oxalic acid at 110° for 2 hrs. It was dried over CaCl<sub>2</sub>, distd. from Na, and from a trap at 0° and condensed at -182°;  $b_{\text{Na}} = 43.5-43.8^\circ$ , vapor pressure 131 mm. at 0°, 1.0 mm. at -77.5°. On polymerization by  $\alpha$ -rays from Rn at 25° each formed a liquid. The  $-M/N$  value of I is 1.7; that of II, 4.5. These values are in agreement with the heats of formation for I, 18.9 kg.-cal., and for II, -18 kg.-cal. and with  $\Delta(H_2 + CH_4)_{100}$

-AHC

Janet E Austin

Photochemistry of the future. G. Sollazzo. *Boll chim farm* 74, 773-9(1935).—A discussion. H. L. G.

The light sensitivity of several reactions. Chr. Winther. *Z. physik. Chem* A174, 41-8(1935).—The "photochem" oxidation of citric acid, tartaric acid, lactic acid and oxalic acid by chromic acid and of KCl by K persulfate was studied first in the dark, then in blue light, where strong absorption takes place, and finally in green light, where absorption is slight. For the total reactions the quantum sensitivity is less than 0.05 so that it must be considered insensitive to the light used.

R. H. Baechler

Diatomic reactions and primary chemiluminescence. R. A. Ogg, Jr. and M. Polányi. *Trans. Faraday Soc.* 31, 1375-84(1935).—A theory is developed for reactions in which atoms are converted into ions. The potential energy surface for such an ionogenic reaction is described for adiabatic and diabatic transitions. In a series of similar reactions the activation energy decreases as the heat of reaction increases. It is predicted that the reaction velocity of  $M + ClR \rightarrow MCl + R$  should increase as M is varied in the order Na, K, Rb and Cs. The luminescence produced when Na vapor reacts with  $SnCl_4$  is attributed to the diabatic process:  $Na + SnCl_4 \rightarrow NaCl + SnCl_3$ , followed by  $2SnCl_3 \rightarrow SnCl_2 + SnCl_4 + h\nu$ . This process is considered to be the inverse of predissociation. E. J. Rosenbaum

Mechanism of reactions between alkali atoms and halogen hydrides. A. G. Evans and M. G. Evans. *Trans. Faraday Soc.* 31, 1400-10(1935).—Theoretical. The method of Ogg and Polányi (preceding abstr.) is applied to these reactions, which are considered as transitions from a homopolar to an ionic energy surface. Activation energies are calculated. The increased probability of the collided state is significant for Na and HCl, and K and HBr or HI, which reactions are the most exothermic of those considered. This increased probability accounts for the abnormal collision diams. found previously for these reactions. The relative rates of reactions of H and D compds. are discussed. The probability of a nonadiabatic transi-

tion with a D atom is 1.4 times as great as for a H atom. E. J. Rosenbaum

Photochemistry of methylene blue. Knut M. Brandt. *Arkiv Kemi, Mineral. Geol.* 12B, No. 7, 5 pp.(1935).—The photosensitized reduction of methylene blue was studied in the presence of EtOH, Fe salts and a Na<sub>2</sub>HPO<sub>4</sub> buffer to keep the pH at 8.6. A considerable dark reaction occurred only when both EtOH and Fe<sup>++</sup> ions were present. In all cases photosensitization was observed. The greatest effect was observed in the solns. which contained both EtOH and Fe<sup>++</sup> ions. The reduced solns. were reoxidized in the dark, as was observed previously with acidic solns. An equil. is established which is displaced toward the leuco-methylene blue side. E. J. R.

Photodissociation of single crystals of some nitrates in polarized light. L. K. Narayanswamy. *Trans. Faraday Soc.* 31, 1411-12(1935); cf. C. A. 29, 3906f.—The degree of disson. of nitrates decreases in the order K, Na, Sr, Ba, Cd, Al, Pb and NH<sub>4</sub>. The long-wave limit of the active radiation is 250 mμ. Nitrate was found only in a thin layer at the surface, since at greater depths the O cannot escape and recombination results. Polarized radiation the vibrations of which are along the normal to the plane of the nitrate ions is much less effective in dissong. the ions than radiation with vibrations in the plane of the ions. E. J. Rosenbaum

Purification of electrode carbons for spectrum analysis (Zürcher, Treadwell) 4. Detn. of U, and the U content of sea water (Hernegger, Karlik) 7. Absorption of the most important cinchona alkaloids in the ultraviolet (Fuchs, Kampitsch) 17. Content of heavy water in the water of crystals of minerals (Riesefeld, Tobian) 8. Structure of heterosides from their absorption in the ultraviolet (Ramart-Lucas, Rabat) 10. Photolytic and thermal decomp. products of azomethane (Heidt, Forbes) 2. Periodic classification of the rare earths (Yagoda) 2. Chem. and phys. characteristics of water of crystals. (Paserini) 2. The change, with time, of the vapor pressure of mixts. of the H isotope at 20.38° abs. (Steiner) 2. Chem. and phys. characteristics of the water contained in colloidal substances (Paserini) 2.

Fluorescent screens. I. G. Farbenindustrie A.-G. Brit. 432,432, July 26, 1935. An intensifying screen for x-ray photography comprises a layer of a fluorescent substance of low mol. wt., e. g., ZnS or a sulfide of an alk. earth, and a layer of a fluorescent substance of high mol. wt., e. g., CaWO<sub>4</sub>, the former layer being arranged nearer the x-ray tube. The photographic film or plate is arranged between the layers of the screen.

Fluorescent screens, particularly for Braun tubes. Radioaktiengesellschaft D. S. Loewe and Kurt Schlesinger. Brit. 431,404, July 8, 1935. See Fr. 768,767 (C. A. 28, 7165f).

## 4—ELECTROCHEMISTRY

COLIN G. FINK

*Elec. Eng.* 54, 1195-9(1935).—See C. A. 29, 6813f.

The recovery of potassium chloride by electrolysis with silver electrodes. N. A. Petrov and M. V. Lazareva. *Trans. Inst. Pure Chem. Reagents* (U. S. S. R.) No. 14, 76-80(1935).—Chloride ion can be completely removed from KCl solns. by deposition on Ag electrodes. The best conditions are a concn. of 1.5 mol./l. and an anode c. d. of 0.02-0.03 milliamp./sq. cm. This method is not suitable for the purification of KOH solns. Lewis W. Batz

Hydro power and metallurgical development in Norway. Carl W. Volz. *Mining and Met.* 16, 453-6(1935).—Electric furnace pig Fe of high purity is replacing charcoal Fe. Special steels are being produced: Cr, Cr-V, W, high-speed tool steel and stainless steels. Sponge Fe is being made by a process primarily dependent on elec. power. A new process for electrolytic refining of Ni from a sulfide

The engineering development of electrochemistry and electrometallurgy. Paul Bunet. *Elec. Eng.* 54, 1320-31(1935).—Some notes on the engineering development of electrochem. and electrometallurgical industries, with particular reference to those in France. The 3-phase and single-phase production of CaCl<sub>2</sub> are compared, and the production of Al, AlN and other electrochem. and electrometallurgical products is discussed. W. H. Boynton

Induction heating at low temperatures. Edgar L. Bailey. *Elec. Eng.* 54, 1210-12(1935).—Inductively heating Fe parts for drying coatings discloses interesting features which may be applied to other low-temp. processes. The elec. design of the oven and currents for induction heating at low temps. are considered and indicate low maintenance and operating costs, cleanliness and reduced processing time. W. H. Boynton

Electric furnaces with carbon radiator. Henri George.

mat contg 45% Ni and 37% Cu has resulted in import of mat from Canada. S and Cu are being produced from chalcopryite. Electrolytic Zn and Cd are being produced from imported ores. Na output is increasing. Norway has made important contributions to Al metallurgy and is the third largest producer. The manuf. of carbide and ferroalloys in the elec. furnace is important. A. H. E.

**Cathode film control and metal deposition.** Colin G. Fink. *J. Chem. Education* 12, 520-4 (1935).—On the basis of explt data a cathode film is found to be necessary for successful metal deposition. Furthermore, this film must be held between fairly definite limits as to thickness and H-ion concn. The film can be maintained within the desired limits by the rotating-cathode method, a definite cathode speed and cathode c. d. corresponding to the desired film thickness and  $p_H$ . The range of film thickness and its  $p_H$  are different but specific for each metal. Curves are given showing the relationship between the film thickness and  $p_H$  as affected by various factors such as temp., speed of cathode, c. d., etc. Some problems in metal deposition that have been solved by the rotating cathode are discussed. By varying the cathode speed, cathode-film  $p_H$  and c. d. the usual order of deposition of the metals can be shifted to meet the requirements of the particular case. The method is recommended for the deposition of those metals and alloys which have not yet been satisfactorily produced from aq. solns. W. G. P.

**Theoretical, experimental, and practical studies of the question of electrolysis in subterranean canalizations.** Gibrat. *Bull. soc. belge ing. ind.* 1935, 577-612.—The math. derivation and practical execution of a differential potentiometer arrangement for predicting the likeness and intensity of electrolysis are given. The elec. methods of protection are discussed and their application in some specific cases is described. Leopold Fessel.

**Power company service to arc furnaces.** L. W. Clark. *Elect. Eng.* 54, 1173-8 (1935).—A general procedure is outlined in planning elec. service for unusual loads such as the elec. arc furnace. W. H. Boynton.

**Black nickel.** Jos. L. Downes. *Monthly Rev. Am. Electroplaters' Soc.*, Aug., 1935, *Platers' Guide* 31, 12-16 (Nov., 1935). W. H. Boynton.

**The theory of fused salt electrolysis.** Energy efficiency of the electrolysis of carnallite. I. G. Scherbakov and A. A. Scherbakov. *J. Phys. Chem.* (U. S. S. R.) 6, 649-63 (1935).—Electrolysis of both natural and artificial carnallite was carried out in an E cell with a graphite anode with a total current of from 100 to 400 amps and the temp. gradually rising from 520° to 700°, voltage dropping from 7.2 to 3.2. On the assumption of losses due to secondary cathode processes the yield as a function of the c. d. is given by  $\eta = 1 - kI^{-1/2}$  and the depolarization voltage by  $E_d = kE_d I^{-1/2}$ . Both the theory and the explt data show the possibility of a max. yield with respect to energy input, for the electrodes used, of 78.7% by theory and 72.8% by explt. The calc'd. and explt. values of the depolarization voltage are 0.117 and 0.130 v. for an av. c. d. of 53 amp./sq. cm. F. H. Rathmann.

**Limiting and interrupting the current by a mercury cathode.** A. I. Nikiforov and T. M. Sviridov. *Z. Physik* 97, 398-401 (1935).—A Hg cathode of small surface area, enclosed in a W metal cell, is exam'd. Only a limited amt. of current will pass through the cell. An explanation is offered. S. Tolansky.

**Industrial applications of electrolysis.** H. J. T. Ellingham. *Chemistry & Industry* 1935, 895-902. E. H.

**Inversion of a sucrose solution in contact with a wall traversed by an electric current.** Philippe Fabre. *Compt. rend. soc. biol.* 120, 179-81 (1935).—When a weak d. c. (60 ma. at 480 v.) was passed through a 20% soln. of sucrose contg. 1% of KCl partial inversion of the sucrose occurred if the cell was divided into 2 compartments by a porous cup. If the cup was removed no inversion took place. The effect is ascribed to a polarization of the surfaces of the porous cup by the current passing through the walls. The electrodes were not placed in the sucrose soln. but were in sep. vessels connected to the 2 compartments

of the reaction cell by glass siphons filled with KCl soln. L. E. Gilson.

**Electrolytic preparation of heavy water.** The relation between electric current density and the isotopic separation coefficient. Anon. *J. Electrochem. Soc. Japan* 3, 127 (1935).—The procedure and actual data of prep. 24 cc. of heavy water of the density of 88% D<sub>2</sub>O and 3.0 cc. of 95% D<sub>2</sub>O are reported. Results are recorded of explts on the relation between elec. c. d. and the isotopic sep. coeff. It is concluded that there is no effect of the c. d. upon the sep. coeff. under the following explt. conditions: process of electrolysis: initial vol. of 20% NaOH soln. is 0.08% D<sub>2</sub>O water is kept const. by continuously supplying water of 0.08% D<sub>2</sub>O free from NaOH. Electrodes are Ni temp. is 18°, vol. contraction by electrolysis 1/5, range of c. d. is 0.05-1.0 amp. per sq. cm. Thus was obtained 0.21% D<sub>2</sub>O repeatedly within the range of the c. d. mentioned. C. G. I.

**Electrolytic preparation of hydrogen peroxide.** R. Sidorsky. *J. S. African Chem. Inst.* 18, 44-61 (1935).—The electrolytic processes involve a vacuum distn. which cannot be controlled to give good yield and satisfactory concn. simultaneously. S. records detailed studies of (a) the reactions occurring during the vacuum distn. of the H<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (and its K and NH<sub>4</sub> salts); (b) the detn. of the possibilities of the direct conversion of (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> into H<sub>2</sub>O<sub>2</sub>. B. E. Anderson.

**Effect of total voltage on breakdown in vacuum.** H. W. Anderson. *Elect. Eng.* 54, 1315-20 (1935).—A review of results of a series of high-voltage breakdown tests between metal electrodes in high vacuum. The inverse relation between cathode gradient at breakdown and gap length leads to the conclusion that the current that flows between electrodes as the conditions for high-voltage breakdown are approached must involve neg. ions from the anode. The deposition of anode material upon the cathode, which would occur in the event of such neg. ion emission, was found to take place. Interelectrode current at const. cathode gradient varied with the voltage, proving the above hypothesis. W. H. Boynton.

**Sparking potential of hydrogen in high frequency discharge.** Raymond Zouckermann. *Compt. rend.* 201, 649-51 (1935); cf. C. A. 27, 4168.—Under similar conditions curves of sparking potential (V) vs. pressure (p) were det'd. for  $\lambda$  101 (I) and 12 m. (II) in pure H<sub>2</sub> (s) and in H contg. traces of Hg vapor (b). Ia and Ib show a min. for  $V = 342$  v.,  $p = 0.2$  mm., and are identical for pressures above the min., but for lower pressures Ib lies above Ia, while the color of the discharge changes from rose to blue when Hg is admitted. In Ia the min. is for  $V = 74$  v.,  $p = 0.002$  mm., as Hg vapor is admitted the min. moves toward lower potential and lower pressure.

**Structure and properties of nickel deposited at high current densities.** Wm. Blum and Charles Kasper. *Trans. Faraday Soc.* 31, 1203-11 (1935); cf. C. A. 29, 7821.—Deposits were made from NiSO<sub>4</sub> and NiCl<sub>2</sub> bathes as such and said with boric acid at c. ds. from 22 to 45 amp./sq. dm. at the boiling temp. of the solns (about 102°) and at a  $p_H$  of 1.0-2.0. Deposits were made in 5-l. glass jars with 5-cm. sep'n. of electrodes. For micrographic etching glacial acetic acid plus < 1% HNO<sub>3</sub> was employed. Deposits from NiCl<sub>2</sub> solns were fine-grained smooth, strong (approx. 7000 kg./sq. cm. tensile), hard (about 200 Vickers) and brittle, and the corresponding NiSO<sub>4</sub> deposits were rough, coarse-grained, soft (approx. 120 Vickers) and ductile (approx. 4300 kg./sq. cm. tensile). Boric acid slightly softens the NiSO<sub>4</sub> deposit. Variation of  $p_H$  from 1.0 to 2.0 makes no definite change in properties. Cathode efficiencies are always higher for chloride (approx. 85%) than for sulfate (approx. 60%) deposits. The need for a great deal more work over a wide range of conditions is remarked as well as need for more discriminating methods of examn. of the deposits. H. A. Smith.

**The thermal theory of cathode sputtering.** II. The elementary process. N. D. Morgulis. *J. Exptl. Theoret. Phys.* (U. S. S. R.) 5, 588-94 (1935), cf. C. A. 29,

7780<sup>1</sup>.—The theory of Hippel (*C. A.* 21, 2425) is further developed. I. H. Rathmann

Changes of temperature with time in arc discharges with alternating current. D. Th. J. ter Horst, H. Brinkman and L. S. Orstein. *Physica* 2, 652-68 (1935), cf. I. H., Diss. Utrecht, 1934.—An a.-c. arc, C electrodes in air, was studied at different frequencies (100 to 500), 2-4 amps, 60-85 v., 5-mm arc, 5-mm. electrodes. The spectrograph slit was perpendicular to the electrodes, halfway. A stroboscopic interrupter was interposed between arc and spectrograph, giving 6° phase intervals. The temp. was detd. from violet CN bands. The temp. variations are given in diagrams, they decrease with increased frequency. From oscillograph diagrams the energy production per vol. unit was detd. and on the basis of this an energy balance made up for the discharge. The output items are heating of arc gas between electrodes, heat losses by conduction, radiation and convection. An equation is derived for the total heat cond. of the arc including mol and atom effects due to dissociation. From the calcul. is derived a phase lag between energy and temp. function in fair agreement with the exptl. data, it varies with the frequency, at 50 cycles the delay angle is 25°, at 250 cycles it is 45°. The thermal theory of the arc discharge (O. and B., *C. A.* 29, 35<sup>9</sup>) is again confirmed. B. J. C. van der Hoeven

Experimental study of the influence of the support or cathode on the structure of electrolytic deposits obtained in aqueous solution. Albert M. Portevin and Michel Cymboliste. *Trans. Faraday Soc.* 31, 1211-18 (1935).—The influence (1) of the rate of formation of crystal nuclei and (2) of the rate of growth of such nuclei is fundamental in detg. the structure of electrolytic deposits. The fineness, orientation and form of the crystals making up the deposit depend on (1) and (2). The fundamental concepts (1) and (2) depend upon the conditions exterior to the cathode and upon the surface and structure of the cathode. The geometry, chem. and cryst. condition of the cathode surface affect markedly the condition of the deposit. The influence of surface irregularities due to scouring, cataphoresis, cracks and holes is illustrated by photomicrographs. The sep. effects of the type of metal of the cathode, oxidation, selective soln., adsorbed colloids and gas bubbles are illustrated for Cr and Ni deposits. To obtain crust. continuity between cathode and deposit the Deily layer as well as any layer of foreign matter must be thoroughly removed. Such continuity is also influenced by (1). If (1) is large the continuity may be entirely lacking. Cryst. continuity of Zn and Cd deposits on cathodes of these metals, resp., is known. Deposits of Ni on Cu cathodes and of Cd on Sn and Pb are also known. H. A. Smith

The cathode bend discharge. H. Beck. *Z. Physik* 7, 370-81 (1935).—The current-voltage characteristic of the discharge is found. A condition for the formation of beads is that the cathode surface shall be electrically clean. The discharge may take place in Zn and Cd. The effect of a magnetic field is investigated. B. Swirles

"Somatoid" elements of structure in electrolytic metal deposits. V. Kohl-Schütter. *Trans. Faraday Soc.* 31, 1181-8 (1935).—Small but microscopically visible particles possessing a definite "organization" acquired in the process of formation are termed somatoids. They are characteristic aggregations of crystallites. The form and nature of somatoid formations do not depend on the compn. and crystallographic properties of the material involved. They are morphological individuals characteristic of definite states realized during their formation rather than definite substances. Colloids play the principal role in somatoid formation. Somatoids are products of the combined action of crystn. forces with surface forces. Unstable forms of some substances acquire a certain stability when existing as somatoids. Electrolytic deposits due to the topochem. nature of the reaction are often somatoid in nature. It is desired especially to call the attention of the electrochemists to the role played by somatoids in electropptn. of metals and to the possibility and value of examg. such deposits by morphological methods. An

extended discussion will appear elsewhere. 15 references. H. A. Smith

Effects of film formation on the structure of electro-deposited metallic coatings. E. Liebreich. *Trans. Faraday Soc.* 31, 1188-94 (1935).—Hydroxides are generally formed at even the lowest c. ds. at the surface of the cathode and profoundly affect the type of deposit obtained. The surfaces of the deposit often react chemically with the OH<sup>-</sup>. In the cathode potential c.-d. curves of Pt on Ag in a soln. of NiSO<sub>4</sub>, Pt shows a slight reaction just prior to the evolution of H<sub>2</sub>, while Ag shows a very marked inflection at this point. Metals showing slight reaction form a thin homogeneous film on the surface not reduced by H<sub>2</sub>. Metals exhibiting a pronounced inflection, and hence tendency to react, form films readily reduced by H<sub>2</sub>, thus presenting an active surface. The formation of films on such a metal depends on the soly. of the products of reaction at the surface. In plating Cr the overvoltage is not due to phys. factors so much as the reduction of the film of oxides formed at the cathode. The peculiarities of the behavior of Cr deposits on various metals from various types of solns. are critically discussed. H. A. Smith

Cathodic film in the electrolytic reduction of aqueous chromic acid solutions. Frick Müller. *Trans. Faraday Soc.* 31, 1194-2302 (1935).—It is remarked that the reason for the failure of Cr to deposit on Pt cathodes in very pure H<sub>2</sub>CrO<sub>4</sub> solns. is due to the formation of a film of Cr chromates on the Pt. Deposition of Cr on C cathodes does proceed, however. C.-d. electrode potential curves for C cathodes rise to a max. at about +0.4 v., thereafter decreasing to a low value at about 0 v., then rising somewhat again. The mechanism of the reaction at the C cathode is worked out in detail based on the supposition that, compared to a Pt surface, a C surface is quite rough and the reaction varies from point to point on the cathode. The mechanism of the action at the cathode in pure H<sub>2</sub>CrO<sub>4</sub> and in H<sub>2</sub>CrO<sub>4</sub> with addnl. sulfate and other ions is presented in considerable detail. The fluctuating type of current obtained in the presence of sulfate is illustrated with typical oscillograms. H. A. Smith

Determination of the structure of electrodeposits by metallurgical methods. D. J. Macnaughtan and A. W. Hotherhall. *Trans. Faraday Soc.* 31, 1168-77 (1935).—I or Ni deposits the Brinell hardness varies inversely with crystal size. After a thorough consideration of methods for making hardness measurements the Brinell impression is considered to be the most generally useful. A deposit thickness of 0.01 in. is considered sufficient to eliminate the influence of base metal on hardness readings of 100-400 Brinell with a 1-mm. ball and a 10-kg. load. No correlation is possible between cathode potential and hardness or microstructure. Co-deposited H does not affect the nature of the Ni deposit. Highest hardness values are assocd. with high *pH*. Alkali metal ions give a harder deposit than chloride ions. Thus, colloidal Ni compds. are considered as important in producing fine grain size and hard deposits. Annealing treatment (4 hrs. at 1000° in *vacuo*) showed the presence of foreign matter in the hard deposits that was lacking in the soft ones. Low-temp. treatments (200°) indicate that the soft metal deposited at low cathode efficiencies is highly stressed and contains considerable amts. of dissolved H while hard deposits at high efficiencies are little stressed and contain little H. H. A. Smith

Chemical and physical properties of electrolytically deposited metals in relation to their structure. M. Schlöter. *Trans. Faraday Soc.* 31, 1177-81 (1935).—I electrolytic metals are never absolutely pure. One of the most important impurities is H. H in Ni deposits is both mechanically and chemically held. Mechanically bound H is liberated on heating to about 400° while chemically bound gas is only liberated above 600°. At a given *pH* the H content of the metal varies inversely with c. d. and at a given c. d. the H content varies directly with the acidity. High overvoltages (corresponding to large anions) are assocd. with high H absorption by the metal. It is found that the lattice consists of Cu and Ag deposited

from solns contg  $I^-$  are larger than for deposition in solns free from  $I^-$ . Thus anions can take part in the deposition and even enter the crystal lattices of the metal. Such Ag deposits will not tarnish in the presence of  $H_2S$ , and they vary in color up to a golden yellow depending on the quantity of  $I^-$  in the deposit. A Cu deposit contg  $I^-$  turns blue in sunlight. "Iodized" Ag deposits seem to have a body-centered tetragonal lattice. It is believed that other anions, halogens, oxides, hydroxides and sulfides may enter the lattice of electrodeposited metals.

H. A. Smith

Theoretical valuation of electrode potentials. Shoji Makubishi. *Z. Elektrochem.* 41, 697-712 (1935).—See C. A. 29, 68451, 78233. G. G.

A new arrangement for measuring polarization voltage. B. Gross. *Physik Z.* 36, 648-9 (1935).—An a.c. rectified by a vacuum tube (half-wave rectification), is used, and the measuring instrument is connected in parallel to the electrolytic cell. The tube is connected in series, with its cathode connected to the cathode of the cell, and its grid is regulated by the polarization current itself, through a rheostat. The current used for heating the tube should be kept as low as possible. In the primary circuit, it can be completely suppressed by using a 3-electrode tube, the grid of which gives a neg. current of a few v. Wiring diagrams are given. E. R. Rushton

Improvement in the quality of tin plate by superimposed electrodeposition of tin. A. W. Hotherhall and W. N. Bradshaw. *J. Soc. Chem. Ind.* 54, 320-6T (1935); cf. C. A. 28, 53441.—The Macraquhart process of electrodeposition of tin coating of Sn on tin plate has been investigated to det. the behavior of diff. acid and alk. plating solns., the effect of preparatory cleaning treatments, c. d. and thickness of deposits, polishing after deposition and the effect of bending the tin plate before and after plating. Optimum results were obtained with alk. solns., low c. d. and cleaning in boiling Na disulfate soln. The polishing process did not increase porosity.

M. McMahon

Inhibitors—safe and dangerous. Ulick R. Evans. *Trans. Electrochem. Soc.* 69, 15 pp. (preprint) (1935).—An attempt to inhibit the anodic reaction of a corrosion-change controlled by the cathodic reaction will usually diminish the corroded area more quickly than it diminishes the total destruction of metal, and thus increase the intensity of corrosion if the addn. has been insufficient to stop attack altogether, such methods of inhibition are dangerous. This intensification of attack will not occur where the corrosion is under anodic control, or where the inhibitor is one which smoothes the cathodic reaction. The principles are applied in discussing the addn. of alkali to hard, soft and saline waters, and also the treatment of brine with chromate. The classification of protective processes into safe and dangerous groups can be extended to protection by oxide films, paints and metallic coats.

C. G. F.

The anodic behavior of copper in aqueous solutions of orthophosphoric acid. P. A. Jacquet. *Trans. Electrochem. Soc.* 69, 22 pp. (preprint) (1935).—A Cu surface anodically etched in aq. solns. of orthophosphoric acid becomes as bright as though it had been polished, if voltage and c. d. are kept within definite limits. If properly controlled, anodic etching will reveal the cryst. structure of the metal. The phenomenon seems to be based on the passivation of the anode and is a function of concn., polarization. The anodic-etch method has been applied to metal sections intended for metallographic examn.; excellent results were obtained. As compared with the usual acid-etching method, the anodic has the advantage of being much more rapid and less expensive. C. G. F.

The electrolytic preparation of anthranic acid. J. W. Shipley and J. M. Calhoun. *Can. J. Research* 13B, 123-32 (1935).—The electrolytic reduction of o-nitrobenzoic acid in alic.  $H_2SO_4$  at a Pb cathode was investigated. The effect of various exptl. conditions was studied and a max. yield of 92% of amine obtained under favorable conditions. No evidence of the reduction of the carboxyl group was observed. J. W. Shipley

Purification of electrode carbons for spectrum analysis. Th. Zürcher and W. D. Treadwell. *Helv. Chim. Acta* 18, 1181-9 (1935); cf. Heyne, C. A. 26, 6074.—The working portions of small electrode carbons are freed from impurities which interfere in emission spectra by passing a 12-amp. arc between them for 2-3 min. in an atm. of  $Cl_2$  together with 5%  $Cl_2$ . No  $Cl_2$  is necessary in most cases, but the presence of B, Cu and Mg in the carbons would require it.  $HCl$  is not effective. Tables are given showing the strength of lines of 15 elements in carbons purified in various ways. W. F. Bruce

Gas reactions in the silent discharge at atmospheric pressure. I. A new screened point-discharge with change from a high-current to a low-current discharge form. P. A. Thuessen and H. Bartel. *Z. tech. Physik* 16, 285-93 (1935).—In order to get this type of discharge a screen is essential. The low-current form resembles a glow-discharge at low pressure. The material of the point, or of the plate cathode, has no influence on the discharge. At low gas pressure the current max. is higher and the transition range is sharper. At increased temp. the current max. is higher but the transition range is broader. Pure gases do not show the effect. A mixt. of N and 15% O shows it very well. J. B. Austin

Gaseous discharge tube especially designed as an intense source of continuous ultraviolet radiation. R. H. Munch. *J. Am. Chem. Soc.* 57, 1863-5 (1935).—A simple low-voltage H<sub>2</sub> discharge tube with thin Pyrex window has been developed which is an intense source of ultraviolet. When filled with rare gases or Hg the tube is useful as a source of line spectra of these elements.

M. McMahon

Anode materials for high-vacuum tubes. E. C. Spitzer. *Elec. Eng.* 54, 1246-51 (1935).—Since the power output of a tube is proportional to the amt. of heat that can be dissipated safely from its anode, the anode is one of the most vital parts of high-vacuum tubes. Materials considered include W, Mo, Ni and C (graphite). The final choice of material depends upon the limitations involved.

W. H. Boynton

The evolution of the x-ray tube. P. Krauss, Sr. *J. Chem. Education* 12, 553-8 (1935). E. H.

Applications of a photoelectric cell. Anthony H. Lamb. *Elec. Eng.* 54, 1186-90 (1935).—The characteristics of one type of dry disk photoelec. cell are described and a no. of typical applications given. W. H. Boynton

New light sources for exposure tests and fluorescence analyses. Walter M. Münzinger. *Anticellulose* 6, 179-80 (1935).—An incandescent lamp contg. Hg vapor is described. F. M. Symmes

Bottom brick for Ajax elec. furnaces (Vakhramiev) 19. Oxide coatings on cathodes for elec. discharge devices (U. S. pat. 2,019,548) 9. Vulcanizing long lengths electrically (Brit. pat. 432,943) 30. Elec. furnace for melting glass (U. S. pat. 2,022,112) 19. Halogenated di- and triphenylmethanes (Brit. pat. 433,072) 10. Turbine blades (Brit. pat. 432,386) 9. Coating Al and its alloys (Brit. pat. 433,367) 9.

Batteries. Accumulatorenfabrik A.-G. Brit. 433,809, Aug. 21, 1935. The gas that collects in the space above the electrolyte while a battery is in use or on open circuit is absorbed by electrodes that are given a potential by connection with the main electrodes.

Batteries. P. R. Mallory & Co., Inc. Brit. 433,771, Aug. 20, 1935. A voltaic couple consists of an electrode of Cd, Al, Zn or other electropos. material in contact with an electrode of solid  $CrO_2$ .

Storage battery. Robert H. Lewis (to Firestone Battery Co.). U. S. 2,019,823, Nov. 5. Structural details. Storage battery. Frank S. Carlisle (to Carlisle & Doughty, Inc.). U. S. 2,021,288, Nov. 19. Structural features.

Storage battery. Walter C. Roberts (to B. F. Goodrich Co.). U. S. 2,022,090, Nov. 26. Structural details.

Storage batteries. Leonard Fuller. Brit. 432,775, Aug. 1, 1935. A dry storage battery is made by placing



a pasted neg. electrode in position in a container and then making the pos. electrode by filling the container with  $\text{PbO}_2$  or  $\text{PbO}$  which has been electrolytically formed and compressing it against a separator covering the neg. electrode. The electrolyte is absorbed in the electrodes and may be introduced into the active material before or after the pos. active material has been placed in the container.

**Storage-battery electrodes.** Karl W. J. Hjelmblad Brit. 433,518, Aug. 16, 1935 Structural features are given

**Condensers.** Porzellan Fabrik Kahla Brit. 432,793, July 30, 1935 A condenser is made with heat-resisting ceramic insulating-material as the dielec., the armatures being produced by burning on layers of precious metal by a process such as that of Brit. 407,509 (C. A. 28, 4987)

**Condensers.** Roger B. Peacock and Imperial Chemical Industries Ltd. Brit. 433,140, Aug. 9, 1935 Chlorinated rubber, with or without a plasticizer such as chlorinated Pb, is used as dielec. The rubber may be coated on the surface of the metal electrodes or used to impregnate a paper separator.

**Electrolytic condenser.** Julius F. Lihensfeld (to Ergon Research Laboratories, Inc.) U. S. 2,021,455, Nov. 19 Structural features

**Electrolytic condensers.** Joseph B. Brennan Brit. 433,818, Aug. 21, 1935 See Fr. 773,812 (C. A. 29, 13314) The solns must be acidified

**Electrolytic condenser.** Siemens & Halske A-G (Walter Michaels, applicant in Ger.) Fr. 785,674, Aug. 16, 1935 Hydroxyalkylamines, e. g., triethanolamine, are used as electrolyte or a constituent of the electrolyte in the formation or use of the condensers

**Electrolytic cells suitable for condensers.** Howard E. Rhodes (to Aerovox Corp.) U. S. 2,019,994, Nov. 5 An Al electrode is formed by impressing a voltage on it in the presence of an electrolyte including borax and boric acid, and then impressing a higher voltage in the presence of an electrolyte including a mixt. formed from boric acid and  $\text{aq. NH}_3$  soln.

**Electrolytic condenser electrodes.** Joseph B. Brennan Brit. 433,491, Aug. 15, 1935. The electrode comprises a base having applied thereto a reticulated surface consisting of fine coherent particles of metal. Preferably molten Al is sprayed onto a base of metal, paper, wax, rubber, glass, etc.

**Mercury turbine condenser and associated apparatus.** Leonard R. Biggs (to General Elec. Co.) U. S. 2,020,097, Nov. 5. Structural and operative details

**Making endless metal bands by electrodeposition.** Seamless Metals Ltd. and Charles Rice Brit. 432,095, July 19, 1935 Metal is deposited on a cathode in the form of an endless band mounted on a frame round which anodes are moved continuously by a conveyor. The cathode may be of Cu coated with Ag to permit detachment of the deposit; the edge portions are coated with a resin. App. is described.

**Depositing metals by electric discharge.** Paul Alexander and La Dispersion Cathodique (en abrégé Disca) Soc. anon. Brit. 432,460, July 26, 1935 The cathode to be disintegrated is first treated for the removal of gases strongly incorporated in the surface by active adsorption or "chemisorption." This preferably is effected by heating in an atm. of a gas, e. g., H, that reacts with the adsorbed gas. Heating may be by a preliminary discharge or otherwise. The heating temp. should be above  $800^\circ$  and in the case of Pt and W should be above  $1300^\circ$ . Liberation of the adsorbed gas is facilitated by ionization of the filling gas by means of x-rays, Ra or by auxiliary electrodes. The formed products are evacuated continuously by pumping. In 432,470, July 26, 1935, articles are coated by cathode disintegration with a hard metal of the Fe or W group, including Mn, Fe, Co, Ni, Mo, W and Cr. The 2nd metal is preferably a precious metal, e. g., Pt, Ir. The articles coated may be of enameled porcelain, fused quartz, enameled metal or glass; they should be heated, e. g., to  $200-800^\circ$ , during deposition. A mirror may be made by deposition of Ag on an initial deposit of Ni or Mo.

1 Plates coated according to the process may be used as elec. heating resistances.

**Plating metals.** Heinz Tichauer, Jean Frasch and Henri Jolivet. Fr. 785,717, Aug. 17, 1935. In covering metals such as Al by electrolysis, particularly for plating with Ni, a basic bath, in which a small amt. of an org. acid such as malonic acid is incorporated, is used. An example of a bath for plating Al contains  $\text{NiCl}_2$ , 18-20, NaOH 6-8, the malonic acid 1-2, NaCl 1-3% and water the rest. The metal may be preliminarily treated in a bath contg. Zn salts

**Chromium plating.** Richard Schneidewind. U. S. 2,020,282, Nov. 12. The "throwing power" of Cr-plating baths is increased by adding a quinone such as 0.5% or less of anthraquinone, hydroquinone or naphthaquinone to the bath

**Apparatus for the electrolytic generation of gases under pressure.** Rudolf A. Erren Brit. 432,698, July 31, 1935

**Electrolytic purification of liquids.** Jean Billiter. Brit. 431,695, July 15, 1935 In the electro-dialytic purification of liquids in a 3-compartment cell, the anode space is not flushed, the anode liquor thereby increasing in acid strength and cond. Pure acid may be added from time to time to maintain an anolyte cond. of 10-3 reciprocal ohms per cc. The cathode liquor may similarly be allowed to increase in strength and its cond. further increased by adding pure bases. Cells are described

**Electrolytic transmission of sodium through glass.** Marconi's Wireless Telegraph Co. Ltd. and Ernest W. B. Gill Brit. 432,898, Aug. 2, 1935 Na is introduced into the interior of an elec. lamp or discharge tube having an envelope made wholly or in part of a substance contg. Na by immersing the device in a molten bath of a Na compd., and passing an elec. discharge in the vessel without the use of internal electrodes. App. is described.

**Refining iron and iron alloys by electrolysis.** Ernst Kelsen and Edgar Aussnit Austrian 142,870, Sept. 25, 1935 (Cl. 48a). Products of reduced S content are obtained by adding an alk. earth salt, e. g.,  $\text{BaCl}_2$ , to the electrolyte, whereby  $\text{SO}_4$  ions derived from S in the crude material are eliminated.

**Treatment of electrolytic iron.** Ernst Kelsen and Edgar Aussnit. Austrian 142,871, Sept. 25, 1935 (Cl. 40b). The magnetic properties of electrolytic Fe are improved by removing as much as possible of the occluded H. This is effected by subjecting the metal to a heat treatment in which the temp. is alternately raised and lowered, under such conditions of reduced pressure that the partial pressure of H is kept below 1 mm.

**Treatment of electrolytic iron and iron alloys.** Ernst Kelsen and Edgar Aussnit. Austrian 142,872, Sept. 25, 1935 (Cl. 48b). The surface of the material is highly polished, and occluded H is then removed by the usual heat treatment. The products have a greater resistance to rusting than those produced by the known process in which the surface is polished after the heat treatment.

**Electrolytic surface treatment of aluminum and its alloys.** Ralph B. Mason (to Aluminum Co. of America). Brit. 433,494, Aug. 15, 1935. An article of Al or Al alloy is provided with a highly reflective surface by anodic or a.-c. treatment in an electrolyte contg. a fluoborate. Suitable electrolytes are solns. of  $\text{HBF}_4$ ,  $\text{NH}_4\text{BF}_4$  or  $\text{Pb}(\text{BF}_4)_2$ . Aceto-tri-fluoroboric acid, obtained by treating  $\text{AcOH}$  with  $\text{BF}_3$ , may be used.  $\text{NH}_4\text{F}$  may be added. The treated surface may be coated with a clear transparent lacquer or may be subjected to further anodic treatment in a  $\text{H}_2\text{SO}_4$ ,  $\text{CrO}_3$  or  $(\text{COOH})_2$  bath to produce a transparent oxide coating.

**Coloring aluminum and its alloys.** Veremigte Aluminium-Werke A.-G. Ger. 619,163, Sept. 23, 1935 (Cl. 48a. 16). Addn to 607,473 (C. A. 29, 17239). Articles of Al or its alloys, before they are colored in known manner, are superficially oxidized by electrolytic treatment with a. c. in a bath contg.  $\text{CrO}_3$  and  $(\text{COOH})_2$ .

**Beryllium.** Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. Fr. 785,072, Aug. 1, 1935. Be is obtained by submitting to electrolysis at  $370-400^\circ$  a mixt. of

Ne chloride (40-60%) and an alkali chloride, the f. p. of the mixt. being below 450°.

Gold Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. Fr. 784,928, July 29, 1935. Au free from Pt or of low-Pt content is obtained by using a product contg. both these metals as anode in an electrolyte free from chlorides of precious metals and composed of HCl or chlorides or both, with c. ds. of about 1200 amps. per sq. m. or more. The resulting mud is afterward treated to sep. other metals such as Cu or Ag, e. g., by treatment with  $\text{HNO}_3$  or  $\text{H}_2\text{SO}_4$ .

Producing fluoride coatings on light metals and their alloys Siemens & Halske A-G (Hellmut Fischer, inventor). Ger. 619,415, Oct. 1, 1935 (Cl. 45a. 16). The metal or alloy is subjected to electrolytic treatment in a fused electrolyte comprising a compd. or mixt. of compds. of F m. at a relatively low temp., e. g., 150-220°. Suitable electrolytes are  $\text{KHF}_2$  or  $\text{NaHF}_2$  or mixts. thereof, and also double fluorides of Na and Bi, K and Mg, Na and Al or an alkali metal and Sb. Anodic treatment with d. c. is preferred, but a c. may also be used. The cathode may be a fused metal or alloy of low m. p. which alloys with the metal of the electrolyte. Various uses for the fluoride-coated light metals or alloys are indicated, e. g., for coating app. to be used in contact with F or its compds., or as supports for light-sensitive materials or cements for uniting metal surfaces. The fluoride coating also facilitates the absorption or adhesion of dyes or lacquers.

Electrochemical treatment of clay. Giovanni Rodio Brit. 432,253, July 23, 1935. Clayey soil is hardened by passage of an elec. current from an electrode of Al to 1 of Cu, Fe or other metal, preferably a metal widely sep'd from Al in the electrochem. series.

Electrolytic furnaces E. I. du Pont de Nemours & Co. Brit. 433,633, Aug. 19, 1935. In the electrolysis of a fused salt mixt., e. g.,  $\text{NaCl}$  and  $\text{CaCl}_2$ , in a cell comprising narrowly spaced electrodes of large area, sep'n. of undesired solid metal is avoided by artificially increasing the rate of circulation of the electrolyte between the electrodes.

Electrolytic furnaces for producing aluminum Siemens-Planiawerke A-G Iur Kohlefabrik Brit. 433,508, Aug. 15, 1935. Contact surfaces are machined on the electrodes during the feeding of the latter by means of a cutting edge of hard metal, e. g., a W-Co alloy, provided on the contact plates. The plates are of soft metal.

Electric furnace, with means for indicating the magnetic condition of the charge Hevi Duty Electric Co. Ger. 619,398, Sept. 30, 1935 (Cl. 15c. 1.50). See Brit. 366,542 (C. A. 27, 1604).

Electric muffle furnace with gas-circulating means Siemens-Schuckertwerke A-G (Johann Schnepf and Otto Günther, inventors) Ger. 619,479, Oct. 1, 1935 (Cl. 15c. 11.10).

Electric induction furnaces for melting metals Gustaf A. Juhlin and Associated Electrical Industries Ltd. Brit. 433,539, Aug. 13, 1935. The charge is subjected to a vertical alternating magnetic flux and simultaneously to a horizontal magnetic field rotating about a vertical axis.

Electric resistance heated furnace. Numan R. Stansel (to General Elec. Co.) U. S. 2,020,127, Nov. 5. Structural features.

Roof construction for electric furnaces Howard A. McPherson (to American Manganese Steel Co.) U. S. 2,021,424, Nov. 19.

Electric heating and conditioning of wire or the like as in wire-drawing operations Wm. H. Wood, Oscar C. Trautman and Wm. F. Benninghoff (to Ira Crouse). U. S. 2,019,555, Nov. 5. Various details of app. and operation are described.

Electrical pyrometer Samuel Ruben (to Vega Mfg. Corp.) U. S. 2,021,491, Nov. 19. A Cu shell carries a layer of cuprous oxide on its inner surfaces and is substantially filled with graphite powder. One electrode is in contact with an unoxidized portion of the Cu shell and another is imbedded in the graphite powder.

Electric-arc apparatus for making artificial gems by fusion of powdered oxides Alfred Schmid Ger. 619,331, Oct. 3, 1935 (Cl. 12m. 6).

Electrical precipitation of suspended particles from gases Walther Deutsch (to International Precipitation Co.) U. S. 2,019,485, Nov. 5. Various details of app. and operation are described.

Electric apparatus for determining the temperature of conductive bodies such as calender rollers. Georg Kemath (to Siemens & Halske A-G) U. S. 2,020,067, Nov. 5. Various structural, elec. and operative details.

System for making electric resistances by depositing carbon on supports Bernhard Benschlag Ger. 619,298, Sept. 25, 1935 (Cl. 21r. 34.05).

Stable contact rectifiers Süddeutsche Apparate-Fabrik G. m. b. H. Brit. 432,619, July 30, 1935. A dry contact rectifier comprises a light metal base, e. g., Al, coated with a layer of a metal of the Fe group, a layer of Se firmly applied and a 2nd electrode pressed or sprayed onto the layer of Se.

Electric rectifying and conversion system employing mercury vapor or a rare gas Jürgen von Isendörff (to Westinghouse Elec. & Mfg. Co.) U. S. 2,020,922, Nov. 12. Various structural, elec. and operative details.

Mercury-arc rectifier Joseph Sierpan and Leon R. Ludwig (to Westinghouse Elec. & Mfg. Co.) U. S. 2,020,915, Nov. 12. Structural, elec. and operative details.

Backfiring indicator for mercury-arc rectifiers Bernard F. Lenehan (to Westinghouse Elec. & Mfg. Co.) U. S. 2,020,952, Nov. 12. Structural, elec. and operative details.

Vapor electric device with a mercury cathode Eugene H. Reed (to General Elec. Co.) U. S. 2,020,962, Nov. 12. Structural and operative details.

Röntgen meter Charles C. Lauritsen (to Calif. Institute of Technology) U. S. 2,022,117, Nov. 20. Structural and elec. details.

Photoelectric cells Electrical Research Products, Inc. Ger. 619,402, Oct. 1, 1935 (Cl. 21g. 29). See Brit. 351,606 (C. A. 27, 4964).

Photoelectric tube Harvey C. Rentchler (to Westinghouse Lamp Co.) U. S. 2,019,634, Nov. 5. An envelope is used having a portion permeable to radiation below 2700 Å. and contg. an anode which may be formed of W and a photosensitive cathode having a W photosensitive surface.

Electric discharge devices such as neon and sodium-vapor lamps Cornelius Bol and Adrianus Lunneman (to General Elec. Co.) U. S. 2,020,718, Nov. 12. An insulating base is used which is formed of asbestos, ceramic and talc and the pores of which are filled with a hardening resin such as a phenol-formaldehyde resin.

Gaseous electric discharge device suitable for cathode glow lighting Ted E. Foulke (to General Elec. Vapor Lamp Co.) U. S. 2,020,722, Nov. 12. One only of a plurality of electrodes is coated with an alk. compd. such as  $\text{BaCl}_2$  or  $\text{SrCO}_3$  which is reducible to the oxide and the electrode is heated sufficiently to effect such reduction to oxide, a gaseous atm. such as Hg vapor, Ne or He is provided about the electrodes, and a steep wave bombardment of the coated electrode is produced to reduce some of the oxide to metal and to sputter the metal onto another electrode U. S. 2,020,723 relates to elec. structural and operative details of an elec. gaseous discharge device suitable for use with Ne, A and Hg.

Gaseous electric discharge device which may contain neon, argon, mercury, etc. Georg Gaudes and Martin Reger (to General Elec. Co.) U. S. 2,020,727, Nov. 12. Various structural, elec. and operative details.

Gaseous electric discharge devices such as those containing metal vapor Otto Fritz and Alfred Rüttemann (to General Elec. Co.) U. S. 2,020,724-5, Nov. 12. Various structural, elec. and operative details.

Gaseous electric discharge device containing sodium vapor and suitable for high-intensity lighting Georg Gaudes (to General Elec. Co.) U. S. 2,020,726, Nov. 12. Various structural, elec. and operative details.

Gaseous electric discharge device which may contain sodium, etc. Marcello Pirani and Martin Reger (to

General Elec. Co.). U. S. 2,020,736, Nov. 12 Structural, elec. and operative details.

Electric discharge devices such as those producing ultraviolet rays containing a gaseous atmosphere such as mercury vapor. Gustav Zecher (to General Elec. Co.). U. S. 2,020,715, Nov. 12. Structural, elec. and operative details.

Gaseous electric discharge lamps for illumination such as those containing metal vapor. Willem Uytendhoeven and Cornelis Verburg (to General Elec. Co.). U. S. 2,020,706, Nov. 12. Various structural, elec. and operative details.

Gaseous electric discharge lamps for illumination such as those containing neon and sodium vapor. Willem Uytendhoeven and Aart de Bruin (to General Elec. Co.). U. S. 2,020,707, Nov. 12. Structural, elec. and operative details.

Gaseous electric discharge arc lamp which may contain argon and mercury or neon and sodium, etc. Marcello Pirani and Alfred Rittenauer (to General Elec. Co.). U. S. 2,020,737, Nov. 12. Structural, elec. and operative details.

Negative glow device containing inert gas such as neon or argon and giving flaming light effects. Andrew P. Henninger, Jr. U. S. 2,020,411, Nov. 12. Structural details.

Electric glow-discharge lamp. Gustav Zecher and Johannes Bruynes (to General Elec. Co.). U. S. 2,020,716, Nov. 12. Structural and elec. details, of Hg-vapor lamps, etc.

Electric lamps. Soc. anon. pour les applications de l'électricité et des gaz rares (L'at. Inventions Claude-Paz & Silva). Fr. 785,139, Aug. 2, 1935. The lamps are filled with a mixt. of Kr and Xe and less than 8% of N, the formation of arcs being avoided by replacing the usual Ni leads by Mo. The persistence of the arc which forms by rupture of the W filament is avoided by placing one or 2 fuses in the foot of the lamp. Fr. 785,140. The Kr and Xe remaining in the conduit after filling the lamps are recovered by drawing the gases by a vacuum pump through a condenser dipping into liquid N, O or air.

Electric lamp containing alkali metal vapor. Harvey C. Rentschler and Donald L. Henry (to Westinghouse Lamp Co.). U. S. 2,019,633, Nov. 5. An envelope contains an atm. of a condensable alkali metal vapor such as that of Na and a thermionically active oxide-coated electrode or the like, together with means such as a mesh metal getter for rendering ineffective gas derived within the envelope which would normally lower the efficiency of the device when expressed in lumens per watt.

Sodium-vapor lamp. John W. Marden (to Westinghouse Lamp Co.). U. S. 2,019,613, Nov. 6. Structural features.

Discharge lamps. Patent-Treuhand-Gesellschaft für elektrische Glühlampen m. b. H. (to The General Electric Co. Ltd.). Brit. 432,320, July 24, 1935. All the electrodes are composed of Te and the envelope is of a transparent material, e. g., quartz, that resists for at least 100 hrs. the attack of Te vapor at a temp. of the lamp corresponding to 0.1 mm. pressure. Fluorescent substances excited by the ultraviolet component of the light may be associated with the lamp.

Discharge lamps. Patent-Treuhand-Gesellschaft für elektrische Glühlampen m. b. H. (to The General Electric Co. Ltd.). Brit. 432,614, July 30, 1935. In high-pressure Hg-vapor lamps having thermionic electrodes and a filling of rare gas, 0.2-5% of Cs is added to the Hg and the glass of the envelope contains not less than 20%  $Al_2O_3$  or more than 60%  $SiO_2$ , blackening of the envelope being thus avoided. The glass may also contain oxides of B, Mg and Ca. The thermionic electrodes may comprise a sintered mixt. of 90% of mixed BaO and CaO and 10% W.

Discharge lamps. Compagnie des lampes. Fr. 785,301, Aug. 7, 1935. The spectrum formed by Hg-vapor lamps is improved by the addn. of K, Li or alloys thereof, correctly proportioned to increase the red radiation.

Electric discharge lamp containing a metal vapor. Willem Uytendhoeven, Martinus van Dam and Cornelis Verburg (to General Elec. Co.). U. S. 2,020,708, Nov. 12. Structural and operative details.

Electric discharge lamp system for illumination and containing sodium vapor. Hendrik A. W. Klinkhamer, Aart de Bruin and Ferdinandus H. A. van Stekelenburg (to General Elec. Co.). U. S. 2,020,786, Nov. 12. Various structural, elec. and operative details.

Luminous gas-discharge tubes such as those containing neon. Edward S. Woolrich (to Flexlume Corp.). U. S. 2,020,393, Nov. 12. Structural features.

Apparatus (with a photometer chamber) for testing and asserting electric incandescent lamps. Caspar Reiter, Walther-Winfried Loebe, Curt Samson, Felix Bolck and Max R. Grossmann (to General Elec. Co.). U. S. 2,020,964, Nov. 12. Various structural, elec. and operative details.

Caps for electric lamps and discharge devices. Patent-Treuhand-Gesellschaft für elektrische Glühlampen m. b. H. (to The General Electric Co. Ltd.). Brit. 433,260, Aug. 12, 1935. A screw-cap comprises a sleeve of Al which has all or part of its surface insulated by oxidizing it electrolytically.

Caps for electric lamps and discharge devices. Patent-Treuhand-Gesellschaft für elektrische Glühlampen m. b. H. (to The General Electric Co. Ltd.). Brit. 433,274, Aug. 12, 1935. In a metal cap having 1 or more contact pins projecting therefrom, each pin fits into a hole in the metal and is insulated from the cap by means only of a layer of oxide formed on the surface of the metal before the pin is placed in the hole. The cap is preferably made of Al, with a layer of oxide formed thereon by electrolytic oxidation.

Activating alkaline earth oxide-coated cathodes. Charles H. Prescott, Jr. (to Bell Telephone Laboratories, Inc.). U. S. 2,019,561, Nov. 5. The cathode is heated in a highly evacuated vessel to which a material such as  $CH_4$  yielding free C upon thermal decompn. is supplied, and activation is completed by chemically combining the free C with the alk. earth oxides on the cathode to form free alk. earth metal. App. is described.

Oxidizing electrodes such as those of cathode-ray tubes. Sanford F. Fessig (to Radio Corp. of America). U. S. 2,020,305, Nov. 12. For producing a uniform oxide layer of desired thickness, electrodes such as those comprising Ag globules are subjected to an atm. of O and an elec. field is developed between the electrode under treatment (which is supported in a container) and an independent exterior electrode movable to vary the effectiveness of the field. App. is described.

Thermionic cathodes. Franz Skaupy. Ger. 619,133, Sept. 23, 1935 (Cl. 21g. 13 03). Addn. to 600,374 (C. d. 28, 65377). A core wire is coated with a suitable metal oxide mixt. by decomg. the mixed vapors of the corresponding metal acetylacetonates, as described in Ger. 600,374. The preferred oxide mixt. comprises approx. equivalent proportions of an emitting oxide, e. g., BaO, and an acidic oxide stable to heat, e. g.,  $Al_2O_3$  or  $ZrO_2$ .

Thermionic cathodes "Flin" A-G für elektrische Industrie. Austrian 142,827, Sept. 25, 1935 (Cl. 21g). Cathodes of relatively long life are made by depositing an emitting oxide layer on a support of an alloy contg. Ni 60-80, Co 15-22, Fe 6-8 and Ti 4-6%, with or without 3-9 6% of another element of the 4th periodic group. A specified alloy contains Ni 67, Co 17, Fe 6, Ti 5, Si 3 and Mn 2%.

## 5—PHOTOGRAPHY

E. R. BULLOCK

Infrared radiation and photography in the dark. A Seyewitz *La Nature*, No 2962, 300-1 (1935) —Photographic plates are sensitized with kryptocyanine and objects to be photographed are irradiated from lamps contg. Kr at 12,000 Å. The industrial application of infrared photography is considered. James C. Munch

The use of infrared photography in navigation. Ragnar Thoren *Nord Tid Fotografi* 19, 1-4 (1935) —Plates with max sensitivity at 7100 Å. were used, without hypersensitizing, for photographing the coast of Denmark at a distance of 20,000 m through a slight fog (visibility 14,000 m). The exposure was  $\frac{1}{16}$  sec. J. W. Holst

Three-color pictures by the Duxochrome process R Weizsaecker *Fot Rundschau* 72, 282-4 (1935) —A review of the operations in the Duxochrome process. Prints are made from color-separation negatives on films bearing dyed Ag halide emulsions. After development, fixing, washing to remove excess dye, and removal of the Ag, the residual dye images are removed from the films and superimposed on a paper support. P. W. Vittum

Color sensitometry. John Eggert *Z. wiss. Phot.* 34, 54 (1935) —By the use of a standard yellow filter over the light source of the DIN sensitometer, a DIN value for the color sensitivity is attained. The difference between general sensitivity and yellow sensitivity gives a "yellow-differential" value in DIN degrees. The simplicity of the measurement is discussed. Some applications noted are: the simple and accurate measurement of emulsion sensitivity, the distinguishing of various yellow filters; and the detn. of filter factors for filter-emulsion combinations merely by simple calcs. from data in tables.

H. A. Kurtzner  
The care of tank developers W. Triepel *Phot Ind* 33, 491-2 (1935) —A comprehensive study includes elimination of impurities and especially the prevention and elimination of bacterial infections in developers.

C. E. Meulendyke  
Tests with the selenium toning bath. E. van Beugen. *Focus* 22, 221-2 (1935) —Se baths, such as are obtained by dissolving Se in an alkali metal sulfite soln., produce images of suitable coloration when acting directly upon fine grain images (chloride and chlorobromide), but not on coarser (bromide) images. On the contrary, the coarser grain is favorable to good color and image quality when a bleach is used before the Se bath to which, in this case,  $\text{Na}_2\text{S}$  has been added.

C. E. Ives  
Topographical relations in image reversals Luppocramer. *Phot. Kor.* 71, 89-92 (1935) —Plates coated with a nearly pure (but not entirely free from  $\text{AgBr}/\text{AgI}$ ) gelatin emulsion, prep'd. as previously described (C. A. 24, 2669), after a preliminary uniform exposure to white light followed by a similar tablet exposure, show on development in alk. amido both ordinary solarization and a "2nd reversal," whereas without the preliminary exposure they show only solarization. The sensitivity of this emulsion for the 2nd reversal, after the preliminary exposure, appears to be much greater than its primary sensitivity. The results are discussed from the same standpoint as was taken in the article on varieties of the Becquerel effect (C. A. 29, 7839).

E. R. Bullock  
Silver halides peptized in gelatin by supersonic waves (Dangers) 2 Films of cellulose derivs. (Fr. pat. 785,639) 23

Color photography. Richard Gschöpf *Brit.* 432,349, July 22, 1935. In the production of multicolor prints upon paper, films, etc., by the imbibition process, colloidal solns. of dye bases are used for coloring the photographic gelatin printing matrices. The colloidal soln. is prep'd. by adding to the soln. of the dye, e. g., thionine blue, an alkali, e. g.,  $\text{NH}_4\text{OH}$ , until the soln. is alk. In certain cases, a slight excess of alkali may be added, a

protective colloid, e. g., dextrin, may be added prior to the alkali to avoid flocculation. Cf. C. A. 29, 7839.

Color photography. Opticolor A-G. *Brit.* 433,088, Aug. 8, 1935. In reproducing lenticular color-record material by developing the images by the reversal process with the addn. to the 1st developer of agents that increase the sharpness of detail, i. e.,  $\text{AgBr}$  solvents, e. g.,  $\text{NH}_4\text{OH}$ ,  $\text{KCNS}$ , the addn. of such agents is kept so small that the prints, and also, if desired, the originals from which such prints are produced, are free from a microscopically fine cloudy structure of the reduced Ag which becomes perceptible during exhibition as smudgy images.

Color photography. Béla Gáspár. *Fr.* 785,523, Aug. 12, 1935. The adhesive layer or the intermediate layers contain agents which destroy directly dyes diffusing into them from adjacent layers, or they participate indirectly in the decoloration of the dye. Thus diffusion of leucostereers is prevented by  $\text{HgCl}_2$ .

Color photography. Béla Gáspár. *Fr.* 785,665, Aug. 16, 1935. Photographic colloids are colored by dyes which contain metal in a complex state, e. g., those known by the names "Neolan" and "Lanasol" color. Diffusion and efflorescence is thereby avoided.

Photographic material. Gerd Heymer (to Agfa Anso Corp.). U. S. 2,020,607, Nov. 12. A support carries at least two emulsion layers dyed to have different colors and colored for the same color to which the respective layer is specially sensitive, at least one of these emulsion layers contg. also a filter dye, sol. in the photographic treating baths, which absorbs at least part of the spectral rays which is transmitted by the color of this layer and to which this layer is sensitive, for equalizing the gradation of the emulsion layers. A blue sensitive emulsion layer dyed green may be used with a red sensitive emulsion layer dyed red and contg. a blue-green filter dye.

Sensitive photographic material Samuel E. Sheppard and Waldemar Vanselow (to Eastman Kodak Co.) U. S. 2,019,737, Nov. 5. Cd, mercuric, mercurous, nickelous, Ag, thallous, quadrivalent Th or stannic orthoarsenates are used as light-sensitive materials with gelatin, etc.

Photographic processes Arthur Ernest Field *Brit.* 431,384, July 8, 1935. A negative, especially for use in the production of photomech. printing surfaces, is treated with a bleaching reagent, e. g., a bath comprising (1) I in an aq. soln. of KI, (2)  $\text{K}_4\text{Fe}(\text{CN})_6$  and  $\text{KBr}$ , or (3)  $\text{KMnO}_4$  acidified with  $\text{AcOH}$  or  $\text{HCl}$ , to produce a photographic image of pos. appearance, retouched, etched or reduced by a Ag halide solvent, e. g., in a bath contg.  $\text{KCN}$ ,  $\text{Na}_2\text{S}_2\text{O}_3$  or  $\text{KI}$ , the portions of the image not to be retouched being coated with cellulose varnish or litho crayon, and finally treated with a soln. that blackens the Ag salt image and reconverts the image to a neg. image. After bleaching with I soln., the excess is removed with a  $\text{KOH}$  bath. After bleaching with  $\text{KMnO}_4$ , stains are removed by treatment with a K metabisulfite bath. After retouching with  $\text{KCN}$ , the seum is removed by dil.  $\text{HNO}_3$  or  $\text{AcOH}$ . The rebleaching bath may comprise  $\text{Na}_2\text{S}$  or a metal-hydroquinone developer, or a mixt. of  $\text{Na}_2\text{S}$  and a developer. After washing, the negative may be intensified with  $\text{HgCl}_2$ ,  $\text{HgBr}_2$  or  $\text{HgI}_2$ , or a redevelopment and intensifying soln. comprising aduol, citric acid,  $\text{AgNO}_3$ ,  $\text{HNO}_3$  and  $\text{H}_2\text{O}$ . The fixing soln. for the original negative may include KI.

Photographic emulsions. I. G. Farbenindustrie A-G *Brit.* 432,909, Aug. 7, 1935. This corresponds to Fr. 768,047 (C. A. 29, 1341). Cyanine dyes contg. an unsubstituted or substituted  $\beta, \beta'$ -naphthoxazole nucleus are also used.

Photographic emulsions Soc. pour l'ind. chim. & Bâle *Ger.* 619,538, Sept. 25, 1935 (CI 57b. 8 01). See *Brit.* 428,305 (C. A. 29, 6520\*).

Sensitizing silver halide emulsions Walter Dietele and Hermann Durr (to Agfa Anso Corp.). U. S. 2,020,

636, Nov. 12. 1,1'-Dimethyl-4,4'-diethylamino-3,3'-bisdimethylindocyanine bromide, etc., are used as sensitizers, emulsions so sensitized being sensitive to a range of wave lengths from about 545 m $\mu$  to about 760 m $\mu$  with a max. at about 695 m $\mu$ .

**Photographic images.** I. G. Farnham, A.-G. Brit. 785,071, Aug. 23, 1953. Ag images of white coloration are obtained by incorporating in the developer derivs. of org. bases or inorg. salts which dissolve Ag, e. g., thiocyanate of guanidine or of K, derivs. of xantogenic acid, NH<sub>4</sub>Cl or NH<sub>4</sub>diethiocarbamate. As these in the concn necessary have a solvent action on the gelatin also, this is made very hard.

**Treating superposed photographic images.** Leopold D. Mannes and Leopold Godowsky, Jr. U. S. 2,019,718, Nov. 5. In the differential treatment of images superposed in different depths of an emulsion, the emulsion is bathed in a strong soln. of I until a complete image is bleached, and the leaching action is then quickly interrupted by bathing the emulsion in a bisulfite soln.

**"Ben Day" photographic tones.** Fayette T. Marsh and Charles H. Andersen U. S. 2,019,760, Nov. 5. Various reflective coloring matters having different reflective values are placed upon the surface of a master drawing to be toned so that they are spaced where "Ben Day" tones are to subsequently appear on a photographic negative of the drawing, the drawing has superposed upon it a transparency contg. a "Ben Day" pattern, and the combination is photographed with the transparency nearest the camera.

**"Ben Day" tones photographically produced.** Fayette T. Marsh and Charles H. Andersen (to Charles V. Price) U. S. 2,021,191, Nov. 10. A negative of a master drawing is made on a white translucent support, there is superimposed on the reverse side of the negative a transparency contg. a "Ben Day" pattern, the face of the transparency contg. the pattern being placed nearest the operator, and on the exposed face of the thus positioned transparency, in selected areas where tones or shades are to subsequently appear on a photo-copy of the drawing, various reflective coloring materials are placed which have different reflective values; the registry positions of the negative and transparency are reversed and the negative is exposed through the stencil to a sensitized film, the transparency is removed and the negative is then exposed alone.

**Photographic films.** Siemens & Halske A.-G. Brit. 433,538, Aug. 12, 1935. Addn. to 407,830 (C. A. 28, 5358). A metallic film of Al, Al alloy or a metal, e. g., Cu, coated with Al, has as a photographic base a porous carrier layer of an Al compd, artificially produced on the Al surface and is impregnated with light-sensitive substances as in 407,830. The porous layer may be produced by plunging the metal into aq. solns. or fused substances, with or without application of d. c. or a. c. Impregnation with light-sensitive substances may be effected by coating, spraying or immersion. Developed Ag pictures may be toned and the porous layers may be recolored.

**Photographic films.** Kodak Ltd. and Raymond I. Crowther, Brit. 433,655, Aug. 10, 1935. Addn. to 204,852 (C. A. 27, 2104). The protective collod layer coated on the antihalation dye layer of the film according to 204,852 is formed with a mat surface, which diminishes specular reflection of light which may pass through the dye layer and, if the layer is not removed during processing, provides a surface for retouching. The collod layer may contain an inert dispersed material, e. g., levigated starch or laryna, having a refractive index different from that of the collod, e. g., gelatin, or the collod layer, e. g., of cellulose nitrate or acetate, may be cast on a mat surface.

**Sensitized films.** N. V. Philips' Gloeilampenfabrieken, Brit. 433,680, Aug. 10, 1935. See I. r. 782,321 (C. A. 29, 7409).

**Light sensitive films.** N. V. Philips' Gloeilampenfabrieken, I. r. 785,041, July 31, 1935. Images are obtained by photochem. means using a diazo compd. and a mercurous salt. The salt is preferably incorporated in the film with the diazo compd. but may also be after exposure of the film contg. the diazo compd. Much greater con-

trasts are obtained. The fixation of the image may be effected by water washing.

**Films for additive color photography.** Gabriel G. Moreno (to C. M. C. Corp.). U. S. 2,020,761, Nov. 12. Two consecutive series of images are recorded on 2 strips of film, pairs of images being recorded simultaneously one on each film, the films having their photographic emulsions in substantial contact, one of the emulsions being preferentially sensitive to green and the other preferentially sensitive to red, the 2 films are developed separately, followed by printing every other pair of isomorphous images from both films upon a single positive film in juxtaposition.

**Splicing films having photographic sound-record bands.** Albert Narath (to General Elec. Co.). U. S. 2,021,010, Nov. 12. Various operative details.

**Photographic films and plates.** I. G. Farnham Industrie A. G. Brit. 433,110, Aug. 15, 1935. A rigid support to which films may be affixed for exposure or liquid treatment comprises a base carrying a permanently adhesive layer consisting of a degraded aluminum colloid, gelatin, sol. in cold H<sub>2</sub>O. The colloid may be applied dissolved in H<sub>2</sub>O or a volatile org. solvent, e. g., glycol monoacetate, AcOH and dioxane admixed with H<sub>2</sub>O. Chloral hydrate and KCN are selected degrading agents. The solns. may be stabilized by adding, e. g., phthalic, salicylic or acetic acids. In an example, a film is pressed, for exposure or development, upon a glass plate bearing an adhesive layer formed from a compn. comprising gelatin, H<sub>2</sub>O, ethylmethylolhydriin, MeOH, phthalic acid and glycerol. The adhesive layer may be given a mat surface by addn. of finely divided agents, e. g., SnO<sub>2</sub>, BaSO<sub>4</sub>, hydrated SiO<sub>2</sub>.

**Color separation negatives for colored reproductions.** Michael Kronschnabl (in part to August Böttger) U. S. 2,020,688, Nov. 12. A plurality of different masking negatives of an original are successively produced through differently colored filters, each negative during its production being masked by the previously completed negative, a light-sensitive plate in a camera is exposed through at least two of the masking negatives in succession and the plate is also exposed directly to the original.

**Photographic plates, etc.** I. G. Farnham Industrie A.-G. Brit. 432,584, July 24, 1935. A Ag halide emulsion is developed in the presence of an arylimidazole of formula R.NS.CH.NH, in which R is a C<sub>11</sub>H<sub>7</sub> or polynuclear ring

residue substituted by a halogen atom and (or) alkyl group, or a residue of an unsubstituted polynuclear system. The arylimidazole may be added to the developer or incorporated during the prepn. of the light-sensitive material in an intermediate layer, a lacking or coating on the emulsion, for photographic paper, it may be incorporated in the paper pulp, laryta coating or sizing agent or in the finished paper. It may also be incorporated in exposed material by bathing prior to development. 5-substituted compds. are 5-chloro-, 5-chloro-6-methyl-, 4,6- and 5,6-dichloro-, 4,6-dibromo- and 5-iodobenzimidazoles, 1,2-naphthylimidazole, 1,2-anthrylimidazole and 4,6-acenaphthylimidazole.

**Photographic plates.** Siemens & Halske A.-G. Brit. 432,084, Aug. 7, 1935. Addn. to 407,830 (C. A. 28, 5358). The carrier layers produced on the surface of Al or Al alloy according to 407,830 are subjected, after formation and before applying the light-sensitive material, to an intermediate chem. and (or) thermal treatment to render them less brittle and more flexible. The treatment may consist in dipping the layers in cold or warm solns. of acids or alkalies or salts having an acid or alk. reaction.

**Storing photolithographic plates.** Morland & Incey Ltd. and Arthur G. Rendall, Brit. 431,415, July 8, 1935. To prevent deterioration, plates coated with ultraviolet collod are stored, before use, in a vacuum or in deoxygenated air or other gas. The desiccating agent may be CaCl<sub>2</sub>, silica gel or P<sub>2</sub>O<sub>5</sub>.

**Reflex prints.** Lodewijk P. J. van der Grinten (to N.-V. Chemische Fabriek L. van der Grinten). U. S. 2,022,014, Nov. 20. On an original, there is applied a

Action of hydrogen sulfide on insoluble chromates. I. Lead chromate and silver chromate. H. B. Dummick and Brahms Prakash. *J. Indian Chem. Soc.* 12, 505-17 (1935); cf. C. A. 23, 1074; 25, 2876; 26, 390 —Analysis of the reduction products of  $PbCrO_4$  indicated that the reaction proceeded as follows at 15-20°:  $63H_2S + 42PbCrO_4 \rightarrow 42PbS + 4Cr_2(SO_4)_3 + Cr_2(S_2O_3)_3 + 2S + 32Cr(OH)_3 + 15H_2O$ . At higher temps. more sulfite was formed at the expense of thiosulfate. The same general products were obtained on the reduction of  $Ag_2CrO_4$  with the exception that sulfite is also found. This indicates that sulfite and not thiosulfate is the precursor of sulfite.

H. E. Phipps

Preparation of bromine bleaching powder. E. J. Williams. *School Sci. Rev.* 17, 297-8 (1935) O. R.

The properties of ferric chlorosulfate. Francesco Scaife. *Gazz. chim. ital.* 65, 588-93 (1935) —To the salt (I) formed by the action of Cl on concd. aq.  $FeSO_4$ , Röhm (C. A. 16, 1714) assigned the formula  $FeSO_4 \cdot Cl \cdot 6H_2O$  (II), and assumed its decompos. to be  $3FeSO_4 \cdot Cl \rightarrow Fe_2(SO_4)_3$  (III) +  $FeCl_3$ . In the original paper, precise directions for its prepn. are lacking, but a communication from R. describes a different method, by which a solid mixt. of  $FeSO_4$ ,  $H_2O$  and  $FeSO_4 \cdot 7H_2O$  (IV) is treated with Cl. By this method R. obtained products, the soly of which differed from that described, and a com. sample differed likewise. Numerous expts. led to a satisfactory method, as follows. Cl is passed into satd. aq. II until all Fe is ferric, the soln. is replenished with more II and treated with Cl, and this process repeated until a thick brown liquid (giving no ppt. with  $K_4Fe(CN)_6$ ) is obtained. This is let stand over CaO and then  $H_2SO_4$  in *vacuo* until partially dry and then dried in air at room temp. It is then stable, has the compn. II, and is insol. in  $Et_2O$  (no uncombined  $FeCl_3$ ). The assertion of R. that III is insol. in water and in  $EtOH$  is erroneous. Tests of the soly of III in water, in  $EtOH$  and in aq.  $EtOH$ , and of a mixt. of III,  $FeCl_3$  and water in proportions to form II in  $EtOH$  showed complete soln. only in the latter case.  $EtOH$  added to a mixt. of I (2 mols.) and Na $SO_4$  10H $2O$  (1 mol.) in a mixt. of water causes to sep. a blood-red oil which crystallizes to the new double sulfate,  $3Na_2SO_4 \cdot Fe_2(SO_4)_3 \cdot 2FeSO_4 \cdot OH \cdot 7H_2O$  (IV), yellow, its aq. solns. are opalescent, decomposes without fusion when heated. The mother liquor contains uncombined III and Na $SO_4$ . Accordingly I behaves as a double salt of III and  $FeCl_3$  rather than as a complex, since III unites with Na $SO_4$  and since  $FeCl_3$  and excess Na $SO_4$  remain uncombined. In an analogous way  $EtOH$  added to I and  $(NH_4)_2SO_4$  gives  $Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$ . The results in general show that I is not a complex salt of the type assumed by Röhm, but is a weak double salt of the compn.:  $Fe_2(SO_4)_3 \cdot FeCl_3 \cdot 18H_2O$ .

C. C. Davis

$Li_2FeF_6$ . A. H. Nielsen. *Z. anorg. allgem. Chem.*

224, 84 (1935) — $Li_2FeF_6$  was prepd by dissolving  $FeCl_3 \cdot 6H_2O$  in HF, and adding an aq. soln. of  $Li_2CO_3$ . The white ppt. was washed by decantation with  $H_2O$ , alc. and  $Et_2O$ , and dried at room temp. It is a white, isotropic powder having  $n$  of about 1.42; difficultly sol. in  $H_2O$ , but dissolved by HCl giving a strong yellow color. R. H. L.

The systems alkaline earth chloride-alkaline earth oxide and the decomposition of alkaline earth chlorides by water vapor. Bernard Neumann, Carl Kröger and Herbert Jüttner. *Z. Elektrochem.* 41, 725-36 (1935) —Cooling curves for the systems were detd. in a dry atm. of  $N_2$  in a Ni crucible at temps. up to 1320°, as high as the Ni would stand. Some  $SrCl_2$  sublimed at 1350°. Temp.-compn. curves showed the compds.  $4SrCl_2 \cdot SrO$ ,  $BaCl_2 \cdot 2BaO$ ,  $BaCl_2 \cdot 3BaO$  and  $4CaCl_2 \cdot CaO$ , the last with a congruent m. p. at 839° and a transition point at 700°. The compd.  $SrCl_2 \cdot 2SrO$  is probably formed; it has an incongruent m. p. and a transition point at 1020°. Decompos. of the chlorides by  $H_2O$  vapor takes place at or above their m. ps. only, i. e., at 930° for  $SrCl_2$ , 910° for  $BaCl_2$  and 780° for  $CaCl_2$ . Decompos. isotherms were detd. from the resp. m. ps. to 1040° for  $SrCl_2$ , to 1145° for  $BaCl_2$  and 1000° for  $CaCl_2$ , and phase diagrams are given.

E. R. Rushton

System water-sulfuric acid-nickel sulfate. Raymond Rohmer. *Compt. rend.* 201, 672-4 (1935), cf. C. A. 28, 2893 —The system was examd. at 25° and 50° and partially at 80° and 90°. At 25° 3 stable hydrates,  $NiSO_4 \cdot nH_2O$ ,  $n = 7, 6$  and 1, with one metastable,  $n = 4$ , and some indication of one with  $n = 2$  were found. Results at 50° were similar, save that  $NiSO_4 \cdot 7H_2O$  was not found. Above 84.7° the only solid phases were  $NiSO_4$ ,  $H_2O$  and  $NiSO_4$ .

C. A. Suberrad

A silver mercuric complex. J. Bougault and E. Cattelain. *J. pharm. chim.* 21, 581-3 (1935) —See C. A. 29, 6860.

S. Waldbott

Organic cupratetraldehydes and cupratetramides. Jean Amiel. *Compt. rend.* 201, 904-6 (1935), cf. C. A. 29, 1355 —By mixing concd. solns. of the constituents and evapd. at 60° in vacuum the compds.  $[CuX_2](YH)_2$  are obtained, where  $X = Cl$  or  $Br$  and  $Y = PhNH_2$ ,  $i-C_4H_9NH_2$ ,  $C_6H_5NH_2$ ,  $C_6H_5N$  and  $C_6H_5N$ . The Cl compds. are yellow, the Br black, both are permanent in the air, insol. in  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ ,  $C_7H_8$ ,  $C_8H_{10}$  and pinene, slightly sol. in  $Me_2CO$  and  $AcOH$ , more so in alc., decomposed by  $H_2O$ ,  $H_2SO_4$  or strong bases; on heating they melt with decompos. With piperazine  $[CuX_2] \cdot C_4H_{10}N_2 \cdot 2H_2O$  is formed, which soon loses  $2H_2O$  in a dry vacuum; excess of  $CuX_2$  or piperazine give  $[CuX_2] \cdot (C_4H_{10}N_2 \cdot H_2)$  and  $CuX_2 \cdot 2[C_4H_{10}N_2 \cdot 2HX] \cdot 4H_2O$ , resp.

C. A. Suberrad

Inorganic photosyntheses (Calcagni) 3.

## 7—ANALYTICAL CHEMISTRY

W. T. HALL

A new electroanalytical method for the determination of alkalis. Karl Abresch. *Angew. Chem.* 48, 683-5 (1935). —The test soln. is electrolyzed in a container having a layer of Hg on the bottom as anode and a cathode which consists of a glass capillary dripping Hg through the soln. During the sepn. of a cation the current strength increases as amalgam formation occurs (as soon as the sepn. potential is reached). With increasing voltage the current strength reaches a const. value and the amt. of this satn. current is proportional to the concn. of the cation in the soln. Constancy of temp. and viscosity of the soln., and of the dripping velocity are essential. The perchlorate and Pt chloride methods of the sepn. of K from Na are described in detail as auxiliary parts of the electrolytic detn. The methods permit the sepn. detn. of alkalis in solns. contg. the cations Fe, Al, Ca, Mg, K, Na and any kind of anions. For the analysis of a K-bearing ash 20 min. is required when an accuracy of  $\pm 1\%$

of the detd. value is desired. A series of 12 detns. requires 1-1.5 hrs. Detailed directions and exptl. results are given. Five references.

Karl Kammermeyer

The use of Aluminum in the determination of small quantities of aluminum. V. M. Peshkova. *Trans. Inst. Pure Chem. Reagents* (U. S. S. R.) No. 14, 42-8 (1935). —To 15 cc. of the neutral soln. add 5 cc. of 1 N HCl and 5 cc. of 3 N  $NH_4OAc$  and 5 cc. of 0.1% Aluminum reagent. After 3 min. add 0.5 cc. 5 N  $NH_4OH$  and 0.5 cc. 5 N  $NH_4$  carbonate as little as 2.5  $\gamma$  Al will give the test, but the color varies somewhat if alkali or alk. earth ions are present: Fe should be absent or added to the standards.

Lewis W. Butz

The determination of small quantities of arsenic. G. G. Karanovich. *Trans. Inst. Pure Chem. Reagents* (U. S. S. R.) No. 14, 93-5 (1935). —The app. of Martin and Pien (C. A. 24, 5253) permits the detn. of 0.0001-0.0005 mg. As in samples of 0.1-1.0 g. The method is

ufficiently accurate to differentiate between 0.1 and 0.2 % of As.

**Determination of chromium. III. Rapid method for determining chromium in chrome steels.** Daniel Brard *Ann chim anal chim appl.* 17, 317-19 (1935); cf. C. A. 28, 7857. —In a 100 ml. Pyrex flask treat 0.5 g. of the alloy with 15 ml. of a mixt. of 10 ml.  $\text{HClO}_4$ , 4.161 and 5 ml. of concd  $\text{HNO}_3$ . Heat gently. Continue heating until after all  $\text{HNO}_3$  is removed and the soln. shows a green color due to  $\text{Cr}^{+++}$ . Then increase the heating somewhat until fumes of  $\text{HClO}_4$  are evolved copiously. Finally boil the acid for a min. to make sure that the oxidation to chromic acid is complete. Cool, add 50 ml. of water and boil 5 min. to expel  $\text{Cl}_2$  resulting from the decomposition of the  $\text{HClO}_4$ . Cool, add an excess of standard  $\text{FeSO}_4$  soln. and titrate the excess with  $\text{KMnO}_4$ . Cf. Willard and Young, C. A. 22, 3111.

**The thiocyanate test for iron.** T. N. Karsko *Trans Inst Pure Chem Reagents (U S S R)* No. 14, 81-7 (1935). —Fe can be detd. in sol. chlorides, nitrates and sulfates of the alkali and alk. earth metals and Mn by means of the thiocyanate test. For Hg and Pb salts and for  $\text{Cd}(\text{NO}_3)_2$  and  $\text{CdSO}_4$  it cannot be used, and it is not entirely satisfactory for Zn salts.

**The determination of iron in sea water.** Thomas G. Thompson and Raymond W. Bremner *J. consel intern exploration mer* 10, 33-8 (1935); cf. C. A. 26, 4551. —To 100 cc. of sea water, add 6 cc. of concd  $\text{H}_2\text{SO}_4$ , and evaporate to strong fumes. Cool, add a little water and oxidize with excess of  $\text{KMnO}_4$  at about 100°, and finally boil with Br. Expel excess  $\text{Br}_2$ , dil. to 83 cc. at room temp., add 10 cc. KCNS soln. and extract the red  $\text{Fe}(\text{CNS})_3$  with 10 cc. of isoamyl alc. Compare the color with that obtained similarly from known quantities of Fe.

**Determination of iron and aluminum by the method of Crisp in natural phosphates.** R. Maurice and P. Martens *Ann chim anal chim appl* 17, 313-14 (1935). —In the method of C., the acid soln. of the phosphate is suitably neutralized and the Fe + Al pptd. as basic phosphates leaving the alk. earths and most of the  $\text{H}_2\text{PO}_4$  in the filtrate. The ppt. is dissolved in  $\text{HNO}_3$ , P is removed by treatment with  $\text{NH}_4\text{Molybdate}$  and the Fe and Al are pptd. with  $\text{NH}_4\text{OH}$ . Even after dissolving and reprecipitating the hydrated oxides of Fe and Al, the ppt. weighs too much owing to the retention of some molybdate. It is better to take the first basic acetate ppt., dissolve it in acid and reprecipitate in the same way. The ignited ppt. will then contain  $\text{Fe}_2\text{O}_3$  +  $\text{Al}_2\text{O}_3$  +  $\text{P}_2\text{O}_5$ . From this ppt. the Fe can be detd. volumetrically by the method of Zimmerman-Reinhardt and the Al by adding pure NaOH to the acid soln., filtering off the  $\text{Fe}(\text{OH})_3$  and detg. the Al in the filtrate as phosphate.

**Hymolal test for potassium.** E. A. Wildman *Proc Indiana Acad. Sci.* 44, 121-3 (1934). —Hymolal ("Gardinal", "Drene", "Dref"), a new detergent which appeared on the market in 1933, is said to be a mixt. of Na alkyl sulfates in which the alkyl radicals are those of the alcs. formed by the reduction of coconut-oil fatty acids. Such alkyl sulfates of Li, NH<sub>4</sub>, Mg, Na and Ca are readily sol. in  $\text{H}_2\text{O}$  at 20°. The K salt is only slightly sol. A 3% soln. of the Na hymolal salt has been found to be an excellent reagent for the qual. detn. of K. The procedure is: Divide the filtrate from group IV into 3 equal parts. Use 2 of the portions for the Mg and Na test before making the K test. Remove  $\text{NH}_4$  salts as usual from the third portion. Take up the residue in about 15 cc. of  $\text{H}_2\text{O}$  that contains a few drops of  $\text{AcOH}$ . Filter if not clear. To 5 cc. of the unknown soln. add 1 cc. of a 3% soln. of the hymolal salt reagent. A white ppt. within 1 min. indicates K. If no ppt. is obtained or if it were previously found that Mg is present cool the test tube in ice  $\text{H}_2\text{O}$ . A significant amt. of K forms a ppt. within 1 min. unless there is a very large quantity of Mg. In the latter case use the cobaltinitrite test.

**A rapid photometric method for the determination of silicon in light metals.** Hans Pusch *Z. Metallkunde* 27, 107-14 (1935), cf. C. A. 29, 3937. —A photometric or colorimetric method is developed whereby from traces to

2% of Si can be accurately detd. in Al or Mg in 15-20 min. Al is dissolved in NaOH, reducing agents are removed by  $\text{H}_2\text{O}_2$  in alkali and then by  $\text{KMnO}_4$  after acidifying with  $\text{HCl}$ . The residual  $\text{KMnO}_4$  color is removed with  $\text{H}_2\text{C}_2\text{O}_4$ . The greenish yellow soln. produced by addn. of  $(\text{NH}_4)_2\text{MoO}_4$  is then compared with suitable water or  $\text{CaCrO}_4$  standards in a colorimeter or photometer. Less than 1% Fe, Zn, Cu, Mn and traces of Sn, Pb, Ti, Cr, V, S and Na do not interfere, and methods of handling larger amts. of Fe, Cu and Ni are given. (Special standards must be used for Al alloys contg. appreciable amts. of Mg.) Ni vessels are recommended. Dil.  $\text{HNO}_3$  is used as a solvent for Mg, otherwise it is treated the same as Al.

G. Derge.

**The determination of very small amounts of uranium, and the uranium content of sea water.** Friedrich Herreger and Berta Karlik. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse Abt. IIa*, 144, 217-26 (1935). —A spectrograph and microphotometer were used to measure the fluorescence of very small amts. of U with NaF. As little as  $10^{-10}$  g. was detd. The method was applied to the detn. of U in sea water;  $0.30-2.3 \times 10^{-6}$  g. U per l. was found.

G. M. P.

**Determination of zinc in copper alloys containing less than 0.5% zinc.** Christo Nikolov. *Przemysl Chem* 19, 137-9 (1935). —Of Al filings 0.6 g. is quite sufficient to displace 1 g. Cu from soln. The soln. should contain 8 ml.  $\text{HCl}$  (1.1 g. per 100 ml). The pptn. should last less than 1 hr. (Sn if present should be removed first by dissolving the sample in  $\text{HNO}_3$ .) Tests in which quant. addns. of Zn were detd. showed this method to be accurate.

A. C. Zachlin.

**Combination of catalysts to reduce digestion time in the determination of nitrogen. II. Dairy products.** Charles I. Poe and R. R. Schafer. *J. Dairy Sci.* 18, 733-39 (1935). — $\text{H}_2\text{O}_2$  when used with Cu, Hg and Se (separately or in combination) reduces the digestion time somewhat and does not impair the accuracy of the detn. The combination of the 3 catalysts showed a reduction in digestion time from 105 to 13 min. compared to that of the Gunning method. When  $\text{H}_2\text{O}_2$  was used with these 3 catalysts the time was reduced to 10.7 min.

P. D. A.

**The iodine question in Westphalia.** Rudolf Balks *Landw. Jahrb.* 81, 939-1002 (1935). —The detn. of I in soils, water, spinach and milk are outlined. The I contents of a no. of minerals from which the soils originated varied from 0.420 p. p. m. (rag limestone) to 0.425 p. p. m. (graywacke) with an av. for the 23 rocks of 1.450 p. p. m. Those materials resulting from the weathering of the rocks showed without exception a higher I content than the original material. The I content of the soil examd. varied from 1.460 p. p. m. to 19.300 and av. 3.765. This was considered high for the heavy rainfall of this district. A meadowland with about 50% higher humus than a plowed land had an I content about 25% higher than the plowed land. The I content of the water varied from 0.00012 to 0.0196 p. p. m. and milk from 0.281 to 0.90 p. p. m. No correlation was found between the I accumulated in beets, potatoes, white cabbage or carrots and the I content of the soil. A higher accumulation of I was found in the leaves of potatoes, beets and carrots than in the other parts of the plant. Spinach took up more I than any other plant, and the accumulation of I was proportional to the I content of the soil. I fertilization increased the I content of spinach. It was established that the soly. of the I was higher in light soils than in heavy soils.

K. C. Beeson.

**Potentiometric determination of the iodine value.** Karol Drewski. *Przemysl Chem* 19, 63-75 (1935). —The app. consists of a glass H<sub>2</sub> vessel into the bottom of 2 legs of which there are sealed Pt wires which connect to the potentiometer. Equal amts. of standardized oxidizing or reducing soln. are placed in each leg of the vessel. In one leg this is then covered with a soln. of the oil in  $\text{CCl}_4$ , and in the other with solvent alone. The solns. are mixed and the p. d. between the 2 legs is read after 5 or 10 min. From that value of the oxidation-reduction potential the corresponding I value is then found on stand-

ardured curves. Solns. of BrI and ClI show a p. d. of 100 mv. when 120 mg. of linseed oil is introduced and involve an error of about 1-1.5%. The precision of this method is not inferior to that of the method of Hannus. Temp. has but little effect on the results. A. C. Z.

**Determination of barium fluosilicate in insecticide powders.** A. Bonis. *Ann. fals.* 28, 461-3 (1935).—Calc. of the Ba fluosilicate from the detn. of F generally gives greatly erroneous results. The following method has been found satisfactory. Fuse 0-5 g. of sample with 8 g. of a 1:1  $\text{Na}_2\text{CO}_3\text{-K}_2\text{CO}_3$  mixt., take up in boiling water, filter and wash the residue (R); digest the alk. filtrate at gentle heat for several hrs. after addn. of 4-5 g.  $(\text{NH}_4)_2\text{CO}_3$ , filter the flocculent  $\text{SiO}_2$  ppt. ( $\text{S}'$ ), to the filtrate add a few tenths of a g. of  $\text{ZnO}$  dissolved in  $\text{NH}_4\text{OH}$ , evap. nearly to dryness on the water bath, take up in hot water, filter, dissolve the residue in HCl, evap. to dryness, take up in dil. HCl, filter and wash the 2nd  $\text{SiO}_2$  ppt. ( $\text{S}''$ ), to the filtrate from the  $\text{NH}_4$  zincate treatment add 1 drop of helminthine, make just acid with dil. HCl and then faintly alk. with 1-2 cc. of 10%  $\text{Na}_2\text{CO}_3$ , heat to boiling, add sufficient  $\text{CaCl}_2$  to ppt. F and the excess of  $\text{Na}_2\text{CO}_3$ , boil till the ppt. is granular, filter, wash, transfer to an evap. dish, digest for a few mins. on the water bath with a few cc. of 10%  $\text{AcOH}$ , filter, wash, dry, ignite and weigh; calc. Ba fluosilicate from the wt. of  $\text{CaF}_2$ . As a check on the results dissolve R in dil. HCl, evap. to dryness on the water bath, treat for a few mins. in the cold with cold HCl, dil. with boiling  $\text{H}_2\text{O}$ , filter, wash, ignite and weigh the 3rd  $\text{SiO}_2$  ppt. ( $\text{S}'''$ ), total  $\text{SiO}_2 = \text{S}' + \text{S}'' + \text{S}'''$ . If the original product was free from  $\text{SiO}_2$ , the result should correspond to the F detd. as above. If other  $\text{SiO}_2$ -contg. materials (tak, clay, kieselsuhr, etc.) are present, in the filtrate from  $\text{S}'''$  ppt. Ba by addn. of 10%  $\text{H}_2\text{SO}_4$  to the boiling soln., filter, wash, ignite and weigh. The Ba thus detd. should correspond to the F detn. A. Papineau-Couture.

**Volumetric determination of carbonyl compounds.** I. Siege Hahnel. *Swiss Chem. J.* 47, 235-48 (1935).—Bismuth methods (acidimetric, alkalimetric or iodometric) are unsatisfactory for acetaldehyde, acetone, etc. Hydrazine can be used (cf. Olsender, C. A. 22, 344) but a better indicator than has been used must be found. The influence of acetyl on analytical reactions has been too often neglected. A. R. Rose.

**Simplified alkalimetric determination of phosphoric acid.** Robert Heubtrum. *Phosphorsäure* 5, 638-40 (1935).—A comparison of the Neumann method and the Bongartz method as modified by Glassmann gave concordant results. K. C. Boeson.

**Chemico-toxicological study of carbon tetrachloride.** Frido Caravino. *Boll. chim. farm.* 74, 741-8 (1935).— $\text{CCl}_4$  is detected by a method similar to that used by Viali (C. A. 6, 2438) for the detn. of  $\text{CHCl}_3$  and chloral. Expts. in which guinea pigs ingested  $\text{CCl}_4$  showed that its presence could be proved by this method in the viscera 12 days after death. The test is sensitive enough to detect 0.0035 g.  $\text{CCl}_4$  in 50 cc. alc. and can be used for detn. by condensing the product of H combustion, passing it into  $\text{NH}_4\text{OH}$ , acidifying with  $\text{HNO}_3$ , and titrating with  $\text{AgNO}_3$ . Helen Lee Gruehl.

**Chemical detection of arson attempts.** Hermann Schmidt-Hebbel. *Pharm. Zentralbl.* 76, 673-5 (1935).—Some general suggestions are offered looking to the possible detection of chemicals or other substances which may have been used in attempts at arson. W. O. E.

**Studies in the field of organic analysis. I. Determination of sulfur in certain organic compounds.** N. N. Mel'nikov. *J. Gen. Chem.* (U. S. S. R.) 5, 839-40 (1935).—Heat 0.2-0.5 g. of the substance with 10-15 cc. of  $\text{H}_2\text{PO}_4$  (d. 1.7). To the hot mixt. add gradually, with stirring, 1.5-4 g. of finely powd.  $\text{KMnO}_4$  (explosions sometimes take place). After 10-15 min. remove excess of  $\text{KMnO}_4$  with  $\text{H}_2\text{O}$ . Dil. the mixt. with 300-400 cc. of water, add HCl and det. the  $\text{H}_2\text{SO}_4$  with  $\text{BaCl}_2$  as usual. The method is not suitable for the detn. of S in volatile compds., thiocyanates and heterocyclic S compds., difficultly oxidized. II. Determination of copper in organic compounds. N. N. Mel'nikov. *Izv.* 841-2.—Proceed with the treatment of a sample with  $\text{H}_2\text{SO}_4$  and  $\text{KMnO}_4$ , as in the detn. of Ti (cf. C. A. 29, 814). After 15-20 min. add 20-5 cc. of water and remove the excess of  $\text{KMnO}_4$  with oxalic acid or  $\text{H}_2\text{O}_2$ , boiling some time if the latter is used. Cool, dil. with water to 100-230 cc., add 0.5 g. of KI, 20-5 cc. of 10%  $\text{KSCN}$  and titrate with  $\text{Na}_2\text{SO}_3$  in the presence of starch soln. by Bruhn's method (C. A. 12, 2263; 13, 939, 15, 351). The method is accurate to 0.15-2%. Chas. Blanc.

**Determination of diolefins by the hydrogenation method.** B. A. Dolgoplosk. *Soviet. Khimika* 1935, No. 5, 11-18.—The detn. was made in a special app. by passing the gas contg. butadiene diid. with H (27-30 cc. of gas to 94-100 cc. of mixt.) over a catalyst and observing the change in vol. The catalysts used were 50% Ni or 30% Pd pptd. on asbestos, preferably the latter. The time of hydrogenation over Ni or Pd in the cold was 5-7 min., for the total analysis 12-15 min. In the presence of 0.03% CO the catalyst became poisoned unless it was heated. Butadiene was detd. by this method in different products obtained in the manuf. of synthetic rubber from alc. and good results were obtained. A. Pestoff.

**Microvolumetric determination of methoxyl.** David T. Gibson and Thomas H. Canfield. *J. Chem. Soc.* 1935, 1419-20.—Viebeck and Schwappach (C. A. 25, 475) devised an iodometric method for detg. methoxyl and Viebeck and Brecher (C. A. 25, 896) based a micro-method upon it which is now modified as follows: As absorbent for  $\text{MeI}$ , mix 0.5 cc. of  $\text{Br}_2$  with 18 cc. of 20%  $\text{KBr}$ , reserve half of the soln. for a blank test and place the rest in absorption tubes. After the absorption, add 45 cc. of 25%  $\text{KAcO}$  and remove excess  $\text{Br}_2$  by treating with 1 cc. of 85%  $\text{HCO}_2\text{H}$ . Then add 2 cc. of 10% KI and 25 cc. of 5 N  $\text{H}_2\text{SO}_4$  and titrate the liberated  $\text{I}_2$  with  $\text{Na}_2\text{S}_2\text{O}_3$ . C. J. West.

**Effect of temp. on the bacterial ammonification of urea (Tandon) 15.** Triple nitrates of the rare earths and urea for Cs (Goswami, Sarkar) 6.

**Quantitative spectrum analysis by discharge apparatus.** W. C. Heraeus G. m. b. H. Ger. 589,633, 615,185 and 613,186, May 15, 1935 (Cl. 42, 348).

## S—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND J. F. SCHAEFER

**Bolivian minerals.** V. Franckite. Friedrich Ahlfeld, Hans Himmel and Willi Kleber. *Zentr. Mineral. Geol.* 1935A, 292-8; cf. C. A. 29, 6339.—Crystallographic. Franckite is orthorhombic, pseudo-tetragonal.

**Strengite from Pierstein.** Hans Himmel and Robert Schroeder. *Zentr. Mineral. Geol.* 1935A, 289-92.—Crystallographic. Michael Fleischer.

**Kaolin and clay.** Their difference in structure and ease of formation. Albert Vassel. *Keram. Rundschau* 43, 457-60 (1935).—Weathering of feldspar leads either to kaolin

or clay, the formulas assigned being  $6\text{Al}_2\text{O}_3 \cdot 12\text{SiO}_2 \cdot 9\text{H}_2\text{O}$  and  $2\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot 3\text{H}_2\text{O}$ , resp. The predominance of clay is due not so much to increased weathering leading to finer subdivision as to the greater ease of formation of the smaller mol. Clay and kaolin were heated at  $180^\circ$  and then subjected to 20% boiling caustic. From the resistance to soln. it was concluded that clay decomposes to muscovite and kaolin to metakaolin. Thirty-two references. P. S. Roller.

**Three kaolins.** D. Belvankin and V. Ivanova. *Zentr. Mineral. Geol.* 1935A, 298-308.—Normal kaoline and



fibrous hydrokaolin from Saghal, Transcaucasia, and halloysite from Awdyrt, Ural, were analyzed and examined optically. Heating curves taken with a temp. increase of 6°/min showed minima at 530-600°, corresponding to the loss of constitutional water, and maxima at 930-960°, corresponding to the exothermic formation of mullite. The optical properties of dehydrated samples indicated the following stages in the dehydration: (1) loss of adsorbed water, (2) loss of water of constitution (300-400°), (3) formation of  $\gamma$ - $\text{Al}_2\text{O}_3$  and (4) reaction of  $\gamma$ - $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  to form mullite.

**Ore deposits—they follow the syndines.** James Bichan *Can Mining J.* 56, 523-5 (1935). W. H. B. A concretionary sulfide deposit at Culm near Haselbach in the Riesengebirge. Adolf Posenenke. *Zentr. Mineral. Geol.* 1935A, 309-10.

**Organic mineral substances.** V. Porphyry in coals. Alfred Trebs *Ann.* 520, 144-50 (1935); cf. *C. A.* 29, 4708<sup>+</sup>.—Examination of the 7 and 20% HCl exts. of various coals show the presence of deuterio-, meso- and desoxyphyllerythron- and etio-porphyrins; porphyrins were seldom present. No coporphyrin was found in various samples of coprolites.

**Geochemical constants of iron.** A. E. Fersman *Uspekhi Khim.* 4, 731-55 (1935).—The at. no., the valences and the ionic radius of Fe are considered in relation to other elements, with the corresponding relations of physicochem. properties. The various chem. compounds which can be formed by replacement and migration under the conditions found in nature are discussed from the standpoint of their relative stabilities, or solubilities in

water and weak acids, etc., and applied to the conditions of formation of Fe ores.

F. H. Rathmann

**Occurrence of selenium in the Colorado River and some of its tributaries.** Kenneth T. Williams and Horace G. Byers *Ind. Eng. Chem., Anal. Ed.* 7, 431-2 (1935).—Analyses of 23 samples of water from the Colorado River, its tributaries and drainage ditches, showed Se contents varying from 0 to as much as 2 (S p p m). The sources of the Se in drainage waters were traced to certain soils, shales and alk. crusts. One of the salt crusts contained as much as 260 p p m of Se. The data together with field observations indicate clearly the possibility of improvement of sclerosed land by irrigation and drainage, where such management is practicable.

M. S. Anderson

**The content of heavy water in the water of crystallization of minerals.** H. E. H. Riesenfeld and M. Tolmanc *Ber.* 63B, 1962-9 (1935); cf. *C. A.* 29, 41<sup>+</sup>.—The  $\text{H}_2\text{O}$  of crystal of polyhalite from Stassfurt, a primary mineral was found by the float method to contain 163.9 mols  $\text{D}_2\text{O}$  per 10<sup>4</sup> mols  $\text{H}_2\text{O} + \text{D}_2\text{O}$ . That of gypsum from Sanb. Harz, a secondary mineral, contained 164.1. These values are lower than the values (185-225) reported by other workers for natural waters and minerals. R. and T. suggest that the  $\text{D}_2\text{O}$  content of the  $\text{H}_2\text{O}$  of crystal of minerals does not differ from that of natural waters because the speed of crystal formation is slow as compared with the speed of exchange of  $\text{D}_2\text{O}$  and  $\text{H}_2\text{O}$  between the crystal and liquid phases.

John E. Milbery

Au in Canada (Danlour-Dumesnil) 9

## 9—METALLURGY AND METALLOGRAPHY

D. J. DEMOREST, OSCAR E. HARDER AND RICHARD RIMBACH

**Metallurgical developments and the mining industry.** L. Sanderson *Can Mining J.* 56, 526-7 (1935).—A brief review of metallurgical developments during the past year. Mn steels, welding high-Mn steels, highly alloyed cast irons, alloy steels, stainless steels and wires, Ni-Mo, Mn-Mo and special C steels are discussed. W. H. B. **The Nundydroog Mine.** H. E. Jeffery. *Mining Mag.* 53, 265-77 (1935).—Operations on this mine on the Kolar goldfield, Southern India are described. The gold-bearing reef is believed to be a derivation of the earliest of a series of granites intruding a belt of hornblende schists. The flow sheet of ore treatment shows sixty 1250-lb. Californian stamps and blankets. The classification plant for the tailings consists of one primary and one secondary Caldecott cone for each of the 5 tube mills, where the ore is ground to about 75% minus 200 mesh. The overflow of secondary cones after agitation for 10 hrs is transferred to the Butters filter plant. The filtrate passes through sand clarifiers to extractors, boxes filled with Zn shavings. The stamp-mill and tube-mill concentrates are stamp milled in 850-lb. stamps with screens of 1200 holes per sq. in. and passed over amalgamating plates and James conc. tables, the heads of the latter are burnished and amalgamated on a latex, while the tailings are returned to the main current. The extn. by stamps, tube mills and cyanidation is 55, 31 and 12%, resp., or an over all extn. of about 88%.

**The Meridian concentrator.** J. P. Dick. *Can Mining J.* 56, 509-13 (1935).—A description of the flotation concentrator of the Meridian Mining Co., Ltd. at Camborne, B. C. A flowsheet is included. W. H. Boynton

**Treatment of precious-metal mat.** C. C. Downie. *Metallurgist* 13, 21-2, 25 (1935).—The treatment consists of a sulfate roast, leaching with 2%  $\text{H}_2\text{SO}_4$ , and pptn. of Ag with Cu.

**Gold in Canada.** H. Maurice Danlour-Dumesnil *Rev. ind. minerale* No. 356, 499-506, No. 357, 523-30 (1935); cf. *C. A.* 29, 5387<sup>+</sup>.—The geology and the working of the mines at the 3 principal centers, Porcupine and Kirkland Lake (Ontario) and Noranda (Quebec), are described.

C. D. West

**Siemensite and its application in the basic open hearth furnace.** E. Greiner. *Rev. universelle mines* 11, 502-6 (1935).—Operating data with Siemensite, composed of 20-40%  $\text{Cr}_2\text{O}_3$ , 25-45%  $\text{Al}_2\text{O}_3$ , 18-30%  $\text{MgO}$  and 8-14% other constituents, are given. The material is obtained by reducing fusion in an elec. arc furnace and cast in 42 molds. The heat resistance is higher than Seger cone 42, sp. gr. 3.2-3.4, it softens under 2 kg./sq. cm. pressure above 1800°. Comparisons with other refractories are compiled in tables.

M. Hartenbaum

**New oil fired assay furnace combines roasting, fusion and cupellation.** G. B. O'Malley. *Chem. Eng. Mining Rev.* 28, 491-2 (1935).—An illustrated description.

W. H. Boynton

**Waste-heat boilers in open-hearth practice—2nd report of the open-hearth committee.** R. Percival Smith, et al. *Iron Steel Ind.* 8, 511-15 (1935).—Mechanically induced draft of the boiler frequently enables an increase in output in the open-hearth production. Capital expenditure, operating costs, steam costs at the main boilers and fuel savings are some of the factors that must be considered in deciding whether waste-heat boiler installations are justifiable.

C. B. Jenni

**Use of coke-oven and town gas in open-hearth furnaces.** J. B. R. Brooke. *Iron Steel Ind.* 9, 3-5 (1935).—Town gas and coke-oven gas analyses are similar except for the CO and methane content; the town gas has been "steamed" and shows a high CO and a low methane content. Either gas can be used in open-hearth furnaces if the cost is not too high. A luminous flame which is more readily controllable and which radiates heat to the bath is used in open-hearth practice. Producer gas gives a luminous flame from the incandescent particles of C liberated by the tar and benzene in the gas at 700-800°. Tar adds, are necessary when burning coke-oven gas to give the luminosity. Since coke-oven gas is about 3½ times as rich as producer gas the checker chambers must be considerably reduced in size and well insulated. Thus preheating may crack the methane and block up the small checkers. The H<sub>2</sub> would also make the flame difficult to control. Continental experience has found that the best

results are obtained by using a gas mixt. with 25-30% coke-oven gas, the remainder blast-furnace gas. The gases must be thoroughly mixed to prevent striation. The coke-oven gas should not be below 25% of the mixt. and may be as high as 40% during the melting down period. The mixt. should be preheated as high as possible; 1150-1250° will suffice. The moisture content of the gases must be kept low. A reduction of gas port area of 27 1/2% gives better results than the area originally designed for producer gas. Furnace life has been increased, better fuel consumption secured, less oxidation of the bath secured and furnace operations are more closely controllable when mixed gases are used. High CO and low methane content of town gas would give a very low luminosity. The calorific value of the gas is also lower than coke-oven gas. This would not be suitable for mixing with blast-furnace gas, but when tar is added town gas could be used, provided the cost is not excessive. C. B. Jenni.

Modern foundry practice W. G. Morgan. *J. Inst. Production Engrs.* 14, 480-94 (1935) M. W. Schwarz.

Production of cupola malleable castings. F. B. Riggan. *Trans. Am. Foundrymen's Assoc.* Preprint No. 35-19, 11 pp (1935).—Malleable Fe melted in a cupola is adapted to the continuous production of light castings and has higher C and lower strength than malleable Fe melted in an air furnace; high fluidity and low shrinkage, 48,000 lb per sq. in. tensile strength, 34,000 lb per sq. in. yield strength and 9% elongation. The operating details and tech. control necessary to produce uniformly satisfactory castings are described. Frank G. Norris.

Notes on roll failures with reference to chilled iron cast rolls containing chromium and molybdenum Selwyn Caswell. *Foundry Trade J.* 53, 360-71 (1935) D. S.

Metallurgical research at McGill Alfred Stansfield. *McGill News* 16, No. 4, 27-31 (1935).—Results are reported of investigations on a penetration of Au, Ag, Pt and Cu into Pb cylinders; electrolytic production of Mg and of Cl; elec. smelting of Zn and Fe ores, elimination of S, softening points of mixts. of SiO<sub>2</sub>, CaO, TiO<sub>2</sub> with Al<sub>2</sub>O<sub>3</sub>; and the production of TiO<sub>2</sub> pigments. J. C. Munch.

Crystal orientations in a nickel casting and their alteration by rolling and recrystallization. G. Tammann and F. Laass. *Z. Metallkunde* 27, 84-8 (1935).—A 4-cm. Ni cube was cut from a casting of the same cross section and orientations were detd. by observation of points of max. light reflection, cubic, dodecahedral and octahedral types were distinguished, the last being further divided into [111] and [211] groups. Orientation distributions are detd. on the casting for faces parallel and perpendicular to the cooling surface, on cold rolled samples cut from the casting, and on these specimens when recrystd., and correlated with similar information on Al and Cu. On the castings [111] surfaces predominate near the cooling face, [100] 12 mm. from the surface, similar to Al, and indicating most rapid grain growth in [111] direction when heat dissipation is fast, in [100] direction when slow. However, Cu showed [100] orientation. Changes in orientation during rolling are the same as those for Al and Cu. With increasing reductions the fraction of octahedral types increases and alignment of these in [111] direction occurs. However, up to 20% reduction Ni showed an anomalous decrease of the octahedral type and increase of cubic orientations, which cannot be explained by slip on [111] planes in the [111] direction. Samples which had been reduced 33% recrystd. at 750°, while recrystn. occurred at 650° after 90% reduction. In both cases grains were too small to permit orientation detns. until after heating to 1300° when octahedral surfaces predominated, as in Al, whereas Cu showed mainly [100]. G. Derge.

Cold rolling L. Weiss. *Z. Metallkunde* 27, 73-5 (1935).—It is shown that by the use of 2 arbitrary factors which were developed previously (*Z. Metallkunde* 20, 280 (1928), 23, 47 (1931)), and which evaluate the effect of dynamic working during the rolling process and of initial thickness of the material upon yield point of the material, it is possible to est. the resistance of the material to deformation. By this means the effects upon rolling

pressure of the roll diam. and of friction between rolls and material are calcd. and shown to be in agreement with exptl. values. A graph is developed which relates possible reduction in thickness to roll diam., original thickness of the material, and frictional conditions. The limitations placed upon rolling pressures by elastic deformation of the rolls are described and related to possible reduction of thickness. G. Derge.

A research into tests for materials used in cold-pressing operations, with special reference to the fluid-pressure cupping test. H. J. Gough and G. A. Hankins. *J. Roy. Aeronaut. Soc.* 39, 1047-80 (1935). M. W. Schwarz.

The apparent increase in the plasticity of metals under the action of reversible twisting. V. D. Kuznetsov. *J. Exptl. Theoret. Phys. (U. S. S. R.)* 5, 550-5 (1935).—Iron rods on reversible twisting break at a tension of 2.5 kg./sq. mm. without increase in length but at 25-30 kg./sq. mm. ordinarily with a relative lengthening of 23 to 29%. F. H. Rathmann.

The characteristic behavior of the elasticity modulus of ferromagnetic materials M. Kersten. *Z. Metallkunde* 27, 97-101 (1935).—The theory of magnetic domains, as developed by Weiss and Heisenberg, is described simply, especially with regard to magnetostriction. It is shown that the deviations from Hooke's law which occur in ferromagnetic materials, even in the absence of an external magnetic field, may be explained upon this basis. Likewise the theory explains the experimentally detd. variations of the elasticity modulus of Ni and their dependence upon cold work and external field. By applying the theory to the temp. coeff. of the elasticity modulus, the change found at the Curie point for Ni is explained, and a reasonable picture is provided for the behavior of Elmvur.

Crystallization in binary systems with eutectics. E. Raub. *Z. Metallkunde* 27, 77-83 (1935).—The grain size and structure in binary systems were observed by metallographic methods. It was found that these are both detd. by the characteristics of the primary constituent, but are also influenced by other factors which affect pure metals, such as size and shape of the casting, pouring temp. and rate of cooling. Expts. have shown that the structure has little effect on the mech. properties when the constituents have widely varying properties, but the structure is determinative when the components have similar properties. With 83% Ag-Cu alloy as an example of the latter type it is shown that recrystn. and deformation processes are similar to those in pure metals and solid solns., the distribution of phases having little importance. For this alloy it is shown that cold rolling leads to anisotropy, as in pure metals, as does subsequent recrystn., both effects being strongest at high reduction and high annealing temp. Demonstrated by data on extension and tensile strength, the first shows a max., the latter a min. in test foils cut 45° to rolling direction. G. Derge.

The preparation of thin wires by freezing liquid metal jets G. Tammann and G. Moritz. *Z. Metallkunde* 27, 114-15 (1935).—By forcing low-melting metals through conical nozzles 0.04 to 0.1 mm. in diam., liquid jets 20 to 100 cm. long, resp. were formed. These solidified into wires which showed a variation in diam. of about 3%. Pressures of 3 to 5 atm. were used. With lower pressures, or liquids more than 40° above the m. p., the jets tended to break into droplets because of the action of surface tension. Smooth wires were obtained from Sn, Bi, Zn, Pb, Al, and the binary eutectics of Bi, Cd, Pb and Zn. Rough surfaces resulted from Cd and Sb. Good jets could not be obtained with Ag or Cu with pressures up to 13 atm. Wires of the pure metals contained single crystals up to 1 cm. long. G. Derge.

Determination of the heats of transformation of cobalt. H. v. Steinwehr and A. Schulze. *Z. Metallkunde* 27, 90-2 (1935).—See C. A. 29, 4253. G. Derge.

Some experiments with austenitic cast irons. P. A. Russell. *Foundry Trade J.* 53, 325-7 (1935). D. S.

Temper brittleness in cast iron and the influence of nickel and molybdenum. J. E. Hurst. *Foundry Trade J.* 53, 572-3 (1935) D. S.

Endurance limit of black heart malleable iron. E. G. Mahin and J. W. Hamilton. *Trans. Am. Foundrymen's Assoc.*, Preprint No. 35-2, 7 pp (1935).—Malleable Fe contg Si 1.00, C 2.44, Mn 0.31, S 0.072, and P 0.168% and having 55,340 lb per sq. in. tensile strength, 35,600 lb. per sq. in. yield point, and 18.6% elongation had an endurance limit of 20,600 lb. per sq. in. and an endurance ratio of 0.54. The few previously reported values for similar material are approx 25,000 lb. per sq. in. The high value for such unsoftened material as malleable Fe is attributed to the good resistance of the metallic phase to repeated stresses, which resistance is not impaired by the graphite nodules because of the absence of sharp projections thereon. The special polishing technic necessary to maintain the true outline of the graphite nodules is described.

Irank G. Norris  
Definitive investigation for the good preservation of older types of iron. K. Daevs. *Naturwissenschaften* 38, 653-6 (1935).—D. thinks, reasoning from recent corrosion expts., that the good corrosion resistant properties of older irons are due (1) to relatively high P and Cu contents, (2) to the protective layer formed after long use in contact with human skin or by annealing in lard oil or (3) to the former purity of the atm. under which conditions a very closely adhering protective film is formed.

H. A. Smith  
Controlled atmosphere—applications to heating of steel. T. B. Bechtel. *Iron Steel Engr.* 12, 1-4 (Nov. 1935).—An illustrated discussion.

W. H. Boynton  
Controlled atmospheres in steel treating. H. W. Gillett. *Metals & Alloys* 6, 195-203, 204-7, 235-40, 293-8, 323-7 (1935).—A discussion of the character, cost and action of different usable gases with a description of the furnaces in which to apply controlled atm., and a final article on "Correlation of Experiments and Experiences." The articles are profusely illustrated with photographs of furnaces and equipment for producing gases. 13 light-six references.

Downs Schaaf  
Thermal treatment of alloy tool steels. R. Saxton. *Heat Treating and Forging* 21, 515-16, 521 (1935). D. S.

Some factors relating to the strength of steels. Georges Delhart. *Rev. ind. minérale* No. 357, 607-22 (1935).—A review. The effects of varying chem. compn., thermal and mech. treatments on the strength and microstructure of steel are discussed and illustrated by numerous figures and photomicrographs.

C. D. West  
Acceleration of structural reaction in steel by mechanical constraint. Pierre Chevenard and Xavier Waché. *Compt. rend.* 201, 877-9 (1935); cf. *C. A.* 29, 6551. Samples of a Cr-Mo-V steel (C 0.1, Cr 7.0, Mo 0.7 and V 0.2%) air-tempered and reheated for 1 hr. at 675° and then subjected to loads of 2.5-40 kg./sq. mm. at 450°, 500°, 550° and 600° were then subjected to an ordinary tensile test at the ordinary temp. (a), after which the resistivity (b), residual magnetism and coercive field (c) and dilatation (d) were detd. and micrograph exam (e) was carried out. (a) shows a distinct softening increasing as the temp. at which the sample was loaded and the load itself was increased. As (b), (c) and (d) showed no physicochem. change this must be due to structural change, i. e., a coalescence of the carbide, though not scarcely visible in (e). Such a steel would only be satisfactory for loads not exceeding 8 kg./sq. mm. and temp. not above 600°.

C. A. Silberrad  
Elastic limits of some structural steels. Georges Delhart and Hervé Francelle. *Rev. ind. minérale* No. 355, 463-72 (1935).—Elastic limits are detd. for various alloy steels (Mn, Ni, Cr-Mo, Cr-Mo-V) after various thermal treatments.

C. D. West  
"Electrical" transfer of carbon in solid steel. W. Seith. *Z. Elektrochem.* 41, 541-6 (1935).—The diffusion of atoms in substitutional and interstitial solid solns may primarily be a function of the differential ionization of the atoms involved in the solid soln. This idea is derived from the electron gas concept of the structure of metals. In the Pd-H system the elec. transfer of H ions in the Pd has been definitely shown. Similarly the transfer of Au ions in a Au-Pb alloy (0.02% Au at 200°) has been less

well established. In the present investigation Arco wires 0.15 cm. in diam. were used. A Cu covering 0.05 mm. thick was plated on a wire 30 cm. long in cones 1 cm. long sepd. by bare wire zones of 2-3 cm. long. These wires were then carburized in city gas for 2 hrs. at 670°. The C content of these sections of the wire not protected by Cu averaged 1%. The wires thus prepd. were then heated *in vacuo* in a glass tube at about 1000°. Under these conditions expts. were made where (a) no current was passed through the wire, (b) a.c. was passed and (c) where a d.c. was passed through the wire for 250-500 amp hrs. Photomicrographs and elec. cond. measurements were made to det. the transfer of C. The relation between the diffusion const.  $D$  and the ionic molality  $Bu$   $D = BRT/nF$ , where  $R$  is the gas const.,  $T$  the abs. temp. and  $F$  the electrochem. equiv.  $D$  was detd. from a-c. measurements of resistance to be 0.083 sq. cm./d. By use of this value of  $D$  and the assumption of singly ionized C atoms,  $B$  is calc'd. to be 0.825  $10^{-4}$  cm./sec. in a field of 1 v./cm. Values of  $B$  calc'd. from cond. and max. of  $C$  moved are 1.6 and 2.2  $10^{-4}$  sq. cm./sec. for the same field. This indicates that, on the av., the C ion is more than singly ionized.

H. A. Smith  
Measurement of thermoelectric forces of some alloys at temperatures from 25° to 17.5° absolute. W. H. Keesom and C. J. Matthijs. *Physica* 2, 623-32 (1935).—The thermoelec. force was measured relative to a normal Ag alloy wire of Ag-Au alloys and alloys of Au with Fe, Co, Ni, Mn or Cr at temps. from 25° to 17.5° abs. In most of the Au-Fe and Au-Ni alloys a neg. max. appears at lowest concn. of admixt. Tables and curves are given for each combination. For practical use in thermometry at liquid He or H<sub>2</sub> temps. the alloy of Au with 1 or 2% Cu against normal Ag + 0.37 at % Au gives the largest thermoelec. force; the Au + 1.87 at % Fe alloy is likewise useful. All alloys tested give neg. potential against the Ag alloy normal. B. J. C. v. d. H.

Surface preparation and painting of magnesium alloys. A. W. Winston, J. H. Reid and W. H. Gross. *Ind. Eng. Chem.* 27, 1333-7 (1935).—Data on preferred procedures for obtaining proper paint adhesion and protection against sea water are given. The HNO<sub>3</sub>-K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> pickle treatment and the effect of various factors in its operation and control upon subsequent paint adhesion are described. Mat. iridescent coatings are preferred to bright yellow ones. The effect of the compn. of primers and finishing coats, especially of inhibitory pigments such as Zn chromate, on adhesion and protection is discussed and typical satisfactory paint schedules are given. Leopold Fessel.

Recent developments in light-alloy pistons. H. J. Mabrey. *J. Inst. Production Engrs.* 14, 495-506 (1935).

M. W. Schwarz  
Oxide film of alloys containing small percentages of aluminum. Ichiro Itaka and Shizuo Miyake. *Proc. Imp. Acad. (Tokyo)* 11, 256-7 (1935); *Nature* 136, 437 (1935).—Cu alloys contg 2-30% Al show practically no oxide formation (scales) when heated for hrs. at red heat. Cathode ray photographs show that the alloy is protected by a film of Al<sub>2</sub>O<sub>3</sub>. William F. Vaughan.

Liquation phenomena in cast bolt and bar brass. Ch. Bernhoeft. *Z. Metallkunde* 27, 115-16 (1935).—A 55% Cu, 40% Zn and 2% Pb brass was cast into uncoiled cylindrical molds 180 mm. in diam. and 800 mm. long. Measurements showed that the Pb content and Brinell hardness were both low in the center of the middle section. A 64% Cu, 36% Zn brass was cast into both water-cooled and uncooled molds 600 X 65 mm. in cross section by 780 mm. high. The Cu content was low in the center at the top, middle and bottom of the casting.

G. Derge  
Hardenable copper nickel-tin bronzes. J. Sandast alloys. F. Petz. *Korrosion u. Metallschutz* 11, 217-29 (1935); cf. *C. A.* 29, 6074.—The field of Sn bronzes capable of improvement by heat treatment is reviewed and 28 references are given. A large no. of cast and casting alloys, contg. 0.25% Mn, 2% Zn, and up to 10% Sn and 40% Ni was investigated, mainly by Rockwell hardness measurements. A new  $\alpha$ -phase boundary is proposed for the system Cu-Ni-Sn, comprising a wide scope of improvable

alloys. All alloys within the limits of 10% Sn and 40% Ni could be rendered mechanically workable by homogenizing. The progress of hardening during tempering periods of 100 hrs. at various temps. was investigated on typical alloys of this group. A max. improvement effect was observed with about equal parts of Sn and Ni. The max. hardness values, contrary to the rate of hardening, were proportional to the degree of supersaturation. Alloys with sufficient supersaturation showed a close relation between the m. p. and the rate of hardening. Ni improved the mech. properties of bronze sand castings, especially the elongation and the proportional limit. With increasing excesses of Ni, the characteristics of Ni-Cu alloys were approached. Economic advantages of these alloys are seen in the improvement of the mech. properties by heat treatment and in the substitution of half of the Sn by Ni.

Leopold Pessel

The hardness of sprayed metal coatings. Kessler and Th. Everts. *Z. Metallkunde* 27, 104-7 (1935).—Sprayed metallic coatings of Zn, steel, V2A, Cu, Al and monel are discussed. The effects of temp. of the basis material, distance of spray from the material, gas pressures, types of spray aim, feeding speeds, and rates of coverage upon structure, scratch hardness, and Brinell no. are discussed and optimum conditions given.

G. Derge

Effect of alkaline detergents upon metals. Aluminum, copper, tin and zinc. Chester L. Baker. *Ind. Eng. Chem.* 27, 1358-64 (1935).—Strips of the metals were immersed in solns. of NaOH, Na<sub>2</sub>CO<sub>3</sub>, trisodium phosphate, Na metasilicate and 6 other Na silicate solns. of various SiO<sub>2</sub>/Na<sub>2</sub>O ratios. The loss of wt. after various periods of immersion at 60° in various concns. of the liquids was detd. and the surface attack observed. The silicates of soda impaired the surfaces less than the other alkalis considered, with the exception of the behavior of the Na<sub>2</sub>CO<sub>3</sub> soln. toward Zn. Among the silicates, an increasing ratio of SiO<sub>2</sub> to Na<sub>2</sub>O seemed to give greater protection. A method of obtaining a more siliceous silicate soln., and thus greater protection, by adding to Na metasilicate a less alk. substance, such as Na acid phosphate, is outlined.

Leopold Pessel

The possibility of preventing corrosion in hot-water systems. L. W. Haase. *Gesundh.-Ing.* 58, 621-4 (1935); cf. C. A. 29, 4496\*, 7263\*.—Factors responsible for corrosion in various types of hot-water systems and means of combating such corrosion are reviewed.

M. G. Moore

The corrosion of aluminum. J. M. Bryan. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 187-95 (1935).—The corrosion of Al by citric acid was studied for 14 days at 25° and for 3 days at 75° by immersing 3 X 1 in. strips for 1/4 of their length in 130 cc. of soln. in the presence of air; daily readings of the gas burets gave an indication of the type of corrosion occurring and, where necessary, a gas analysis was carried out at the end of the tests. In the absence of citric acid, a 2% NaCl soln. and a similar soln. contg. 25% sucrose had little or no effect in contact with Al, either at 25° or at 75°; in presence of citric acid, NaCl greatly acted as an inhibitor. A purified Al (99.5%) corroded more rapidly in 1% citric acid than an ordinary com. sample (99.2% Al), and an alloy contg. 3.5% Mg, 0.5% Mn and 0.7% impurities corroded at about the same rate as the purified Al; fine abrasion of the surface with emery increased the rate of corrosion about 10-13%. A study of the effects of variation in pH at 25° and 75° showed that NaCl has a much greater effect at 25° than at 75°; at 25° the rate of corrosion increased greatly with increase in H-ion concn., and was mainly of the oxidation type; at 75° corrosion was stimulated only below pH 3.0, there being inhibition at higher pH values and the corrosion being mainly of the H-evolution type over the whole pH range. At 25°, on varying the NaCl concn. from 0 to 3% and keeping the citric acid const. at 0.5%, there was marked stimulation of the H-evolution type of corrosion when the concn. of NaCl exceeded about 1%; when the NaCl concn. was kept const. at 1.5% and the citric acid varied from 0 to 2%, the H-evolution type of corrosion was practically a linear function of the citric acid concn. At 75° an increase in

the amt. of NaCl in unbuffered 0.5% citric acid soln. greatly increased the rate of corrosion; in solns. buffered to pH 4.0 with 1.5% of Na citrate, the effect was relatively small, there being a slight retardation of corrosion with concns. less than about 2% NaCl and a slight stimulation at higher concns. The relative rates of corrosion of Sn and Al in citric acid for 3 days at 75° were approx. the same for pH 4-5.5, but below pH 4 the corrosion of Sn was much faster for the limited period, being nearly double that of Al at pH 2.4; the corrosion of the Sn was mainly of the oxidative type and would cease when the O was exhausted, while that of Al was of the H-evolution type and would continue until the acid was neutralized. A study of the corrosion of Al by fruit exts. showed considerable corrosion at 75° by gooseberry and black currant, moderate corrosion by greengage, red currant, strawberry and raspberry; at 25° corrosion was relatively slow, and little or no H was evolved; the results indicate that factors other than H-ion concn. and total acidity influence the rate of corrosion. From results obtained with blackberries and cherries it seems likely that these fruits tend to form a film on Al at 75°, but this was not sufficiently protective to prevent the formation of H-swells in an actual canning test.

A. Papineau-Couture

Influence of certain organic hydroxy compounds on the corrosion of iron in oxygenated salt solutions. W. Stewart Patterson and R. C. A. Culbert. *J. Soc. Chem. Ind.* 54, 327-31T (1935). cf. C. A. 28, 6650\*.—The corrosion of mild steel strip contg. 0.15% C in 0.01 N K<sub>2</sub>SO<sub>4</sub> soln. was observed for a period of 340 hrs. At intervals, the specimens were weighed and the O<sub>2</sub> content of the soln. was detd. The corrosion product was Fe(OH)<sub>2</sub> and a thin, strongly adhering film of ferrous-feric oxide which furthered corrosion by differential aeration and was due to the reducing action of the metal. At a low O<sub>2</sub> concn., the rate of corrosion was directly proportional to it. At higher concns. the corrosion was more rapid and became ultimately const. and independent from further increases in the O<sub>2</sub> concn. In another series, lasting 72 hrs., erythritol and mannitol were added in varying amts. Up to a crit. concn. of these addns., corrosion was retarded apparently by adsorption of the inhibitor to the corrosion product, which thus became softer, less adherent, and therefore less conducive to differential aeration. At higher concns., beyond the crit. point, the inhibitor's retarding action was due to peptization of the corrosion product and further elimination of differential aeration. No evidence of inhibition due to adsorption on the metal surface could be observed. In other series, extending for 72 hrs., the effects of 5 inhibitors, i. e., mannitol, mesoerythritol, sorbitol, glycerol and glycol, were compared. The amt. of retardation was controlled by the quantity of inhibitor present, the no. of OH groups per mol. of inhibitor, and the space arrangement of the OH groups within the mol.

Leopold Pessel

Corrosion of wrought iron pipes by water. Wilhelm Rädker. *Gas- u. Wasserfach* 78, 793-4 (1935).—The conditions under which ferrous metal pipes are used are as important as the compn. of the metal. Analyses of the corrosion products are valuable in detg. the causes of corrosion. In one case, deposits from a pipe which had failed in a short time showed high carbonate content, because of the high CO<sub>2</sub> content of the water. In the absence of O<sub>2</sub>, deposits consist of equal parts of FeO and Fe<sub>2</sub>O<sub>3</sub>, in the presence of dissolved O<sub>2</sub> the latter predominates. A case of local corrosion of a pipe due to O is discussed.

R. W. Ryan

Development of specifications for protection of underground pipes. K. H. Logan. *Ind. Eng. Chem.* 27, 1354-7 (1935); cf. C. A. 28, 85\*.—The more important results of pipe-coating research are summarized and the essentials of an adequate protective coating are given. The need for quant. data on the properties of pipe coatings and for specifications for coatings suitable for various soil conditions is stressed. Such specifications can be developed from available and obtainable data.

Leopold Pessel

Effect of various inorganic substances on the corrosion

of mild steel by citric acid at about  $pH$  3.5 T. N. Morris *Dept Sci Ind Research, Rept Food Investigation Board 1934, 180* (1935) —Aq solns were prep'd contg 10 g citric acid and 5 g Na citrate per l, together with 0.01 and 0.05 M  $NaH_2PO_4$ , 0.01 and 0.05 M  $NaNO_3$ , 0.01 and 0.05 M NaCl, and 0.01 mg per l  $K_2Cr_2O_7$ , resp. Corrosion tests carried out on 3 X 1 in strips of steel at 25° for 3 days showed that  $Na_2HPO_4$  had a much greater buffering effect than any of the other salts. This effect was shown to be responsible for the diminished corrosion observed.

A Papineau-Couture  
The corrosion of tin J. M. Bryan *Dept Sci Ind, Research, Rept Food Investigation Board 1934, 177-9* (1935) —Expts were carried out to ascertain the effects of NaCl and sucrose, resp., on the rates of corrosion of Sn by solns of citric acid at 25° and 75° in presence of a limited amt of air. Addn of 2% NaCl had a slight inhibiting effect in corrosion, which was more pronounced on untreated specimens than on those in which the surfaces of the metal had been abraded with fine emery. Addn of 1% NaCl to a 0.5% citric acid soln of  $pH$  2.4 at 25° had no effect, but as the  $pH$  increased to 5.5 (by addn of Na citrate buffer) there was a slightly increasing inhibiting effect. Addn of 25% sucrose at 25° produced an appreciable inhibition over the whole  $pH$  range studied, the rate of corrosion being reduced to 1/2 at  $pH$  2.4 and to 1/2 at  $pH$  5.5. At 25°, addn of varying amts of sucrose to 0.5% citric acid soln progressively retarded the rate of corrosion as the concn increased. This inhibition seems to be due mainly to the effect of sugar in reducing the soly of air in the solns, since the corrosion (like that in plain citric acid solns) was entirely of the oxidative type. At 75°, concns of less than 15% sucrose inhibited corrosion, while greater concns stimulated it, the latter effect, however, may have been due to the action of one or more of the decomn products formed by caramelization. Addn of Sb to the Sn produced a small reduction in corrosion, amounting to 8% with 0.25% Sb and to 11% with 0.5%, larger addns did not give further reduction.

A. Papineau-Couture  
Arc welding in argon gas Gilbert E. Doan and Wm C. Schulte *Elec Eng 54, 1144-9* (1935) —Research work in A gas reveals 3 phenomena not previously assoc'd with the welding arc: (1) the impossibility of maintaining a stable arc in highly purified A under ordinary conditions, (2) the absence of all crater formation under pure Fe in pure A, and (3) the absence of all observable "pinch effect" accompanying the detachment of the Fe globules from the electrode wire. The melting rates per kw.-hr in A and in air are approx. equal. Pure Fe welds made in A possess high ductility, 90% reduction of area and 30% elongation. The app for exptl welding in pure gases is illustrated and welding methods are described. W. H. Boynton

Metallic contaminations of foods (Datta) 12 Al in the food industries (Akers) 12 Steel alloys in food manu. (Mitschell) 12 Ni and its alloys (LaQue) 12 Cu, brass and bronze in the food industries (Cole) 12 Cu alloys in food manu. (Kemp) 12 Sn as an anticorrosion coating (Gonser) 12 Pb in the food industries (Wormser) 12 Inhibitors [of corrosion] (Evans) 4 Corrosion of metals by contact with leather (Innes) 29 Thickness of the amorphous layer on polished metals (Hopkins) 2 Oxidation of metals (Preston, Bircumshaw) 2 Analytl triethoxycarbonates [for use as ore flotation agents] (U. S. pat 2,021,726) 10 Coloring Al and its alloys (Ger. pat 619,163) 4 Tunnel kiln (Ger. pat 612,524) 19

Froth-flotation concentration Carl G. R. Melzer. Brit 432,105, July 22, 1935 Addn to 407,963 (C. A. 28, 5030). Precious metals or their compds such as selenides or tellurides are conc'd from their ores by froth flotation in a pulp contg a small amt of 1 or more of the sulfides and (or) tho salts of Cu, Ag, Pb or Hg, formed by coagulating, e. g., by heating or by slow agitation, very finely divided or colloidal particles of the sulfide or tho salt. Examples are given of the contrn. of the precious metal in a pyritic Au ore and in an ore free from pyrites.

1 Apparatus for concentrating gold ores Eldred A. Knapp and Wm. R. Bates. Brit. 432,677, July 31, 1935 Au or Au ore is conc'd by causing the pulp to flow down a stationary inclined table or strake that is adjustable between 2 limiting inclined positions in 1 of which it communicates with a tailings launder and in the other with a concentrates launder, the upper surface of the table or strake being covered by a detachably mounted blanket consisting of a sheet of rubber having a rifled, undulating, pitted or other irregular surface.

Gold for dental use Baker & Co., Inc. Brit 432,881, July 30, 1935 Spongy, cohesive Au for fillings is prep'd by pptg Au from soln, heating the ppt in a high-boiling liquid, e. g., conc'd  $H_2PO_4$  or com  $H_2SO_4$ , at 150-250° and washing and drying. Preferably, a  $AuCl_3$  soln is neutralized by an alkali, heated to 80° and the Au pptd by (COOH), or  $NaNO_3$  added in excess, the soln and ppt are then boiled and any Au remaining in soln is pptd by (COOH), if  $NaNO_3$  was used for the 1st pptn or by (COONa), or an alkali if (COOH), was used. The  $pH$  should be 6-7 at this point. The liquid is decanted and the ppt washed in  $H_2O$ , heated to 150-250° in com.  $H_2SO_4$ , repeatedly washed with dist'd  $H_2O$  and dried below 100°.

Obtaining metals such as tin, zinc or iron from ores Hendrik J. J. Janssen (to Shell Development Co.). U. S. 2,019,785, Nov. 5 An ore such as an oxide of Sn, Zn or Fe is suspended in a liquid fuel such as oil and a suspension stabilizer such as a soap is added, the resulting slurry is atomized and burned with air in such quantity that only incomplete combustion takes place and the ore is reduced. App is described.

Iron ores Georges Gredt. Fr. 785,085, Aug. 16, 1935 Oolitic Fe ores are enriched by calcining the ground ore at a temp near the fritting point of the ore sufficiently long to transform the oolitic grain to  $Fe_2O_3$  and (or) to obtain sufficient hardness and d of the oolites. The ore is then cooled with water.

Treating lithium-bearing ore. Raymond J. Kepner and Robert Pfanzl (to Grasselli Chemical Co.). U. S. 2,022,003, Nov. 20 An ore such as a lipulidite ore is calcined with at least one-third as much  $CaCl_2$  at a temp of about 700-930°, and Li values are ext'd with water.

Zinc ores Associated Metals & Minerals Corp. Fr. 784,884, July 27, 1935. Impurities such as Pb and Cd are removed from ores of Zn by adding chlorinating agents and heating to 1300-1500°. The chlorinating agent (NaCl) is added to the charge in the form of solid particles and is mixed with water in such amt., or is heated so rapidly that the chlorinating agent remains practically undissolved.

Apparatus for vaporizing zinc. The New Jersey Zinc Co. Ger. 616,891, Aug. 5, 1935 (Cl 40a. 34 80).

Apparatus for fractional condensation of metal vapor such as zinc and cadmium in a condenser rotating about a horizontal axis Arthur Leynser (to Fried. Krupp Grusonwerk A.-G.). U. S. 2,021,365, Nov. 19. Various structural and operative details.

Refining metals. Victor Hertl Austrian 142,886, Sept. 25, 1935 (Cl 18). Pused metals are degassed by the action of supersonic vibrations of a frequency of 60,000 or more. Methods of procedure are indicated.

Refining nickel Societ  d'electrochimie, d'electrometallurgie et des aceries electriques d'Ugine. Brit 432,439, July 26, 1935 See Fr. 705,165 (C. A. 28, 6037) and Ger. 594,651 (C. A. 28, 4355).

Apparatus for fritting or roasting ores Allmannia Ingenieursbyran H. G. Torulf. Fr. 785,382, Aug. 8, 1935

Roasting sulfide ores Verein fur chem und metallurgische Produktion Ger. 619,313, Sept. 27, 1935 (Cl 40a. 2 01).

Oscillating tubular furnace with horizontal hearth, particularly for roasting ores Metaliges A.-G. (Hans Klencke and Manfred Sachs, inventors). Ger. 619,364, Sept. 28, 1935 (Cl 40a. 6 01).

Oscillating furnace with an elongated hearth suitable for ore treatment Hans Klencke and Manfred Sachs (to American Lurgi Corp.). U. S. 2,019,912, Nov. 5 Structural and mech. features

Siemens-Martin furnace. "Terzi" Società per l'Industria e l'Elettricità. Ger. 616,593, Aug. 1, 1935 (Cl. 188 14 05). Details of the furnace head are given.

Sintering plant. Humboldt-Deutymotoren A.-G. Ger. 616,511, July 20, 1935 (Cl. 40a. 3 60). Details of a traveling grate are given.

Rotating-drum smelting furnace. Wm. F. Wiltshire. Ger. 616,732, Aug. 9, 1935 (Cl. 31a. 2 30).

Apparatus for melting metals such as various non-ferrous metals. John W. Brown. U. S. 2,020,101, Nov. 5.

Melting steel scrap in shaft furnaces. Mathias Frankl. Ger. 619,457, Oct. 1, 1935 (Cl. 19a. 6 01). A method of charging the furnace is described.

Blast furnace top. Karl F. Juengling. U. S. 2,021,555, Nov. 19.

Heat storer for blast furnaces. Stein- und Thon-Industriegesellschaft "Brodtal" (Eduard Pohl, inventor). Ger. 616,518, July 20, 1935 (Cl. 19a. 14).

Apparatus for regenerative heating of gases such as in blast-furnace operation. Percy H. Royster (to Research Corp.). U. S. reissue 19,757, Nov. 12. A reissue of original 1,840,371 (Cl. A. 28, 1318).

Apparatus (with a selenium cell) for determining the temperature of molten metals. Ired L. Collins and Carl Osland. U. S. 2,020,019, Nov. 5. Light which serves to affect a Se. cell or the like passes through a tube one end of which is immersed in the molten metal.

Melting and casting iron. Wm. A. Brown (to Carborundum Co.). U. S. 2,020,171, Nov. 5. In remelting iron and forming castings from it, SiC is brought into contact with the molten iron in the presence of a substantial quantity of slag, and serves to facilitate obtaining of sound castings.

Increasing the carbon content of cupola-melted iron. Theodore Tafel, Jr. (to Marie C. Tafel). U. S. 2,021,189, Nov. 19. For increasing the C in cupola-melted iron having a relatively high C content, there is added to the molten metal, after it leaves the cupola, a mixt. of carbonaceous material such as graphite or charcoal about 80 and NaNO<sub>3</sub> about 20%.

Apparatus for manufacture of hollow ingots such as those of steel. Ernest F. Jones (to Fraser-Jones Ltd.). U. S. 2,019,820, Nov. 5. Structural and mech. features.

Isolating unsound metal from ingots. Geo. A. Dornin. U. S. 2,021,227, Nov. 19. A gap or crevice is formed between a core including the unsound metal to be isolated and the surrounding zone of metal, without substantial displacement of the core axially of the ingot to divide the core from such zone, and the zone is then displaced away from the core. App. is described.

Casting magnesium. Österreichisch Amerikanische Magnesit A.-G. Austrian 142,935, Oct. 10, 1935 (Cl. 31b). Molds for casting Mg are coated with a sulfide of Sb or Bi on the surfaces with which the Mg comes into contact. Pore-free castings of smooth, corrosion-resisting surface are obtained.

Metal containing compositions. Heinrich Borofski. Brit. 432,278, July 21, 1935. A colloidal mixt. of a metal with a non-alloyable additive substance, e. g., graphite, is made by wetting the latter, before its addn. to the molten metal, with a liquid, e. g., H<sub>2</sub>O, glycerol, oil, that completely evaps. in the metal. In a modification, a mixt. of the metal, additive substance and wetting liquid is heated until the metal is melted and the liquid completely evaps. The invention is suitable for the production of bearing metals or alloys having Cu, Pb, Zn, Sn or Al bases. In the manuf. of bronzes and like alloys, a component, e. g., Zn, Sn, may be added to the additive substance as a fine powder before or after the wetting but prior to the addn. of the wetted substance to the molten metal. App. is described.

Recovering light metals. F. I. du Pont de Nemours & Co. Brit. 432,810, Aug. 2, 1935. To recover Na, etc., from the sludges produced in the production of light metals by the electrolysis of fused metal salts or to recover a substantially light metal from a mixt., the sludge is introduced into a bath of molten salt of sp. gr. higher than that of the

metal or alloy to be recovered and of a compn. such that the metallic constituents of the sludge do not react with it or such that 1 or more of such constituents react with 1 or more of the constituents of the bath to form a metal or metals, the bath being maintained above the m. p. of the metal or alloy to be recovered, such metal or alloy being floated off. App. is described.

Aluminum. I. G. Iarbenud. A.-G. (Frich Noack, inventor). Ger. 602,811, May 25, 1935 (Cl. 12i. 20). Al or its alloys are used in app., etc., which comes into contact with halo derivatives of H<sub>2</sub>SO<sub>4</sub>, such as SOCl<sub>2</sub>, SO<sub>2</sub>Cl<sub>2</sub>, and ClHSO<sub>3</sub>, or with SO<sub>2</sub>.

Aluminum. Karl Mitterbiller and Walter Frühwein. Ger. 607,601, July 0, 1935 (Cl. 40a. 2 01). Al is obtained from Al<sub>2</sub>O<sub>3</sub> by treating a mixt. of Al<sub>2</sub>O<sub>3</sub> and C with AlH<sub>3</sub>, AlH<sub>3</sub> or AlH<sub>3</sub> and decomposing the product to Al halide and metallic Al by heating. An example is given. Cl. C. A. 29, 717.

Copper. The American Metal Co. Ltd. 1r. 784,923, July 29, 1935. In casting deoxidized Cu contg. a small amt. of deoxidant such as P a layer of water is added to the mold and further water is added at the same time as the metal in amt. sufficient to maintain a layer of water above the metal in the mold. An improved surface on the casting is thereby obtained.

Apparatus for hardening iron rails by treatment with liquids. Les petits fils de Francoeur de Wendel. Ger. 619,219, Sept. 26, 1935 (Cl. 18c. 2 23).

Hardening steel. Irma Adolf Thöl. Ger. 619,158, Oct. 1, 1935 (Cl. 18c. 1 70). Phosphate-contg. residues from the treatment of soybeans are added to oil baths used for hardening steel. The inflammability of the baths is thus reduced, and their hardening action is modified.

Tempering and cementation baths. Artigo Consonni. 1r. 785,033, Aug. 13, 1935. The bath is composed of NaCN 42-60 (preferably 37), NiCl<sub>2</sub> 1, H<sub>2</sub>Cl<sub>2</sub> 45, Na<sub>2</sub>CO<sub>3</sub> 7, NaCl 10 and graphite 0.95 part.

Pickling acid. Vereinigte Stahlwerke A.-G. (Wilhelm Mühlendyck and Heinrich Maunz, inventors). Ger. 618,842, Aug. 8, 1935 (Cl. 484. 2 02). Steam is fed into waste H<sub>2</sub>SO<sub>4</sub> from C<sub>2</sub>H<sub>4</sub> and mineral-oil refineries at 90° for 1/2 to 1 hr., or the waste acid is diluted to 35-40 BE, and heated to 90° for the same time, to give H<sub>2</sub>SO<sub>4</sub> suitable for use in pickling or derusting Fe or steel.

Treating acid solutions such as waste pickling liquors. Thomas C. Oliver and Samuel F. Sprangler (to Chemical Construction Corp.). U. S. 2,021,807, Nov. 19. A soln. contg. free H<sub>2</sub>SO<sub>4</sub> and FeSO<sub>4</sub> is mixed with Fe oxide material mainly comprising Fe<sub>2</sub>O<sub>3</sub> and the mixt. is heated to a sufficiently high temp. (suitably about 112° or higher) to cause combination of the oxide with the acid and the mixt. is evapd. to dryness and heated with a reducing agent such as coal to remove the S content as SO<sub>2</sub> and produce an Fe oxide residue. App. is described.

Quenching of wire such as bed spring "water-wire." Josef Gassen (to Schloemann A.-G.). U. S. 2,020,910, Nov. 12. In wire manuf., quenching is effected by directing jets of cooling liquid from points within a coil of wire outwardly against the inner side of the coil and simultaneously rotating the coil rapidly in its own plane to increase the coil-penetrating effect of the liquid. App. is described.

Hardening inner walls of cast-iron cylinders and valve-seat portions of engine blocks. Donald J. Campbell (to Campbell, Wyant & Cannon Laundry Co.). U. S. 2,019,480, Nov. 5. The cast block is heated to above 700° and a relatively large cooling stream such as one of water, steam or air is passed through the cylinders and against the valve-seat portions and a 2nd cooling stream of air or the like is caused to flow through the valve-seat portions. App. is described.

Apparatus for the continuous heat-treatment of ferrous articles as in carburizing and nitrogenizing. Adolph W. Machlet. U. S. 2,021,072, Nov. 12. Various structural, mech. and operative details.

Heat-treating chromium alloy steels. Robert H. Aborn and John J. B. Rutherford (to United States Steel Corp.). Brit. 432,648, July 22, 1935. Corrosion and (or) heat-

of mild steel by citric acid at about  $pH$  3.5. T. N. Morris *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934*, 180(1935).—Aq. solns were prep'd contg 10 g citric acid and 5.8 g Na citrate per l, together with 0.01 and 0.05 M  $Na_2HPO_4$ , 0.01 and 0.05 M  $NaNO_3$ , 0.01 and 0.05 M NaCl, and 0.1 mg. per l  $K_2CrO_4$ , resp. Corrosion tests carried out on 3 X 1 in strips of steel at 25° for 3 days showed that  $Na_2HPO_4$  had a much greater buffering effect than any of the other salts. This effect was shown to be responsible for the diminished corrosion observed.

A. Papineau-Couture  
The corrosion of tin. J. M. Bryan *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934*, 177-9 (1935).—Expts were carried out to ascertain the effects of NaCl and sucrose, resp., on the rates of corrosion of Sn by solns of citric acid at 25° and 75° in presence of a limited amt of air. Addn of 2% NaCl had a slight inhibiting effect in corrosion, which was more pronounced on untreated specimens than on those in which the surfaces of the metal had been abraded with fine emery. Addn. of 1% NaCl to a 0.5% citric acid soln of  $pH$  2.4 at 25° had no effect, but as the  $pH$  increased to 5.5 (by addn of Na citrate buffer) there was a slightly increasing inhibiting effect. Addn of 25% sucrose at 25° produced an appreciable inhibition over the whole  $pH$  range studied, the rate of corrosion being reduced to  $1/4$  at  $pH$  2.4 and to  $1/5$  at  $pH$  5.5. At 25°, addn. of varying amts of sucrose to 0.5% citric acid soln progressively retarded the rate of corrosion as the concn increased. This inhibition seems to be due mainly to the effect of sugar in reducing the soly of air in the solns, since the corrosion (like that in plain citric acid solns) was entirely of the oxidative type. At 75°, concns of less than 15% sucrose inhibited corrosion, while greater concns stimulated it, the latter effect, however, may have been due to the action of one or more of the decompn products formed by caramelization. Addn of Sb to the Sn produced a small reduction in corrosion, amounting to 8% with 0.25% Sb and to 11% with 0.5%, larger addns did not give further reduction.

A. Papineau-Couture  
Arc welding in argon gas. Gilbert J. Doan and Wm. C. Schulte *Elec. Eng.* 54, 1144-9 (1935).—Research work in A gas reveals 3 phenomena not previously assoc'd with the welding arc. (1) the impossibility of maintaining a stable arc in highly purified A under ordinary conditions; (2) the absence of all crater formation under pure Fe in pure A; and (3) the absence of all observable "pinch effect" accompanying the detachment of the Fe globules from the electrode wire. The melting rates per kw.-hr in A and in air are approx. equal. Pure Fe welds made in A possess high ductility, 90% reduction of area and 30% elongation. The app for exptl welding in pure gases is illustrated and welding methods are described. W. H. Boynton

Metallic contaminations of foods (Datta) 12. Al in the food industries (Akers) 12. Steel alloys in food manuf. (Mitchell) 12. Ni and its alloys (LaQue) 12. Cu, brass and bronze in the food industries (Cole) 12. Cu alloys in food manuf. (Kemp) 12. Sn as an anticorrosion coating (Conser) 12. Pb in the food industries (Wormser) 12. Inhibitors [of corrosion] (Evans) 4. Corrosion of metals by contact with leather (Innes) 29. Thickness of the amorphous layer on polished metals (Hopkins) 2. Oxidation of metals (Preston, Bircumshaw) 2. Aralkyl trihydrocarbonates [for use as ore-floatation agents] (U. S. pat. 2,021,726) 10. Coloring Al and its alloys (Ger. pat. 619,163) 4. Tunnel kiln (Ger. pat. 612,524) 19.

Froth-flotation concentration. Carl G. R. Meizer *Brit.* 432,105, July 22, 1935. Addn to 407,963 (C. A. 28, 5030\*9). Precious metals or their compds, such as selenides or tellurides are conc'd from their ores by froth flotation in a pulp contg. a small amt of 1 or more of the sulfides and (or) tho salts of Cu, Ag, Pb or Hg, formed by coagulating, e. g., by heating or by slow agitation, very finely divided or colloidal particles of the sulfide or tho salt. Examples are given of the concn. of the precious metal in a pyritic Au ore and in an ore free from pyrites.

1 Apparatus for concentrating gold ores. Eldred A. Knapp and Wm. R. Bates *Brit.* 432,677, July 31, 1935. Au or Au ore is conc'd by causing the pulp to flow down a stationary inclined table or strake that is adjustable between 2 limiting inclined positions in 1 of which it communicates with a tailings launder and in the other with a concentrates launder, the upper surface of the table or strake being covered by a detachably mounted blanket consisting of a sheet of rubber having a rifled, undulating, pitted or other irregular surface.

Gold for dental use. Baker & Co., Inc. *Brit.* 432,881, July 30, 1935. Spongy, cohesive Au for fillings is prep'd by pptg Au from soln, heating the ppt in a high-boiling liquid, e. g., conc'd  $H_2PO_4$  or com  $H_2SO_4$ , at 150-250° and washing and drying. Preferably, a AuCl<sub>3</sub> soln is neutralized by an alkali, heated to 80° and the Au pptd by (COOH)<sub>2</sub> or NaNO<sub>2</sub> added in excess, the soln and ppt. are then boiled and any Au remaining in soln is pptd by (COOH)<sub>2</sub>; if NaNO<sub>2</sub> was used for the 1st pptn or by (COONa), or an alkali if (COOH)<sub>2</sub> was used. The  $pH$  should be 6-7 at this point. The liquid is decanted and the ppt washed in  $H_2O$ , heated to 150-250° in com.  $H_2SO_4$ , repeatedly washed with distd  $H_2O$  and dried below 100°.

Obtaining metals such as tin, zinc or iron from ores. Hendrik J. J. Jaussen (to Shell Development Co.) U. S. 2,019,785, Nov. 5. An ore such as an oxide of Sn, Zn or Fe is suspended in a liquid fuel such as oil and a suspension stabilizer such as a soap is added, the resulting slurry is atomized and burned with air in such quantity that only incomplete combustion takes place and the ore is reduced. App is described.

Iron ores. Georges Gredt. *Fr.* 785,685, Aug. 16, 1935. Oolitic Fe ores are enriched by calcining the ground ore at a temp. near the frating point of the ore sufficiently long to transform the oolitic grain to  $Fe_2O_3$  and (or) to obtain sufficient hardness and d. of the coillotes. The ore is then cooled with water.

Treating lithium-bearing ore. Raymond J. Kepfer and Robert Planstiel (to Grasselli Chemical Co.) U. S. 2,022,093, Nov. 20. An ore such as a lipulidite ore is calcined with at least one-third as much  $CaCl_2$  at a temp. of about 700-930°, and Li values are ex'd with water.

Zinc ores. Associated Metals & Minerals Corp. *Fr.* 784,884, July 27, 1935. Impurities such as Pb and Cd are removed from ores of Zn by adding chlorinating agents and heating to 1300-1500°. The chlorinating agent (NaCl) is added to the charge in the form of solid particles and is mixed with water in such amt, or is heated so rapidly that the chlorinating agent remains practically undissolved.

Apparatus for vaporizing zinc. The New Jersey Zinc Co. *Ger.* 616,801, Aug. 5, 1935 (Cl. 40a. 34 80).

Apparatus for fractional condensation of metal vapor such as zinc and cadmium in a condenser rotating about a horizontal axis. Arthur Leysner (to Fried. Krupp Grusonwerk A.-G.) U. S. 2,021,365, Nov. 19. Various structural and operative details.

Refining metals. Victor Hertl. *Austrian* 142,886, Sept. 25, 1935 (Cl. 18). Fused metals are degassed by the action of supersonic vibrations of a frequency of 50,000 or more. Methods of procedure are indicated.

Refining nickel. Société d'Electrochimie, d'Electrometallurgie et des usines électriques d'Ugine. *Brit.* 432,439, July 26, 1935. See *Fr.* 765,165 (C. A. 28, 663\*9) and *Ger.* 591,651 (C. A. 28, 4365\*9).

Apparatus for fritting or roasting ores. Allmanna Ingeniörsbyrå H. G. Torulf. *Fr.* 785,382, Aug. 8, 1935.

Roasting sulfide ores. Verein für chem. und metallurgische Produktion. *Ger.* 619,313, Sept. 27, 1935 (Cl. 40a. 2 01).

Roasting tubular furnace with horizontal hearth, particularly for roasting ores. Metallgesellschaft A.-G. (Hans Klencke and Manfred Sachs, inventors). *Ger.* 619,364, Sept. 28, 1935 (Cl. 40a. 6 01).

Oscillating furnace with an elongated hearth suitable for ore treatment. Hans Klencke and Manfred Sachs (to American Lurgi Corp.) U. S. 2,019,912, Nov. 5. Structural and mech. features.

Siemens-Martin furnace. "Terni" Società per l'Industria e l'Elettricità. Ger. 616,598, Aug. 1, 1935 (Cl. 184 14 05). Details of the furnace head are given.

Smelting plant. Humboldt-Deutzmotoren A.-G. Ger. 616,511, July 20, 1935 (Cl. 40a. 3 60). Details of a traveling grate are given.

Rotating-drum smelting furnace. Wm F. Wiltshire. Ger. 616,732, Aug. 8, 1935 (Cl. 31a. 2 20).

Apparatus for melting metals such as various non-ferrous metals. John W. Brown. U. S. 2,020,101, Nov. 5.

Structural and operative details.

Melting steel scrap in shaft furnaces. Mathias Frankl. Ger. 619,457, Oct. 1, 1935 (Cl. 18a. 6 01). A method of charging the furnace is described.

Blast furnace top. Karl F. Juengling. U. S. 2,021,555, Nov. 19.

Heat storer for blast furnaces. Stein- und Thon-Industriegesellschaft "Broththal" (Eduard Pohl, inventor). Ger. 616,518, July 20, 1935 (Cl. 18a. 14).

Apparatus for regenerative heating of gases such as in blast furnace operation. Percy H. Royster (to Research Corp.). U. S. reissue 19,757, Nov. 12. A reissue of original 1,940,371 (C. 28, 13187).

Apparatus (with a selenium cell) for determining the temperature of molten metals. Fred L. Collins and Carl Oseland. U. S. 2,020,019, Nov. 5. Light which serves to affect a Se cell or the like passes through a tube one end of which is immersed in the molten metal.

Melting and casting iron. Wm A. Brown (to Carborundum Co.). U. S. 2,020,171, Nov. 5. In remelting iron and forming castings from it, SiC is brought into contact with the molten iron in the presence of a substantial quantity of slag, and serves to facilitate obtaining of sound castings.

Increasing the carbon content of cupola-melted iron. Theodore Tafel, Jr. (to Marie C. Tafel). U. S. 2,021,159, Nov. 19. For increasing the C in cupola-melted iron having a relatively high-C content, there is added to the molten metal, after it leaves the cupola, a mixt. of carbonaceous material such as graphite or charcoal about 80 and NaNO<sub>2</sub> about 20%.

Apparatus for manufacture of hollow ingots such as those of steel. Ernest F. Jones (to Fraser-Jones Ltd.). U. S. 2,019,820, Nov. 5. Structural and mech. features.

Isolating unsound metal from ingots. Geo. A. Dornin. U. S. 2,021,227, Nov. 19. A gap or crevice is formed between the core including the unsound metal to be isolated and the surrounding zone of metal, without substantial displacement of the core axially of the ingot to divide the core from such zone, and the zone is then displaced away from the core. App. is described.

Casting magnesium. Österreichisch Amerikanische Magnesium A.-G. Austrian 142,835, Oct. 10, 1935 (Cl. 31b). Molds for casting Mg are coated with a sulfide of Sb or Bi on the surfaces with which the Mg comes into contact. Pore-free castings of smooth, corrosion-resisting surface are obtained.

Metal-containing compositions. Heinrich Borofski. Brit. 432,278, July 21, 1935. A colloidal mixt. of a metal with a non-alloyable additive substance, e. g., graphite, is made by wetting the latter, before its addn. to the molten metal, with a liquid, e. g., H<sub>2</sub>O, glycerol, oil, that completely evaps in the metal. In a modification, a mixt. of the metal, additive substance and wetting liquid is heated until the metal is melted and the liquid completely evapd. The invention is suitable for the production of bearing metals or alloys having Cu, Pb, Zn, Sn or Al bases. In the manuf. of bronzes and like alloys, a component, e. g., Zn, Sn, may be added to the additive substance as a fine powder before or after the wetting but prior to the addn. of the wetted substance to the molten metal. App. is described.

Recovering light metals. E. I. du Pont de Nemours & Co. Brit. 432,810, Aug. 2, 1935. To recover Na, etc., from the sludges produced in the production of light metals by the electrolysis of fused metal salts or to recover a substantially light metal from a mixt., the sludge is introduced into a bath of molten salt of sp. gr. higher than that of the

metal or alloy to be recovered and of a compn. such that the metallic constituents of the sludge do not react with it or such that 1 or more of such constituents react with 1 or more of the constituents of the bath to form a metal or metals, the bath being maintained above the m. p. of the metal or alloy to be recovered, such metal or alloy being floated off. App. is described.

Aluminum. I. G. Farbenund A.-G. (Erich Noack, inventor). Ger. 602,814, May 25, 1935 (Cl. 12i. 30). Al or its alloys are used in app., etc., which comes into contact with halo derivs of H<sub>2</sub>SO<sub>4</sub>, such as SOCl<sub>2</sub>, SO<sub>2</sub>Cl<sub>2</sub> and CH<sub>3</sub>SO<sub>3</sub>, or with SO<sub>2</sub>.

Aluminum. Karl Mittelbiller and Walter Frühwein. Ger. 607,561, July 9, 1935 (Cl. 40a. 50 01). Al is obtained from Al<sub>2</sub>O<sub>3</sub> by treating a mixt. of Al<sub>2</sub>O<sub>3</sub> and C with Al<sub>2</sub>, Al<sub>2</sub> or AlBr<sub>3</sub>, and decomposing the product to Al halide and metallic Al by heating. An example is given. Cl. C. 29, 717<sup>a</sup>.

Copper. The American Metal Co. Ltd. Fr. 784,923, July 29, 1935. In casting deoxidized Cu contg. a small amt. of deoxidant such as P a layer of water is added to the mold and further water is added at the same time as the metal in amt. sufficient to maintain a layer of water above the metal in the mold. An improved surface on the casting is thereby obtained.

Apparatus for hardening iron rails by treatment with liquids. Les petits fils de François de Wendel. Ger. 619,249, Sept. 26, 1935 (Cl. 18c. 2 23).

Hardening steel. Firma Adolf Thöl. Ger. 619,458, Oct. 1, 1935 (Cl. 18c. 1 70). Phosphatide-contg. residues from the treatment of soybeans are added to oil baths used for hardening steel. The inflammability of the baths is thus reduced, and their hardening action is modified.

Tempering and cementation baths. Arrigo Consolini. Fr. 785,563, Aug. 13, 1935. The bath is composed of NaCN 32-60 (preferably 37), NH<sub>4</sub>Cl 1, BaCl<sub>2</sub> 45, Na<sub>2</sub>CO<sub>3</sub> 7, NaCl 10 and graphite 0.95 part.

Pickling acid. Vereinigte Stahlwerke A.-G. (Wilhelm Mühlendyck and Heinrich Manz, inventors). Ger. 616,802, Aug. 8, 1935 (Cl. 48d. 2 02). Steam is led into waste H<sub>2</sub>SO<sub>4</sub> from CaH<sub>2</sub> and mineral-oil refineries at 90° for 1/2 to 1 hr., or the waste acid is diluted to 35-40 B<sub>e</sub>, and heated to 90° for the same time, to give H<sub>2</sub>SO<sub>4</sub> suitable for use in pickling or desulfurizing Fe or steel.

Treating acid solutions such as waste pickling liquors. Thomas C. Oliver and Samuel F. Spangler (to Chemical Construction Corp.). U. S. 2,021,867, Nov. 19. A soln. contg. free H<sub>2</sub>SO<sub>4</sub> and FeSO<sub>4</sub> is mixed with Fe oxide material mainly comprising Fe<sub>2</sub>O<sub>3</sub> and the mixt. is heated to a sufficiently high temp. (suitably about 112° or higher) to cause combination of the oxide with the acid and the mixt. is evapd. to dryness and heated with a reducing agent such as coal to remove the S content as SO<sub>2</sub> and produce an Fe oxide residue. App. is described.

Quenching of wire such as bed spring "water-wire." Josef Gassen (to Schloemann A.-G.). U. S. 2,020,940, Nov. 12. In wire manuf., quenching is effected by directing jets of cooling liquid from points within a coil of wire outwardly against the inner side of the coil and simultaneously rotating the coil rapidly in its own plane to increase the coil-penetrating effect of the liquid. App. is described.

Hardening inner walls of cast-iron cylinders and valve-seat portions of engine blocks. Donald J. Campbell (to Campbell, Wyant & Cannon Foundry Co.). U. S. 2,019,480, Nov. 5. The cast block is heated to above 700° and a relatively large cooling stream such as one of water, steam or air is passed through the cylinders and against the valve-seat portions and a 2nd cooling stream of air or the like is caused to flow through the valve-seat portions. App. is described.

Apparatus for the continuous heat-treatment of ferrous articles as in carburizing and nitrogenizing. Adolph W. Machlet. U. S. 2,021,072, Nov. 12. Various structural, mech. and operative details.

Heat-treating chromium alloy steels. Robert H. Ahorn and John J. B. Rutherford (to United States Steel Corp.). Brit. 432,548, July 22, 1935. Corrosion and (or) heat-



resisting alloy steels contg Cr 1-60, C up to 0.25 and Ti or Nb 0.5-3% are heat-treated to prevent loss of corrosion resistance at temps below about 1470°F by heating to between about 1346°F and 1740°F. For 0.1-10 hrs, the time being sufficient to obtain a substantial conversion of the C into carbides of Ti or Nb. The steels may be heated to 2150°F and quenched to room temp before the heating just described. The heat treatment may also be applied to austenitic Ni-Fe alloys contg Cr, C and Ti. In an example, a steel contg C 0.1, Cr 18, Ni 8 and Ti 0.5% is heated either for 1 hr at 1600°F or for 4 hrs. at 1400°F.

Apparatus for carburizing metal articles in a heat treating box Harry W. McQuaid (to Timken-Detroit Axle Co.) U. S. 2,021,666, Nov. 19. Various structural and operative features.

Apparatus for pack annealing of steel bars Erik W. Mikaelson and Alexander K. Hamilton (to Treadwell Engineering Co.) U. S. 2,019,576, Nov. 5. Structural, mech and operative details.

Rolling metal sheets Norman C. Rendleman (to Jones & Laughlin Steel Corp.) U. S. 2,021,328, Nov. 19. Various operative details.

Apparatus for degreasing articles such as those of metal by use of solvent vapors Joseph Savage (to Imperial Chemical Industries Ltd.) U. S. 2,020,335, Nov. 12. Various structural and operative details.

Apparatus for degreasing metal and other nonabsorbent articles with solvent vapors Robert A. Eastwood and Imperial Chemical Industries Ltd. Brit. 433,472, Aug. 15, 1935.

Making clad metals as in coating tungsten x-ray anodes with copper Frank H. Driggs (to Westinghouse Lamp Co.) U. S. 2,019,599, Nov. 5. Successive coatings of metal such as Cu are applied to an underlying metal such as W in reducing atmospheres such as CO and H<sub>2</sub>, which, resp., have different solubilities in the metal used. App and various operative details are described.

Electrotype production Joseph H. Shea (to Frederick Griswold, Jr., as trustee) U. S. 2,022,156, Nov. 26. A sheet of Sn foil is superimposed upon a plate of metal such as Cu and a metal plate bearing indicia is superimposed on the foil, and regulated heat is applied to opposite faces of the metal plates, with pressure, and the assembly is cooled under pressure. App. is described.

Turbine blades The English Electric Co. Ltd., John P. Chittenden and Frank Buckley Brit. 432,386, July 25, 1935. Erosion resisting edges are formed by depositing a harder material, e. g., Mn steel, by a mech. process, e. g., by elec. welding, on the blade, further hardening this material by mech. working, e. g., cold rolling or hammering, and then electrolytically depositing a coating of a still harder material, e. g., Cr.

Steel Vereinigte Stahlwerke A.-G. Ger. 612,996, Aug. 20, 1935 (Cl. 185 20). A P-steel with a low Cr content is obtained by the Thomas process, the steel being blown till a P content of about 0.08% is obtained, and Cr, as Fe-Cr, being added. The whole is then subjected to a short blasting.

Steel Walter Eilender and Nicolaus Wark. Ger. 619,334, Sept. 28, 1935 (Cl. 185 16 01). In the manu. of steel by the basic process, Mn ore or Mn slag is added to the converter charge, simultaneously with the lime, and also at a later stage if desired. The proportion of Mn ore or slag must be such that a basic slag of high Mn content is present throughout the process, and more than 6% of MnO is present in the final slag. The Mn ore or slag exerts a deoxidizing effect.

Steel alloy Fried Krupp A.-G. Ger. 616,599, Aug. 1, 1935 (Cl. 185 1.30). A steel alloy for withstanding high temps and pressure in steam generators consists of C 0.3, Mn 0.2-0.5, Al 0.05-0.2% and the rest Fe. Amts of Mn, Si, P and S usual in steel manu. may be added.

Steel alloys. Vereinigte Stahlwerke A.-G. (Hermann Schulz, inventor). Ger. 616,712, Aug. 5, 1935 (Cl. 185 2.20). A Cu-Si-steel alloy for constructional uses contains C up to 0.2, Si 0.5-1.5, Mn 0.45-0.90 and Cu 0.2-0.5%. Examples are given.

Alloy steel boiler tubes, etc. Walter G. Hildorf, Albert

E. White and Claude L. Clark (to Timken Roller Bearing Co.). U. S. 2,021,781, Nov. 19. Boiler tubes, etc., for use at high temp. are formed of an alloy steel of high creep strength at temps. above 400° and of good resistance to oxidation at high temps., contg. C about 0.01-0.2, Cr about 0.75-1.5, Mn about 0.2-1.0, Si about 0.5-1.0 and W about 0.8-1.0%, the remainder being substantially all Fe. U. S. 2,021,782 relates to the similar use of steels contg. Fe together with C 0.2-0.6, Cr 0.75-1.5, Mn 0.2-1.0, Si 0.5-0.9 and W 0.5-1.5%. U. S. 2,021,783 relates to grate bars, stoker parts, carburizing boxes, valve and furnace parts, etc., of good resistance to oxidation at temps. of 540° or higher formed of steel contg. Fe together with C 0.01-0.6, Cr 1.5-3.0, Mn 0.2-1.0, Si 1-3 and W 0.25-1.0%.

Compositions for steel-cutting tools, drawing dies, etc. Philip M. McKenna (to Vanadium-Alloys Steel Co.) U. S. 2,021,576, Nov. 19. Ta carbide 20-85% is used with W carbide and Zr or Hf which are proportioned to react with the W carbide and produce W and Zr or Hf carbide during the formation of the compn. App. is described. Cf. C. A. 29, 65027.

Alloys The Mond Nickel Co. Ltd. and John McNeil. Brit. 432,484, July 29, 1935. Elec. contacts and electrodes, particularly for spark plugs, are made from an alloy of Al 0.1-8% with Ni or Ni and Cr, Mn and (or) Fe. A low-Ni content, e. g., 15%, should correspond to a low-Al content, e. g., 4%. Suitable proportions are Ni 93, 77; Al 3, 3; Mn 4, 0; Cr 0, 20%. Alloys contg. 80% Fe may be used, the Al in the case of the ferrous alloys being partly replaced by Si. Small quantities of Mo, Ti, Zr, W, V, Ta, Cu, Be, C, Ca, Ba, Sr and Li may be used. The alloys may contain up to 0.25% C.

Alloys Stahlwerke Röchling Buderus A.-G. and Alfred Kropf. Ger. 616,858, Aug. 6, 1935 (Cl. 408 14). An alloy capable of hot or cold working consists of Cr 10-40, Ta 3-50, C 0.1-1.0 and Ni 40-60%. Si, V or Zr in amts. up to 5% may be added as deoxidizing agents.

Hard alloys. Compagnie Lorraine de charbons pour l'électricité. Fr. 785,576, Aug. 13, 1935. A more fusible auxiliary metal or alloy is fixed separately on each of the hard constituents, the fractions thus prep. are united, homogenized and submitted to fritting by the action of heat and pressure.

Hard alloys containing tungsten, etc. Alan R. Powell and Ernest R. Box (to Johnson Matthey & Co. Ltd.) U. S. 2,021,021, Nov. 12. Alloys which are suitable for the points of gold nibs or compass needles, etc., are obtained by melting a mixt. of W 50-75, B 0.5-5.0, Co or Ni 5-25 and Pt, Ru or Os 2-40%, the proportion of B to W being not greater than 11 to 184 and the proportion of Co or Ni to W being at least 9 to 184.

Surface hardening alloys Max Botton. Fr. 785,683, Aug. 16, 1935. Fe alloys are heated in the presence of NH<sub>3</sub> and a solid substance, such as powd. Al, Mg, B, Fe-Cr, Fe-Mo, Fe-W, Ca, Cr and Ni, which does not melt during the treatment and which is capable of forming with NH<sub>3</sub> at least 1 unstable compd. favoring the formation of nitrides of at least 1 of the metals of the alloy. The alloy may be submitted during the treatment to a high-frequency current capable of modifying the cryst. texture of the alloy. The alloy may be tempered before or after the treatment.

Alloys for electric resistances Établissements Driver-Harris. Fr. 785,701, Aug. 17, 1935. Alloys contain Ni 50-90, Cr 9-30, Ca 0.01-0.25, Zr 0.1-1% and Fe the rest if present. Two examples are Ni 78 and 65, Cr 20.5 and 15, Ca 0.06 and 0.06, Zr 0.15 and 0.15 and Fe 0.4 and 16-17%.

Alloys for permanent magnets Soc. industrielle et commerciale des aciers. Fr. 785,732, Aug. 17, 1935. Alloys contain Fe and Pd or Fe and Pt, the metals being present in the ratio approx. corresponding to whole multiples of their at. wts., e. g., Fe 30-40 and Pd 70-60% or Fe 28-18 and Pt 72-82%. The alloys are cooled from a high temp. and reheated to a lower temp. or cooled slowly from a high temp. (600°).

Changing the physical properties of light alloys I. G. Farbenindustrie A.-G. Brit. 432,828, Aug. 2, 1935.

The mech. properties of Mg and other light metal alloys, which have been plastically deformed, e. g., by rolling or pressing, and exhibit in the direction of flow lower tensile strength, elongation, yield point, elastic limit, etc., than in a direction transverse thereto, are modified to destroy the original uniform orientation of the crystal structure and set up therein a twinning of the crystals by subjecting said alloys to a succession of at least 10 straightening operations, each involving several bending or twisting operations.

**Aluminum alloys.** I. G. Farbenindustrie A.-G. Brit. 432,351, July 22, 1935. This corresponds to Fr. 766,071 (C. A. 28, 7249).

**Aluminum alloys.** Paul Briske and Viktor Prohl (trading as Briske & Prohl) and Alexander Lushenovsky Brit. 432,064, Aug. 2, 1935. See Fr. 772,586 (C. A. 29, 1389).

**Aluminum alloy.** Franz Jordan (Otto Kamps, inventor). Ger. 613,014, May 10, 1935 (Cl. 43, 18). Addn. to 595,531 (C. A. 28, 5635). The Al-Cu-Light metal alloy of 595,531 is varied by replacing the V by 0.1-0.2% of Zr.

**Heat-treating aluminum alloys.** Horace C. Hall Brit. 432,815, Aug. 2, 1935. To remove internal stresses from an Al alloy piece after soln. treatment, i. e., heating to 320° and quenching, but before aging, if carried out, the piece is plunged into, and agitated in, a bath of oil, molten salts or metal, the temp. of which is 190-250°.

**Treatment of aluminum-silicon alloys.** Metallgesellschaft A.-G. (Abdur Paz, inventor). Ger. 619,409, Oct. 1, 1935 (Cl. 43, 19). Alloys consisting wholly or mainly of Al and Si are improved in their mech. properties by treating the fused alloy first with a small proportion of an alkali metal, e. g., 0.1% of Na, and then with such a proportion of Mg (0.05-0.35%) that 0.045-0.3% of Mg remains in the alloy. The alkali metal may be added as such or as a reagent which liberates it under the conditions of the process. The Mg may be added as such or as an alloy.

**Columbium alloys.** Electro Metallurgical Co. Fr. 753,667, Aug. 16, 1935. Sn is extd. from Cb alloys by adding Sn in amt. sufficient to produce Cb-Sn and preferably sufficient to obtain a ratio of Sn to Cb of about 1 to 6.5 and then leaching with HCl of about 11° Bé. Cf. C. A. 29, 5004.

**Refining copper alloys.** Theodor Laible. Ger. 616,914, Aug. 7, 1935 (Cl. 43, 15 01). Al and Fe are removed from Cu alloys by fusing the alloys and adding Na<sub>2</sub>SO<sub>3</sub>. This causes the Al and Fe to form a removable scum.

**Iron alloys.** Steepbridge Stokes Centrifugal Castings Co. Ltd. and James B. Hurst. Brit. 432,974, Aug. 7, 1935. The seats and other parts of the valves of internal-combustion engines are made of cast Fe alloyed with 0.5-2% of both Al and Cr, together with 2-3% of both C and Si. A preferred compn. is C 2.65, Si 2.5, Al 1.25 and Cr 1.55%. Mn, S and P may be present in the usual amts. for cast Fe.

**Iron alloy.** Röschling'sche Eisen- und Stahlwerke A.-G. (Hans Zieher, inventor). Ger. 616,897, Aug. 1, 1935 (Cl. 43, 10). Crude Fe with a high-V content for making Thomas steel and V or V compounds, is obtained by heating converter discharge masses (magnetically sep'd.) rich in untrapped Fe and V, in a blast furnace or other reducing furnace, with or without coke. The converter masses may be mixed with Fe or Fe ore cont'g. V. Examples are given.

**Iron alloys.** I. G. Farbenindustrie A.-G. Fr. 784,700, July 22, 1935. Fe alloys contg. more than 12, preferably about 25-30% Cr are found to be resistant to most mixts. of Cl and O.

**Nickel-chromium alloys.** James M. Lehr (to Driver-Harris Co.). U. S. 2,019,680, Nov. 5. Alloys which are suitable for elec. resistance units contain Ni together with Cr 15-25, Ca 0.01-0.20 and Zr 0.01-0.50%. U. S. 2,019,681 relates to alloys containing Ni together with Cr 7-22, Fe 8-20, Ca 0.01-0.20 and Zr 0.01-0.50%. U. S. 2,019,688 relates to alloys cont'g. Ni together with Cr 7-22, Fe 8-20 and Zr 0.01-0.50%.

**Nickel-chromium-iron alloys.** Heraeus-Vacuum-schmelze A.-G. Fr. 783,078, Aug. 1, 1935. Alloys resistant to high temps., particularly 1000-1200°, contg. Ni, Cr 1-50, and Fe up to 50% also contain rare earth metals, 0.02-1.2%. The alloys may also contain Co, Mo or W up to 20% and small amts. of Mn, Mg or Si.

**Silver alloys suitable for soldering.** Robert H. Leach (to Hander & Harrison). U. S. 2,019,984, Nov. 5. Alloys are used contg. Ag 30-70, Cu 10-35, P 0.5-5.0 and Zn about 10-40%.

**Silver-beryllium-copper alloys.** Robert H. Leach (to Hander & Harrison). U. S. 2,020,949, Nov. 12. Alloys which are suitable for elec. contacts contain Ag about 5-50 and Be 0.5-2.5%, the remainder being Cu.

**Composite bearings.** General Motors Corp. Brit. 433,954, Aug. 23, 1935. A composite member for the main of bearings is produced by heating an Fe or steel trav approx. to the m. p. of plastic bronze, pouring molten plastic bronze into the trav and rapidly cooling to below the m. p. thereof. The bronze may contain 50-60% Cu and the rest Pb, with small quantities of Sn, Ni or other metals, the m. p. being approx. 1700°F. A flux, e. g., H<sub>2</sub>BO<sub>3</sub>, is introduced with the trav into the furnace and the heating is preferably in a neutral or reducing atm. App. is described.

**Composite metal product suitable for use in aircraft construction.** Fred Keller and George Frederick Saer (to Aluminum Ltd.) Brit. 432,617, July 30, 1935. See U. S. 1,975,105 (C. A. 28, 7241).

**Protecting metals.** Anciens établissements Soc-born, Lang-Ferrv & Cie. Fr. 741,948, July 30, 1935. Sheets of metal such as Fe are coated in any manner by Zn or Pb and then covered with a varnish which is baked on.

**Protecting metals against corrosion by alcohol.** L'Etat Français représenté par le Ministre de la Marine. Fr. 783,117, Aug. 2, 1935. NH<sub>4</sub>Et or NH<sub>4</sub>Et<sub>2</sub>, at least 0.03% by wt., is added to EtOH, pure or aq., to prevent corrosion of steel, without, however, giving a soln. which will attack Cu, this being the case when NH<sub>3</sub> is used.

**Rust preventing coatings.** Metallgesellschaft A.-G. (Hans Weidmann, Gerhard Roemer and Wilhelm Overath, inventors). Ger. 616,982, Aug. 9, 1935 (Cl. 43, 4 01). These coatings are formed on Fe or steel objects by hanging the objects in a bath contg. phosphates of metals. Any Al present in the bath is removed before immersion of the Fe or steel objects. In an example, a bath of phosphate salts contg. Mn, Fe, PbO<sub>2</sub> and Al is used. The Al is removed by adding colloidal Fe(OH)<sub>3</sub>. Other examples are given.

**Protective coating of carbides such as those of tool cutting edges.** John A. Zablum. U. S. 2,021,043, Nov. 12. In attaching a piece of metallic carbide such as W carbide to a metallic body such as a cutting or digging tool, the piece of carbide is coated with substantially pure Ni which protects it against partial decomposition of alloying with the metallic body, and the coated piece is then welded to the body.

**Oxide coatings on cathodes for electric discharge devices.** Emil Theist (to Egyesült Izzolampas és Villamosági Reszvénytársaság). U. S. 2,019,546, Nov. 5. A "trocen" such as a Ra-Mo bronze is applied as a coating on a core such as one formed of W or Mo and a layer of alk. earth metal oxide is applied to this coating.

**Depositing metals.** Carl Levst-Kuchemester (to Ludwig A. Seimert). Brit. 432,466, July 23, 1935. Metals or nonmetallic substances are coated with metal by exposure to metallic vapor produced by melting the coating metal and evap. it in a high vacuum corresponding to a pressure of 10<sup>-4</sup> to 10<sup>-6</sup> mm. Hg. App. is described.

**Coating with metals.** Anselmo Ortiz Rodriguez. Brit. 432,212, July 23, 1935. A metallic coating free from pores, spots, etc., is obtained on ferrous metal articles by heating them in an atm. of reducing and decarbonizing gases or vapors in the nascent state, passing the articles through a bath of molten metal the surface of which is not in contact with said gas or vapors and finally cooling and smoothing the coating. App. is described.

Coating metal surfaces Dunlop Plantations Ltd Tr 784,880, July 27, 1935 Substances such as waxes or resins which are fusible are projected as rain onto metals to be coated The powder deposit thus obtained is caused to fuse and to solidify by cooling to give a thin uniform coating

Coating strands of material as in coating wires with tin Carl M Underwood (to Western Elec. Co.). U. S. reissue 19,758, Nov. 12 A reissue of original pat no 1,981,130 (C A 29,440)

Coating aluminum and its alloys Pentall Soc anon Brit 413,367, Aug 13, 1935 See Swiss 175,365 (C A 30,77)

Coloring aluminum and its alloys Siemens & Halske A-G (Nikolai Budiloff and Alexander Jenny, inventors) Ger 619,450, Oct 1, 1935 (Cl 57b, 14 01). Addn to 607,012 (C A 29,1766) An oxide coating on Al or Al alloy is impregnated with a salt of Ag and then exposed either completely or under a negative or a diapositive or a stencil The Ag image is fixed and converted wholly or partly into an Au image by means of a toning bath, and the Au image is dried and then strongly heated, e. g., to 500-600°. The Al alloy is thus colored red. Cf. C. A. 30,754

Apparatus for coating pipes by dipping into fused metal I G Farbennd A-G (Fritz Steinbach, inventor). Ger 619,321, Sept. 27, 1935 (Cl 48b, 4)

Inhibiting attack of sulfur dioxide on metals. Frank D. Burke U. S. 2,019,559, Nov. 5. A small amount of an aldehyde such as  $\text{CH}_3\text{CHO}$  is added to  $\text{SO}_2$  such that employed in refrigerating systems

Imparting a high luster to cadmium Geo Dubpernell and Karl G Soderberg (to Udyllite Process Co.). U. S. 2,021,592, Nov. 19 The Cd is immersed in a soln of chromic acid and a sulfate radical and the ratio in g per l of chromic acid to sulfate radical is maintained above a crit ratio of about 20 The soln may be formed from  $\text{CrO}_3$  and  $\text{H}_2\text{SO}_4$

Electric system for preventing corrosion of metal surfaces such as condensers used on aircraft Herbert S Polin (to Polin, Inc.). U. S. 2,021,519, Nov. 19

Preventing corrosion of condensate-return lines of steam-heating systems Robert L Holmes U. S. 2,020,359, Nov. 12 A portion of the water contained in the boiler is directed into the condensate-return line near a point where the condensate water enters the return line. App is described

Electric arc apparatus for fusing metals as in the manufacture of alloy welding rods John A. Zublin U. S. 2,022,171, Nov. 26 Various structural, elec. and operative details

Arc welding electrode Louis J Larson (to A. O. Smith Corp.). U. S. 2,021,628, Nov. 19 An electrode with a core of ferrous metal has a covering including carbonaceous material such as charcoal and at least one

ingredient which contains O, such as Fe oxide and  $\text{SiO}_2$  so proportioned that the ratio of C atoms to effective O atoms is from about 0.54 to about 0.66. Various other ingredients also are used

Welding rods. Oxweld Acetylene Co. Brit 433,097, Aug. 8, 1935. See U. S. 2,010,605 (C. A. 29,6566)

Welding metals Lloyd T. Jones, Harry E. Kennedy and Maynard A. Rotermund (to Union Carbide and Carbon Research Laboratories, Inc.). Brit. 433,723, Aug. 20, 1935

In the elec-fusion welding or deposition of metals with a fusible metal electrode, 1 end of the electrode is placed in contact with or submerged in a mass placed over the joint and consisting of unbonded finely-divided material which is electrically conductive when molten and composed mainly of fusible silicates and contg no uncombined Fe oxide or substances evolving gases, a portion of the mass near the electrode is melted and welding heat is generated by passing an elec. current from the electrode through the mass to the work. In an example, the mass is previously made by melting  $\text{CaCO}_3$ , 50,  $\text{MgO}$  9.4 and  $\text{SiO}_2$ , 40.5 lb, cooling, granulating and subsequently adding 1 oz of fluorspar to 1 lb. of product.

Welding steels Frederick M. Decket and Russell Franks (to Electro Metallurgical Co.). Brit. 433,585, Aug. 16, 1935. Cr-Ti steels contg. about 3-30% Cr or Cr-Ni-Ti steels contg. about 3-30% Cr and 5-35% Ni are welded by the oxyacetylene or elec. arc process with the use of a steel welding rod contg. about 3-30% Cr, 5-35% Ni and a substantial amt. of Cb to produce a ductile corrosion-resisting joint. The filler material contains C up to 0.3%, the Cb being preferably 4-10 times the C but not over 5%. The C content of the base metal may be up to about 0.3% with Ti at least twice the C but not over 5%.

Fluxes Hanson-Van Winkle-Munning Co. Brit 432,746, Aug. 1, 1935 A flux for use in galvanizing consists of 1 or more fluxing salts, e. g.,  $\text{NH}_4\text{Cl}$ ,  $\text{ZnCl}_2$ ,  $\text{Zn NH}_4$  chloride, and 1 or more substances, selected from the glycosides, e. g., licorice root, saponin, soapbark, amygdalin, phloridzin, populin, salicin, soap wort root, tannin and the partly or wholly  $\text{H}_2\text{O}$ -sol carbhydrates, e. g., xylene, dextrose, cane sugar, starch, galactose, lactose, maltose, which will stabilize the froth formed by the gas evolved from the fluxing salt or salts. In 432,747, Aug. 1, 1935, the surface of a metal to be galvanized is prepd by passing the metal through a flux wash comprising a dil aq soln of a fluxing compd., e. g.,  $\text{HCl}$ ,  $\text{NH}_4\text{Cl}$  and a froth stabilizer that reduces the surface tension and increases the viscosity of the soln, e. g., glycerol, tallow, oatmeal, bran, a glycoside or a carbhydrate.

Solder-extruding apparatus Arthur S. Berry (to Berry Solder Co.). U. S. 2,021,828, Nov. 19 Various mech features are described of app suitable for extruding solder in the form of strands with a flux core.

## 10—ORGANIC CHEMISTRY

CLAS. A ROULLIER AND CLARENCE J WEST

The mechanism of some important organic reactions. The dissociation of carbon bonds Otto Schmidt *Chem Rev* 17, 137-54 (1935), cf. C. A. 27, 3910—A discussion of the disscion of C bonds in the scission of the sugar mol and in the cracking process L K

Stereochemistry of solid substances Wilhelm Rdtz, *Ber* 68A, 91-103 (1935)—An address J F M.

Characterization of hydrocarbons Ernst Galt, Rudolf Klatt and Walter Friedl *Montan Rundschau* 27, No 18, 14 pp (1935)—To distinguish paraffin from naphthene hydrocarbons phys methods are more convenient than chem methods Differences in fluorescence in ultraviolet light (Fe or Hg arc) appeared in paraffin, ceresin, carnauba wax, montan wax, beeswax, petr. ether, petroleum and mineral oil, but when measured by Ostwald's method were too small to permit the use of this property in the study of mixts No fluorescence was found in cyclohexane (I),  $\text{C}_7\text{H}_{14}$  (II),  $\text{C}_8\text{H}_{18}$  (III),  $\text{C}_9\text{H}_{20}$  (IV) and

low-boiling fractions of Pennsylvania oil (V). On soln in concd  $\text{H}_2\text{SO}_4$  V gave strong fluorescence, light blue, dark blue, green and yellow-green with increasing b. p. Xylene also gave yellow-green fluorescence in concd.  $\text{H}_2\text{SO}_4$ . On crystn. from  $\text{Et}_2\text{O}$  the waxes can be distinguished by their cryst form (photomicrographs), which appears the same in either ordinary or ultraviolet light. I and IV on slow crystn give star-shaped crystals which soon change to a blunt needle form. The soly. of II in  $[\text{C}(\text{ClH}_3)_3]\text{O}$  at  $-10^\circ$ ,  $-17^\circ$  and  $-80^\circ$  was detd by measuring  $n_D^{20}$  (graph for known compns). The best sepn. occurs when equal vols. are used. At  $-80^\circ$  III is sol in 3 vols of VI and can be sepd. from I, II and V are insol in VI, IV is sol. Quant. data for these mixts are given Paraffin and naphthene oils can be sepd. from aromatic and unsatd. oils, but not from each other, by chlorox (VI). W. F. Bruce

Synthesis of alkyl acetylenes from calcium acetylide.

Thomas H. Vaughn and James P. Danehy. *Proc. Indiana Acad. Sci.* 44, 141 (1931).—Ca acetylide (I) is obtained either by the passage of  $\text{HCl}/\text{CH}$  into  $\text{Ca-NH}_2$  solns. or by the addn. of  $\text{Ca-NH}_2$  solns. to  $\text{HC}/\text{CH}$   $\text{NH}_2$  solns., the 2nd scheme being the most rapid. I (50%), reacts vigorously with  $\text{I}_2/\text{NH}_3$  to give a theoretical yield of pure I butyne.  $\text{AlCl}_3$ ,  $\text{AlBr}_3$  and  $\text{BuLi}$  react with I in  $\text{NH}_3$ , producing the corresponding acetylides in yields of 50–31 and 45%, resp.

W. J. Peterson  
The choice of optimal pressure of absorption of butadiene. Ya. L. Zhigalin. *Soviet Kauchuk* 1935, No. 5, 22–6.—Detailed calcs. of the power used at different pressures of absorption of butadiene in alc. at 30°, are given. The comparative costs of power used, of desorption of alc., and of rectification of alc. at different pressures are given. The optimal pressure should be 5 atm.

Δ. P. Stoff  
The chlorination of propane. II. The heterogeneous reaction. L. H. Meyerson and Samuel Yuster. *J. Phys. Chem.* 39, 1111–23 (1935), cf. *C. A.* 29, 6522.—The reaction of  $\text{C}_3\text{H}_8$  with  $\text{Cl}_2$  was studied under various conditions and a mechanism postulated. High partial pressures of  $\text{Cl}_2$  tend to poison the catalyst. Secondary chlorination in part was shown to be due to the addn. of  $\text{Cl}_2$  to  $\text{C}_3\text{H}_7$  formed by the pyrolysis of  $\text{Pr}$  chlorides. The high percentage of 1,2-dichloropropane in the products supports this view. High  $\text{Cl}_2$  partial pressures, high temps. and high rates of flow all favored the formation of more highly chlorinated products. Unsat. was found in all products collected when the  $\text{Cl}_2$  employed was 100%. II. A.

1,1-Diodoethene or vinylidene iodide. I. The supposed "1,1-diodoethene" of H. P. Kaufmann and Th. Uetzel. Guy I. Mechwiller. *Bull. soc. chim.* [5], 2, 1025–31 (1935), cf. *C. A.* 28, 3000<sup>1</sup>. K and U (cf. *A.* 16, 2302) claim to have prep. 1,1-diodoethene,  $\text{C}_2\text{H}_2\text{I}_2$  (I), by the isomerization of ethereal acetylene iodide,  $\text{C}_2\text{H}_2\text{I}_2$  (II) in the presence of metallic Na. Repetition of this work has shown that the product is, in reality, a mixt. of *trans*-II and diiodoethene,  $\text{C}_2\text{H}_4\text{I}_2$  (III). Finely divided Na (10 g.) was introduced into a soln. of 30 g. of II in 70 cc. of anhyd.  $\text{I}_2/\text{O}$ . The mixt. was refluxed on a shaking machine for 21 days. Every 3rd day, 2 g. of Na was added. The product was filtered and the filtrate, combined with the  $\text{I}_2/\text{O}$  washings of the Na, was evapd., yielding 3 g. (10%) of white needles, m. 70–5°. Repetition of the exp. over a period of 6 days gave 18 g. of a mixt. of *trans*-II, m. 72°, and a product, III, m. 70–5°, with a characteristic odor. From the m-p curve it was evident that K's product contained 70% of unchanged *trans*-II and 30% of III, converted by the action of ultraviolet light into C and  $\text{C}_2\text{H}_4\text{I}_2$ , m. 184°. The treatment of 20 g. of II in 50 cc. of abs.  $\text{I}_2/\text{O}$  with 10 g. of dry NaOH gave no decomp. on refluxing the mixt. for 7 days but, on the addn. of 5 cc. of abs. alc., 800 cc. of  $\text{CH}_3\text{CH}$  and 8 g. of III were produced. I. identically the  $\text{NaI}$  produced in K's procedure was hydrolyzed to NaOH by the damp air introduced on opening the flask to admit the Na at intervals with the resulting decomp. of II to  $\text{CH}_3\text{CH}$  and III. I has not yet been repd. C. R. A.

Ethylene nitriles. R. Vandeweyer. *Bull. soc. chim. Belg.* 44, 376–86 (1935).—Sepn. of the *cis* and *trans* isomers of  $\alpha$ -hexene- and  $\alpha$ -heptenenitriles has been accomplished by 13 fractionations of the resp. mixts. at atm. pressure. *cis*- $\alpha$ -Hexenenitrile, b. 140–6–8°, d<sub>4</sub> 0.82410, *trans*-, b<sub>10</sub> 165–8–0°, d<sub>4</sub> 0.82010. *cis*- $\alpha$ -Heptenenitrile, b<sub>10</sub> 127–3–7.5°, d<sub>4</sub> 0.82161; *trans*-, b<sub>10</sub> 141–7–4–0°, d<sub>4</sub> 0.82311.

W. J. Peterson  
The alkyl and aryl sulfonic esters. P. Carré and D. Liebermann. *Bull. soc. chim.* [5], 2, 1700–3 (1935).—See *C. A.* 29, 6200<sup>1</sup>.

A review on the organic compounds of phosphorus. V. M. Metz. *Uspekhi Khim.* 4, 573–609 (1935).—The prepn., properties, reactions and interrelations of the various classes of org. Ph compds. with I to several alkyl or aryl groups and with various anionic groups, as halogens, etc., as well as of compds. contg. a P–P, P–N, P–S or P–O bond are discussed. As yet unpublished is the reaction of  $\text{Ph}_3\text{N}/\text{Cl}$  in the presence of Cu with  $\text{PCl}_5$  and

$\text{PhPCl}_2$  to give  $\text{N}_2$  +  $\text{PhPCl}_4$  and  $\text{Ph}_2\text{PCl}_2$ , resp. One hundred and seventy other references. T. H. Rathmann

Optical rotation of configurationally related aldehydes. P. A. Levene, Alexandre Rothman and Martin Kama. *J. Biol. Chem.* 111, 759–47 (1935). Active  $\text{AlOH}$  oxidized by the method of I. H. G. gave 1- $\text{CHMeCH}_2\text{CHO}$ , b. 90–2°,  $[\alpha]_D^{20}$  20.5°. 1- $\text{CHMeCH}_2\text{CH}_2\text{CHO}$  from 31 g. of active  $\text{AlOH}$  in  $\text{I}_2/\text{O}$  (85% ) was drop. (d into boiling  $\text{CHCl}_3$  (100 g.). Hydrolysis of the product with 5 N  $\text{H}_2\text{SO}_4$  for 15 min. and purification over the  $\text{NaHSO}_4$  compd. gave 1- $\text{CHMeCH}_2\text{CH}_2\text{CHO}$ , b. 122°, d<sub>4</sub> 0.8010, n<sub>D</sub> 1.4001,  $[\alpha]_D^{20}$  8.7°. Similarly 1- $\text{CHMeC}(\text{CH}_3)\text{CHO}$ , b. 141°, d<sub>4</sub> 0.8112, n<sub>D</sub> 1.4081,  $[\alpha]_D^{20}$  12.0°, and 1- $\text{CHMeC}(\text{CH}_3)\text{CH}_2\text{CHO}$ , b<sub>10</sub> 72°, d<sub>4</sub> 0.8161, n<sub>D</sub> 1.4114,  $[\alpha]_D^{20}$  12.0°, were prepd. Values for  $[\alpha]_D^{20}$  are mixt. and taken in heptane. Due to the proximity of the first absorption band in the ultraviolet of aldehydes the analysis of their rotatory dispersion curves can be made with greater rigor than that of the acids. The partial rotation of the  $\text{CHO}$  group (due to the absorption band at  $\lambda 2900$ ) changes its sign when  $n$  passes from 0 to 1 in substances of the type 1- $\text{CHMeC}(\text{CH}_3)\text{CHO}$  and the direction of this partial rotation remains constant when  $n = 2$  and 3. An unmistakable periodicity does occur, however. The assumption previously made in regard to the configuration relationships of the primary and secondary azides is thus strengthened.

R. C. I. Herfield  
Preparation of mixed formal by direct acetalization. II. Wuyts and P. Docquier. *Bull. soc. chim. Belg.* 44, 297–308 (1935), cf. *C. A.* 18, 1970–1. A mixt. of 320 g.  $\text{MeOH}$ , 460 g.  $\text{PrOH}$ , 300 g. dry paraformaldehyde and 7.8 g.  $\text{H}_2\text{SO}_4$  is refluxed 12 hrs. with a 0° condenser and  $\text{CaCl}_2$  guard tube, washed with  $\text{NaOH}$ ,  $\text{NaHSO}_4$  and  $\text{NaOH}$  in turn, and fractionally distd. The fractions b. 50–61.5° (318.5 g.), mixed with 500 g.  $\text{CS}_2$  and refractionated, yield 120.7 g. pure  $\text{MeOC}_2\text{H}_4\text{O} \cdot \text{I}$  (I), b<sub>10</sub> 65.90° (d<sub>7</sub>/d<sub>4</sub> = 0.940), d<sub>4</sub> 0.8641, d<sub>20</sub> 0.8415. Binary azeotropic mixts. with  $\text{H}_2\text{O}$ ,  $\text{MeOH}$  and  $\text{I}$  OH contg., resp., about 95.0, 74.7 and 86.7% I, b<sub>10</sub> 61.25°, b<sub>10</sub> 77.1° and b<sub>10</sub> 83.95°, resp.; no ternary mixts. of these are found.  $\text{CH}_3\text{OH}$  (I) (60%) with  $\text{PrOH}$  forms a binary azeotrope, b<sub>10</sub> 80.15°, addn. of  $\text{H}_2\text{O}$  does not give a ternary azeotrope. A similar prepn. yields 1- $\text{OCH}_2\text{OPr}$  (II), b<sub>10</sub> 113.7°, d<sub>4</sub> 0.8512, d<sub>20</sub> 0.8316. The azeotrope of 81.6% II with  $\text{PrOH}$  b<sub>10</sub> 85.9°, that of 59.5% II and 22.5%  $\text{PrOH}$  with  $\text{H}_2\text{O}$  b<sub>10</sub> 83.8°, no others are found.

H. A. B.  
New method for the general preparation of *N*-carbethoxyketimines  $\text{RR}'\text{C}(\text{NCO})\text{C}_2\text{H}_5$ . Joseph Hoch. *Compt. rend.* 201, 660–2 (1935).— $\text{RR}'\text{C}(\text{O})\text{I}$  (I), treated with  $\text{NH}_4\text{CO}_2\text{Et}$  (I) and a trace of  $\text{PhNH}_2$ ,  $\text{HCl}$  at 100–100° gave  $\text{RR}'\text{C}(\text{NCO})\text{C}_2\text{H}_5$ , where R and R' are I, b. 105–6° (50% yield); Pr, b. 122–4° (80% yield);  $\text{MeC}_2\text{H}_5$ , b<sub>10</sub> 170–2° (45% yield);  $\text{C}_2\text{H}_5$ , b<sub>10</sub> 135° (76% yield);  $\text{CHMeC}(\text{CH}_3)$ , b<sub>10</sub> 141–5° (81% yield); and  $\text{MePh}$ , b<sub>10</sub> 162–3° (50% yield). When  $\text{RR}'$  was  $\text{Me}$ ,  $\text{Me}_2\text{C}(\text{NHCO})\text{C}_2\text{H}_5$ , b<sub>10</sub> 145–55°, was obtained and no acetonecarboxyethylamine. All are decomposed by  $\text{HCl}$  to give the original ketone and I.  $\text{MeC}(\text{OEt})\text{CHCl}_2$  (I), treated with I, gave  $\text{MeC}(\text{NHCO})\text{C}_2\text{H}_5$  (I)  $\text{CHCl}_2\text{Pr}$ , b<sub>10</sub> 137° (85% yield).

Julius White  
Molecular compounds in the systems: acid-salt, anhydride-salt and acid ester (acetic acid, butyric acid, phenylacetic acid). Maria Rakunin and T. T. Vitale. *Gazz. chim. ital.* 65, 503–616 (1935).—The present work deals with the thermic analysis of binary systems composed of (I)  $\text{AcOH}$  with  $\text{AcONa}$  and  $\text{AcOK}$ ; (2)  $\text{PrCO}_2\text{H}$  with  $\text{PrCO}_2\text{Na}$ ; (3)  $\text{PhCH}_2\text{CO}_2\text{H}$  with its  $\text{Rb}$ ,  $\text{K}$ ,  $\text{Na}$ ,  $\text{Li}$ ,  $\text{Ba}$ ,  $\text{PhNH}_4$ , and tertiary base salts; (4)  $\text{PhCH}_2\text{CO}_2\text{H}$  with  $\text{PhCH}_2\text{CO}_2\text{K}$  and  $\text{PhCH}_2\text{CO}_2\text{Na}$ ; (5)  $\text{PhCH}_2\text{CO}_2\text{H}$  with some of its esters and (6)  $\text{PhCH}_2\text{CO}_2\text{H}$  with  $\text{PhCH}_2\text{CO}_2\text{Pr}$ . The exptl. technique is described in detail, and the diagrams of state of each combination are given. The following important results were obtained on the various systems:  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{Li}$ .—No compd. One eutectic around 64° for approx. 19%  $\text{PhCH}_2\text{CO}_2\text{Li}$  ( $\text{PhCH}_2\text{CO}_2\text{O}$ )  $\text{PhCH}_2\text{CO}_2\text{Li}$ .—No compd. One eutectic around 67° for approx. 18%  $\text{PhCH}_2\text{CO}_2\text{Li}$ .

$\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{Na}$ —Two stable *comps.* ( $\text{PhCH}_2\text{CO}_2\text{H}$ ),  $\text{PhCH}_2\text{CO}_2\text{Na}$ , *m.* approx 95°, and  $\text{PhCH}_2\text{CO}_2\text{H}$  ( $\text{PhCH}_2\text{CO}_2\text{Na}$ ), *m.* approx 116°. Three eutectics at approx 67°, 62° and 102° for approx 13, 52 and 71%  $\text{PhCH}_2\text{CO}_2\text{Na}$ , resp. The diagram also shows in the region of high salt an infection characteristic of a 3rd *compd.*, which *dissoc.* below its *m.* *p.* and contains over 80% of salt. ( $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{Na}$ )—One unstable *compd.*, probably ( $\text{PhCH}_2\text{CO}_2\text{H}$ ) ( $\text{PhCH}_2\text{CO}_2\text{Na}$ ), which *dissoc.* around its *m.* *p.* at 110°. One definite eutectic around 50° for approx 39%  $\text{PhCH}_2\text{CO}_2\text{Na}$ , with indications of another eutectic at a higher proportion of salt.  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{K}$ —One stable *compd.*,  $\text{PhCH}_2\text{CO}_2\text{H}$   $\text{PhCH}_2\text{CO}_2\text{K}$ , *m.* around 142°. Two eutectics at around 50° and 130° for approx 17 and 64%  $\text{PhCH}_2\text{CO}_2\text{K}$ , resp.  $\text{PhCH}_2\text{CO}_2\text{H}$   $\text{PhCH}_2\text{CO}_2\text{Rb}$ —One stable *compd.*,  $\text{PhCH}_2\text{CO}_2\text{H}$   $\text{PhCH}_2\text{CO}_2\text{Rb}$ , *m.* around 128°. Two eutectics at 18° and 57° for approx 18 and 68%  $\text{PhCH}_2\text{CO}_2\text{Rb}$ , resp.  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{Ba}$ —Mixts *contg.* over 20% of salt showed marked vitrification, and even at low temps *crystn.* could not be induced.  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{NH}_4\text{Ph}$ —No *compd.* One eutectic at around 46° for approx 36%  $\text{PhCH}_2\text{CO}_2\text{NH}_4\text{Ph}$ .  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{NHMe}$ —Up to approx 40% of salt, *crystn.* began around -22°, but at higher concns no *crystn.* could be induced even at -90°, and the mixts became more viscous and ultimately vitreous.  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{Me}$ —A slight infection, indicating the probable formation of the *compd.*  $\text{PhCH}_2\text{CO}_2\text{H}$   $\text{PhCH}_2\text{CO}_2\text{Me}$ , dissociable below its *m.* *p.* One eutectic at approx -67° for approx 91%  $\text{PhCH}_2\text{CO}_2\text{Me}$ .  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{Et}$ —A point of infection indicating a *compd.* of the probable *compn.* ( $\text{PhCH}_2\text{CO}_2\text{H}$ ),  $\text{PhCH}_2\text{CO}_2\text{Et}$ , which *dissoc.* around 46°, below its *m.* *p.* One eutectic around -45° for approx 20%  $\text{PhCH}_2\text{CO}_2\text{Et}$ .  $\text{PhCH}_2\text{CO}_2\text{Na}-\text{PhCH}_2\text{CO}_2\text{Et}$ —One unstable *compd.*,  $\text{PhCH}_2\text{CO}_2\text{Na}$   $\text{PhCH}_2\text{CO}_2\text{Et}$ . One eutectic around -60° for approx 5%  $\text{PhCH}_2\text{CO}_2\text{Et}$ .  $\text{PhCH}_2\text{CO}_2\text{H}-\text{PhCH}_2\text{CO}_2\text{CH}_2\text{Ph}$ —No *compd.* One eutectic around -13° for approx 80%  $\text{PhCH}_2\text{CO}_2\text{CH}_2\text{Ph}$ .  $\text{AcOH}-\text{AcONa}$ —One unstable *compd.* of the probable *compn.* ( $\text{AcOH}$ ),  $\text{AcONa}$ , which *dissoc.* around 93°, below its *m.* *p.* Two *comps.* were reported by Kendall and Davison (C. A. 15, 2398). One eutectic around 4° for approx 6%  $\text{AcONa}$ .  $\text{AcOH}-\text{AcOK}$ —Two unstable *comps.* of the probable *comps.*  $\text{AcOH}$  ( $\text{AcOK}$ ), and ( $\text{AcOH}$ ),  $\text{AcOK}$ , both of which *dissoc.* below their *m.* *ps.* One eutectic at approx 0° for approx 15% of  $\text{AcOK}$ . A diagram by Davidson and McAllister (C. A. 24, 5205) is similar but less clearly defined.  $\text{PrCO}_2\text{H}-\text{PrCO}_2\text{Na}$ —Two unstable *comps.*, one of which has the *compn.*  $\text{PrCO}_2\text{H}$   $\text{PrCO}_2\text{Na}$ , and *dissoc.* at approx 48°, the other of which has an uncertain *compn.* but contains several mols. of  $\text{PrCO}_2\text{Na}$  per  $\text{PrCO}_2\text{H}$  mol. One eutectic around -30° for approx 25%  $\text{PrCO}_2\text{Na}$ . The behavior of the various *comps.* shows the influence of the cations of a salt on the formation and on the stability of its mol. addn. *comps.* Among the alk. metal group, the tendency to form addn. *comps.* increases with increase in the at. wt. In systems of esters and acids, addn. *comps.* are either not formed at all, or they are very unstable. The influence of the addn. *comps.* on the Perkin-Ogialoro synthesis (cf. B, loc. cit.) have been studied systematically, and will be the object of a later paper. C. C. Davis.

Vinylacetic acid C. Mannich *Arch. Pharm.* 273, 415-17 (1935)—This acid, prep'd by sapon of allyl cyanide with concd. HCl and subsequent rectification, *b.p.* 70-2°, the Et ester *b.p.* 119°, it yields on oxidation with  $\text{Br}_2/\text{O}$   $\beta$ -hydroxybutyrolactone, *b.p.* 110-15°.

W. O. E.

Duturic acid. Ralph W. Clark. *J. Am. Pharm. Assoc.* 24, 843 72 (1935)—The literature regarding duturic (margaric) acid published since 1811 is briefly reviewed. Curves are given of the *m.* *ps.* of fatty acids having an even no. of C atoms and of those having an odd no. of C atoms, and also of their Me esters. Ftn. of the ground seeds of *Datura stramonium* and treatment of the oil by the methods generally used in the past for the investiga-

tion of the *compn.* of the fatty acids (sapon, sepn. of the solid acids by means of the Pb salt- $\text{H}_2\text{O}$  method and sepn. of the solid fatty acids by fractional *crystn.* from alc, fractional pptn. of the Mg salts, and vacuum distn. of the Me esters) gave neg. results. It is concluded that studies on the identification of synthetic fatty acids should be carried out by means of other derivs. such as were used by Reid and co-workers, Whitby, etc., and that x-rays could probably be used to advantage in the study of duturic acid, the identity of which is not yet firmly established in the minds of all investigators. A. P. C.

Mechanism of three-carbon tautomerism D. J. G. Ives and H. N. Rydon. *Nature* 136, 476-7 (1935)—Vinyl-acetic (I), crotonic (II) and butyric (III) acids were, resp., treated with 1.05 mol. NaOH in *dd.*  $\text{D}_2\text{O}$  at 100°. No isotopic interchange was detectable with II or III but a marked one in I. These results indicate a parallelism between isotopic interchange and isomerization and hence the theories for the mechanism of 3-C isomerization as being purely an intramol. one are unacceptable. I. W.

The removal of alcohol from ethyl acetate. III. V. Longinov and V. Duzkal. *Trans. Inst. Pure Chem. Reagents* (U. S. S. R.) No. 14, 42-8 (1935)—By distn. of the crude ternary azeotropic mixt. of  $\text{AcOEt}$  (I),  $\text{H}_2\text{O}$  and alc. with excess  $\text{H}_2\text{O}$ , it has been found possible to obtain a binary azeotropic mixt. of I and  $\text{H}_2\text{O}$  (C. A. 24, 3755). The content of alc. was thus reduced from 8.4 to 4.5%. It is now reported that when such a ternary mixt. is washed in the vapor state with  $\text{H}_2\text{O}$ , a product *contg.* only 0.3% alc. is obtained. Two starting materials were used: (A) A purified *com.* I and (B) a crude *com.* I. A was 94% I, 6% alc. and 0.1%  $\text{AcOH}$ , B was 71.6% I, 17.7% alc., 7.5%  $\text{H}_2\text{O}$  and 3.2%  $\text{AcOH}$ . Typical *expt.*—Three I. B, distd. at a rate of 558 cc. per hr, the vapors being washed with 2.5 l. of  $\text{H}_2\text{O}$  flowing at a rate of 700 cc. per hr, gave 2 l. condensate (fraction 2, *b.* 70-1°) *contg.* 94.4% I, 0.3% alc. and 5.3%  $\text{H}_2\text{O}$ . The proportion of alc. in the condensate was greater if the  $\text{H}_2\text{O}$ /condensate ratio was less than that shown above. The amt. of alc. removed was not inversely proportional to the speed of distn. The yield of pure I was greatest (88%) with the conditions shown. Similar results were obtained with a Barlet still (10 l.), although with the most favorable  $\text{H}_2\text{O}$ /condensate ratio of 1.0 or more, flooding of the app. occurred. Therefore a ratio of 0.85 was employed, giving lower yields of a product *contg.* 0.4-0.5% alc. Fractionation of this to remove  $\text{H}_2\text{O}$  gave abs. I *contg.* 0.1-0.2% alc. in 50% yield based on the I in B. By recovering the head and tail fractions the yield is raised to 75%, and by starting with A it is 80% or more. Lewis W. Butz.

Amino alcohols with secondary alcohol groupings. Jean Matti and Emile Barman. *Bull. soc. chim.* [5], 2, 1742-4 (1935)—3-Diethylamino 2,2-dimethylpropanol (I) reacted with  $\text{PhMgBr}$  to give 1-diethylamino-2,2-dimethyl-3-phenyl-3-propanol, *b.p.* 123.5°, *n*<sub>D</sub><sup>20</sup> 1.5400, *d*<sub>4</sub><sup>20</sup> 0.96391; HCl salt, *m.* 196°; benzoate-HCl, *m.* 180°. 1-Diethylamino 2,2-dimethylpropanol (II) and  $\text{PhMgBr}$  gave dimethylamino-2,2-dimethyl-1-phenyl-3-propanol, *b.p.* 115°, *n*<sub>D</sub><sup>20</sup> 1.5182, *d*<sub>4</sub><sup>20</sup> 0.9742, HCl salt, *m.* 181°; benzoate-HCl, *m.* 208°. II and 1-tMgI gave 1-dimethylamino-2,2-dimethyl-3-pentanol, *b.p.* 83° *n*<sub>D</sub><sup>20</sup> 1.4789, *d*<sub>4</sub><sup>20</sup> 0.8610, benzoate-HCl, *m.* 163.5°, *m.* aminobenzoate-HCl, *m.* 184.5°; *p*-aminobenzoate-HCl, *m.* 194°; emmanate-HCl, *m.* 177°. II and BuMgBr gave 1-dimethylamino-2,2-dimethyl-3-heptanol, *b.p.* 119.5°, *n*<sub>D</sub><sup>20</sup> 1.4418, *d*<sub>4</sub><sup>20</sup> 0.8417, benzoate-HCl, 115°, *p*-aminobenzoate-HCl, *m.* 182°.

R. Baltzy.

The preparation of ethers of hydroxy aldehydes by oxidation of  $\alpha$  ethers of glycerol with lead tetracetate. Mme. S. Sabatay. *Bull. soc. chim.* [5], 2, 1744-6 (1935)— $\alpha$ -Ethers of glycerol were prep'd by warming allyl or aryl halides with  $\text{NaOCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OH}$ , and the ethers oxidized in benzene soln. by  $\text{Pb}(\text{AcO})_4$  in the cold. The prep'n of benzoyloxyacetaldehyde is described. R. D.

Aminolysis of leucine. Emil Baur and G. Schindler. *Helv. Chim. Acta* 18, 1147-9 (1935), cf. Wunderly, C. A. 27, 2136, 3975—In order to show that hydrolytic as well as reductive decarboxylation occurs in nature, leucine (I) was

changed to AmOH (II) by boiling with 10 parts of air-free bone black. From 10 g. of I, 1.5 g. of II was isolated by steam distn. followed by salting out with  $K_2CO_3$  and redistn. II,  $b_p$  55–55° (0.7 g.), gave the odor of AmOAc with  $H_2SO_4$  and  $HIO_4$ , and in alc. free from fusel oil gave the red-violet color reaction for fusel oil with  $H_2SO_4$  and furfural. The high b. p. is attributed to superheating and the presence of impurities. W. F. Bruce

Combination of thiol acids with methylglyoxal. Maxwell P. Schubert, *J. Biol. Chem.* 111, 671–8 (1935). Methylglyoxal forms 1:1 addn. products with glutathione and thioglycolic acid and its amide and phenylglyoxal forms similar compds. with these and with cysteine, taurine, thiosuccinic acid and thioacetamide. Both form condensation products with cysteine and pseudohomocysteine which possess a nearby  $NH_2$  group, by elimination of a mole of  $H_2O$ . The prepn. of cysteine and cysteine betaine is described. Cysteine betaine *farinariae*, m. 230°, cysteine *bovine farinariae*, m. 210°. It has been suggested that in the body glutathione forms a compd. with methylglyoxal and that this compd. yields lactic acid under the influence of glyoxalase. A. P. Lothrop

Synthesis of *l*-ribo-3-ketoheptonic acid lactone (*l*-allo-ascorbic acid). Marguerite Steiger, *Helv. Chim. Acta* 18, 1252–4 (1935); cf. *C. A.* 29, 6213. *l*-Allo-ascorbic acid treated with HCN and then hydrolyzed gave *l*-allo-ascorbic acid,  $O=C(OH)C(OH)CHCH(OH)CH(OH)CH_2OH$ , m.

176–7°,  $n_D^{20}$  1.50 ± 0.01 (0.01 N HCl). Julius White, *Acetylthymylcarbinol*. James R. Young and Allan M. Wilson, *J. Phys. Chem.* 39, 1135–8 (1935). Samples of acetylthymylcarbinol (I), m. –72°, prep'd from the polymer (II) by melting or distg., were kept at –10°, room temp., 30°, 100° and 130° and the  $n$  was det'd. at intervals. The initial value of  $n$  varied with the method of prep'n but the min. may be 1.4175.  $n$  increases with time more rapidly at higher temps. The  $d$  and viscosities of I kept at 30° varied similarly. By extrapolation freshly dist'd. I would have  $d_4^{20}$  0.9060,  $n_D^{20}$  0.0175. The vapor pressures of I increase from 162 mm. at 0° to 760 at 144°. Crystals of II,  $d$  1.26, are deposited in 2–9 days at room temp. Although in general it m. about 124°, II has no definite m. p. but passes to liquid above 30°. The transition temp. is 16° (dilatometer) but in the absence of I, II is stable at higher temps. II is monomol. in  $H_2O$ , AcOH and paraldehyde. In contact with Zn at –10°–0° I became solid in 24 hrs. The crystals are transparent, flat and monoclinic with angles 127°, 108° and 125° on the chloroformic acid face. Janet E. Austin

Formolins II. P. Karrer and Carlo Mucante, *Helv. Chim. Acta* 18, 1140–3 (1935); cf. *C. A.* 29, 5672. In a study of the effect of *p*-substituted Ph groups on the enolization of benzoylformolins the following amts. of enol were found. Benzoylformolin, 57%; *p,p'*-dimethoxybenzoylformolin, 40%; *p,p'*-dichlorobenzoylformolin (I), 100%; *p,p'*-dibromobenzoylformolin, 95–100%. By refluxing *p,p'*-dichlorobenzoylformolin with  $SeO_2$ , *p*-chlorophenylglyoxal was prep'd as the monohydrate,  $C_6H_4Cl_2C(OH)O$ , m. 122°. With KCN this gave I,  $C_6H_4Cl_2C(OH)CN$ , red plates, m. 190°. *p*-Bromophenylglyoxal hydrate,  $C_6H_4Br_2C(OH)O$ , m. 125°, similarly prep'd, gave *p,p'*-dibromobenzoylformolin,  $C_6H_4Br_2C(OH)O$ , m. 196°. W. F. Bruce

Synthesis with glyoxal semiacetal Nitroaldehyde III. Glyoxal, Hermann O. L. Fischer, Frick Baer and Heinrich Nidecker, *Helv. Chim. Acta* 18, 1079–87 (1935); cf. *C. A.* 29, 5075. (I)  $O=C(OH)CH_2OH$ , treated with  $MeNO_2$ , gave (I')  $CH_2(OH)CH_2NO_2$  (I), b.p. 136–38°,  $d_4^{20}$  1.132,  $n_D^{20}$  1.4410. I, warmed with 0.1 N  $H_2SO_4$  at 80–85° for 30 min., gave  $O_2NCH_2CH(OH)CH_2OH$  (II), m. 160° (decomn.). II, treated with dimedon, gave the dimedon complex, m. 180° (decomn.). II can be depolymerized by heating at 100–50° at 0.01 mm. II, treated with 2,4 ( $NO_2$ ),  $C_6H_3N_2NH_2$ , gave the hydrazide, m. 167–68°, osazone, m. 294–7°. I, treated with (CO-H), and reduced with Pd-BaSO<sub>4</sub>, gave (I')  $CH_2(OH)CH_2CH_2NH_2$  (II'), m. 113–5°. I, treated with activated Al and reduced, gave (I')  $CH_2(OH)CH_2CH_2NH_2$  (III), m. 43°,

$n_D^{20}$  1.4463. III on hydrolysis with fuming HCl gave  $NH_2CH_2CH(OH)CHO$ , m. 143–5°. Julius White

Hydrolysis velocity of cyclic acetals. II. Rudolf Leutner, *Moravsk. 66*, 222–48 (1935), cf. *C. A.* 26, 5820. The following values are reported for  $k$ , (velocity of acid hydrolysis in  $H_2O$  at 25°). pinacone formal,  $Me_3C(O)CH_2O CMe_2$ ,  $k$ , 0.000027; 1,3-butylene glycol formal,

$MeCH(O)CH_2O CMe_2$ ,  $b_m$  113–14°,  $k$ , 0.000048;

1,3-butylene glycol acetal,  $b_m$  118°,  $k$ , 0.21; 1,3-butylene glycol acetonal,  $MeCH(O) CMe_2O CMe_2$ ,  $b_m$  129–5–

30.5°,  $k$ , 338; pinacone acetonal,  $Me_3C(O) CMe_2O CMe_2$ ,

$b_m$  147.5–5°,  $k$ , 0.0120. The relative ease of splitting of the 6-membered ring of 1,3-butylene glycol acetals of  $HCHO$ ,  $AcOH$  and  $Me_3CO$  is about  $1.4 \times 10^3 : 7 \times 10^4$ .

C. J. West

Anhydrous glyoxylic acid Arvi Talvite, *Swen. Kemistilshs* 8B, 32 (1935) (in German). A preliminary report. A dil. aq. soln. of glyoxylic acid, prep'd by adding the calcd. quantity of oxalic acid to Ca glyoxylate, is evaporated in a vacuum desiccator over  $H_2SO_4$  at room temp. The resulting colorless sirup is treated with abs. ether to remove traces of Ca oxalate and evaporated (about 3 weeks under a pressure of 1 mm. Hg over  $H_2SO_4$ ). The anhyd. glyoxylic acid is very hygroscopic. F. E. Jukkola

The formation of "acetylacetic ester" II. F. Seidel, W. Thier, A. Uher and J. Dittmer, *Ber.* 68B, 1913 21 (1935), cf. *C. A.* 26, 5075. It has been reported that in the prep'n of  $AcCHCO_2Et$  according to Claisen there is also formed  $Ac_2C(O_2Et)_2$  (I), since the reaction product gave with  $NH_3$ , a product (II) assumed to be 1,3,5-dimethylpyrazole-4-acetyl-4-carboxylate (III). v. Auwers (*C. A.* 27, 162) doubts the existence of I and the correctness of the structure III for II. It has now been found that II changes above its m. p. into  $\beta$ -(5-hydroxy-3-methyl-4-pyrazolyl)crotonolactone (IV), and hence II is not a condensation product of I with  $NH_3$ , but  $\beta$ -(5-hydroxy-3-methyl-4-pyrazolyl)crotonolactone (V). The  $NH_3$ , first deacetylates the  $AcCHCO_2Et$  in the reaction mixt., forming  $AcCH_2CO_2Et$  and  $AcNHNH_2$  (the greater part of the  $NH_3$ , thus escaping further reaction), and the unchanged  $NH_3$  condenses with a part of the  $AcCH_2CO_2Et$  to 5-methyl-5-pyrazolone which combines with the rest of the  $AcCH_2CO_2Et$  to form V with loss of water. All attempts to prep. the true III (which is isomeric with IV) were unsuccessful, but they yielded hitherto unknown intermediate products which not only contributed to the knowledge of the reactivity of the pyrazoles and pyrazolones but also helped to establish the structure of V with certainty. 1,3,5-dimethylpyrazolone-4-carboxylate (VI), from  $AcCHCO_2Et$  (reacting in the mono-enolic form, of which the equil. mixt. contains 91.85% and  $NH_3$ , gives with  $AcCl$  the 1-acetyl-3,5-dimethylpyrazole (VIII). The structure of VIII is established: (1) by condensing  $AcCH_2$  (76% enol) with  $NH_3$  to 3,5-dimethylpyrazole (IX) and acetylating IX to VIII with  $AcCl$  in pyridine; (2) by synthesis of VIII from  $AcCH_2$  and  $NH_2NHAc$ . Direct replacement of the 4-H atom on the pyrazole nucleus by acyl is not possible; to obtain 3,5-dimethyl-4-acetylpyrazole (X),  $CH_3C(=O)CH_2$  (88.3% enol at equil.) must be condensed with  $NH_3$ . It reacts with  $AcCl$  to form the 1,4-di-*Ac* derivative (XI). It was hoped that IV (or the free acid) might be obtained by saponifying isodihydraetic ester to  $HO_2CCH_2CMe_2C(O_2Et)_2$  (C) (II) and condensing the latter with  $NH_3$ , but the product was always only 3-methyl-5-pyrazolone, the isodihydraetic ester evidently breaking down into  $AcCH_2CO_2Et$  under even the mildest conditions.

Definite proof that 3-methyl-5-pyrazolone reacts at the 4-position with  $AcCH_2CO_2Et$  to form V was obtained indirectly. Reaction through the  $NH$  group is excluded, for 3,4,4-trimethyl-5-pyrazolone does not react with  $AcCH_2CO_2Et$  but 1-phenyl-3-methylpyrazolone does, yielding  $\beta$ -(5-hydroxy-1-phenyl-3-methyl-4-pyrazolyl)crotonolactone (XII), which splits off  $EtOH$  at 165° to form the lactone

(XIII) Further evidence that the NH group is not involved in the condensation is afforded by the fact that IV forms a Bz deriv. V, obtained in 24 g. yield from a charge of 638 g.  $\text{AcCH}_2\text{CO}_2\text{Et}$ , 88.6 g. Na and 600 g.  $\text{AcCl}$ , m 188°, is also obtained readily by Wolff's method (*Ber.* 38, 3038 (1905)), it is stable toward boiling MeOH and alc. NH, but with  $\text{PhNH}_2$  and concd. KOH gives 3-methyl-5-pyrazolone, m 246°. Free acid, from IV and 10% NaOH at 15–20°, m 154° (decompn.), regenerates IV on heating *in vacuo* at 15–20° or in hot alc. VII (6.2 g. from 7.0 g. VI), m 69°, free acid, similarly obtained (6.0 g.) from the free acid (5.0 g.) of VI, m 164°. VIII, b<sub>p</sub> 70°, b<sub>p</sub> 81°. 1-Bz analog, b<sub>p</sub> 158°. X, needles with 1 H<sub>2</sub>O, m 121° (decompn.) and (anhyd.) 128°. XI, m 50°. XII, m 88°. XIII, light yellow, m. 132°. 1-Bz deriv. of VI, m 181°. C A R

Oxidation of monoethyl dimethylmalonate with potassium persulfate. F. Fichter and Jules Heer *Helv. Chim. Acta* 18, 1276–80 (1935), cf. C. A. 29, 5077°.  $\text{Me}_2\text{C}(\text{CO}_2\text{Et})_2$  (I), partially saponified with KOH, gave after acidification  $\text{Me}_2\text{C}(\text{CO}_2\text{Et})\text{CO}_2\text{H}$  (II), b<sub>p</sub> 101–2°, b<sub>w</sub> 80°. II, heated with 1.5 N KOH and  $\text{K}_2\text{S}_2\text{O}_8$  for 30 min. on a water bath, gave I and  $(\text{Me}_2\text{C}(\text{CO}_2\text{Et})_2)$  (III), b 218–20° ( $\text{Me}_2\text{C}(\text{CO}_2\text{H})_2$ ) (IV), m 188.5°. III, treated with  $\text{PhNH}_2$ , gave the phenylimide, m 88°. Similarly  $\text{Me}_2\text{CHCO}_2\text{H}$  and  $\text{Me}_2\text{CHCO}_2\text{Et}$  gave resp. IV and III.

Parachor and chemical constitution. III. Structure of urea and thiourea. Susil Kumar Ray *J. Indian Chem. Soc.* 12, 404–9 (1935), cf. C. A. 29, 6227°. The surface tensions of urea (I) and thiourea (II) have been detd. by the method of max. bubble pressure. Xylene was used as the manometric liquid. The compds. decompose on fusion and consequently the surface tension and d were detd. in soln. The parachor values calcd. from the results obtained for I and II in aq. solns. and for I in  $\text{HCONH}_2$  agree with the carbamide structure universally accepted. The low parachor value of 125 for I in EtOH and MeOH is supposed to be due to assocn. between the solute mols. or between the solute and solvent mols. This was corroborated by detns. of the mol. wt. of I in MeOH and EtOH by the vapor-pressure-lowering method. The apparent mol. wt. of I in alc. soln. increases rapidly with increasing concn. The required values were obtained by plotting the parachor results against concns. and finding the intersection point of the curve with the parachor axis. C R Addinal

Crystalline hypoxanthine. H. Stendel *Z. physiol. Chem.* 236, 228–9 (1935) —Hypoxanthine and xanthine, obtained in the usual manner, were purified by repeated recrystn., hypoxanthine as the HCl salt and xanthine as the sulfate and the  $\text{NH}_4$  compd. By liberation of the base from its HCl salt by treatment with NaOAc, hypoxanthine was obtained in large rhombic plates, while xanthine yielded only small prisms which did not alter their appearance after 10–12 recrystns. The illustrations of xanthine crystals in numerous textbooks are undoubtedly of hypoxanthine, and the error has been perpetuated for more than 2 decades. A W. Dox

Chromium-bisulfonate complexes. Priyadarshan Ray and Haribabu Saha *J. Indian Chem. Soc.* 12, 621 (1935) —The constitution of complex compds. of bisulfonate with bivalent Cu, Co and Ni is uncertain. Sol. trivalent Cr-bisulfonate complexes have been prepd. The free base (I), obtained as ruby red crystals, has the compn.  $[\text{Cr}(\text{C}_6\text{H}_5\text{SO}_3)_3]\text{H}_2\text{O}$ , the bisulfonate mol. acting as a bidentate group and occupying 2 coordinate positions. I is expected to give rise to optically active enantiomers. In aq. solns. it behaves as a triacidic base. Similar compds. with trivalent Cu have also been prepd. C R A

The structure of heterosides from their absorption in the ultraviolet. Mme. Ramart-Lucas and J. Rakaté *Bull. soc. chim.* [5], 2, 1596–1625 (1935) —Since the common sugars and their alkylsides have no absorption in the ultraviolet for  $\lambda$  less than 2400 Å, the absorption of heterosides in this part of the spectrum should be a function of the prosthetic group, more or less modified by the displacement of the H on the HIO by the sugar. Absorp-

tion spectra were made of the following natural and synthetic heterosides and of most of their prosthetic groups:  $\beta$ -Me glucoside, Me mannose, aucuboside, amygdaloid, benzyl glucoside, benzyl galactoside, amygdalitrile glucoside, phenylethyl glucoside, betuloside, salicyl glucoside, phenol glucoside,  $\alpha$ -,  $m$ - and  $p$ -cresol glucosides, tetraacetyl- $p$ -cresylglucoside, guaiaacil glucoside, resorcinol glucoside and its tetraacetate, arbutoside, Me arbutoside, salicoside, conferoside, helicoside, monotroposide, lusitanoside, geoside, piceoside, chloropiceoside and cinnamyl glucoside. From these it appears that when the prosthetic group is combined with the sugar through an alc. HO the absorption curves of the prosthetic group and of the heteroside are almost, if not entirely, identical. Where the HO of the prosthetic group is phenolic the absorption curves are similar in shape but that of the heteroside is shifted toward the shorter wave lengths and the absorption is usually less intense. This shift is usually 40–90 Å with simple phenols, but is over 200 Å in some cases where the prosthetic group contains aldehyde or other groups strongly affecting the absorption. R. Baltzy

Catalytic oxidation of the carbohydrates and related compounds by oxygen in the presence of iron pyrophosphates. IV. Methyl alcohol, formaldehyde, formic acid, sodium formate, ethyl alcohol, acetaldehyde, acetic acid, sodium acetate, glycol, glycolic acid, sodium glycolate, oxalic acid and sodium oxalate. E. F. Degering. *Proc. Indiana Acad. Sci.* 44, 129–31 (1934); cf. C. A. 28, 7244°.  $\text{MeOH}$ ,  $\text{CH}_2\text{O}$ ,  $\text{HCO}_2\text{H}$ ,  $\text{HCO}_2\text{Na}$ ,  $\text{EtOH}$ ,  $\text{AcOH}$ ,  $\text{AcONa}$ ,  $\text{HOCH}_2\text{CH}_2\text{OH}$ ,  $\text{CH}_3(\text{OH})\text{CO}_2\text{H}$ , Na glycolate, and Na oxalate are not oxidized to give  $\text{CO}_2$  and could therefore be identified as end products in the oxidation of the simple sugars under the conditions of these expts.  $\text{AcH}$  is oxidized to give  $\text{CO}_2$ , but  $\text{AcOH}$  cannot be an intermediate step in the oxidation of  $\text{AcH}$  to  $\text{CO}_2$ . At 75° ( $\text{CO}_2\text{H}$ ), yields  $\text{CO}_2$ , but little, if any, at 50°. W. J. P.

Chemical studies on sulfuric acid esters of sugars. I. A rough estimation of proportion of glucose polysulfates in their mixture. Tokuro Soda and Willy Nagai *J. Chem. Soc. Japan* 56, 1258–62 (1935) —By estg. the velocity const. of the hydrolysis of mono- and poly-sulfates of glucose, an approx. estn. of the mono-, di- or poly-sulfates can be made. K. Katsuta

Cyclohexyl sulfite. W. Voss and W. Wachs *Zer.* 68B, 1939–41 (1935) —Carré and Liebermann (C. A. 29, 3314) claim that neither the method of Voss and Blanke (C. A. 25, 1797) nor that of Katsuta and Sone (C. A. 25, 3964) gives cyclohexyl sulfite. According to them the product obtained by the V and B method from cyclohexanol and  $\text{SOCl}_2$  is only cyclohexene. Thus V. and W. deny unqualifiedly, a repetition of the work has confirmed the earlier results in all respects except as to the yield, which is 45% instead of 54%. The product prepd. by V. and B. 7 yrs. ago showed the same const., after purification, as a freshly prepd. sample. Complete analysis, mol.-wt. detns. and the amt. of cyclohexanol isolated after alk. hydrolysis point to the substance as being the neutral cyclohexyl sulfite,  $\text{C}_6\text{H}_{11}\text{O}_3\text{S}$  (I). Special importance is attached to the satisfactory agreement between the observed and calcd. values for mol. refraction. Any possibility of its being cyclohexene is eliminated; the differences as respects  $\alpha_D$ ,  $n_D$ ,  $d_4$ ,  $n_D$  and  $n_D$  are in part considerable. I. b<sub>p</sub> 173–3.5°, mol. wt. in benzene 238,  $n_D^{20}$  1.4809,  $d_4^{20}$  1.0697,  $d_4^{25}$  1.0690,  $n_D^{40}$  1.48402, 1.48497, 1.49362 for  $\text{H}_a$  (17.6°), D (17.3°) and  $\text{H}_b$  (17.5°). Cyclohexene, b<sub>p</sub> 85–5–6.5°,  $n_D^{20}$  1.48321,  $n_D^{40}$  1.44512, 1.44655, 1.45480 for  $\text{H}_a$  (17.3°), D (17.3°) and  $\text{H}_b$  (17.5°). C A R

Multiplanar cyclohexane rings. R. D. Desai and R. I. Hunter *Nature* 136, 608–9 (1935), cf. Quadri-Khudi. C. A. 29, 7294°. —D and H conclude that there are only 2 stereoisomeric forms of 4-methylcyclohexane-1-carboxyl-1-acetic acid. They have likewise been able to isolate only 1 form of carboxy-3,3-dimethylcyclohexane-1-acetic acid. E. O. Wug

Isomers and derivatives of hexetone. R. Wegler and W. Frank *Arch. Pharm.* 273, 408–14 (1935). —In an

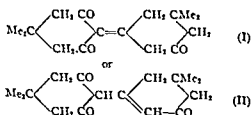
attempt to find an ideal substitute for camphor as a heart stimulant—so far negative—certain derivs., notably reduction products of hexetone, and also amines were prepd. Of more general interest is the manner of specific reduction. Thus with Pd on animal charcoal only the double-bonded C in the nucleus was hydrogenated, leaving the CO group unaffected. Pt, on the other hand, reduces the CO group to an alc., and finally to a hydrocarbon with animal charcoal. This behavior, whereby Pt very frequently reduces more energetically or unspecifically, was likewise observed in the case of Schiff bases of hexetone, which, although unaffected by Pd were readily reduced with Pt. *d*-Hexetone-phenylhydrazine-*p*-sulfonic acid, m. 228°, is split by 10%  $H_2SO_4$  into hexetone and PhNHNH<sub>2</sub>. The  $\alpha$ -phenylethylamine compd. yielded on treatment with tartaric acid 2 antipodes having  $\alpha_D^{20}$  -38.7° and  $\alpha_D^{25}$  37.8°. The said ketone, obtained with PdCl<sub>2</sub> and animal charcoal, b<sub>11</sub> 96–8°, and possessed a typical menthol odor, as also the corresponding alc. (obtained from the ketone by treatment with Pt and K in aq. MeOH), b<sub>11</sub> 152°. The hydrogenated hexetone yielded with ethanolamine the Schiff base b<sub>11</sub> 125°, which, reduced with Pd and animal charcoal, gave 1-methyl-3-isopropyl-5-aminocyclohexene, b<sub>11</sub> 90°, a strong smelling amine, gives cryst. salts with tartaric, quinic and camphorsulfonic acids, it may be split into its antipodes by recourse to diphenylsuccinic acid; a coramine-like compd., in the form of a bright yellow oil, b<sub>11</sub> 230°, on treatment with nicotinic acid-HCl.

W. O. F.

The constitution of the methylketones II Koster *Rieckhoff-Ind.* 10, 150 (1935).—By the condensation of citral with MeCOEt and cyclization of the mixt. of methyl-pseudoionone (V) and isomethylpseudoionone (VI) arises 4 isomers: (I)  $\alpha$ -methylionone, b<sub>11</sub> 125–6° (semicarbazone, m. 146°), (II)  $\beta$ -methylionone, b<sub>11</sub> 133° (semicarbazone, m. 140°), (III)  $\alpha$ -isomethylionone, b<sub>11</sub> 121–2° (semicarbazone, m. 202°) (IV)  $\beta$ -isomethylionone, b<sub>11</sub> 124–5° (semicarbazone, m. 182°). III and VI arise by the condensation of  $\alpha$ -cyclocitral with MeCOEt and also as the chief products when the mixt. of V and VI are cyclized with  $H_2PO_4$ . III and IV are valuable in perfumery and known as Iraldein Gamma and Iraldein Delta.

H. M. Buriag.

Anhydrosilmethene (bithione). N. J. Tuovonen, A. Tauno Fjäder and Armas Heikel. *Suomen Kemistilehti* 8B, 32–3 (1935) (in German); cf. *Suomen Kemistilehti* 6A, 72 (1933).—Heating of methone (dimethylcyclohexadione) at 150–70° gives a condensation product, bithione, having the structure



It is formed as a reddish yellow syrup on the surface of an aq. alc. or slightly acid-NH<sub>4</sub>Cl soln. of methone on long heating. Metallic K added to methone in benzene gives a deep red K salt. Colorless crystals are formed from the syrup after washing with ether or benzene. The product recryst. from boiling water m. 155–7°; the mixed m. p. with methone is 148–50°. It is sol in alc., AcOH, acetone and CHCl<sub>3</sub>; difficultly sol. in benzene, ether, water and petr. ether. The slightly yellowish aq. soln. gives a deep violet color with FeCl<sub>3</sub>. Long boiling in water gives some methone. Analysis gave C 72.90, H 8.51; calcd. for C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>, C 73.24, H 8.51%; mol. wt. 267, calcd. 202.2, 0.2253 g. required 8.66 cc. of 0.1 N NaOH, calcd. 8.61. Analysis of the Ag and Cu salts gave Ag 29.10, calcd. 29.23; Cu 10.93, calcd. 10.85%. The self-condensation of  $\beta$ -diketones in aq. soln. appears to be general. E. E. J.

The self-condensation of acetylacetone. Armas Heikel. *Suomen Kemistilehti* 8B, 33–4 (1935) (in German); cf.

preceding abstr.—Forty g. CH<sub>3</sub>Ac, 40 ml. 2 N NaOH and 200 ml. water were refluxed for 24 hrs. and then distilled. The distillate sepd. into 2 layers, each contg. the condensation product. The lower aq. part was concd. by renewed distn., the dried oily portion distilled and the residue remaining at 145° was crystd. from water. The recrystd. white needles, m. 58°, were sol in the common solvents, also petroleum, gave a yellow soln. with alkalis and concd. H<sub>2</sub>SO<sub>4</sub>. The substance was easily brominated, gave a pos. Lieberman reaction and a violet color with aq. FeCl<sub>3</sub>. The analysis was C 72.80, H 7.61%, mol. wt. 116.2. The properties check those of 3,5-dimethyl-2-acetylphenol. E. E. Jukkola.

Optical rotation of methyloctylphenylmethane. P. A. Levene and Stanton A. Harris. *J. Biol. Chem.* 111, 735–8 (1935).—Ph(CH<sub>3</sub>)<sub>2</sub>CHMeCH<sub>2</sub>MgBr and CH<sub>3</sub>CHO gave Ph(CH<sub>3</sub>)<sub>2</sub>CHMeCH<sub>2</sub>CH(OH)C<sub>2</sub>H<sub>5</sub>, b<sub>11</sub> 182–4°, d<sub>20</sub> 0.9131, n<sub>D</sub><sup>20</sup> 1.4952, [M]<sub>D</sub><sup>20</sup> -29.68°. This with anhyd. HCl and reduction with Raney's catalyst gave Ph(CH<sub>3</sub>)<sub>2</sub>CHMeC<sub>2</sub>H<sub>5</sub>, b<sub>11</sub> 143–5°, d<sub>20</sub> 0.8590, n<sub>D</sub><sup>20</sup> 1.4832, [M]<sub>D</sub><sup>20</sup> -12.59° which, reduced with PtO<sub>2</sub>, gave C<sub>11</sub>H<sub>19</sub>(CH<sub>3</sub>)<sub>2</sub>CHMeC<sub>2</sub>H<sub>5</sub>, b<sub>11</sub> 147°, d<sub>20</sub> 0.8260, n<sub>D</sub><sup>20</sup> 1.4553, [M]<sub>D</sub><sup>20</sup> -5.27°. From this the cyclohexyl group furnishes a much higher partial rotation than the iso-*p*-propyl group.

R. C. Elderfield

The preparation of nitrobenzene with a maximum specific resistance. L. Zepalova-Mikhailova. *Trans. Inst. Pure Chem. Reagents* (U. S. S. R.) No. 14, 49, 57 (1935).—Nitrobenzene (I) with a sp. resistance ( $\rho$ ) of about 10<sup>11</sup> ohms per cc. was desired for special purposes in television app. A freshly distd. sample of I having phys. consts. as given in the literature had  $\rho = 4.29 \times 10^8$  ohms/cc. at a potential of 1035.5 v.,  $2.23 \times 10^7$  (817 v.) and  $9.74 \times 10^7$  (65.5 v.), before distn. it had  $7.13 \times 10^7$  (570 v.) and  $3.62 \times 10^7$  (66.5 v.). After treatment with Al<sub>2</sub>O<sub>3</sub>, drying over P<sub>2</sub>O<sub>5</sub> and distn., it had  $1.86 \times 10^8$  (722 v.) and  $1.27 \times 10^8$  (133 v.), after treatment with Ag<sub>2</sub>O, drying over CaCl<sub>2</sub> and distn.,  $6.4 \times 10^7$  (570 v.) and  $1.75 \times 10^7$  (133 v.) and this last when redist. once more,  $6.56 \times 10^7$  (920 v.) and  $4.31 \times 10^8$  (190.5 v.). When I was steam-distd. in the presence of N NaOH the sample of I obtained had  $\rho 10^9$ . However, when CaH<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> is absent, the passage of an elec. current through I rapidly increased  $\rho$ . At 18°,  $\rho$  varied from  $2.27 \times 10^8$  (1358.3 v.) to  $2.73 \times 10^8$  (90.25 v.). When I was partially frozen, the liquid portion had a higher  $\rho$  than the solid ( $1.15 \times 10^8$  and  $0.89 \times 10^8$  at 1197 v.). On storing for more than 4 days,  $\rho$  fell to as low as 10<sup>6</sup>. From I prepd. from PhH under mild conditions (30°, excess H<sub>2</sub>SO<sub>4</sub> (98%), theoretical quantity HNO<sub>3</sub>) the numerous samples obtained by various methods of purification were always unstable in that  $\rho$  decreased on storage. The use of different types of glass in the app. for the prepn. did not solve the difficulty. I prepd. from *p*-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NH-NH<sub>2</sub> in boiling AcOH, contg. AcONa equiv. to H<sub>2</sub>SO<sub>4</sub> formed by the addn. of CuSO<sub>4</sub> had  $\rho = 1.06 \times 10^8$  (81.7 v.),  $1.95 \times 10^8$  (473 v.) and  $1.30 \times 10^8$  (1371.8 v.). The  $\rho$  of this product also decreased on standing.

Lewis W. Dutz

Nitration. V. Nitration of monohalogenated derivatives of xylenes. Phuldeo Sahay Varmā and K. S. Venkat Raman. *J. Indian Chem. Soc.* 12, 540–1 (1935); cf. *C. A.* 24, 5714.—The O<sub>2</sub>N derivs. of the halogenated xylenes have been obtained in good yields by nitrating the resp. monohalogenated deriv. in the presence of nitrosulfonic acid mixt. (I) contg. about 50% of fuming HNO<sub>3</sub>. The addn. of 9 cc. of I to a cooled mixt. of 7 cc. of 4,3-CIMeC<sub>6</sub>H<sub>3</sub>Me and 5 cc. of Ac<sub>2</sub>O, and refluxing of the mixt. for 4 hrs. gave a product which, when washed with H<sub>2</sub>O and NaHCO<sub>3</sub> and extd. with benzene, yielded 6.8 g. of 4,3,6-CIMe(NO<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>Me, m. 42° after recrystn. from Et<sub>2</sub>O. The nitration of 5 cc. of 2,4-CIMeC<sub>6</sub>H<sub>3</sub>Me gave 5.6 g. of 2,4,5-CIMe(NO<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>Me, m. 78°, and 0.4 g. of 3,4-CIMeC<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>H, m. 200–2°. The following xylenes, 4-bromo-6-nitro-, m. 57°, and 4-iodo-6-nitro-m-xylene, m. 86°, and 2-bromo-5-nitro-, m. 70°, and 2-iodo-5-nitro-*p*-xylene, C<sub>8</sub>H<sub>7</sub>INO<sub>2</sub>, m. 83°, were similarly obtained. C. R. Addinall



**Preparation of *o*-, *m*- and *p*-hydroxyphenoxyalkylamines** Jean Druey. *Bull. soc. chim.* [5], 2, 1737-41 (1935).—A soln of 23 g. of Na in 350 cc of EtOH and 126.5 g. of PhCH<sub>2</sub>Cl was added to 110 g. of pyrocatechol (I) in 250 cc of alc. in a N atm., and the mixt. was refluxed in the presence of a current of N. The cold acidified soln. was filtered free from NaCl and, on standing overnight, formed 25 g. of *O*-dibenzylpyrocatechol (II). The filtered mother liquor was evapd. and extd. with Et<sub>2</sub>O. The washed, dried and evapd. ext. was distd. free from I and gave 84 g. of *O*-monobenzylpyrocatechol (III), *b*<sub>p</sub> 173-4°, *d*<sub>4</sub><sup>20</sup> 1.154, *n*<sub>D</sub><sup>20</sup> 1.585, *M* R. 58.31 (theor. 58.20). Crystn. of the residue from alc. gave 30 g. of II, *m* 62°. By heating a mixt. of 20 g. of II and 56 g. of (CH<sub>3</sub>Br), in 40 cc. of alc. with 16.5 g. of KOH in 140 cc. of alc. added dropwise over 1 hr. and further heating until neutral, a mixt. was obtained which, on extn. with Et<sub>2</sub>O, yielded 64% of *O*-benzyl-*O*-(*β*-bromoethyl)pyrocatechol (IV), *b*<sub>p</sub> 45-6°, *b*<sub>1</sub> 186-7°, and a residue contg. bis(2-benzylphenoxy)ethyl ether, *m* 88-9°. A mixt. of 6.2 g. of IV with 3 mols. of Me<sub>2</sub>NH was heated in a sealed tube for 12 hrs. at 115° and, on working up, yielded 4.35 g. (80%) of 1-benzyl-2-dimethylaminoethoxybenzene, *b*<sub>p</sub> 153-6°, HCl salt, *m* 142-4°. Similar condensations with Et<sub>2</sub>NH and Me<sub>2</sub>NH, gave the corresponding *di*-*Et* deriv., *b*<sub>p</sub> 170-2°, and *Me* deriv., *b*<sub>p</sub> 152-5° (HCl salt, *m* 102-3°). These amines were almost quant. hydrolyzed by boiling for 2 hrs. with alc. G N HCl to the corresponding *o*-hydroxyphenoxyethylamine HCl salts *di*-*Me*, *m* 215.5-6.5°, *di*-*Et*, *m* 162-3°, and *Me*, *m* 235-6°. A similar series of transformations was carried out starting with resorcinol and giving successively *O*-monobenzylresorcinol, *b*<sub>p</sub> 180-5°, *O*-dibenzylresorcinol, *m* 73-4°, *O*-benzyl-*O*-(*β*-bromoethyl)resorcinol, *b*<sub>p</sub> 180-5°, *O*-benzyl-*O*-(*β*-dimethylaminoethoxy)resorcinol, *b*<sub>p</sub> 166-9° (HCl salt, *m* 125-30°); and *m*-hydroxy(*β*-dimethylaminoethoxy)benzene. The corresponding hydroquinone series was produced *O*-monobenzyl, *m* 121.2°, dibenzyl, *m* 128-9°; benzyl-*β*-bromoethyl, *m* 84.0-4.5°; (PhCH<sub>2</sub>OCH<sub>2</sub>CH<sub>2</sub>O)<sub>2</sub>, *m* 172-2.5°, *O*-benzyl-*O*-(*β*-dimethylaminoethyl), *m* 171-2°, and *p*-*β*-dimethylaminoethoxyphenol, *m* 174-6°.

C. R. Addinall

***β*-Substituted ethylamines II. Catalytic hydrogenation of oximes** Otto Schales. *Ber.* 68B, 1943-5 (1935), cf. C. A. 28, 1491.—In spite of the many studies which have been made of the question, there is as yet no known method for the smooth reduction of oximes to primary amines. According to V. Braun, *et al.*, as confirmed by Winans and Adkins (C. A. 26, 969), the reduction of oximes proceeds according to the scheme RCH NOH (+ H<sub>2</sub>) → RCH<sub>2</sub>(NH) (+ H<sub>2</sub>) → RCH<sub>2</sub>NH<sub>2</sub>(A); RCH: NH + RCH<sub>2</sub>NH<sub>2</sub> (- NH<sub>3</sub>) → RCH NCH<sub>2</sub>R (+ H<sub>2</sub>) → (RCH<sub>2</sub>)<sub>2</sub>NH(B). The intermediate aldime of reaction (A), reacting with primary amine already formed, would explain the undesirable formation of secondary amine according to (B). The formation of secondary amine should therefore be hindered or completely prevented if the aldime is not allowed to accumulate at any point and the reaction is carried out in strongly acid medium. This was confirmed experimentally. If, instead of bringing the oxime in contact with the catalyst before the reduction is begun, the oxime soln. is slowly dropped into the reduction chamber on the catalyst, the small amt. present in each drop is quantitatively reduced to the primary amine by the excess of catalyst before the next drop falls. By dropping AcOH solns. of hydroxyphenylacetaldoximes into an AcOH-H<sub>2</sub>SO<sub>4</sub> suspension of the Adams Pt catalyst, 80-5% yields of the corresponding *p*-phenylethylamines were obtained at 20° and 760 mm. Two examples are given: the prepn. of *p*-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, *b*<sub>p</sub> 136°, and of homopiperonylamine, *b*<sub>p</sub> 148°.

C. A. R.

**Condensation of *o*-bromacetophenone with 1-*o*-amino-phenyl-3-phenylthiocarbamide** Kumud Behari Pathak. *J. Indian Chem. Soc.* 12, 463-5 (1935).—The reaction mechanism suggested by Ghosh (C. A. 25, 3001) to explain the formation of well-defined condensation products from the action of BrCH<sub>2</sub>Br (I) on *o*-H<sub>2</sub>NCH<sub>2</sub>HNHC(SNHPh

(II) is considered as improbable since the Br atom of I is always eliminated as HBr in condensation with a compd. contg. the NHCSNHPh group and the final product is the HBr salt of a S-contg. cyclic base. A repetition of G's expt. in 95% AcOH gave a poor yield of a cryst. product (III), C<sub>11</sub>H<sub>11</sub>BrN<sub>2</sub>OS, *m* 223° (decompn.), acidic in Me<sub>2</sub>CO-H<sub>2</sub>O soln. to Congo red, liberating CO<sub>2</sub> on titration with NaHCO<sub>3</sub> and yielding a base (IV), C<sub>11</sub>H<sub>10</sub>N<sub>2</sub>OS, *m* 164°. III was obtained by condensation in alc. and, on treatment with pyridine, gave IV which may have the

constitution  $\begin{array}{c} \text{CH} \quad \text{CH} \quad \text{C} \cdot \text{NH} \cdot \text{CO} \cdot \text{S} \\ | \quad | \quad | \\ \text{CH} \quad \text{CH} \quad \text{C} \cdot \text{NH} \quad \text{C}(\text{NPh}) \quad \text{S} \end{array}$ . The elimination of PhNH<sub>2</sub> during the condensation is explained by the formation of an intermediate with the structure  $\begin{array}{c} \text{CH} \quad \text{CH} \quad \text{C} \cdot \text{NH} \quad \text{C}(\text{NPh}) \quad \text{S} \\ | \quad | \quad | \\ \text{CH} \quad \text{CH} \quad \text{C} \cdot \text{NH} \quad \text{C}(\text{NPh}) \quad \text{S} \end{array}$ .

The non-elimination of allylamine in the condensation of I with *o*-H<sub>2</sub>NCH<sub>2</sub>HNHC(SNHCH<sub>2</sub>CH=CH<sub>2</sub>), (V) is attributed to the reaction of V in the isomeric form *o*-H<sub>2</sub>NCH<sub>2</sub>HN(C(SH)NHCH<sub>2</sub>CH=CH<sub>2</sub>) in which the allylamine group is attached to the ring by a single bond and in which form no hydrolysis takes place.

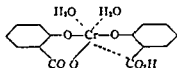
C. R. Addinall

**The behavior of benzylamine tartrate in acetic acid** F. Damosos and I. Peyches. *Bull. soc. chim.* [5], 2, 1656-68 (1935).—The optical rotation of benzylamine tartrate and of *d*-tartaric acid have been detd. in AcOH solns. between 1 and 0.001 *M*. The yellow, green and blue lines of Hg were used. Between 1 and 0.01 *M* the rotation conformed to the law  $[\alpha]_D^{25} = 2 + 5.25\sqrt{C}$  (*C* = concn). For lower concns, however, the observed values fell too low. Theoretical considerations showed that reaction with AcOH to liberate tartaric acid could not account for all of the difference between observed and calcd. values, since addn. of tartaric acid to more concd. solns. did not make the necessary reduction of  $\alpha$ . The authors conclude that there is formation of amide between AcOH and benzylamine, liberating H<sub>2</sub>O which solvates the ions. In evidence they found that addn. of H<sub>2</sub>O in small amts. produced the same effect, and that no such phenomenon could be observed with tetramethylamine tartrate.

R. Balzly

**The chromium compounds of salicylic acid azo dyes and of *o*-methoxybenzoic acid** Kurt Brass and Walter Wittenberger. *Ber.* 68B, 1905-12 (1935).—Preliminary expts. with the azo dye (I) obtained from *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> and salicylic acid showed that either chromation after dyeing (*Nachchromierung*) or dyeing on wool previously mordanted with Cr salts improves the fastness of the dye to the same extent, and that in both processes bichromate can be completely replaced by chromic salts. In passing from the technical processes, or from the system fiber-dye, to the dye (I) itself, however, it was found that Cr cannot be introduced into I with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> alone without a reducing agent also being present, but it can be introduced with chromic salts. After the necessary conditions had been established the Cr compd. of I was prepd. with chrome alum (1 atom Cr per mol salicylic acid), the pure dye acid in the theoretical amt. of NaOH being added hot to the chrome alum. The red Cr compd. is composed of 2 mols. dye + 1 atom Cr + 2 mols. H<sub>2</sub>O, and its compn. does not alter at 70-130°. Cr compds. of similar compn. were obtained from the corresponding dyes obtained from PhNH<sub>2</sub> (dye II) and *o*-toluidine (dye III) while that of the benzidine dye (IV) contained only 1 mol dye per atom of Cr. The Cr can be removed with boiling AcOH, the unchanged dye acid being recovered in very good yield. In the prepn. of the Cr compd. of II the course of the introduction of the Cr into the yellow dye acid and its dependence on the temp. and time were followed. After 30 min at room temp. the brown product contained hardly 2% Cr, but at the boiling temp. there was rapidly formed a crimson labile precursor with almost 14% Cr, after 1 hr. the ppt. contained 9.57% Cr, but the formation of the light red Cr compd. (9.14% Cr) of the dye was complete only after 3 hrs. The Cr compd. of I, which begins to decompose at 200°, lost the calcd. amt. of H<sub>2</sub>O (5.47%)

in 4 hrs. at 190° or in 13 hrs. *in vacuo* when gradually heated to this temp., but the C content of the dehydrated Cr lake was 1-1.5% low, indicating that dehydration cannot be effected without partial decompn. Attempts to replace the 2 mols. H<sub>2</sub>O by 2 mols. NH<sub>3</sub> resulted in the addn. of 2 mols. NH<sub>3</sub> (to the Cr compds. of I and II), which were very firmly held; neither with dil nor with concd hot HCl could the products be so broken down that all the NH<sub>3</sub> passed into the filtrate and the NH<sub>3</sub>-free Cr compds. remained unchanged. This shows that the Cr compds. can only be complex compds of coordinately 6-valent Cr (8-valent in the NH<sub>3</sub> compds.) of the general type



Morgan and Smith (C. A. 19, 219) assigned an entirely different structure (that of a dichromic-m-nitrobenzenesulfonylalkylate with 2 atoms Cr (10.8%) to 3 mols. dye and no H<sub>2</sub>O) to the Cr compd of I, but in a careful repetition of their work it was found that the crude product contained 6.6% Cr and that, while exhaustive extn with benzene removed unchanged I, the residue contained only 8.1% Cr, corresponding to the formula given above. Some expts. were also made to det. the role which the 2 functional groups of the salicylic acid play in the building up of the complex. All attempts to methylate the Cr compd of I with Me<sub>2</sub>SO, or to ethylate it with EtI, were unsuccessful, as was the attempt to obtain the methylated dye by coupling *o*-MeOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H with diazotized *m*-ON<sub>2</sub>CH<sub>4</sub>NH<sub>2</sub>. Neither could chromic-salicylic acid *O* CO CaH<sub>4</sub>, *O* CrO<sub>2</sub>CaH<sub>4</sub>CO<sub>2</sub>H 2H<sub>2</sub>O (Barbieri, C. A. 9,

2819, gives 3 H<sub>2</sub>O), be methylated, as it is decomposed by CH<sub>3</sub>N<sub>3</sub>, but *o*-MeOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H gave with chrome alum a complex, corresponding in compn. to the formula *HO*Cr(*O*<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>OMe)<sub>2</sub>H<sub>2</sub>O. Similar structures might be ascribed to the Cr compds. of the dyes of type I, but this is not in harmony with their difficult soly. in alkali. Moreover, it has not been det. whether such dyes react in the same way as *o*-MeOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H with chromic salts. The investigation is being continued. C. A. R.

5,5-Diphenylbarbituric acid, 5-phenyl-5-cyclohexylbarbituric acid and 5-phenyl-5-cyclohexenylbarbituric acid. H. J. Morasman. *Helv. Chim. Acta* 18, 1254-61 (1935), cf. C. A. 29, 6522; —Ph<sub>2</sub>C(COCl)<sub>2</sub> (I), treated with NH<sub>3</sub>, gave Ph<sub>2</sub>C(CONH<sub>2</sub>)<sub>2</sub> (II), m. 243-4°. II, treated with (COCl)<sub>2</sub>, gave 5,5-diphenylbarbituric acid (III), m. 298-9°. III, treated with CH<sub>3</sub>N<sub>3</sub>, gave 1,3-dimethyl-5,5-diphenylbarbituric acid (IV), m. 206-7°. IV was also obtained from I and (MeNH)<sub>2</sub>CO. PhCH<sub>2</sub>(CONH<sub>2</sub>)<sub>2</sub>, treated with (COCl)<sub>2</sub>, gave 5-phenylbarbituric acid, m. 264-6°. PhCH<sub>2</sub>(CO<sub>2</sub>Me)<sub>2</sub>, treated with CH<sub>3</sub>I and KOEt, gave PhCH<sub>2</sub>C(CO<sub>2</sub>Me)<sub>2</sub> (V), m. 70-80°. V on sapon gave PhCH<sub>2</sub>CH(CO<sub>2</sub>H)<sub>2</sub> (VI), m. 123-3.5°. PhCH<sub>2</sub>C(CO<sub>2</sub>H)<sub>2</sub> could not be isolated as an intermediate. VI, treated with NH<sub>3</sub>, gave PhCH<sub>2</sub>CH(CONH<sub>2</sub>)<sub>2</sub>, m. 167-8°. V, heated in a sealed tube with NaOEt and urea for 15 hrs. at 105-8° and subsequently acidified, gave phenylcyclohexenylbarbituric acid (VII), m. 209-10°. V, reduced with Pt and H<sub>2</sub>, gave PhCH<sub>2</sub>CH(CO<sub>2</sub>Me)<sub>2</sub>, m. 89-90°. VII gave phenylcyclohexenylbarbituric acid, m. 226°.

Organic magnesium compounds. V. Reaction between the alkyl esters of *p*-toluenesulfonic acid and ROMgX. Kunio Mine. *J. Chem. Soc. Japan* 56, 1112-17 (1935); cf. C. A. 29, 7910°. The reaction between *p*-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>R' (I, R' = alkyl) and ROMgX (II) proceeds according to the equation: 2I + 2II → (CH<sub>3</sub>)<sub>2</sub>SO<sub>2</sub> + 2R'SO<sub>2</sub>R' (III) and it does not proceed in 2 steps, R'SO<sub>2</sub>R' + ROMgX → ROR' + R'SO<sub>2</sub>MeX, R'SO<sub>2</sub>R' + R'SO<sub>2</sub>MeX → (R'SO<sub>2</sub>)<sub>2</sub>Me + III, as indicated by Gilman and Fleck (C. A. 22, 3156), for the yield of III reaches 70-90% if III is distd. off from the reaction system in order to allow the reaction to proceed to the right. If

the reaction proceeded in 2 steps, only 0.5 of the ester would change to III and the yield of III should not be over 50% (R is an alkyl or aryl group). The products of the reaction between Ph<sub>2</sub>CHOMgI and *p*-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>R' are Ph<sub>2</sub>Br, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>SO<sub>2</sub>Me, (CH<sub>3</sub>)<sub>2</sub>SO<sub>2</sub> (IV) and Ph<sub>2</sub>CO (V). IV and V are not direct reaction products but they may be derived from the thermal decompn. of benzoylhydrol during the distn. K. Kutsuta.

Mineral-ol sulfonic acids. V. Reactions of the mineral ol sulfonic acids with aromatic amines. Jaroslav Sereďa, K. Macura and A. Udrycki. *Ber.* 68B, 1933-9 (1935); cf. C. A. 29, 70577. It had been found that heating the pure mineral ol sulfonic acids with PhNH<sub>2</sub> gave, instead of the PhNH<sub>2</sub> salt or amide, sulfamic acid (I), along with a hydrocarbon, as chief product: RSO<sub>3</sub>H + PhNH<sub>2</sub> = H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H + RH. A more thorough study has been made of this reaction, in which the SO<sub>3</sub>H group migrates from 1 compd. to another. The formation of I might be due to the splitting off of SO<sub>3</sub>H or to the presence of H<sub>2</sub>SO, or its esters. To eliminate these possibilities the mineral ol sulfonic acids were set free from their Na salts with H<sub>2</sub>SO, free HCl and the purified Na sulfonates were heated a long time with water. The solns of the sep'd sulfonic acids contained no SO<sub>3</sub> ions, even after boiling with alkali, but the acids still reacted with PhNH<sub>2</sub> to form I and hydrocarbons. PhNH<sub>2</sub>, H<sub>2</sub>SO, was isolated from the mixt. when the reaction was carried out at 160° and I when it was carried out above 160°.

As no H<sub>2</sub>SO, or its esters were present, the PhNH<sub>2</sub>, H<sub>2</sub>SO, can have been formed only secondarily through the aid of water, always present in the hydrophilic mineral ol sulfonic acids. This is possible only on the assumption that there is first formed PhNH<sub>2</sub>SO<sub>3</sub>H which at higher temps. rearranges into I while heating with water breaks it down into PhNH<sub>2</sub> and H<sub>2</sub>SO. The reaction followed the same course with *o*- and *p*-toluidine. The former gave 3,4-Me(H<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>SO<sub>3</sub>H, whereas the *p*-toluidine does not split off the SO<sub>3</sub>H group from the sulfonic acids even on long boiling. Reaction sets in only at 220-30° under pressure. The formation of amine sulfates is a proof, as yet indirect, that the reaction proceeds through the arylsulfamic acids. The isolation of PhNH<sub>2</sub>, H<sub>2</sub>SO, as an intermediate product in the reaction leading finally to I is also an indirect confirmation of the view that the com. taking place (Backprozess) of prep. I from PhNH<sub>2</sub>, H<sub>2</sub>SO, also proceeds through PhNH<sub>2</sub>SO<sub>3</sub>H, although the presence of the latter has not as yet been detected in this process.

Attempts to isolate the arylsulfamic acids in the reaction between mineral ol sulfonic acids and amines have likewise thus far been without success. When dry *p*-toluidine was heated with the absolutely anhyd. sulfonic acids at 220-30°, 5,2-Me(H<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>SO<sub>3</sub>H was obtained, whence it is to be assumed that when the *p*-position to the NH<sub>2</sub> group is occupied, the SO<sub>3</sub>H group is directed to the *o*-position. The evolution of NH<sub>3</sub> was repeatedly observed when the sulfonic acids-amine reaction products were dissolved in alcohols, and in the expts. with *p*-toluidine (NH<sub>3</sub>)<sub>2</sub>SO, and PhNH<sub>2</sub> were isolated. Study of individual groups of the mineral ol sulfonic acids showed that aromatic amines split out the SO<sub>3</sub>H group only from the oil-sol.  $\beta$ - and  $\delta$ -acids. All the acids, however, split off the SO<sub>3</sub>H group when boiled with 20% HCl. C. A. R.

Acylation of *o*-aminophenol. C. E. Sparks and R. L. Nelson. *Proc. Indiana Acad. Sci.* 44, 132-4 (1931). Acylation of *o*-hydroxynonylamino-phenol (I) with ClCO<sub>2</sub>Me yields a diacyl compd (II), m. 10.8-61.5°. Acylation of *o*-hydroxymethyl carbanilate with hydrocyanic chloride gives a diacyl compd identical with II, indicating that a rearrangement occurs during acylation. Sapon. of II gives I, m. 130.5-1°. Similarly *o*-isovalerylamino-phenol (III), acylated with ClCO<sub>2</sub>Me, gives a diacyl compd. (IV), m. 68.9°, also readily obtained by acylation of *o*-hydroxymethyl carbanilate. Sapon. of IV gives III. W. J. Peterson.

Condensation of alcohols with aromatic hydrocarbons in the presence of anhydrous aluminum chloride. II. Condensation of secondary alcohols with benzene and toluene. I. Tzukurvanik and K. Tokareva. *J. Gen. Chem.*

(U S S R) 5, 764-6(1935), cf. C. A. 29, 4746'—Condensation of  $\text{Me}_2\text{CHOH}$  (I) and  $\text{EtCH}(\text{OH})\text{Me}$  (II) with  $\text{C}_6\text{H}_6$  and  $\text{PhMe}$  by the method previously described gave up to 70% of alkylated hydrocarbons. An excess of  $\text{AlCl}_3$  results in the formation of some dialkyl derivs.  $\text{PhMe}$  gave only  $\beta$ -substituted derivs. A mixt. of 30 g I, 150 cc  $\text{C}_6\text{H}_6$  and 43 g  $\text{AlCl}_3$  gave 35 g of a fraction, b. 147-200°, consisting of cumene, b. 150-4°, d<sub>4</sub> 0.8738, n<sub>D</sub> 1.4946, and  $\text{C}_{11}\text{H}_{14}(\text{CHMe})_2$ , b. 205-10°.  $\beta$ -Cymene, b. 173-5°, d<sub>4</sub> 0.8597, n<sub>D</sub> 1.4948, was obtained in 35 g yield from 30 g I, 100 cc  $\text{PhMe}$  and 43 g  $\text{AlCl}_3$ .  $\text{PhCH}_3$  (I), b. 182-5°, d<sub>4</sub> 0.8625, n<sub>D</sub> 1.487, resulted from 20 g II, 100 cc  $\text{C}_6\text{H}_6$  and 30 g  $\text{AlCl}_3$ .  $\text{MeC}_6\text{H}_4\text{CH}_2\text{CHMe}$ , b. 193-7°, d<sub>4</sub> 0.8611, n<sub>D</sub> 1.4903, was obtained from 20 g II, 100 cc  $\text{PhMe}$  and 25 g  $\text{AlCl}_3$ . Chas. Blanc.

New method of alkylation of phenols. I. Tzankrevanik and Z. Nazarova. *J. Gen. Chem. (U. S. S. R.)* 5, 767-70 (1935), cf. preceding abstr.—The method of alkylation of aromatic hydrocarbons in the ring by condensation with secondary and tertiary alcs. in the presence of  $\text{AlCl}_3$  was applied to the alkylation of phenols. The condensation of  $\text{Me}_2\text{COH}$  and  $\text{Me}_2\text{C}(\text{OH})\text{Et}$  with  $\text{PhOH}$  proceeds analogously to that with  $\text{C}_6\text{H}_6$  and  $\text{PhMe}$ , giving 60-70% of alkyl phenols. As by-products there are obtained alkylated hydrocarbons ( $\text{PhCMe}_2$  and  $\text{MeC}_6\text{H}_4\text{CMe}_2\text{Me}$ ) formed evidently as secondary products of the reaction of alkyl phenols with the excess of  $\text{AlCl}_3$ . The condensation of secondary alcs. ( $\text{Me}_2\text{CHOH}$  and  $\text{EtCH}(\text{OH})\text{Me}$ ) with  $\text{PhOH}$  produced only insignificant amts of alkyl phenols. The chief products of condensation were simple esters of alkylated phenols.  $\text{C}_6\text{H}_5\text{CH}_2\text{OC}_2\text{H}_5$  and  $\text{C}_6\text{H}_5\text{CH}_2\text{OC}_2\text{H}_5$ . Similar results were obtained by Dauns and Bathrock by condensation of secondary alcs. with phenols in the presence of  $\text{ZnCl}_2$ . In general, the mechanism of the condensation of secondary and tertiary alcs. with phenols is different. The secondary alcs. in distinction from tertiary alcs. do not give considerable amts of olefins and alkyl halides. The condensation proceeds evidently according to the scheme  $\text{ROH} + \text{AlCl}_3 \rightarrow \text{AlCl}_2(\text{OR}) + \text{HCl}$ ,  $\text{PhOH} + 2\text{AlCl}_3(\text{OR}) \rightarrow \text{RC}_6\text{H}_4\text{OR} + 2\text{AlCl}_3\text{OH}$ . This is confirmed by the normal reaction of condensation of  $\text{Me}_2\text{CHOH}$  with anisole, giving 70% of a mixt. of *o*- and *p*-isopropylanisole. A mixt. of 22 g  $\text{Me}_2\text{C}(\text{OH})\text{Et}$ , 23 g  $\text{PhOH}$  and 40 cc of petr. ether was treated with 25 g  $\text{AlCl}_3$  on a water bath for 3-4 hrs. The reaction mixt. was decompd. with ice and exd. with  $\text{Et}_2\text{O}$ . The  $\text{Et}_2\text{O}$  extn. was treated with 10%  $\text{NaOH}$ , giving 70% of  $\text{HOC}_6\text{H}_4\text{CMe}_2\text{Me}$ , b. 91.5-2°. From the nonphenolic fraction was isolated  $\text{PhCMe}_2\text{Me}$ , b. 188°. Condensation of 22 g  $\text{Me}_2\text{COH}$ , 20 g  $\text{PhOH}$  in 50 cc of petr. ether with 20 g  $\text{AlCl}_3$  gave 34 g (75.5%) of  $\text{HOC}_6\text{H}_4\text{CMe}_2\text{Me}$ , b. 99°. With increased addn. of  $\text{AlCl}_3$  in the reaction, the formation of alkylated hydrocarbons increases. Of these  $\text{PhCMe}_2$ , b. 165-7°, and  $\text{C}_6\text{H}_5\text{CH}_2\text{CMe}_2$ , b. 78°, were isolated. The condensation of 27 g anisole, 15 g  $\text{Me}_2\text{CHOH}$  and 33 g  $\text{AlCl}_3$  produced a little  $\text{HOC}_6\text{H}_4\text{CMe}_2\text{Me}$ , b. 210-15°, and 25 g of a mixt. of *o*- $\text{MeOC}_6\text{H}_4\text{CMe}_2\text{Me}$ , b. 198-200°, and *p*- $\text{MeOC}_6\text{H}_4\text{CMe}_2\text{Me}$ , b. 222-15°, and  $\text{MeOC}_6\text{H}_4\text{CMe}_2\text{Me}$ , b. 220-3°. Chas. Blanc.

Methylenepyrocatechol. Marcel Mottier. *Arch. sci. phys. nat.* 17, 280-91(1935)—A 20% yield of methylene-pyrocatechol is obtained by heating a suspension of 55 g pyrocatechol in 200 cc of  $\text{H}_2\text{O}$ , 40 g  $\text{KOH}$ , 140 g  $\text{CH}_2\text{Cl}_2$  and 125 cc  $\text{EtOH}$  in an autoclave at 120° for 24 hrs, b<sub>1</sub> 57°, d<sub>4</sub> 1.185, n<sub>D</sub> 1.53867. W. J. Peterson.

2,6-Dibenzoylhydroquinone. Otto Dschendorfer and August Verdino. *Monatsh.* 66, 255-84(1935); cf. Doerner, *Ber.* 12, 661(1879); *Ann.* 210, 264(1881); Bogert and Howells, C. A. 24, 1630—While the compd. has not actually been synthesized, the evidence is definitely in favor of the fact that the compd. from *m*- $\text{C}_6\text{H}_4(\text{OBr})_2$  and  $\text{BzCl}$  with  $\text{AlCl}_3$  is 2,6-dibenzoylhydroquinone (I) and not the 2,5-isomer. I, m. 210°, results in 12% yield, diacetate, m. 146° (72% yield); dibenzoate, m. 146°,  $\text{Br}$  deriv., yellow, m. 140-1°. I with alk.  $\text{Me}_2\text{SO}$ , gives a mono-Me ether (2-hydroxy-5-methoxy-1,3-dibenzoylbenzene), light yellow, m. 101° (*Ac* deriv., m. 105°). Oxidation of I gives 2,6-dibenzoylquinone, orange-yellow, m. 162°.  $\text{Ph}_3\text{CO}$  and  $\text{BzCl}$  with  $\text{AlCl}_3$  at 200-20° give a poor yield of 1,3-dibenzoylbenzene (II), m. 100° (with a very small amt. of 1,3,5- $\text{C}_6\text{H}_3\text{Br}_3$ , m. 118°). Isophthalic acid (III) was transformed into the 5- $\text{NO}_2$  deriv., the acid chloride of which yields with  $\text{C}_6\text{H}_5$  and  $\text{AlCl}_3$  the 5- $\text{NO}_2$  deriv. of II, m. 130° (75% yield, based on III);  $\text{MeONa}$  gives 3,5,3',5'-tetrahydroxydibenzoylbenzene, bright red, m. 202° and 210° (liquid crystals).  $\text{SnCl}_4$  and  $\text{HCl}$  in  $\text{AcOH}$  give 80% of the 5- $\text{NH}_2$  deriv. (IV) of II, yellow, m. 129-30°, soly in boiling  $\text{H}_2\text{O}$  about 1 g/100;  $\text{HCl}$  salt, m. 185°; sulfate, benzylidene ether (V), pale yellow, m. 124° (92% yield), *Ac* deriv., m. 147°, *Bz* deriv., m. 152°; 2,4,6-tri- $\text{Br}$  deriv., m. 235° (81% yield) (*Ac* deriv., m. 215°). Nitration of V in concd.  $\text{H}_2\text{SO}_4$  gives 31% of the 2,4(or 4,6)-di- $\text{NO}_2$  deriv. of IV, orange-yellow, m. 197°. IV through the diazo reaction yields the 5- $\text{IO}$  deriv. (VI) of II, m. 135°; *Ac* deriv., m. 139-9°, 2,4,6-tri- $\text{Br}$  deriv., m. 216°. Oxidation of  $\alpha$ -cresotic acid with  $\text{K}_2\text{S}_2\text{O}_8$  yields 3,2,5-Me( $\text{OH}$ ),  $\text{C}_6\text{H}_3\text{CO}_2\text{H}$  (diacetate, m. 150°); VI gives 2,6-dibenzoylquinone, m. 164°, 2,5-HO( $\text{MeO}$ ) $\text{C}_6\text{H}_3\text{Br}_2$  and  $\text{PhCH}(\text{OH})\text{Br}$  with 75%  $\text{H}_2\text{SO}_4$  give 27% of 4-methoxy-6-benzoyl-1,2-diphenylcoumarone (VII), m. 155°, and about 10.5% of 2-hydroxy-5-methoxy-4(or 6)-dibenzoylbenzene, m. 232° (*Ac* deriv., m. 168°).  $\text{CrO}_3$  in  $\text{AcOH}$  oxidizes VII to 2-benzoyl-5-methoxy-1,3-dibenzoylbenzene, m. 121° (75% yield); hydrolysis gives the mono-Me ether of Doelner's compd. (I, e, I).

C. J. West.

Certain bases derived from pyrogallol ethers. Mme. Y. de Lestrang. *Bull. soc. chim.* [5], 2, 1078-84(1935)—Pyrogallol reacted with  $\text{Et}_2\text{SO}$  and  $\text{NaOH}$  to give the 4 possible mono- and di-Et ethers which were sep'd by vacuum distn. and identified largely by color reactions (in comparison with resorcinol and pyrocatechol). The 1,2-di-Et ether (I) b<sub>1</sub> 120°; the 1,3-di-Et ether (II) m. 80°. The 1-Et ether (III) b<sub>1</sub> 95.5° and its diacetate m. 75°. The 2-Et ether (IV) m. 102° and its diacetate m. 77°. These and the corresponding Me ethers or their acetates reacted with glycerol dichlorohydrin and alkali to give benzodioxan derivatives or linear ethers. The following were prep'd: 2-(and 3)-Hydroxymethyl-8-methoxybenzodioxan, from the Me analog of III. The compds. were not sep'd, they b<sub>1</sub> 193° 2-(and 3)-Chloromethyl-8-methoxybenzodioxan, b<sub>1</sub> 167° 2-(and 3)-Diethylaminomethyl-8-methoxybenzodioxan, b<sub>1</sub> 172-5° 2-(and 3)-Hydroxymethyl-8-ethoxybenzodioxan, b<sub>1</sub> 190-3° 2-(and 3)-Chloromethyl-8-ethoxybenzodioxan, b<sub>1</sub> 173° 2-(and 3)-Diethylaminomethyl-8-ethoxybenzodioxan, b<sub>1</sub> 188-80°. I yielded  $\text{C}_6\text{H}_3(\text{OEt})_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{Cl}$ , *o*, b<sub>1</sub> 172°, which

reacted with  $\text{NH}_4\text{Et}$  to form 2,3-( $\text{EtO}$ ) $\text{C}_6\text{H}_3\text{OCH}_2\text{CH}_2\text{CH}_2\text{OH}$  ( $\text{OH}$ ) $\text{CH}_2\text{NHEt}$ , b<sub>1</sub> 198°.  $\text{HCl}$  salt, m. 78-80°. II yields analogous compounds; the oxide b<sub>1</sub> 172°, and the base b<sub>1</sub> 193°. II was also treated with  $\text{CICH}_2\text{CH}_2\text{NHEt}$  to form 2-diethylaminomethoxy-1,3-diethoxybenzene;  $\text{HCl}$  salt, m. 134-5°. I with  $\text{CICH}_2\text{CH}_2\text{NHEt}$  gave 3-diethylaminomethoxy-1,2-diethoxybenzene, b<sub>1</sub> 180°;  $\text{HCl}$  salt, m. 112-13°. R. Baltzly.

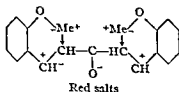
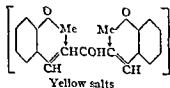
Contact changes of safrole. Yasuji Fujita. *J. Chem. Soc. Japan* 56, 1205-9(1935)—Passing safrole (I) with water through a Cu tube contg. active C at 450-500° gives the following products: I  $\rightarrow$  isosafrole (II), II  $\rightarrow$  pyrocatechol, 4-propylpyrocatechol and cresol. Or I  $\rightarrow$  ethylpyrocatechol methylene ether  $\rightarrow$  *p*-ethylphenol. For obtaining II from I, use of  $\text{CaO}$  or other alkalies gives better results than active C. K. Katsuta.

Contact changes of camphor. Yasuji Fujita. *J. Chem. Soc. Japan* 56, 1210-12(1935)—Passing camphor with water through a Cu tube contg. active C at 450-500° gave carvenone (I). Subsequent changes are: I  $\rightarrow$  carvacrol  $\rightarrow$   $\alpha$ -cresol. Or I  $\rightarrow$  *p*-cymene  $\rightarrow$  cumene. K. Katsuta.

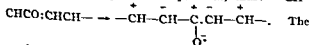
The molecular constitution of qumhydrone. J. Palacios and O. R. Fox. *Anales soc. esp. fis. quim.* 33,

627-42(1935), cf. C. A. 26, 4517.—Further work by magnetochem methods shows that the constitution of *p*-benzoquinhydrone does not correspond to the hypothesis of free radicals, but to the classical hypothesis of additive combination of quinone and hydroquinone. E. M. S.

Unsaturated chromophores II. The effect of the ketovinyl chain on the color of the metal salts of hydroxyvinyl ketones 1. Salts of dialcylalacetone. Angelo Mangini. *Gazz. chim. ital.* 65, 474-86(1935); cf. C. A. 29, 6682<sup>1</sup>.—A review of the literature shows that in spite of some knowledge of the behavior of unsatd ketones toward acids and their halochromism, there have been no systematic researches on the bathochromic effect of the salification of unsatd. HO ketones other than the work of Vorländer (C. A. 19, 1413); Pfeiffer, (*Org. Molekulverbindungen*, Stuttgart 1927, p. 91, C. A. 21, 3625). The past work indicates that the greater the no. of vinyl groups the more intense is the color of compds of vinyl ketones (salts, acids, metal salts). For this reason it was of interest to study the bathochromic effect produced by salification of the OH of unsatd phenolic ketones, to what extent this bathochromism, i. e., the true halochromism of Vorländer, is preserved, whether the nature of the substituent cation of the hydroxy I influences the effect, and if it is possible to obtain 2 forms of salts, a colored salt and a colorless salt. The present work deals with dialcylalacetone (I), and shows that salification gives 2 series of salts. (1) yellow salts, corresponding to the color of I and its ethers, and (2) red salts, like the Na salts of analogous unsatd phenolic ketones, i. e., like the halochromes =CO HX. The stability of the 2 forms depends on the particular cation, e. g., some salts which ppt. in red forms change to stable yellow forms, indicating that the red form is stable only in the case of certain cations. The red forms are stable in the case of cations with pronounced metallic character, e. g., alkalis, alk. earths, Pb, Cd, NH<sub>4</sub>-Cu, etc., whereas with most other metals the initial red forms change to the yellow forms, e. g., with Co, Ni and with still weaker cations, e. g., Al, Cr, ferric Fe, etc., the original ppts are yellow. These red and yellow forms probably have some isomeric relation, and may be considered analogous to "Addukt A" and "Addukt B" of Vorländer (*loc. cit.*). In this case the red and yellow salts would correspond to the activation or the non-activation of the unsatd chromophores in the salified HO ketone mols., so that the red forms correspond to the halochrome "Addukt A" compds and the yellow forms to the colorless "Addukt B" compds. The constitutions of the 2 forms are represented thus.

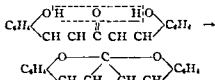


The first corresponds to complex internal salts of the ordinary kind; the 2nd to the probable structure of "intramol. ionides." These structures conform to the development of a dipole ketovinyl chain, thus: —CH



The resultant effects are complex, and this interpretation, though in harmony with exptl results, is tentative. The salts were prepd from the alc. Na salt of I (cf. Decker

and Felsner, C. A. 3, 61) and an excess of metal salt, or from I in benzene and moist metal hydrosulfide. Three types were obtained: (1) ppts. stable in the red form; (2) ppts. stable in the yellow form, and (3) red ppts. not stable in this form. The change in group (3) does not depend on hydrolysis or on a change in the degree of hydration. All salts are very stable in air, but with dil. acids they decompose with formation of I, while decomposition leads to formation of the halochromes and thence to closing of the nucleus



The action of alkalis and NH<sub>4</sub>OH is complicated; these do not decompose the salts completely when cold. With respect to optical behavior, the yellow salts show the same absorption, regardless of the nature of the cation and of the solvent, absorption is complete up to 570  $\mu$ . The red salts also show identical spectra, which are practically independent of the metal and the solvent; they absorb completely up to 500  $\mu$ , a little less in green-yellow than do the yellow salts. The absorption of I in various solvents is the same as that of the yellow salts, that of solns of I in acids the same as that of the red salts. Probably therefore I, its acid derivs and its red and yellow salts, have the same constitution or are related isomerically. With the exception of the Ag salt, all salts contain H<sub>2</sub>O of crystn (dependent perhaps on the coordinate unsatd of the ketodiviny chain), and the dehydration curves and the high temps of completion (180-200°) indicate that at least some of the H<sub>2</sub>O mols. are bound coordinatively to the complexes. There seems to be no relation between the H<sub>2</sub>O of crystn and the colors, as with the already known Na salt (cf. *Orient. Pharm. Post* 36, 101; Fabiny, *German pat.* 110,521). *Group (1)*—Mg salt, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Mg 4H<sub>2</sub>O, dark pomegranate-red, dehydrated at 210° to the anhyd salt, chocolate color; its cold C<sub>17</sub>H<sub>12</sub>N solns give when boiled a violet-red ppt and yellow mother liquor. *Ca salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Ca 6H<sub>2</sub>O (II), intense carmine-red with metallic reflection. *anhyd. salt*, maroon. *Sr salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Sr 7H<sub>2</sub>O (III), wine-red; *anhyd salt*, maroon. *Ba salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Ba 5H<sub>2</sub>O (IV), scarlet with golden reflection. *anhyd. salt*, wine-red. *Cd salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Cd 2H<sub>2</sub>O, red, *anhyd salt*, maroon. *Pb salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Pb 2H<sub>2</sub>O, carmine, when pptd. from basic Pb acetate it is violet-red. *Ammoniacal cupric salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Cu 5H<sub>2</sub>O·NH<sub>3</sub>, carmine, decomposes at 90-100°. *Ag salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Ag, gray-black with golden yellow reflection. *Li salt*, from very concd. solns, purple-red. *Rb salt*, from very concd. solns, purple-red. *NH<sub>4</sub> salt*, from I in NH<sub>4</sub>OH and NH<sub>4</sub>Cl, bright red. *Thallous salt*, maroon-black with lustrous reddish green reflection. *Group (2)*.—*Cupric salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Cu H<sub>2</sub>O, silky yellow; *anhyd salt*, greenish yellow. *Zn salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Zn 2H<sub>2</sub>O, chrome-yellow; *anhyd salt*, greenish yellow. *Mercuric salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Hg·H<sub>2</sub>O, greenish yellow. *anhyd. salt*, olive color. *Ferric salt*, (C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>)<sub>2</sub>·Fe<sub>2</sub> 6H<sub>2</sub>O, dirty yellow; *anhyd. salt*, dark maroon. *Al salt*, (C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>)<sub>3</sub>·Al<sub>3</sub> 6H<sub>2</sub>O, yellow; *anhyd salt*, yellowish green. *Be salt*, yellow. *Cr salt*, yellow. *Group (3)*.—*Mn salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Mn H<sub>2</sub>O, hyvered when moist, greenish yellow or brown when dry; *anhyd. salt*, maroon-yellow. *Ferrous salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Fe·3H<sub>2</sub>O, brilliant dark red when pptd, but turns maroon-yellow, and when dry is dirty yellow; *anhyd. salt*, maroon. *Ni salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Ni 3H<sub>2</sub>O, bright scarlet when pptd., but turns yellow, and when dry is intense yellow; *anhyd salt*, greenish maroon. *Co salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Co 2H<sub>2</sub>O, dark red when pptd, brick-orange when dry, remains red when kept in water; its C<sub>17</sub>H<sub>12</sub>N solns. are red. *Mercurous salt*, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>·Hg<sub>2</sub> 6H<sub>2</sub>O, dark red when pptd, maroon when dry, *anhyd salt*.

C. C. Davis

Preparation of acetoanthranil derivatives Mosuke Hayashi, Ikuro Morikawa and Hirochika Namikawa. *J. Chem. Soc. Japan* 56, 1102-5(1935) — 6-Methyl-2-anthranil and its 2,3-isomer, 5-methyl-2-acetoanthranil and 4-methyl-2-acetoanthranil are prepd. K. Katsuta

Condensation of acetoanthranil derivatives with benzene Mosuke Hayashi, Hirochika Namikawa and Ikuro Morikawa. *J. Chem. Soc. Japan* 56, 1106-11(1935) — Condensation of acetoanthranil and  $C_6H_6$  with  $AlCl_3$  gives 2-aminodiphenyl ketone, m 109-10°, and 2-phenylamino-diphenylketone, m 121.5-2°, 3-methyl-2-acetoanthranil, in a similar manner, gives a small amt of 2-phenylamino-3-methyldiphenyl ketone, m 123-3.5°; 5-methyl-2-acetoanthranil gives 2-amino-5-methyldiphenyl ketone, m 64-4.5°, and 2-phenylamino-5-methyldiphenyl ketone, m 163.5° K. Katsuta

New synthesis of ephedrine derivatives V Bruckner and A. Kraml. *Arch. Pharm.* 273, 372-84(1935) — In a former communication (C. A. 29, 5825<sup>5</sup>) it is shown that from the  $\nu$ -nitrosites of methyl isoeugenol and -isosalfore the acetates of  $\alpha$ -(3,4-dimethoxyphenyl)- $\beta$ -nitropropanol (I) and  $\alpha$ -(3,4-methylenedioxyphenyl)- $\beta$ -nitropropanol (II), resp, can be prepd. in good yield. Both nitro compds can be electrolytically reduced to the corresponding hydroxylamine derivs,  $RCH(OAc)CH(NHOH)Me$  (III and IV). These bases are stable only in the form of their salts (i. e., hydrochlorides), since, when liberated therefrom, an alkyl migration immediately occurs from O to N, thus leading to the alkali-sol., very well cryst. N-Ac derivs,  $RCH(OH)CH(N(OH)Ac)Me$  (V and VI). On treatment with dil. HCl, however, the alkyl migration occurs in the opposite direction, in other words, the HCl salts of the O-acetylated bases (III and IV) are again recovered. Since the prepn of the hydroxylamine bases (V and VI) can be effected in good yield, the authors have undertaken, by the aid of Bamberger's reaction—involving interaction of hydroxylamine bases with aldehydes—to prep. through the nitrones various ephedrine derivs. by further reduction of the former. In the last 2 phases of the synthesis the procedure of Nagai and Kanoo (C. A. 23, 3629) was followed. In order to do this it is above all necessary in reducing the  $NO_2$  compds (I and II) to isolate the hydroxylamine derivs as N-Ac compds. If the formation of nitrones is desired the Ac group must first migrate from N to O. Furthermore, it should be noted that only the free hydroxylamine bases, not their salts, are able to react with aldehydes. The problem then to prep. nitrones from the reduction products (V and VI) was nevertheless practically solved in 1 phase, since the alkyl migration from O to N is apparently not immediate. E. g., on treating the acetoxyhydroxylamine compds (III and IV) in concd aq. solns. with an aldehyde and thereupon liberating the hydroxylamine bases by addn of  $Na_2CO_3$ , nitrone formation follows immediately. Naturally, in the case of many aldehydes the rapidity of nitrone formation cannot reach the rapidity of the alkyl migration from O to N, so that in such, by no means infrequent, cases, the reaction product consists predominantly of the original N-acetylhydroxylamine compd (V, resp., VI). In accordance with the above-described principle there have been prepd. from the 2 hydroxylamine compds. (V and VI) the corresponding methylene-, benzylidene-, m-nitrobenzylidene-, salicylidene-, veratrylidene- and piperonylidene nitrones. During these expts. it developed that the adherence of the Ac group is markedly influenced on the one hand by substituents of the  $\alpha$ -aryl group, on the other hand by the character of the radical in combination with the nitrone ring. It was observed among other things that as a rule the adherence of the Ac group in methylisoeugenol derivs is much less than in the corresponding isosalfore derivs; certain substituents of the nitrone ring weaken the ester union in such cases so much that it is split during the nitrone formation by the action of the  $Na_2CO_3$  as applied in slight excess. In the series of methylisoeugenol derivs only in the case of the methylenenitronone,  $(MeO)_2C_6H_4CH(OH)CHMe-N-O-CH_3$  (VII) and m-nitrobenzylidenenitronone (VIII) did

the AcO group remain unchanged, while with veratrylidene-nitronone,  $(MeO)_2C_6H_4CH(OH)CHMe-N-O-CH_2C_6H_4(OMe)_2$

(IX), salicylidene-nitronone (X) and benzylidenenitronone (XI) it was saponid. to a HIO group by the action of  $Na_2CO_3$ . On the other hand it developed that all isosalfore derivs, i. e., in the methylene- (XII), benzylidene- (XIII), salicylidene- (XIV), m-nitrobenzylidene- (XV) and piperonylidene-nitronone (XVI), the AcO group remained unchanged. That the AcO group of certain nitrones of the methylisoeugenol type is actually saponid. during the formation can be proved by a somewhat modified synthesis of the des-acetylated nitrones. Thus, on boiling the N-acetylated hydroxylamine derivs. (V and VI) and with aq.  $H_2SO_4$ , the Ac group after migration from N to O is smoothly split off. If one thereupon suspends in a thus prepd.  $H_2SO_4$  soln. of  $\alpha$ -(3,4-dimethoxyphenyl)- $\beta$ -hydroxylaminopropanol (XVII) either  $BaH_2$ , salicylaldehyde, veratraldehyde or m- $O_2NC_6H_4CHO$ , there are formed in the 1st 3 cases the same nitrones, which were also previously obtained from the hydroxylamine compd (V). Similarly, it could be shown that the dil.  $H_2SO_4$  soln. of  $\alpha$ -(3,4-methylenedioxyphenyl)- $\beta$ -hydroxylaminopropanol (XIX), which was prepd. by the acidic sapon. of VI, yielded with the corresponding aldehydes in weakly alk. soln. products as benzylidene- (XX), m-nitrobenzylidene- (XXI), piperonylidene- (XXII) or salicylidene-nitrones (XXIII), resp., not identical with the compds XIII, XIV, XV and XVI. Notable differences are apparent in the soly. of the various nitrones in acids. Very striking is the fact that many desacetylated nitrones slowly dissolve in dil. HCl, while the acetylated nitrones possess no observable soly. in this acid. In this connection it should be noted that the nitrones of both types are almost completely insol. in  $H_2O$ . Accordingly, the supposition arises that the des-acetylated nitrones, in contrast to their Ac derivs, possess a weakly basic character, which may possibly find explanation in a radically different constitution of the 2 types. If one formulates the acetylated nitrones in accordance with the general formula  $RCH(OAc)CHMe-N-O-CH_2R$  (XXIV) in the strictest sense of the word as

nitrones, the desacetylated compds, however, as dihydro-oxazoline derivs,  $RCHCHMe-N(OH)CH_2R$  O (XXV),

then the neutral character of the acetylated derivs. and the basic character of the desacetylated products would not be easily intelligible. The final decision of this question could not for the present be added; further investigations will be necessary to assure more definite insight. Accordingly the present nomenclature of the desacetylated nitrones has been retained. Benzylidenenitronone (XI) of XVII, leaflets from  $C_{11}H_9$ -ligron, m 160°. Salicylidene-nitronone (X), yellowish white fine needles, m 148-50°. m-Nitrobenzylidenenitronone (VIII) of III, yellow needles from alc., m 196°. m-Nitrobenzylidenenitronone (XVIII) of XVII, yellow microscopic prisms from alc., m 174°. Benzylidenenitronone (XIII) of IV, prisms, m 158-9°. Benzylidenenitronone (XX), of XIX, leaflets from alc., m 140°. Salicylidene-nitronone (XIV) of IV, yellow needles, m 140°. Salicylidene-nitronone (XXIII) of XIX, faintly yellow cryst. powder, unstable in the air and daylight, m 117-18°. m-Nitrobenzylidenenitronone (XV) of IV, lemon yellow needles, m 190°. m-Nitrobenzylidenenitronone (XXI) of XIX, lemon yellow microscopic crystals, m 171°. Piperonylidene-nitronone (XVI) of IV, prisms, m 158-60°. 3,4-Dimethoxy-N-benzylideneephedrine (XXVI), feily needles, m 162-3.5°. 3,4-Dimethoxy-N-homoveratrylideneephedrine (XXVII), leaflets, m 127-7°. 3,4-Methylenedioxy-N-benzylideneephedrine (XXVIII), feily needles, m 117.6-19.5°. 3,4-Methylenedioxy-N-homopiperonylideneephedrine (XXIX),  $C_{11}H_9ON$ , leaflets, m 123.5-125°. W. O. E.

New derivatives of dihydrodivanillin and method for the catalytic reduction of nitrostyrenes Kurt Maurer and Bruno Schiedt. *J. prakt. Chem.* 144, 41-8(1935) — Divanillin (I) (20 g.),  $Ac_2O$  and  $H_2SO_4$  give 25-7 g. of the hexaacetate, m 160°, on hydrolysis there results

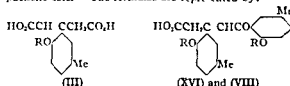
60% I. I (30 g), 12 g. NaOH, 80 cc. H<sub>2</sub>O and 60 g. Me<sub>2</sub>SO<sub>4</sub> (added in portions) give 25–30 g. dimeric aldehyde (II), m. 138° (dioxime, m. 184°). II (10 g) with MeNO<sub>2</sub> in MeOH-KOH gives 6 g. bis-3,4-dimethoxy-nitrostyrene, yellow, m. 206°; this is catalytically reduced in AcOH-H<sub>2</sub>SO<sub>4</sub> at 3 atm. pressure to dihomoveratrylamine (35% yield), a pale yellow thick oil, whose *Ac deriv.* m. 78°. 3,4-Methylenedioxy-nitrostyrene gives 70% homopiperonylamine (*Ac deriv.*, m. 101°); with (CO<sub>2</sub>Et)<sub>2</sub> this gives *Et homopiperonyloxamate*, m. 131°. 3,4-Dimethoxy-piperonylnitrostyrene gives 23% of homoveratrylamine (the remainder consists of polymerization products). The *Me ether* of  $\alpha$ -(3,4-dimethoxyphenyl)- $\omega$ -nitroethanol gives 74% of the corresponding amine.

**C J West**  
Dehydrogenation of phenols Holger Erdtman *Swensk Kem. Tids.* 47, 223–20 (1935) (in German), cf. C. A. 28, 1337; 29, 2938. Some expts. were made to det. the optimum acidity for dehydrogenation, the following gave the best yield: 20 g. ferulic acid in 120 cc EtOH and 160 cc. 12% aq. FeCl<sub>3</sub> were let stand 30 min then 40 cc. concd. HCl was added. Dilg. this to 900 cc and filtering after 12 hrs. gave 1.8 g. dehydrogenation product, m. 202–5°. Vanillic acid ester oxidized with Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub> gave the corresponding dehydro compd., m. 231°. Improved method for prep. 5-iodovanillin 3 g. vanillin in 10 cc. 0.2 N NaOH is dild. to 25 cc., 50 cc. 10% I soln is added very slowly. This soln. contains the least amt KI to hold the I. Add a little NaHSO<sub>3</sub> and wash the ppt. which forms with water. Recrystallize from AcOH. Yield, 4 g., m. 180°. It is less easily methylated than vanillin. 5-Iodo-veratraldehyde, m. 73°, is oxidized with KMnO<sub>4</sub> to the acid, m. 185°. The acid, boiled 10 hrs. under a reflux with 10% MeOH, gives the ester, m. 106°, which, heated 5 min at 220° and extd with boiling acetone and the acetone dild with H<sub>2</sub>O, gives dimeric di-Me ester, m. 130.2°, the same m. p. as that of dehydrodeugenol. Dehydrodivanillin in alk. soln. was treated with NH<sub>4</sub>OH HCl for 3 hrs on the water bath. The oxime heated 6 hrs. with Ac<sub>2</sub>O gave dehydrodivanillonitrile diacetate, m. 220°. This last compd. was also made by treating vanillin oxime with FeCl<sub>3</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and from vanillonitrile in the same way. Five g. *o*-xym-xenol in alc. dehydrogenated with 200 cc. 4.5% FeCl<sub>3</sub> by agitating with a strong current of air gave 3.1 g. dehydrodi-*o*-xym-xenol, m. 137°. The acetylated product, m. 109°. *Me gallate* in MeOH made alk. with a satd. Ba(OH)<sub>2</sub> soln. gives a copious ppt. which after air current agitation for 2.5 hrs. and acidification with dil. HCl gives 30% of ellagic acid. Gallic acid did not give ellagic acid and Ba(OH)<sub>2</sub> is more effective than other alkalis.

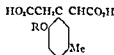
**A. R. R.**  
Maximum rotations of carboxylic acids containing a phenethyl group. P. A. Levene and Stanton A. Harris *J. Biol. Chem.* 111, 725–33 (1935) —  $\text{Ph}(\text{CH}_2)_n\text{CHMeEt}$  (I) can be prepd. from active AmOH without racemization and, on the basis of the max. rotation of I, the max. rotations of other members of the phenethyl series can be calcd. Using this data as a basis, substances of the type  $\text{Me}(\text{Ph}(\text{CH}_2)_n\text{CH}(\text{CH}_2)_m\text{CO}_2\text{H})_n$ , where  $n = 0$  or an integer, were prepd. Periodic fluctuations of the rotatory values as  $n$  increased were noted, thus paralleling the observations with the analogous Ph series and with carboxylic acids of the normal series.  $\text{Ph}(\text{CH}_2)_n\text{CHMeCO}_2\text{H}$ , [M]<sub>D</sub><sup>20</sup> = 51.20°, was prepd by the  $\text{CH}_3(\text{CO})\text{Et}$ , (II) method and resolved by the cinchonidine salt from Me<sub>2</sub>CO.  $\text{Ph}(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{OH}$  and anhyd. HI followed by reduction with Raney's catalyst in alk. MeOH gave I, b<sub>1</sub> 66–7°, b<sub>2</sub> 96°, d<sub>1</sub> 0.8563, n<sub>D</sub><sup>20</sup> 1.4860, [M]<sub>D</sub><sup>20</sup> = 17.2°. I was also prepd. from BrH and active Am-MgBr via the intermediate  $\text{PACH}(\text{OH})(\text{CH}_2)_n\text{CHMeEt}$ , b<sub>1</sub> 90°, d<sub>1</sub> 0.9623, n<sub>D</sub><sup>20</sup> 1.5097, [M]<sub>D</sub><sup>20</sup> = 5.09°, which with anhyd. HI overnight at –70° and 48 hrs. at room temp. gave I, [M]<sub>D</sub><sup>20</sup> = 35.1°. The values for [M] of the following compds. are based on the parent AcOH, and values based on the parent hydrocarbon are found by using the factor 1.981 obtained from the above.  $\text{Ph}(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{MgBr}$  and  $\text{CO}_2\text{H}$  gave  $\text{Ph}(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{CO}_2\text{H}$  (III), b<sub>1</sub> 162–4°. III was also prepd. from  $\text{Ph}(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{Br}$  and II via the intermediate  $\text{Ph}(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{CH}(\text{CO}_2\text{H})_2$ , m. 96–8°, which on heating at 180° gave III, b<sub>1</sub> 162°, d<sub>1</sub> 1.018, n<sub>D</sub><sup>20</sup> 1.5666, [M]<sub>D</sub><sup>20</sup> = 3.29°. *Et ester* (IV), b<sub>1</sub> 122°, d<sub>1</sub> 0.965, n<sub>D</sub><sup>20</sup> 1.4872, [M]<sub>D</sub><sup>20</sup> = 17.20°. IV with Na and EtOH gave  $\text{Ph}(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{OH}$ , b<sub>1</sub> 155°, d<sub>1</sub> 0.9477, n<sub>D</sub><sup>20</sup> 1.5080, [M]<sub>D</sub><sup>20</sup> = 10.13°. Similarly  $\text{Ph}(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{CO}_2\text{H}$  (V), b<sub>1</sub> 172°, d<sub>1</sub> 1.0014, n<sub>D</sub><sup>20</sup> 1.5039, [M]<sub>D</sub><sup>20</sup> = 7.20°, was prepd. III reduced with FeO<sub>3</sub> in AcOH gave  $\text{C}_6\text{H}_5(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{CO}_2\text{H}$ , b<sub>1</sub> 180°, d<sub>1</sub> 0.9523, n<sub>D</sub><sup>20</sup> 1.4657, [M]<sub>D</sub><sup>20</sup> = 2.51°. V and V gave  $\text{C}_6\text{H}_5(\text{CH}_2)_n\text{CHMe}(\text{CH}_2)_m\text{CO}_2\text{H}$ , b<sub>1</sub> 136–40°, d<sub>1</sub> 0.9455, n<sub>D</sub><sup>20</sup> 1.4635, [M]<sub>D</sub><sup>20</sup> = 3.18°. All values for [M] are max and for the homogeneous substance.

**R. C. Elderfield**  
*cis*-Cinnamic acid Julius Meyer. *Z. physik. Chem.* A174, 77–8 (1935) — The theory that the 3 *cis*-cinnamic acids are chem. isomers is supported inadequately and has been abandoned by most workers. Everything points to chem. identity and polymorphism. R. H. B.  
**Chemistry of  $\beta$ -arylglutamic acids.** G. R. Gogte. *Proc. Indian Acad. Sci.* 2A, 185–98 (1935), cf. C. A. 29, 1795. — A yield of 47 g. of crude acids (I) was obtained by the addn of 65 cc. of *p*-MeC<sub>6</sub>H<sub>4</sub>OEt (II) to the cooled reaction mixt. of 200 g. of citric acid and 240 cc. of concd. H<sub>2</sub>SO<sub>4</sub> and 80 cc. fuming H<sub>2</sub>SO<sub>4</sub>. The H<sub>2</sub>O-sol. portion of I contained  $\beta$ -(2-ethoxy-5-methylphenyl)glutamic acid (III), m. 153° (decompn.); HO anhydride, m. 112°; semianilide, m. 136°; HO anil, m. 163°. III with H<sub>2</sub>SO<sub>4</sub> gave  $\beta$ -methylcoumarin- $\alpha$ -acetic acid (IV), m. 180° (decompn.). The H<sub>2</sub>O-insol. residue of I on recrystn. from alc. m. 190–200° and gave 2 *Et* esters, m. 124° (V) and 110–15° (VI). Hydrolysis of 15 g. of V gave 8 g. of the monolactone of  $\beta$ , $\beta'$ -(2,2'-diethoxy-5,5'-dimethyldiphenyl)glutamic acid (VII), m. 205°. Hydrolysis of VI gave 2 g. of 2,2'-diethoxy-5,5'-dimethylchalcone- $\alpha$ -acetic acid (VIII), m. 232°, *Et* ester, m. 133°, semicarbazone, m. 264° (decompn.); semicarbazone of *Et* ester, m. 171°. III (10 g) in 50 cc. cold 80% H<sub>2</sub>SO<sub>4</sub> gave with 20 cc. of II 3.5 g. mixed acids (IX). On esterification IX gave 2 g. of VII, 1 g. of VIII and IV. H<sub>2</sub>SO<sub>4</sub> (80%), allowed to act on 2.5 g. VIII at room temp., yielded 1 g. 7-ethoxy-4-methyl-3-ketodienacetic acid (X), m. 216° (decompn.); *Et* ester, m. 169°; semicarbazone, m. 247° (decompn.); semicarbazone of *Et* ester, m. 208° (decompn.). X could not be obtained from III. Concd. H<sub>2</sub>SO<sub>4</sub> (10 cc.) reacting at 60° with 2 g. VIII gave 1.5 g. of a neutral compd., m. 165°; semicarbazone, m. 245° (decompn.). VII (5 g), heated at 60° for 1 hr. in 20 cc. concd. H<sub>2</sub>SO<sub>4</sub> gave 1 g. IV and 3 g. of the neutral dilactone (XI), m. 184°, of  $\beta$ , $\beta'$ -(2,2'-diethoxy-5,5'-dimethyldiphenyl)glutamic acid. XI could not be obtained by the treatment of VII with H<sub>2</sub>SO<sub>4</sub>. XI (1.5 g) on hydrolysis and ethylation gave 1.7 g. of the di-*Et* ester, m. 82°, which on hydrolysis gave  $\beta$ , $\beta'$ -(2,2'-diethoxy-5,5'-dimethyldiphenyl)glutamic acid (XII), m. 219° (decompn.); di-Me ester, m. 105°; anhydride, m. 189°; acid anilide, m. 193° (decompn.); anil, m. 216°. XII, warmed with H<sub>2</sub>SO<sub>4</sub> gave IV and a small amt. of VII. The di-Me and di-*Et* esters of XII did not condense with aromatic aldehydes.  $\beta$ , $\beta'$ -(2,2'-Dimethoxy-5,5'-dimethyldiphenyl)glutamic acid was prepd. by the hydrolysis and methylation of XI. A yield of 52 g. of mixed acids (XIII) was obtained by the addn of 80 cc. *p*-MeC<sub>6</sub>H<sub>4</sub>OMe (XIV) to a reaction mixt. of 200 g. citric acid, 200 cc. concd. H<sub>2</sub>SO<sub>4</sub> and 120 cc. fuming H<sub>2</sub>SO<sub>4</sub>. The HOAc-insol. portion of XIII gave  $\beta$ -(2-methoxy-5-methylphenyl)glutamic acid (XV), m. 167° (decompn.). The HOAc-insol. portion of XIII on recrystn. from alc. gave 12 g. 2,2'-dimethoxy-5,5'-dimethylchalcone- $\alpha$ -acetic acid (XVI), m. 252°; *Et* ester, m. 122°; semicarbazone, m. 277° (decompn.); semicarbazone of Me ester, m. 219° (decompn.). XV (5 g) in 4 cc. 80% H<sub>2</sub>SO<sub>4</sub>, treated with 10 cc. XIV, gave 1.5 g. XVI and IV. XVI (2 g) in 15 cc. 80% H<sub>2</sub>SO<sub>4</sub> at room temp. for overnight gave 1.1 g. of the indoneacetic acid (XVII), m. 218° (decompn.). Concd. H<sub>2</sub>SO<sub>4</sub> (10 cc.), acting for 1 hr. at 60° on XV, gave 0.6 g. of XVII and 0.8 g. of a neutral compd., m. 214°; semicarbazone, m. 263° (decompn.); benzylidene deriv., m. 174° (de-

compn) The Et ester of XVI with concd.  $H_2SO_4$  gave the Et ester of XVII, m 158°, and some XVIII. Conc.  $HNO_3$  transformed XVI to XVII and the phthalic anhydride, m 186°. Since XV by the action of  $H_2SO_4$  produces XVII as well as IV, whereas III gives only IV and no trace of an indone acid, and both XVI and VIII give only the indone acids and no IV, it appears that in III, XVI and VIII there is a restricted rotation round the bond joining the  $\beta$  C atom of the glutamic acid to the phenolic ester. The formulas are represented by:



In the case of condensation with II, III must have rearranged to



since it alone is capable of producing X. The difference in reactivity between II and XIV is noteworthy

Rachel Brown  
The tyrosine tyrosinase reaction in the presence of l ascorbic acid Fritz Schaaf *Helv. Chim. Acta* 18, 1017-21(1935); cf. *C. A.* 29, 5903<sup>a</sup>—The addn. of ascorbic acid to the tyrosine-tyrosinase reaction has very little effect on the formation of 3,4-dihydroxyphenylalanine (I). Ascorbic acid in the presence of tyrosine with no tyrosinase will cause the formation of I but to a smaller degree.

Julius White  
Ozone as an oxidation catalyst IX Ozonization of anisaldehyde, vanillin and heliotropin F. Bruner and A. Gelbert. *Helv. Chim. Acta* 18, 1239-42(1935); cf. *C. A.* 27, 17—The amt. of  $O_3$  consumed was compared with the amt. of  $O_2$  fixed, less that used in autooxidation, in a study of the oxidation in  $CCl_4$  of anisaldehyde, vanillin and heliotropin (piperonal), of which the last 2 were prepd by the ozonization of isoeugenol and isosafrole, resp. The less the concn. of  $O_3$ , the less was the mobilizing effect of  $O_3$  on  $O_2$ , and the greater the concn. of  $O_3$  the greater was the mobilizing effect of  $O_2$ . The catalytic effect of  $O_3$  on  $O_2$  in the oxidation of vanillin and its Me and Et derivs. does not appear until the concn. of  $O_3$  is less than 2%. This study shows that prolonged ozonization leads to the formation of acids because  $O_3$  catalyzes oxidation by  $O_2$ .

W. F. Bruce  
Substitution synthesis of mellitic acid, and the replaceability of chlorine in the benzene nucleus Franz Feist. *Ber.* 68B, 1941-3(1935)—The Rosenmund and Struck method of replacing nucleus halogen with  $CO_2H$  by heating with  $KCN + CuCN +$  water under pressure is not universally applicable, as claimed by them (*C. A.* 14, 1446; Ger pat 327,049). While it gives satisfactory results with brominated aromatic compds., the replacement of Cl is already tedious in  $PhCl$  and the yield of  $BzOH$  is moderate, and with polychlorobenzenes the method fails completely. Neither 1,2,4- $C_6H_3Cl_3$  nor  $C_6Cl_6$  can be converted into the corresponding tri- and hexacarboxylic acid, even when the conditions are modified in the most varied manner. A loosening of the Cl atoms is made possible only by the presence of neg o-substituents, mellitic acid can be obtained (in 60% yield) by their method from tetrachloro-o- or p-phthalic acid.

C. A. R.  
Anomalous rotatory dispersion of l- $\beta$ -pinene. I. R. Padmanabhan and S. K. Kulkarni *J. Indian Chem. Soc.* 12, 518-26(1935); cf. *C. A.* 29, 1708<sup>a</sup>—The only substances among the optically active hydrocarbons known to have anomalous rotatory dispersion are d-cabinene and l- $\beta$ -pinene (I). The optical rotatory

power of I is neg in the visible region of the spectrum, becomes zero at about  $\lambda$  3700 and attains high pos values with shorter wave lengths. The anomaly is not due to the Cotton effect since an examn. of alc I by the Hartley-Baly method shows no absorption in the anomalous region. The proximity of the double bond to an asym C atom is not responsible for the anomaly since camphene with the same characteristics shows no anomalous rotatory dispersion up to  $\lambda$  3100. The anomaly is not due to the presence of nopinene but the necessary expns showed that elaborate purification is necessary to free I from assoc impurities. After drying over anhyd.  $MgSO_4$ , 500 g. of I,  $n_D^{20}$  -16.8,  $n_D^{25}$  -17.1,  $n_D^{30}$  -18.2,  $n_D^{35}$  -18.8, was fractionated over Na at 21 mm, using a Widmer-Schenck column. The first 8 fractions were redistd in order, a fraction being added when nearly half the previous fraction had distd over. The sepn, though gradual, was quite perceptible by measurement of the rotation for  $\lambda$  5780, 5461 and 4358. The rotatory dispersions of a pure sample and of head and tail fractions were measured up to  $\lambda$  2780 by a modification of the usual photographic method. This was found necessary on account of the smallness of the rotatory dispersion in the visible and near ultraviolet regions of the spectrum. The anomaly at  $\lambda$  2800 (*C. A.* 26, 3185) is due to a closely assoc impurity which is separable from I only with great difficulty. A Darbous diagram of the various fractions showed, however, that anomalous dispersion in the near ultraviolet still remains a characteristic of I. This conclusion was supported by Raman effect data of the various fractions. The anomalous dispersion is due to a superposition caused by a 2nd rotation of opposite sign and having a different dispersion. This 2nd rotation cannot be attributed to the "induced dissymmetry" of the semicyclic double bond since the similarly constituted camphor has normal dispersion.

C. R. Addinall  
The slow combustion of pinane D. Shochet *Bull. soc. chim. Belg.* 44, 387-94(1935).—Oxidation of 1052 g. of pinane with 150 l. of  $O_2$  gas at 110° and subsequent neutralization with  $Ba(OH)_2$  yields 612 g. of a ketone product (I),  $C_{15}H_{24}O$  upon distn. I gives a semicarbazone m 196°, which is not identical with the semicarbazone of tetrahydrocarvone, m 185°. The formula  $MeCOCH(CMe)_2CHEt$  is suggested for I. W. J. Peterson

Metallic uranium in organic synthesis I. Jagaraj Behari Lal and Sukhbihushan Dutt *J. Indian Chem. Soc.* 12, 380-94(1935)—Friedel and Crafts reaction with U powder gave small yields of biphenyl from  $C_6H_6$  and  $PhBr$ ,  $PhCl$  and  $PhI$ . No reaction took place between  $CHCl_3$  and  $C_6H_6$ , but 0.9 g. of  $Ph_3CH$  was produced by refluxing 30 g. of  $C_6H_6$  with 12 g. of  $PhCHCl_2$  in the presence of 4 g. of U dust for 8 hrs. By heating a mixt. of 15 g. of  $PhCCl_2$  and 50 g. of  $C_6H_6$  with U for 8.5 hrs. at 115-23° and distg. the reaction mixt. 4.2 g. of  $Ph_2CCl_2$  and 1.2 g. of  $Ph_3CCl$  were isolated. Similarly from  $C_6H_6$  with  $AcCl$  and  $BzCl$  small yields of  $AcPh$  and  $BzPh$  were recovered. The reaction was unsuccessful with mixts. of  $CCl_4$  and  $C_6H_6$  and  $PhI$  and  $PhNH_2$ . Attempts to carry out Ullmann's reaction in the presence of U were not very encouraging. No result was obtained with  $CCl_4$  and only small yields resulted on refluxing  $PhBr$ ,  $PhCl$ ,  $PhI$ ,  $ICH_2CH_2CO_2H$ , mixts. of  $BrCH_2CO_2H$  and  $AcOEt$ ,  $PhOH$  and  $PhBr$ , and  $ClCH_2CO_2Na$  and anhyd  $AcONa$  with U powder. More satisfactory results were obtained in Zincke's reaction. By the action of  $PhCH_2Cl$  on  $C_6H_6$  dried over Na, in the presence of U, 40% of pure  $Ph_3CH$  was formed in addn to sym triphenylmethane and a mixt. of o- and p-dibenzylbenzenes. A small amt. of a S-yellow hydrocarbon, m 71°, was also recovered. Treatment of  $PhOFt$  and  $PhOMe$  with  $PhCH_2Cl$  gave 80% yields of p-ethoxydiphenylmethane and 55% anisylphenylmethane, together with some dibenzylanisole. Reformatsky's reaction with a mixt. of 12 g. of  $AcPh$ , 17 g. of  $BrCH_2CO_2Et$  and 70 cc. of dry  $C_6H_6$  in the presence of 35 g. of U dust gave a feeble reaction on warming. A crystal of I was added, and, after refluxing for 10 hrs. over an oil bath at 120-30°, the

reaction mixt. was decompd. with ice-cold dil. HCl. The washed and dried  $\text{CaH}_2$  layer was fractionally distilled at reduced pressure and yielded 2.4 g. of phenylmethyl-hydroxypropionic ester,  $b_p$  118–21°. An unsuccessful attempt was made to make a Grignard reagent from U and PhBr. II. Jagaraj Behari Lal. *Ibid.* 616 21.—The action of  $\text{PhCH}_2\text{Cl}$  (I) on  $\text{MePh}$ ,  $\text{PhOH}$ ,  $p\text{-MeO-C}_6\text{H}_4\text{OMe}$  and on the higher hydrocarbons, such as  $\text{C}_{10}\text{H}_{18}$ , acenaphthene and Ph<sub>2</sub>, in the presence of metallic U has been investigated and the various isomeric mono- and dibenzyl derivs have been isolated and characterized. Attempts were also made to prep. hexanitrophenyl from picryl chloride and 4,4-dinitrobiphenyl from  $p\text{-O}_2\text{NC}_6\text{H}_4\text{Cl}$ . It was found that U cannot be used as a neutral reducing agent in either aq. or aq. alc. solns. since attempts to reduce  $\text{Ph}_2\text{CO}$ ,  $p\text{-O}_2\text{NC}_6\text{H}_4\text{Me}$  and 2,4,6-( $\text{O}_2\text{N}$ )<sub>3</sub> $\text{C}_6\text{H}_2\text{OH}$  were unsuccessful. A mixt. of  $p\text{-MeOC}_6\text{H}_4\text{OMe}$  (16 g.), I (10 g.) and U (3 g.) was gradually heated to 70°. After the vigorous evolution of HCl the product was heated at 110–20° for 4 hrs. The material was extd. with *tert*-ene and the filtered, washed, dried and evapd. ext. was fractionated, yielding 12 g. (66.3%) of benzylsulfinyl di-*Me* ether,  $b_p$  353–5°, and 0.8 g. of rhombic crystals,  $m$  104–5°. Similar condensation of I (16 g.) with  $\text{PhOH}$  (21 g.) gave  $\alpha$ - and  $\beta$ -benzylphenols (3 g. and 18.7 g.), together with 2.2 g. of  $p$ -benzylphenol benzyl ether,  $b_p$  367–7°. A mixt. of 25 g. of I, 46 g. of  $\text{MePh}$  and 4 g. of U yielded 20 g. of  $p\text{-PhCH}_2\text{C}_6\text{H}_4\text{Me}$  and 11 g. of dibenzyltoluene but no bibenzyl was formed. Condensation of I with  $\text{C}_{10}\text{H}_{18}$  gave  $\alpha$ - and  $\beta$ -benzylacenaphthalene. None of the  $\alpha$ -,  $\beta$ - and  $\gamma$ -dibenzylacenaphthalenes could be isolated from the higher-boiling fractions. Acenaphthene gave 5-benzylacenaphthene together with  $\beta$ -benzylacenaphthene,  $m$  45–6°; picrate,  $m$  101–2°. The  $\alpha$ - and  $\beta$ -benzylbiphenyls,  $m$  44–5° and 51–6° (isobenzylbiphenyl,  $m$  54°; cf. Goldschmidt, *Monatsh.* 2, 472 (1881)).

## C. R. Addinall

**Biphenyl series. I. Synthesis of unsymmetrical derivatives of biphenyl.** Narendranath Chatterjee *J. Indian Chem. Soc.* 12, 410–17 (1935).—An extension and modification of Hirsch's method (*Ber.* 23, 3705 (1890)) for the synthesis of Ph<sub>2</sub> derivs. is described. The method involves the reaction of an aromatic diazonium salt with a phenol under conditions in which N is eliminated to yield a Ph<sub>2</sub> deriv. and a di-Ph ether as a by-product. Treatment of a cold soln. of 40 g. of  $\text{PhNH}_2$  in 80 g. of  $\text{H}_2\text{SO}_4$  and 40 cc. of  $\text{H}_2\text{O}$  with a soln. of 36 g. of  $\text{NaNO}_2$  in 80 cc. of  $\text{H}_2\text{O}$  gave a soln. of  $\text{PhN}_2\text{SO}_3\text{H}$  which was gradually added with const. shaking to 50 g. of  $\alpha\text{-MeC}_6\text{H}_4\text{OH}$  heated to about 95°. The black oil produced was steam-distd., yielding  $\text{Ph}_2\text{O}$  and 4-hydroxy-3-methylbiphenyl,  $\text{C}_{12}\text{H}_{10}\text{O}$ ,  $m$  114.1°. *MeO* deriv.,  $m$  76°, oxidized by  $\text{KMnO}_4$  to the corresponding acid,  $m$  172°, decarboxylated by heating with  $\text{Ca(OH)}_2$  to 4-*MeO-C}\_6\text{H}\_4\text{Ph}. A series of 9 hydroxydimethylbiphenyls in 10–15% yields and the oxidation products of their *MeO* derivs. were similarly obtained from the diazonium salts of  $\alpha$ -,  $m$ - and  $p$ - $\text{MeC}_6\text{H}_4\text{NH}_2$  and  $\alpha$ -,  $m$ - and  $p$ - $\text{MeC}_6\text{H}_4\text{OH}$ . The yields of the dimethylbiphenyl ethers is high and this method is equally suitable for their prepn. The hydroxydimethylbiphenyls are described for the 1st time and 3 of the Ph ethers are new. Though these Ph<sub>2</sub> derivs. throw little light on the stereochem. of the biphenyls they should be more easily resolvable since the  $p,p'$ -positions are definitely known to be occupied in some of them and these, on nitration or halogenation, would attach these groups only in the  $o,o'$ -positions. **Dimethylbiphenyl ethers.**  $\text{C}_{12}\text{H}_{10}\text{O}$ , 2,3',  $b_p$  170°; 2,4',  $b_p$  180°; 3,4',  $b_p$  175°. **Hydroxydimethylbiphenyls.**  $\text{C}_{12}\text{H}_{10}\text{O}$ , 4,3,2',  $b_p$  160° (*Ac* deriv.,  $b_p$  130°; *MeO* deriv.,  $b_p$  125°, oxidized to 4-methoxy-3,2-dicarboxybiphenyl,  $\text{C}_{12}\text{H}_8\text{O}_4$ ,  $m$  210°); 4,3,3',  $b_p$  175° (*Ac* deriv.,  $b_p$  158°; *MeO* deriv.,  $b_p$  160°, oxidized to the diacid,  $m$  232°); 4,3,4',  $b_p$  136° (*Ac* deriv.,  $m$  74°; *MeO* deriv.,  $m$  80°, oxidized to the diacid,  $m$  275°); 4,2,2',  $b_p$  185° (*Ac* deriv.,  $b_p$  175°; *MeO* deriv.,  $b_p$  170°, and diacid,  $m$  210°); 4,2,1',  $b_p$  193° (*Ac* deriv.,  $b_p$  186°; *MeO* deriv.,  $b_p$  185°, and diacid,  $m$  240°); 4,2,4',  $b_p$  225° (*Ac* deriv.,  $b_p$  210°; *MeO**

*deriv.*,  $b_p$  205°, and diacid,  $m$  280°); 2,2,5',  $b_p$  195° (*Ac* deriv.,  $b_p$  186°; *MeO* deriv.,  $b_p$  185°, and diacid,  $m$  above 300°); 2,5,3',  $b_p$  210° (*Ac* deriv., 201°; *MeO* deriv.,  $b_p$  200°, and diacid,  $m$  above 300°); 2,5,4',  $b_p$  225° (*Ac* deriv.,  $b_p$  215°; *MeO* deriv.,  $b_p$  205°, and diacid,  $m$  above 300°), and from  $\text{PhNH}_2$  and  $m\text{-MeC}_6\text{H}_4\text{OH}$ , 1-hydroxy-2-methylbiphenyl,  $\text{C}_{12}\text{H}_{10}\text{O}$ ,  $b_p$  180° (*Ac* deriv.,  $b_p$  164°; *MeO* deriv.,  $b_p$  164°, oxidized to 4-methoxy-2-carboxybiphenyl,  $\text{C}_{12}\text{H}_8\text{O}_4$ ,  $m$  135°). II. New method for the synthesis of 9-hydroxyphenanthrene. *Ibid.* 418–20.—A method of synthesis of phenanthrenes from Ph<sub>2</sub> derivs. which is capable of considerable extension is described. To a mechanically stirred soln. of 40 g. of diphenic anhydride in 100 cc. of alc., Na-Hg (5%) was added portionwise for 24 hrs. The soln. was kept acidic by addn. of a slight excess of dil.  $\text{H}_2\text{SO}_4$ . The final alc. soln. was filtered, and by fractional pptn. with  $\text{a.c.}$  extn. with dil.  $\text{NaHCO}_3$  and fractional crystn. from 1  $\text{t}$   $\text{c}$  crystalline needles of the pure lactone of 2-hydroxyethyl-2'-carboxybiphenyl (I),  $\text{C}_{12}\text{H}_{10}\text{O}_2$ ,  $m$  136°, were obtained in 5–6% yield, free from  $o,o'$ -diphenic acid and the monomer. By heating 0.5 g. of I with 0.3 g. of KCN for 3–4 hrs. in an oil bath, digesting the product with  $\text{H}_2\text{O}$  and extg. with  $\text{Et}_2\text{O}$ , filtering, acidifying the aq. layer and recrystg. the free acid from benzene, light brown needles of 2'-cyanomethyl-2-carboxybiphenyl (II),  $\text{C}_{12}\text{H}_8\text{N}_2\text{O}_2$ ,  $m$  210°, were obtained. Boiling in 30% KOH, filtering and acidifying gave 2'-carboxymethyl-2-carboxybiphenyl (III),  $\text{C}_{12}\text{H}_8\text{O}_4$ ,  $m$  295°. The Ca salt of III was distd. and a satd. soln. of picric acid was added to the alc. soln. of the distillate. Recrystn. of the red picrate from alc. gave a cryst. compd.,  $m$  185°, from which pure 9-hydroxyphenanthrene,  $m$  153°, was regenerated. III. A new route to phenanthrene. *Ibid.* 691–4.—The Na salt from a mixt. of 25 g. of 1  $\text{t}$  cyclohexanone-2-carboxylate and 3.4 g. of Na in 31 cc. of  $\text{EtOH}$  was heated under reflux for 6 hrs. with 10 g. of  $\text{CICH}_2\text{CO}_2\text{Et}$ , and, after diln. and extn. with  $\text{Et}_2\text{O}$ , gave 18 g. of di-*Et* cyclohexanone-2-carboxylate-2-acetate (I),  $\text{C}_{12}\text{H}_{20}\text{O}_4$ ,  $b_p$  168–75°, hydrolyzed by boiling with concd. HCl for 7 hrs. to cyclohexanone-2-acetic acid (II),  $\text{C}_{11}\text{H}_{18}\text{O}_3$ ,  $b_p$  163°; *Et* ester (III),  $b_p$  122° (semicarbazone,  $\text{C}_{11}\text{H}_{18}\text{N}_2\text{O}_3$ ,  $m$  190–7°). The action of  $\text{PhMgBr}$  on III produced 1  $\text{t}$  1-hydroxyhexahydrobiphenyl-2-acetate (IV),  $\text{C}_{12}\text{H}_{18}\text{O}_2$ ,  $b_p$  170–80°, hydrolyzed to the corresponding acid,  $\text{C}_{12}\text{H}_{16}\text{O}_3$ ,  $m$  129°. Dehydrogenation of IV by means of S yielded *Et* biphenyl-2-acetate,  $\text{C}_{12}\text{H}_{10}\text{O}_2$ ,  $b_p$  165–75° which, on hydrolysis with alc. KOH, yielded crude 9-hydroxyphenanthrene,  $b_p$  220–60°, purified through the picrate,  $m$  185°, to give a product,  $m$  153°, from which phenanthrene can be obtained by heating with  $\text{Se}$ . Treatment of I with  $\text{PhMgBr}$  gave the *Et* ester of a lactone,  $\text{C}_{12}\text{H}_{18}\text{O}_4$ ,  $m$  97°, which on hydrolysis yielded the free lactic acid, 1-hydroxyhexahydrobiphenyl-2-carboxy-2-acetic acid,  $\text{C}_{12}\text{H}_{16}\text{O}_5$ ,  $m$  141°, which may be utilized for the synthesis of phenanthrene.

## C. R. Addinall

**Furfural, its properties, uses, and commercial manufacture.** R. Heublyum. *Nitrocellulose* 6, 41–3, 63–4, 83–5 (1935).—A review.

## E. M. Symmes

**Molecular compounds of pyrroles.** Mladen Deželčić. *Ann.* 520, 290–300 (1935).—The following data were obtained by means of f. p. detns. In each case the eutectic point is given in mole % of the 2nd component; T. P. = transition point. 2,4-Dimethyl-3-acetyl-5-carbethoxypyrrole (I): *AcOH*, no solid compd.; succinic acid (II), 25%; 126.5°; *BzOH* (III), 63%; 87.5°;  $\text{CICH}_2\text{CO}_2\text{H}$  (IV), 91%; 52° (T. P. 55%, 85.3°);  $\text{PhOH}$  (V), 90%; 27° (T. P. 55%, 63°); salicylic acid (VI), 58%; 106° (2 T. P., 38%, 113°, and 60%, 107°); therefore 2 compds. 2  $\text{VI}$  and 1  $\text{VI}$ ; picric acid (VII), 60%; 84° (T. P., 50%, 97.8°);  $o\text{-C}_6\text{H}_4(\text{OH})_2$  (VIII), 57%; 71° (T. P., 41%, 108.5°);  $m\text{-C}_6\text{H}_4(\text{OH})_2$  (IX), 2 eutectics, 10%, 132.5° and 78%, 44° (max. at 33.3% and 139°);  $p\text{-C}_6\text{H}_4(\text{OH})_2$  (X), 2 eutectics, 10%, 138.2° and 62%, 135° (max. at 33.3%, 153°). 2,4-Dimethyl-5-carbethoxypyrrole (XI): IV, 80%; 49°; V, 86%; 23°; VI, 40%; VII, 66%; 71°; IX, 56.5%; 74.5°; X, 32%; 106°; XI, 66%; 91° (T. P., 33.3%, 100°; compd., 2XI.VII).



2,4-Dimethyl-5-carbethoxypyrrrole-3-aldehyde (XII). IV, 86%, 51° (T. P., 60°, 74.5°; compd. XIIIV); III, max at 50° and 135°, 2 eutectics; VII, 60%, 95.5° (T. P., 50°, 97°), VIII forms the compd. XII VIII, m 114° (2 eutectics); X forms the compd. XXII X, m 142° (2 eutectics); 2,5-Dimethyl-3-carbethoxypyrrrole-4-aldehyde (XIII). VIII, 67%, 56°; IX, 61%, 80°, compd. XIII IX, m 98°, compd. XIII X, m 117.5°, VI forms 2 compds, XXIII VI, and XXIV VI, eutectics 42°, 109° and 57.5°, 109° (T. P., 36°, 111°).

C. J. West

The synthesis of pyrrole alcohols. Quintino Mingos. *Gazz. chim. ital.* 65, 459-61 (1935).—In view of a paper by Taggart and Richter (C. A. 28, 4415') in which the synthesis of *N*-pyrrylcarbinol is described, earlier work by M. (cf. C. A. 27, 503) is discussed, and it is shown that in the reaction of magnesiumpyrrole and HCHO there is formed, besides *N*-pyrrylcarbinol, 2,3- or 2,5 pyrrole glycol. Under the proper exptl. conditions, *N*-pyrrylcarbinol may even be only a secondary product.

C. C. Davis

Synthesis of hexahydro- $\alpha$ -coumaranone. Ranajit Ghosh. *J. Indian Chem. Soc.* 12, 601-3 (1935).—To provide material for a study of the Friedel-Crafts reaction of aromatic hydrocarbons, phenols, phenol ethers, etc., with bicyclic  $\gamma$ -lactones with a view to the conversion of the products to phenanthrene derivs., hexahydro- $\alpha$ -coumaranone (I) has been prepd. Di-Et cyclohexanone-2-acetate-2-carboxylate,  $C_{12}H_{18}O_4$ , b<sub>1</sub> 161-2°, obtained by the action of  $ClCH_2CO_2Et$  on Et cyclohexanone-2-carboxylate (*Ann.* 350, 210 (1906)), was hydrolyzed by heating with concd. HCl for 7 hrs to cyclohexanone-2-acetic acid,  $C_6H_{10}O_3$ , b<sub>1</sub> 169-70° (semicarbazone, m. 199-200°); Et ester (from Na salt and EtBr), b<sub>1</sub> 130° (semicarbazone, m. 196°). Reduction of 20 g. of the acid with 400 g. of 30% NaHg and dehydration of the product with  $H_2SO_4$  gave 10 g. of hexahydro- $\alpha$ -coumaranone, b<sub>1</sub> 137°, identical with the lactone obtained by the treatment of 1,2-cyclohexeneacetic acid (C. A. 1, 1006) according to the method of Linstead and Meade (C. A. 28, 6115').

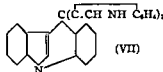
C. R. Addams

Reaction products of indoles with diazo esters. R. W. Jackson and R. H. Manske. *Can. J. Research* 13, 170-4 (1935).—In conformity with findings of previous work on indoles and in contradistinction to more recent work on pyrroles, the action of diazo esters on indole leads to 3-substituted as well as a small amt. of 1,3-disubstituted derivs. The formation of 2-substituted derivs. could not be demonstrated. The reaction discussed is a convenient one for the synthesis of a wide diversity of indole compds. Indole and  $N$ -CHCO<sub>2</sub>Et in dry Et<sub>2</sub>O with a trace of Cu powder yield a product which on hydrolysis gives indolyl-3-acetic acid (I) and indolene-1,3-diacetic acid (II), m. 242°. II on heating at 240° under reduced pressure gives CO<sub>2</sub> and 3-methylindolyl-1-acetic acid, m. 178°. Di-Et diazo-succinate in dry Et<sub>2</sub>O with indole and Cu powder gives a product which on hydrolysis yields indolyl-3-succinic acid (III), m. 199°; di-Et ester, m. 79-80°. III on heating by itself gives  $\beta$ -3-indolylpropionic acid. The prepn of I from  $CN(CH_3)CH(OEt)_2$  is described. R. B. Sandin

Action of the chloride of acetylsalicylic acid on magnesiumindoles. II. Cesco Toffoli. *Gazz. chim. ital.* 65, 487-97 (1935); cf. C. A. 28, 6437'.— $\alpha$ -AcOC<sub>6</sub>H<sub>4</sub>COCl (I) and magnesiumindole (II) (2 mols.) in anhyd Et<sub>2</sub>O, let stand overnight, heated 4 hrs at 100°, decomposed with ice, acidified with  $H_2SO_4$ , filtered, the Et<sub>2</sub>O layer extd with concd. aq. Na<sub>2</sub>CO<sub>3</sub> and the ext. acidified, ppts  $\alpha$ -HOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, while the Et<sub>2</sub>O evapd., the residue washed with petr. ether and boiling water, yields, after purification with  $C_6H_6$ ,  $\alpha$ -hydroxyphenyl  $\beta$ -indolyl ketone ( $\beta$ -salicylsalicylindole) (III), yellow, m. 171°, insol in dil and concd. HCl, alk. carbonates and alk. bicarbonates; its solns in concd.  $H_2SO_4$  are intense yellow; it is sol in alk. hydroxides (reppd. by bicarbonates and acids), with alk. FeCl<sub>3</sub> it gives an intense brown color which disappears on diln with water; it is stable on prolonged boiling in 0.5 N NaOH. III in cold anhyd Et<sub>2</sub>O treated with HCl gas ppts the HCl salt,  $C_{12}H_{11}O_3N$  HCl, intense

yellow, m. around 170° (in a sealed tube), hydrolyzed immediately by water. In boiling Ac<sub>2</sub>O with AcONa, III forms the di-Et deriv,  $C_{14}H_{19}O_4N$ , m. 60°, insol in aq. alkalis; in boiling concd. alkalis it reverts to III. By the method of Schotten-Baumann, III gives a di-Et deriv,  $C_{14}H_{19}O_4N$ , lustrous, m. 182°, shows a distinct superfusion phenomenon; with alkalis it forms III. By ordinary methods, including alk. PhNH<sub>2</sub> (IV), IV in AcOH, aq. alc. alk. NH<sub>2</sub>OH, etc., III does not react but III and IV (1.5 parts) in boiling dil EtOH for 50 hrs yield the phenylhydrazone,  $C_{14}H_{17}O_3N_2$ , m. 166°.

The mother liquor from the crystn. of III in its prepn. evapd., extd with 10% aq. NaOH, and the ext. treated with CO<sub>2</sub> yields  $\alpha$ -salicylsalicylindole (V), light yellow, m. 142°, insol in hot HCl, sol in aq. alk. hydroxides (reppd. by bicarbonates), not saponified by concd. alkalis. The part insol in 10% NaOH treated with picric acid (VI) in  $C_6H_6$  yields the picrate,  $C_{14}H_{11}O_8N$ ,  $C_6H_5O_7N_2$ , of VII (see later), brown-red, m. 150-5° (decompn.), decomposed by NH<sub>4</sub>OH into VI and anhydrosalicylsalicylindolyl- $\alpha$ -hydroxyphenylmethane (VII), dark cocoa color, m. 145-50° (to a thick black liquid), insol. in aq. alkalis.



The petr. ether wash liquor from the prepn. of III yields a little III, and an acetylsalicylsalicylindole,  $C_{14}H_{17}O_4N$ , light yellow, m. 148° (to an intensely red liquid), sol in aq. alkalis (reppd. by bicarbonates), insol in concd. HCl, gives yellow solns in concd.  $H_2SO_4$ . The boiling aq. wash liquor from the prepn. of III yields  $\beta$ -acetylsalicylindole, m. 187-9° (cf. Oddo and Sessa, C. A. 5, 2638). On repeating the reaction of I and II, but with equimol. parts, the same products were obtained, and also an unidentified black resinous substance, m. around 200°, probably formed by the condensation of V with itself by the action of excess I. The same reaction with I and magnesyalsalicylindole (VIII) (2 mols.) yields  $N$ -salicylsalicylindole (IX), m. 151°, sol in aq. alk. hydroxides (reppd. by bicarbonates); its solns in  $H_2SO_4$  are yellow when cold and intensely red-violet when hot; it is decomposed by boiling alk. hydroxides into skatole and  $\alpha$ -HOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H. Mono-Bz deriv.,  $C_{14}H_{17}O_3N$ , m. 89° (shows superfusion phenomenon). The mother liquor from IX yields on distn. *in vacuo*  $N$ -acetylsalicylindole (cf. Oddo, C. A. 8, 85). To obtain the deriv. corresponding to III, I and VIII were made to react under the conditions described by Oddo (*loc. cit.*) for prepn.  $\alpha$ -acetylsalicylindole. By then steam-distg. the Et<sub>2</sub>O-insol. product, extg. the new residue with 10% aq. NaOH, and satg. the ext. with CO<sub>2</sub>, there was obtained  $\alpha$ -salicylsalicylindole, yellow, m. 124°, sol in aq. alk. hydroxides (reppd. by bicarbonates), insol in concd. HCl; its solns in concd.  $H_2SO_4$  are orange when cold and violet-red when hot. The product insol in aq. NaOH yields an unidentified compd., dark brown-gray, m. around 200°; it probably has a high mol. wt. and is formed by condensation of the primary products. The reactions between I and magnesyalsalicylindoles throw light on the influence of the phenolic OH group on the course of the reaction. When the  $C_6H_5OMgBr$  group first formed does not participate in subsequent reactions, the product is that of the normal reaction, i.e., the  $\alpha$ -acyl or  $N$ -acyl deriv. When, however, the  $C_6H_5OMgBr$  does react, there are 2 courses to the reaction. If the temp. is not high, the products are of relatively simple constitutions, and are formed thus:  $C_6H_5OMgBr + HN \rightarrow C_6H_5N + MgBrOH$ . On the other hand when the Et<sub>2</sub>O is eliminated and the temp. is increased, more complex products are formed, which are dark, difficult to crystallize and have high mol. wts. The traces of Cu in the Grignard reagent probably facilitate the entrance into the reactions of the OMgBr group.

C. C. Davis

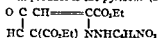
Preparation of  $\alpha$ -nitrophenyl aryl ketones. Max



10.53% yield of  $\alpha$ -benzallacetic acid (proved to be  $\alpha$ , and not  $\beta$  or  $\delta$  by mixed m-p depts with authentic samples); PhMeC<sub>2</sub>CHCOCl, 140-50°, 3.5 hrs in a closed tube or 4 days at 50°, then 0.5 hr. at 100-110° in a stoppered flask, failed to yield a condensation product. To det. the effect of catalysts on the reaction, 1.5 g. of AlCl<sub>3</sub> and 2 g. of BzH were allowed to react 8 hrs in closed tubes at 170-80° with 1.5 mol equivs of the following bases, with yields of PhCH<sub>2</sub>CHCO<sub>2</sub>H as listed: no catalyst, trace, C<sub>2</sub>H<sub>5</sub>N, 0.72%, quinoline, 0.14%, piperidine, 1.79%, Et<sub>3</sub>NH, 2.15%, m-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>), none (7 hrs at 140-50°), PhNH<sub>2</sub>, 0.11% (6 hrs at 130-40°). AcOH and BzH as above gave no reaction product; AlCl<sub>3</sub> and BzH treated similarly, followed by 3 hrs heating with a 20% MeOH soln. of KOH, gave no PhCH<sub>2</sub>CHCO<sub>2</sub>H, but BzOH instead. G. R. Yohe

Citronellal terpene I. Existence of a new terpene C<sub>15</sub>H<sub>24</sub>O (Hiroshi Otsuka, J. Chem. Soc. Japan 56, 1213-20 (1935)).—Shaking of citronellal with 10 vols. 50% H<sub>2</sub>SO<sub>4</sub> for 12 hrs. at room temp. and distn. gives a new terpene, monogene (I), C<sub>15</sub>H<sub>24</sub>, b<sub>m</sub> 184-6°, d<sub>4</sub><sup>20</sup> 0.8624, n<sub>D</sub><sup>20</sup> 1.5026, [α]<sub>D</sub><sup>20</sup> 49.11°. Nitrosate of I, m. 154.5-5°. The structure of I seems to be most close to Δ<sup>4</sup>(10)-p-menthadiene. The lower-boiling fraction gives α-terpinene. K. Kutsuta

Reactivity of carbonyl group in γ-pyrones and in γ-pyridones D. N. Bedekar, R. P. Kaushal and S. S. Deshpande, J. Indian Chem. Soc. 12, 465-9 (1935).—In general the γ-pyrones and γ-pyridones do not form oximes or phenylhydrazones but the more reactive p-O<sub>2</sub>NCH<sub>2</sub>CHNHNH<sub>2</sub> (I) acts on Et chelidonate (II) to give the nitrophenylhydrazone (III), C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>, m. 216°, hydrolyzed by HCl in AcOH to II. The nitrophenylhydrazone of Et 3,5-dibromochelidonate, C<sub>15</sub>H<sub>10</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub>, m. 120°, was similarly prepd. On heating equimol proportions of I and Et xanthochelidonate in abs. alc. for a short time the main product is the pyridone (III)



m. 146°, together with some of its nitrophenylhydrazone (IV), m. 210°. The yield of IV increases with the duration of heating to totality in 1 hr. Similarly equimol. proportions of (AcCH<sub>2</sub>)<sub>2</sub>CO and I yielded a pyridone, C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>, m. 136°, which on treatment with excess I gave the nitrophenylhydrazone, m. 215°. The reaction of 2 mols of I with pyrone produced a product C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>, m. 242°, in which both O atoms have reacted. Chelidonic acid and Et comanate similarly reacted with 2 mols. of I to give pyridone nitrophenylhydrazones, C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>, m. 210°, and C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>, m. 220°. In no case could any definite product of the reaction of 1 mol of I be isolated. C. R. Addinall

Constitution of fustun V. Synthesis of 4'-methoxy-3-hydroxyflavanone, Taichiro Oyama, J. Chem. Soc. Japan 56, 980-3 (1935), cf. C. A. 29, 762<sup>1</sup>—4'-Methoxy-3-hydroxyflavanone made by synthesis showed all the characteristics of methylflutin. K. Kutsuta

Chemical examination of *Cuscuta reflexa*, Roxb. I. The constituents. Radha Raman Agarwal and Sukhshushan Dutt, J. Indian Chem. Soc. 12, 384-8 (1935).—*Cuscuta reflexa* (Roxb.), the Hindustani *akashbel*, is a golden yellow parasitic plant, common throughout India, considered to have purgative properties by native writers. Extn. of 2.5 kg. of sun-dried material from 30 kg. of the freshly collected plant, with boiling alc. gave 25 g. of crude dry cryst. substance contg. a small amt. of yellowish brown wax. Recrystn. from large vols. of EtOH produced white cryst. flakes of *cuscutalin* (I), C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>, m. 68°, sol. in caustic alkalis. It reduces Tollen's reagent slowly, gives a reddish brown color with alk. K<sub>2</sub>Fe(CN)<sub>6</sub>/NO. It has no aldehydic or ketone groups and is evidently an α,β-unsatd lactone (C. A. 21, 99). Pharmacologically, I appears to be a potent drug. After the removal of I, the combined alc. exts. were concd. to a greenish red syrup which was extd. with benzene. The ext. was evapd. to dryness and, after washing with cold alc., was crystd.

from boiling alc., yielding white flakes of I. The residue was extd. with AcOH and was then extd. with ice-cold H<sub>2</sub>O with const. stirring. The brown, difficultly sol. residue was filtered off and dissolved in H<sub>2</sub>O at room temp. to a golden yellow soln. from which yellow crystals of *cuscutin* (II), C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>, m. 208-9° (decompn.), sepd. II is sol. in NaHCO<sub>3</sub> and forms a Pb salt, and a *di-Ac* deriv., m. 140°. By treatment with ClCO<sub>2</sub>Et in pyridine and by methylation with Me<sub>2</sub>SO<sub>4</sub> in the presence of concd. NaOH, *dicarboethoxycuscutin*, m. 151° (decompn.), and *dimethoxycuscutin*, m. 193°, were prepd. II gives a neg. test for flavones and is completely decolorized on boiling with Ni(OH)<sub>2</sub> and Zn dust. It is not glucosidic in character. The above aq. ext. was concd. and was found to contain a large amt. of reducing sugars. II. Constitution of *cuscutalin* Ibid. 586-80.—The white cryst. lactone-like material isolated in 1.5% yields from *Cuscuta reflexa* gave, on recrystn. from MeOH and EtOH, colorless flakes of *cuscutalin* (I), C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>, m. 68°, [α]<sub>D</sub><sup>20</sup> 8° (Ac deriv., C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>, m. 74°, carboethoxy deriv., C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>, m. 105°). Methylation of I with alk. Me<sub>2</sub>SO<sub>4</sub> produced *dimethoxycuscutin mono-Me ester*, C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>, m. 78°, and warming with concd. HCl transformed I into *anhydrocuscutalin* (II), m. 71-2°. Bromination and reduction with Zn and AcOH formed *dibromocuscutalin bromide*, C<sub>15</sub>H<sub>12</sub>Br<sub>2</sub>O<sub>4</sub>, m. 64°, and *dihydrocuscutalin*, m. 91°, resp. Volumetric estn. of the unsatn. of I showed the presence of 2 double bonds in I. Although I has no CO or CHO groups, it gradually reduces Tollen's reagent. It dissolves in alc. KOH and gives a reddish brown color with alk. K<sub>2</sub>Fe(NO<sub>3</sub>)(CN). I is probably an α,β-unsatd. lactone (C. A. 21, 99), and of the 4 O atoms 2 are in the lactone ring and 1 in the OH group. In the formation of II the OH group is lost since II gives no Ac deriv. I dissolved in boiling alc. KOH and on acidification liberated an isomeric α lactone, m. 103°. Fusion of I with KOH gave HCO<sub>2</sub>H, PhCH<sub>2</sub>CHCO<sub>2</sub>H and an aromatic unsatd. hydrocarbon, m. 82°. I was oxidized by 3% alk. KMnO<sub>4</sub> to produce some (CO<sub>2</sub>H) and BzH (probably due to the oxidation of the intermediate PhCH<sub>2</sub>CHCO<sub>2</sub>H). Dry distn. of I gave unchanged material, some II and unidentified hydrocarbons. C. R. Addinall

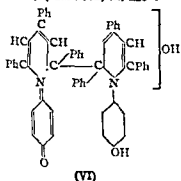
Anthocyanins from the red and violet flowers of *Pharbitis nil* Choisy. Takeo Kataoka, J. Chem. Soc. Japan 56, 1037-42 (1935).—Ext. 1 part dried red morning-glory flowers with 10-12 parts MeOH contg. 2% HCl for 2 hrs., filter through cloth and ext. the residue twice with 6-6 parts solvent for 2 hrs. Unite the exts., pass through the filter paper, keep the filtrate at 55-60° for 3 hrs. and distil *in vacuo* at 60° to 1/3-1/4 of the original vol. After addn. of 0.5 vol. of alc. and 3-4 vols. of ether to obtain a red ppt., let stand for several days. Decant the clear soln., treat the residue with MeOH contg. 1% HCl, filter off the crystals and wash with MeOH contg. 2% HCl. Recrystn. from MeOH contg. 2% HCl gives fine red needles of pelargonium chloride, C<sub>15</sub>H<sub>10</sub>O<sub>4</sub>Cl<sub>2</sub> 5H<sub>2</sub>O. The sugar obtained by hydrolysis is glucose. Similar treatment of pinkish violet flowers gives picein chloride, needles, C<sub>15</sub>H<sub>10</sub>O<sub>4</sub>Cl<sub>2</sub> 5H<sub>2</sub>O, m. 163° (anhyd, m. 174°). K. Kutsuta

Anthocyanin pigment of the flowers of deep-blue *hyacinth* Kozo Hayashi, J. Chem. Soc. Japan 56, 1050-4 (1935).—The pigment consists of delphinidin diglycoside. K. Kutsuta

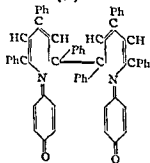
Constitution of the anthocyanins L. F. Levy, J. S. African Chem. Inst. 18, 27-35 (1935).—A review of the literature, with 32 references. B. E. Anderson

Arylated pyridines IX. W. Diltbey and H. Dienrich, J. prakt. Chem. 144, 1-31 (1935); cf. C. A. 19, 989—2,4,6-Triphenylpyridium perchlorate and PhNH<sub>2</sub>, heated a short time on the water bath, MeOH added and then refluxed 1 hr., give a nearly quant. yield of 1,2,4,6-tetraphenylpyridinium perchlorate (I), m. 200°; p-MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gives the 1-(4-methylphenyl) analog (II), pale yellow, m. 243-4°; with NH<sub>2</sub>OH II (after refluxing 1.5 hrs in dil. EtOH) gives the compd. PhC(NH<sub>2</sub>OH)(CH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>CH<sub>2</sub>C(O)Ph II<sub>2</sub>O, m. 163°. The 1-(2-methylphenyl) analog of I m. 254-5°; 1-(4-ethoxyphenyl)

analog, m. 274°; 1-(2-methoxyphenyl) analog, m. 242-3°; 4 MeO isomer (III), m. 243° (sodium, yellow, m. 300-10°), III and NH<sub>4</sub>OH give the compd. PhC(CH<sub>3</sub>)CHPh(NO<sub>2</sub>)CH PhNHCH<sub>3</sub>OMe, m. 145-6°. The 1-(4-hydroxyphenyl) analog (IV) of I m. 170°, then 245°, the salt is about 23% assoc. (mol. wt. 307); this also results from III with HCl, HBr or HI; chloride, m. 350-1°, bromide, m. 382-3°; periodate, brown; iodide, yellow, m. about 340°; nitrate, pale yellow, m. 323-4° (decompn.), picrate, yellow, m. 214-15°. The Ac deriv. of IV m. 288° (periodate, red, m. 185°). The red base from IV and H<sub>2</sub>O<sub>2</sub> give 1-(4-hydroxyphenyl)-2,4,6-triphenylpyridinium hydroperoxide (V), m. 201° (decompn.), HClO<sub>4</sub> gives IV. The above salts with MeOH-KOH give yellow solids in the cold, becoming deep red on heating and yellow again on cooling IV gives a deep red color in the cold. In CHCl<sub>3</sub> or C<sub>6</sub>H<sub>5</sub>N, cold MeOH-KOH gives a deep blue color, becoming red on further addn. of alkali. AcOH gives with the chloride from IV a red compd., sol. in MeOH, changing to the blue base on addn. of H<sub>2</sub>O IV (1 g.) in 70 cc. MeOH, treated with 10 cc. MeOH-KOH (1:4), boiled 30-45 min and filtered into 200 cc. 10% NaOH or KOH, gives 0.8-1 g. of VI, red, with 12 mols. H<sub>2</sub>O; drying over CaCl<sub>2</sub> or P<sub>2</sub>O<sub>5</sub> gives the blue-violet anhydro base (VII). VI may be recrystd. from H<sub>2</sub>O, from MeOH-Et<sub>2</sub>O, VI contains MeOH of crystn, which is lost in the air, giving VII, 100 cc. H<sub>2</sub>O at 18° dissolves 0.080 g. VI VI with HClO<sub>4</sub> gives 91-9% V, excess of nitrate gives the nitrate from IV; H<sub>2</sub>O<sub>2</sub> gives V. VII, on standing in the air some weeks, is oxidized and cannot be regenerated on drying. Reduction of VI gives PhOH and triphenylpyridine. A soln. of VII in piperidine is also oxidized in the air. VII gives only 0.02% active H<sub>2</sub> VI loses 11 mols. H<sub>2</sub>O over P<sub>2</sub>O<sub>5</sub> in vacuo; the last mol. is lost in a PhMe bath at 2-3 mm., VI from MeOH-Et<sub>2</sub>O contains 2 mols. H<sub>2</sub>O. VII takes up 4 mols. H<sub>2</sub>O in the air in 12 hrs. The perchlorate of VI, red, m. 225°, with 1.5 mols. H<sub>2</sub>O; picrate, with 1 mol. H<sub>2</sub>O, red, m. 240°; nitrate, with 3 mols. H<sub>2</sub>O, m. 222-3°, chloride, with 3 mols. H<sub>2</sub>O, dark red, m. 214-15°, bromide, with 4 mols. H<sub>2</sub>O, dark red, m. 225-8°, iodide, with 3 mols. H<sub>2</sub>O, dark red, m. 220°.



(VI)

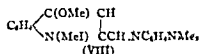
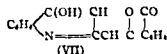
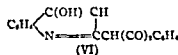
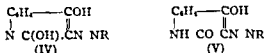
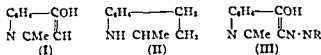


(VII)

2-Methyl-4-hydroxyquinoline and its derivatives. Madeline Maun. Ann. chim. [11], 4, 301-63 (1935).—4-Hydroxyquinoline is 2-methyl-4-hydroxyquinoline and is prepd. by mixing at ordinary temp. equal mol. proportions of PhNH<sub>2</sub> and AcCH<sub>3</sub>CO<sub>2</sub>Et. By heating this compd.

C. J. West

1 rapidly to 240°, EtOH is evolved and I remains. It loses 2 H<sub>2</sub>O of crystn. at 110°, m. 210°, is sol. in EtOH, boiling H<sub>2</sub>O, and insol. in Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, and CHCl<sub>3</sub>. (C<sub>6</sub>H<sub>5</sub>ON), H<sub>2</sub>SO<sub>4</sub> is pptd. from H<sub>2</sub>SO<sub>4</sub> solus. by Et<sub>2</sub>O. The 2% aq. solns. of this salt is 2:4. On reduction by Na in abs. EtOH, II is obtained. The picrate m. 153-4°. The azo compd. III was prepd. by coupling I with diazonium salts. 2,4-Dihydroxyquinoline yields an azo compd. which is tautomeric as follows (IV ⇌ V). By reaction with phthalic anhydride, 1 of the compds. VI or VII results. Studies were made of the reaction of hydroxyquinoline with aldehydes, and aromatic nitrosamines. Methoxyquinoline-MeI and p-ONC<sub>6</sub>H<sub>4</sub>NMe<sub>2</sub> gave VIII.



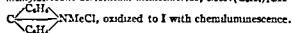
H. E. Messmore

3,6-Dialkoxy-10-alkylacridinium derivatives with various kinds of amino groups on the 9-carbon atom. XV. Synthesis of 3,6-diethoxy-9-alkylamino-10-ethylacridinium derivatives. Kiyoyuki Ishihara. J. Chem. Soc. Japan 56, 1164-73 (1935); cf. C. A. 29, 79841a. — Consts. of 3,6-diethoxy-9-alkylamino-10-ethylacridinium iodides: The alkyl groups are Me, Et, Pr, iso-Bu and iso-Am in the order named. (n) is the no. of mols. of HCl and (m) that of mols. H<sub>2</sub>O per mol. (n): 1, 1 1/2, 0.5, 1, 0.5. (n) after drying at 120°: 1, 1, 0.5, 0.5, 0.5. M. ps. (decompn.): 227°, 224°, 223-5° (m. 207°), 230°, 227°. Soly. (vol. %) in H<sub>2</sub>O at 15°: 0.012, 0.006, 0.008, 0.006, 0.002. 3,6-Diethoxy-9-alkylamino-10-ethylacridinium hydroxides: (m): 0, 0, 0, 1, 1. (n): 1, 1, 1, 0.5, 0. (m) after drying at 120°: 0, 0, 0, 0, 1. (n) after drying at 120°: 1, 1, 1, 0. M. ps.: 126°, 115°, 105°, 122°, 101°. 3,6-Diethoxy-9-alkylamino-10-ethylacridinium chlorides: (m): 0, 0.25, 0, 0.2, 0.6. (n): 1 1/2, 2.2, 1.5, 2.25, 3. (m) after drying at 120°: 0, 0, 0, 0, 0. (n) after drying at 120°: 0.5, 1, 1, 1.25, 1. M. ps. (decompn.): 225°, 216°, 230°, 194°, 190° (m. 152°). Soly. (vol. %) in H<sub>2</sub>O at 15°: 0.507, 1.125, 1.507, 0.605, 0.350. 3,6-Diethoxy-9-alkylamino-10-ethylacridinium oxalates: (m): 0.3, 0.5, 0.5, 0.5, 0.5. (n) 0.5, 0.5, 0, 1, 1. (n) after drying at 120°: 0.1, 0.5, 0, 1, 1. M. ps. (decompn.): 195°, 180°, 174°, 199°, 172°. Soly. (vol. %) in H<sub>2</sub>O at 15°: 0.298, 0.664, 0.750, 0.170, 0.389.

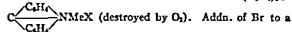
K. Kitsuta

Biacridyl and its derived radicals and luminescent salts, luzigenin salts. Herman Decker and Werner Petsch. J. prakt. Chem. 143, 211-35 (1935).—The chemiluminescent reactions of N,N'-dimethylbiacridylum (luzigenin) salts (I), of related compds. and of the derived free radicals are described and discussed in relation to structure and nomenclature (used below) is suggested. N,N'-Dimethylbiacridylum nitrate (II) and excess concd. aq. KBr give the bromide (III), yellow, sol. in H<sub>2</sub>O with green fluorescence, which splits off MeBr at 230°, giving biacridyl. KCN gives 9,9'-dicyanobiacridan (biacridan

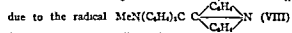
$\approx 9,10,9',10'$ -tetrahydrobiacridyl). The action of Zn on III or of Zn-AcOH on *N*-methylacridone gives dimethylbiacridene,  $[\text{MeN}(\text{C}_6\text{H}_4)_2\text{C}]_2$  (IV), m. 385°, decomp. 390° (cor.), which is stable to air or to reduction in acid or alk. soln. IV is an en-onium conjugated pseudo-base, converted, with loss of fluorescence, by  $\text{HCl-C}_6\text{H}_5$  into methylacridone-acridinium methochloride,  $\text{MeN}(\text{C}_6\text{H}_4)_2\text{CH-}$



oxidized to I with chemiluminescence. Its aq. soln., shaken with  $\text{C}_6\text{H}_6$ , gives a rose color due to the formation of the dimethylbiacridanyl radical,  $\text{MeN}(\text{C}_6\text{H}_4)_2\text{CH}(\text{C}_6\text{H}_4)_2\text{NMe}$ ; addn. of NaOH does not regenerate IV but gives the colorless carbinol base,  $\text{MeN}(\text{C}_6\text{H}_4)_2\text{C}(\text{OH})\text{CH}(\text{C}_6\text{H}_4)_2\text{NMe}$ ; a picrate, m. 280°, decomp. 295°, is isolated from the  $\text{C}_6\text{H}_5$  soln.; the aq. NaOH is colored violet due to the radical  $\text{MeN}(\text{C}_6\text{H}_4)_2\text{C-}$



(destroyed by  $\text{O}_2$ ). Addn. of Br to a  $\text{C}_6\text{H}_5$  soln. of IV gives III. With  $\text{Me}_2\text{SO}$ , in hot PhMe biacridyl (V) gives only the mono-Me sulfate, yellow, m. above 350°, aq. KBr gives 10-*N*-methylbiacridylum bromide (VI), yellow, m. above 350°; picrate, m. 305° (decomp.); iodide, dark red; ferricyanide, yellow, m. 250° (decomp.); with hot  $\text{K}_2\text{Fe}(\text{CN})_6$ , VI gives a compd.  $(\text{C}_6\text{H}_5)_2\text{N}_2\text{O}$ , m. 345°, probably an internal ether. VI, shaken with Zn- $\text{C}_6\text{H}_5$  gives a violet soln., stable to  $\text{O}_2$ ,



due to the radical  $\text{MeN}(\text{C}_6\text{H}_4)_2\text{C-}$  (VIII) (picrate, yellow, m. 220°), but in  $\text{HCl-C}_6\text{H}_5$  with exclusion of  $\text{O}_2$ , monomethylbiacridene is obtained; it is readily oxidized by air to VIII and is converted by I to VII. V is similarly converted into biacridene, m. 392°, which reacts in a similar manner to its Me deriv. V must be heated with  $\text{Me}_2\text{SO}$ , without solvent to give I. The final product of the chemiluminescent reaction of  $\text{KOH-H}_2\text{O}_2$  on I is *N*-methylacridone C. J. West

Racemization of diethylaminomethylbenzodioxan J. Tréfiou, Mme. J. Tréfiou and Y. Dumas. *Bull. sci. pharm.* 42, 459-66(1935).—Prepn. of hydroxymethylbenzodioxan monohydrate (I): boil for 3 hrs. 25 g. hydroxymethylbenzodioxan (m. p. 83°), 22 g.  $\text{C}_6\text{H}_5(\text{CO})_2\text{O}$  and 80 cc. xylene. Keep overnight at 0°, wash first with 50 cc.  $\text{PhH}$  and crystallize twice from  $\text{PhH}$ . It m. 116°; yield 25g. Prepn. of the racemic salt with *d*-ephedrine: Mix the hot solns. of 37.74 g. I in 300 cc.  $\text{PhH}$  and 22 g. *d*-ephedrine in 200 cc.  $\text{PhH}$ . Cool and filter off the crystals. Sepn. of *d*- and *l*-isomers: Boil 51 g. of the salt with 604 cc.  $\text{PhH}$  for 10-12 min. Filter by suction and wash with cold  $\text{PhH}$ . Repeat this treatment. Recrystallize twice from  $\text{MeOH}$  and then twice from  $\text{EtOH}$ , finally again from  $\text{MeOH}$ . Yield 7 g. of the *l*-salt,  $[\alpha]_D^{25} -1.56^\circ$ . The free *l*-I is obtained by removal of the ephedrine with  $\text{NaOH}$  soln. and  $\text{Et}_2\text{O}$  and decompn. of the Na salt with  $\text{HCl}$ . The free ester is sepd. with  $\text{Et}_2\text{O}$ . Sapon. with  $\text{NaOH}$  at 100° gives needles of 1-hydroxymethylbenzodioxan (II), m. 80°,  $[\alpha]_D^{25} -1.90^\circ$ . The use of *l*-ephedrine gives the corresponding *d*-compd. Treatment of II in pyridine with  $\text{SOCl}_2$  at 100°, addn. of  $\text{H}_2\text{O}$  and extn. with  $\text{Et}_2\text{O}$  gives 1-chloromethylbenzodioxan. Heat 2.85 g. of the latter with 3.38 g.  $\text{HNEt}_3$  in a closed tube to 175° for 2 hrs., take up with  $\text{Et}_2\text{O}$ , ext. with  $\text{HCl}$ , add  $\text{NaOH}$  and distil. Yield, 1.44 g. 1-dimethylaminomethylbenzodioxan, b.p. 164°,  $[\alpha]_D^{25} -29.10^\circ$ . The  $\text{HCl}$  salt m. 123-30°. A. E. Meyer

Vasine. Mohan Lal Beri, Kartar Singh Narang and Jnanendra Nath Ray. *J. Indian Chem. Soc.* 12, 395-9 (1935).—Attempts to synthesize vasine (I) have been made on the basis of a structure previously proposed (C. A. 27, 510) on the grounds that I gave 4-quinazoline (II) by oxidation and anthranilic acid by  $\text{KOH}$  fusion. A mixt. of 5.4 g. of II, 1.5 mol. of allyl iodide and 1.2 mols. of 10%  $\text{KOH}$  in 54 cc. alc. was refluxed on the steam bath for 3 hrs. After concn. to 15 cc. and treatment with excess  $\text{KOH}$  an oil sepd. which, on extn. with  $\text{Et}_2\text{O}$  and crystn. from petr. ether, gave 3-allyl-4-hydroxyquinazoline,

$\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}$ , m. 67°, stable to steam distn. and heating with  $\text{HCl}$ , reduced by Na in  $\text{AmOH}$  to a liquid, b.p. 105-8°, which is not identical with the base obtained by Späth (C. A. 29, 4015). Unsuccessful attempts were made to condense  $\text{EtCHBrCONHPh}$  (III) with  $\text{H}_2\text{NCOEt}$  to form a quinazoline but hot  $\text{PhNMe}$ , converted III into crotonanilide in poor yields. The action of alc.  $\text{KOH}$  transformed III into delicate colorless needles of  $\beta$ -ethylindolenone,  $\text{C}_{10}\text{H}_9\text{NO}$ , m. 254-5°. By refluxing an  $\text{Me}_2\text{CO}$  soln. of III with  $\text{Ag}_2\text{O}$  for 9 hrs.,  $\text{EtCH}(\text{OH})\text{CONHPh}$ , m. 89-90°, was formed, which could not be condensed with  $\text{H}_2\text{NCOEt}$ . Treatment of *o*- $\text{O}_2\text{NC}_6\text{H}_4\text{COCl}$  (6 g.) with 16 g. of  $(\text{NH}_4)_2\text{CO}_3$  gave 5 g. of the amide, m. 178°, which was reduced by  $\text{SnCl}_2$  in  $\text{HCl}$  to *o*- $\text{H}_2\text{NC}_6\text{H}_4\text{CONH}_2$  (IV), m. 110°. Condensation of IV with  $\text{PrCHBrCOBr}$  gave  $\text{C}_{11}\text{H}_{13}\text{BrN}_2\text{O}_2$ , m. 144°, which on treatment with  $\text{NaOH}$  and acidification with  $\text{AcOH}$  yielded 2-bromopropyl-4-hydroxyquinazoline, m. 218°, reduced by Zn dust in  $\text{NaOH}$  to 2-propyl-4-hydroxyquinazoline, m. 200°. Condensation of IV with itaconic anhydride gave a colorless substance,  $\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}_4$ , m. 174-6°, which was cyclized by treatment with  $\text{NaOH}$  and subsequent acidification with  $\text{AcOH}$  to a cyclic carboxylic compd.,  $\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}_5$ , m. 197-8°, but whose decarboxylation to the required substance with the proposed structure for I did not proceed satisfactorily. C. R. Adnall

Synthesis of lactoflavin (vitamin B<sub>2</sub>) Richard Kuhn, Karl Reinmund, Friedrich Weygand and Rudolf Sträbele. *Ber.* 68B, 1765-74(1935); cf. C. A. 29, 37077.—The condensation of 1,2-dimethyl-4-amino-5-(*d*-1'-ribitylamino)benzene (I) with alloxan (III) gives a flavin identical with lactoflavin. By carrying out this condensation in  $\text{AcOH}$  in the presence of  $\text{H}_2\text{BO}_3$  at 15-20° the yield of cryst. material can be increased to 90-95% since the necessity of purification by adsorption and elution is eliminated. The degradation of 2 kg. of Ca *d*-gluconate (C. A. 28, 5047) with  $\text{H}_2\text{O}_2$  and  $\text{Fe}_2(\text{SO}_4)_3$  in the presence of  $(\text{AcO})_2\text{Ba}$  gave 520 g. of pure *d*-arabinose of which 500 g. was converted into 320 g. of acetobromo-*d*-arabinose, 140 g. of diacetyl-*d*-arabinal, 80 g. of *d*-arabinal and, finally, into 53 g. of *d*-(-)-ribose (III), m. 87°,  $[\alpha]_D^{25} 23.7^\circ$ ; oxime (IV), m. 141°,  $[\alpha]_D^{25} 6.3^\circ$  (final value after mutarotation from 54° (after 10 min.)). Reduction of IV with Na-Hg gave *d*-ribanose which was condensed with 1,2-dimethyl-4,5-dinitrobenzene (V), m. 115-16°, by heating in 80% alc. for 6 hrs. at 130° to 1,2-dimethyl-4-nitro-5-(*d*-1'-ribitylamino)benzene (VI),  $\text{C}_{18}\text{H}_{20}\text{N}_4\text{O}_6$ , m. 127°. A soln. of 0.60 g. of VI in 75 cc. of 80% alc. was shaken with 0.20 g.  $\text{PtO}_2$  and  $\text{H}_2$  (3 mols.) for 20 min. The filtered colorless soln. was evaporated, taken up in 30 cc. of  $\text{AcOH}$  and was run into a soln. prepd. by heating 0.46 g. of alloxan tetrahydrate and 0.90 g. of  $\text{H}_2\text{BO}_3$  with 30 cc.  $\text{AcOH}$  and cooling to 30°. After shaking to complete soln., crystals sepd. which were extd. with abs. alc. and produced 91% yields of felled, golden yellow fine needles of 6,7-dimethyl-9-*d*-riboflavin, m. 231-2° (tetra-Ac deriv., m. 242°), identical in all its chem., phys., and physiol. properties with the naturally occurring vitamin. Lactoflavin is related therefore to 4 different classes of org. compds. whose various properties it combines; to the sugars through its pentose chain, to the pyrimidines through its alkali labile ring, to the azine dyes to which belong the natural dyestuffs, chlorophyll and pyocyanine, and finally to the benzene derivs. in which the  $\alpha$ -position of the 2 Me groups in natural products is a new phenomenon. The nonglucoside attachment of the pentose chain differentiates the vitamin from the nucleosides and may account for its stability to heat and dil. mineral acids. It is suggested that the flavin may be produced in plants by the reduction of a glucoside intermediate, a true *d*-ribose. The diamine from 50 mg. of VI was added dropwise to a soln. of 100 mg. of methylalloxan,  $\text{NaHSO}_4$ , and 200 mg. of  $\text{H}_2\text{BO}_3$  in 10 cc.  $\text{AcOH}$  and the mixt. was shaken 1 hr. at 20-5°. Working up by the usual procedure gave 25 mg. (38%) of 3,6,7-trimethyl-9-*d*-riboflavin, m. 272° (decomp.). The specificity of the lactoflavins was further demonstrated by the lack of activity of this 3-methyl lactoflavin. The free NH group

in the 3 position, together with the stereometric arrangement of the pentose chain, is of significance for the vitamin activity of the flavin. I was also prepd. in 2 other ways. V was transformed by treatment with  $\text{NH}_4\text{OH}$  to  $\text{NH}_4^+$ , 1,2-dimethyl-4-nitro-5-aminobenzoate (VII) (Ber. 35, 628 (1902)) and converted by the addn of 20%  $\text{COCl}_2$  in toluene to 2-nitro-4,5-dimethylphenyl isocyanate,  $\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2$ , m. 87.5°, which, by heating with alc., gave an almost theoretical yield of 1,2-dimethyl-4-nitro-5-(carbethoxylamino)benzene,  $\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_4$ , m. 55°. This was catalytically hydrogenated to the aminourethan which was condensed with III in the presence of Pd and  $\text{H}_2$  (C. A. 22, 4502) to 1,2-dimethyl-4-(carbethoxylamino)-5-(d-1'-ribitylamino)benzene, m. 169°, and hydrolyzed to I by NaOH and  $\text{Ba}(\text{OH})_2$  (C. A. 29, 21007). The simplest synthesis of I was carried out by heating 0.5 g. of l-arabinose and 2.2 g. of VII with 30 cc. abs. alc. for 6 hrs. at 110–20°. The cooled orange-red soln. was absorbed through a pillar of  $\text{Al}_2\text{O}_3$ . The excess VII was removed with abs. alc. and then the broad orange zone was eluted with a mixt. of 4 parts of MeOH and 1 of  $\text{H}_2\text{O}$ . The recovered condensation product, m. 166°, was hydrolyzed and on condensation with II and  $\text{H}_2\text{BO}_3$  gave lactoflavin. This simple 2-step process is of general application and will lend itself to the conversion of esters of reduced sugars into flavin esters since there is no step involving alk. hydrolysis.

C. R. Addinall

Heterocyclic compounds containing arsenic in the ring. Preliminary note. Hirendra Nath Das-Gupta, *J. Indian Chem. Soc.* 12, 627–8 (1935), cf. C. A. 27, 408—Mannich (C. A. 29, 4764) has prep'd a comp'd designated as 2-(diethylaminomethyl)-1,3-dichloroarsindole. In an attempt to prep. an As analog of indole, D-G. prep'd 1-chloroarsindole (I) by direct synthesis in an investigation undertaken in November, 1933. The formula assigned to the product resulting from the action of  $\text{ClCH}_2\text{CHAsCl}_2$  on  $\text{C}_6\text{H}_5$  in the presence of  $\text{AlCl}_3$  has been proved by a synthesis which is outlined. Compds. of the type  $(\text{PhCH}:\text{CH})_2\text{AsCl}$ ,  $\text{PhCH}:\text{CHAsCl}_2$  and  $(\text{PhCH}:\text{CH})_2\text{As}$  have been prep'd. as intermediates for the prep'n of I.

C. R. Addinall

γ-Triazines. XXIV. Melamine. Adriano Ostrogovich, *Gazz. chim. ital.* 65, 866–88 (1935); cf. O. and Medan, C. A. 29, 2167°.—Though various hydrocarbon derivs. of melamine (I) have been described, no acyl derivs. have been reported, and, moreover, part of the earlier literature is erroneous, as shown by the present exps. Pure I, m. 347° (354° corr.) (decompn.) (cf. *J. prakt. Chem.* [2], 3, 332 (1876)). The following new salts were prep'd. *Picrate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7$ , when first ppt'd. it is gelatinous, and must be heated to become cryst., light yellow, m. 316–7°. *Trisulfate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_6\text{S}_3$  (II), from I and glacial  $\text{HCO}_2\text{H}$  (III), does not fuse, but loses all its  $\text{HCO}_2\text{H}$  when heated; over soda lime or in conc'd aq. soln. it forms the monoformate (cf. Ann. 10, 22 (1834)). *Triacetate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_9$  (IV), from I and glacial  $\text{AcOH}$ , does not fuse but volatilizes completely, over soda lime, or on long standing it forms the monoacetate (cf. Ann. 10, 23 (1834)). *Tetrachloroarsinate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7\text{AsCl}_4$ , from I-HCl and conc'd aq.  $\text{HAsCl}_4$ , yellow or orange-ochre, m. 285–6 (uncor.) (to a red liquid). *Bis(tetrachloroarsinate)*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7\text{As}_2\text{Cl}_8$ , 4H<sub>2</sub>O, from I-HCl and a great excess of conc'd  $\text{HAsCl}_4$ , yellow, m. 290–1° (uncor.) (decompn.). Under different conditions there is obtained another *bis(tetrachloroarsinate)*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7\text{As}_2\text{Cl}_8$ , 2H<sub>2</sub>O, yellow, m. 271–2° (uncor.) (decompn.). *Semioxalate*,  $(\text{C}_4\text{H}_5\text{N}_3\text{O}_7)(\text{CO}_2\text{H})$ , already known (cf. Ann. 10, 23 (1834)). *Monoxalate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7(\text{CO}_2\text{H})$  (V), from cold aq. I and aq.  $(\text{CO}_2\text{H})_2$ . If instead of filtering V from the conc'd.  $(\text{CO}_2\text{H})_2$  soln. in which it is ppt'd., it is dissolved by heating and then cooled, there rapidly ppt's a *dioxalate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7(\text{CO}_2\text{H})_2$  (VI), needles, whereas when cooled slowly there ppt's a *dioxalate* (VII) in lustrous platelets. IV and V form II when recrystd. from water. In boiling water VI is transformed into VII. *Acyl derivs.* I and  $\text{HCONH}_2$  (8 parts), heated up to 160–5°, evolve  $\text{NH}_3$ , and the product purified with 20%  $\text{AcOH}$ ,

1 EtOH and water, yields 80% of *monoformylmelamine*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7\text{CHO}$  (VIII), does not fuse, insol. in hot glacial  $\text{AcOH}$ , sol. in glacial  $\text{HCO}_2\text{H}$  with formation of II. It also hydrolyzes in mineral acids, with formation of the corresponding I salts; alkalis also hydrolyze it. I (1 g.) and  $\text{HCO}_2\text{Et}$  (5–6 cc.), heated in a sealed tube for 8 hrs. at 230–5°, yield 10–15% of VIII. I and glacial  $\text{HCO}_2\text{H}$  form not even a trace of VIII but only II, which conforms with the fact that VIII and IV form II directly. I (1 g.) and  $\text{Ac}_2\text{O}$  (35 cc.), boiled 2 min., yields 1.60 g. of *diacetylmelamine*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7(\text{OAc})_2$  (IX), m. 298–9° (decompn.) (with a short thermometer the m. p. was 305–6°), sol. in aq. alk. hydroxides (repp'd. unchanged by  $\text{AcOH}$ ). IX and aq. picric acid (X) and a little  $(\text{CO}_2\text{H})_2$  (which facilitates the reaction) form the *picrate*  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7\text{AsCl}_4\text{O}_7$ , dark yellow, m. 209–10° (uncor.) (decompn.). IX in aq. HCl forms with  $\text{HAsCl}_4$  the *tetrachloroarsinate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7(\text{OAc})_2\text{AsCl}_4$ , light yellow, decomposes without fusion. IX in aq. NaOH, a little  $\text{NH}_3$  and excess  $\text{AgNO}_3$ , gives a gelatinous ppt of the *di-acyl deriv.*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7(\text{OAc})_2$ , grayish, turns brown when exposed to light. I (1 g.) and  $\text{MeCOSiH}$  (15–20 cc.), refluxed 2 hrs. ( $\text{H}_2\text{S}$  is evolved), yields IX, and when only 5 cc. of  $\text{MeCOSiH}$  in  $\text{AcOH}$  is used, no mono  $\text{Ac}$  deriv. (XI) is formed but only a little IX and a trace of IV. Even with the calc'd. proportion of  $\text{Ac}_2\text{O}$ , no XI was obtained, but only IV. 4 With an excess of  $\text{Ac}_2\text{O}$ , the chief product is IV, with a little IX, but no XI. Though the reaction between  $\text{HCO}_2\text{NH}_2$  and I goes easily,  $\text{AcNH}_2$  and I do not react on prolonged heating, even with excess  $\text{AcNH}_2$  (I 0.5 g.) and  $\text{Ac}_2\text{O}$  (100 cc.), refluxed until all IX is formed (loc. cit.), then with more  $\text{Ac}_2\text{O}$  (100–120 cc.), yield *triacetylmelamine*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7(\text{OAc})_3$  (XII), m. 289–91° (long thermometer and 298–300° (short thermometer) (decompn.)), sol. in cold dil. aq. alkalis (with slow hydrolysis). The 3rd  $\text{Ac}$  group is less stable than the other  $\text{Ac}$  groups, and the *picrate*, *tetrachloroarsinate* and *tri-acyl salt* could not be prep'd. by the Baumann-Schotten reaction, I gives no trace of any  $\text{Bz}$  deriv., even in  $\text{C}_6\text{H}_5\text{N}$ , nor was any  $\text{Bz}$  deriv. obtained by heating with  $\text{BzCl}$  or fusion with  $\text{BzNH}_2$ . On the other hand, I (2 g.) and  $\text{Bz}_2\text{O}$  (10 g.), heated at 130–5° for 4 hrs., the difficultly removable  $\text{BzOH}$  eliminated with  $\text{Et}_2\text{O}$ -petr. ether or petr. ether alone, yield almost 100% of *tribenzoylmelamine* (XIII), m. 142–3°, rhombic prisms from  $\text{AcOEt}$  or  $\text{Me}_2\text{CO}$  under certain conditions, needles of the same m. p. under other conditions, crystallizes from  $\text{PrOH}$  as the comp'd.  $\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_7$ , 2PrOH (XIV), which gives up its PrOH slowly at room temp. and completely in *vacuo* at 85–90°. XIII is repp'd. from its cold alk. hydroxide solns. by  $\text{CO}_2$ . In boiling 95% EtOH contg.  $\text{H}_2\text{SO}_4$ , XIII forms  $\text{BzOEt}$ ,  $\text{BzOH}$  and I semisulfate. Hot alc.  $\text{NH}_3$  also decomposes XIII to I. XIII and PhOH, heated at 130° and extd. with Et<sub>2</sub>O-petr. ether, leaves the comp'd.  $\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_7$ , 3PhOH (XV), m. 192.5–3.5°; in  $\text{AcOEt}$  it decomposes to XIII. By the usual methods of prep. *picrates*, XIII does not react, showing that the 3  $\text{Bz}$  groups destroy its basicity, but in boiling PhMe with excess X (8–9 mols.) it forms the *picrate*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7\text{AsCl}_4\text{O}_7$ , dark yellow, m. 222–3° (long thermometer), and 223–9° (short thermometer). No higher *picrate* was obtained at 180°. A soln. of XIII in a min. of cold aq. dil. NaOH or KOH contg. an excess of  $\text{NH}_4\text{OH}$  poured into aq.  $\text{AgNO}_3$  or  $\text{NH}_4\text{AgNO}_3$  ppt's the *di-acyl deriv.*,  $\text{C}_4\text{H}_5\text{N}_3\text{O}_7(\text{OAc})_2$ , 2H<sub>2</sub>O. Even with a great excess of  $\text{AgNO}_3$ , it is formed exclusively. Mulder (Ber. 7, 1632 (1874)) prep'd. from  $(\text{CO}_2\text{Et})_2$  and  $\text{NCNH}_2$ , a comp'd. which he thought was VIII, but his evidence was dubious and description meager, and a repetition of his exps. by O. gave a yellowish comp'd. (XVI) which gave no I salts on hydrolysis. With hot conc'd.  $\text{H}_2\text{SO}_4$  it gave a little CO. It was partially sol. in hot dil. NaOH, from which an unidentified gelatinous comp'd. was ppt'd. by  $\text{HNO}_3$ . Unlike VIII, a soln. of XVI in hot III ppt's a gelatinous comp'd. on cooling, and with X a *picrate*, different from that of VIII, is formed. The product of Mulder is probably a mixt. of ammeline and ammellides, perhaps with small proportions of other compds. Gerlich (cf. *J. prakt. Chem.* [2], 13, 280 (1876)) prep'd. from  $\text{NCN}$ .

$\text{Na}_2$  and  $\text{BzCl}$  a compd which he thought was XIII, but the present work shows that it was not XIII, as is also indicated by the work of Buddés (J. prakt. Chem., 42, 99 (1890)). The m. p. of XIII prep. by O is the same as that of  $\text{NCNH}_2\text{Bz}$  (XVII), not  $126^\circ$  as reported by Buddés (loc. cit.), but a mixt of XIII and XVII m. several degrees lower, and XIII fuses without decompn whereas XVII decomposes at its m. p. From  $\text{PrOH}$  and from  $\text{PhOH}$  XIII crystallizes as XIV and XV, resp., whereas XVII crystallizes without  $\text{PrOH}$  or  $\text{PhOH}$ . XVII can be heated with alc.  $\text{NH}_3$  in a sealed tube at  $100^\circ$  with only partial decompn, approx  $1/3$  of the XVII being recovered as the  $\text{NH}_3$  salt, whereas XIII decomposes completely into I and  $\text{BzNH}_2$ . XIII also decomposes in aq. alc.  $\text{NH}_3$  at  $50^\circ$ . Other minor differences between XIII and XVII are noted. The various new salts of I show that it can function either as a bivalent or as a trivalent base. This is further supported by the series of  $\text{HCl}$  salts of Barnett (C. A. 24, 4508). However the tetracyclic formula of B is unnecessary, for the stepwise addn. of  $\text{HCl}$  can as readily be explained by the 2 old tautomeric formulas (triaminotriazine and triaminotriazine). It is assumed that free I has the iso structure, or more probably an

intermediate mono-amino form  $\text{HN} \cdot \text{C}(\cdot\text{NH}_2) \cdot \text{N} \cdot \text{C}(\text{NH}_2) \cdot$

$\text{NH} \cdot \text{C} \cdot \text{NH}_2$ , and that when the  $\text{NH}_2$  is shifted, a 2nd group is transformed into an  $\text{NH}_2$  group, and similarly a further transformation takes place. The tetranuclear formula of B. fails also to explain the 2 isomeric series of alkyl melamines and their hydrolysis with acids (cf. Ber. 6, 1372(1873)), and the soly. of the new acyl melamines in alkalies and the formation of Ag salts. C. C. Davis

The optical activity of lactoflavin. P. Karrer and H. Fritzsche. *Helv. Chim. Acta* 18, 1026-7(1935); cf. C. A. 29, 3679<sup>1</sup>, 4363<sup>1</sup>.

Methylalloxazine. P. Karrer and Carlo Musante. *Helv. Chim. Acta* 18, 1134-40(1935); cf. C. A. 29, 1089<sup>1</sup>.—The effect of Me groups on the fluorescence of lumichrome was studied by prep. tri- and tetra-methylalloxazines (cf. Kuhnig, Ber. 28, 1968(1895)). From 2,4,5-trimethyl-6-nitroaniline, m.  $40^\circ$  (cf. Elder, Ber. 18, 620(1891)) the diamine was formed by catalytic reduction in alc. and was condensed with alloxan (II) to give 5,7,8-trimethylalloxazine,  $\text{C}_{12}\text{H}_{12}\text{N}_6\text{O}_2$  (possibly 5,6,8-trimethylalloxazine), decomp. at  $290^\circ$  and giving yellow fluorescence. From 3,4,5-Me<sub>3</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>2</sub> (cf. Noeltling and Forst, Ber. 18, 620(1891)) the diamine was likewise obtained and condensed with I to give 6,7,8-trimethylalloxazine,  $\text{C}_{12}\text{H}_{12}\text{N}_6\text{O}_2$ , with greenish yellow fluorescence. From 1,2,3,4-tetramethyl-5,6-dinitrobenzene,  $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_4$ , m.  $171^\circ$  (cf. Jacobsen, Ber. 19, 1214(1892)) the diamine was similarly prep. and condensed with I to give 5,6,7,8-tetramethylalloxazine,  $\text{C}_{12}\text{H}_{12}\text{N}_6\text{O}_2$ , with purplish yellow fluorescence. From 2-amino-3-nitrotoluene (II) (cf. Cohen and Dakin, J. Chem. Soc. 79, 1127(1901)) 2-chloro-3-nitrotoluene was prep. and condensed with ethanolaniline to give 2-hydroxyethylamino-3-nitrotoluene which upon reduction to the amine was condensed with I, giving 8-methyl-9-hydroxyethylalloxazine,  $\text{C}_{12}\text{H}_{12}\text{N}_6\text{O}_2$ , (III), m.  $294^\circ$ . Photodecompn. of III in 75% MeOH readily gave 8-methylalloxazine,  $\text{C}_{12}\text{H}_{12}\text{N}_6\text{O}_2$ , (IV), with green blue fluorescence. By reduction of II to 2,3-diaminotoluene and condensation with I either 8- or 5-methylalloxazine or a mixt of the 2 with the same fluorescence as IV was prep. W. F. Bruce

Synthetic flavins. VII. P. Karrer, H. Salomon, K. Schöpf and F. Benz. *Helv. Chim. Acta* 18, 1143-6 (1935); cf. C. A. 29, 5847<sup>1</sup>.—Because both lactoflavin and yeast nucleic acid contain d-ribose, and because thymus-nucleic acid contains d-2-ribose (I), the flavin contg. I was prep. to be tested for its triamin  $\text{B}_2$  action. I was condensed with 1-amino-2-carbethoxyamino-4,5-dimethylbenzene with  $\text{N}_2$  and  $\text{H}_2$  at 25 atm to give 4,5-dimethyl-2-carbethoxyaminophenyl-d-desoxyriboside,  $\text{C}_{18}\text{H}_{22}\text{N}_6\text{O}_5$  (II), m.  $115^\circ$ ,  $[\alpha]_D^{25} -21$  ( $\text{H}_2\text{O}$ ), in quant. yield. Hydrolysis of II by KOH and condensation with alloxan (III) gave 6,7-dimethyl-9-(d-1'-desoxy-

ribityl)isalloxazine,  $\text{C}_{20}\text{H}_{24}\text{N}_6\text{O}_4$ , m.  $283^\circ$ ,  $[\alpha]_D^{25} -78^\circ$ . From d-ribose and 1-amino-2-carbethoxyaminobenzene was prep. 2-carbethoxyaminophenyl-d-araboside,  $\text{C}_{18}\text{H}_{22}\text{N}_6\text{O}_5$ , m.  $158^\circ$ . By hydrolysis, condensation with III and acetylation 9-(d-1'-ribityl)isalloxazine tetraacetate,  $\text{C}_{28}\text{H}_{32}\text{N}_6\text{O}_{10}$ , m.  $237^\circ$ , was obtained. Upon hydrolysis this gave 9-(d-1'-ribityl)isalloxazine,  $\text{C}_{20}\text{H}_{24}\text{N}_6\text{O}_4$ , m.  $283^\circ$ . Condensation of d-ribose and 1-amino-2-carbethoxyamino-5-methylbenzene gave 5-methyl-2-carbethoxyaminophenyl-d-riboside,  $\text{C}_{18}\text{H}_{22}\text{N}_6\text{O}_5$ , m.  $150^\circ$ . Upon hydrolysis and condensation with III this gave 7-methyl-9-(d-1'-ribityl)isalloxazine,  $\text{C}_{20}\text{H}_{24}\text{N}_6\text{O}_4$ , m.  $285-6^\circ$ , tetraacetate,  $\text{C}_{28}\text{H}_{32}\text{N}_6\text{O}_{10}$ , m.  $215^\circ$ . W. F. Bruce

A modified flavin synthesis. P. Karrer and Hans F. Meerwein. *Helv. Chim. Acta* 18, 1130-4(1935); cf. C. A. 29, 5347<sup>1</sup>, 6237<sup>1</sup>.—A new synthesis of flavins is accomplished by coupling the condensation product of a sugar and 3,4-dimethylamine (I) obtained under reducing conditions, with a diazonium salt to form an azo dye in quant. yields. On reduction the dye yields a 3,4-dimethyl-6-aminophenyl sugar deriv. which can be transformed to a flavin. This synthesis is particularly suited to the prep. of flavins which contain readily hydrolyzable groups. From I and l-araboside by this procedure 3,4-dimethylphenyl-l-araboside,  $\text{C}_{18}\text{H}_{22}\text{N}_6\text{O}_5$  (II), m.  $123^\circ$ , was obtained (yield 60-70%). From diazotized p-nitroaniline and II an azo dye mixt. resulted which contained (3,4-dimethyl-6-p-nitrophenylazo)phenyl-l-araboside,  $\text{C}_{17}\text{H}_{17}\text{N}_5\text{O}_5$ . Reduction by Zn and HIOAc gave 6,7-dimethyl-9-(l-1'-arabityl)isalloxazine, m.  $299^\circ$ . By a similar synthesis with ribose the following compds were obtained in nearly quant. yield: 3,4-dimethylphenyl-d-riboside, m.  $143^\circ$  (yield 80-90%),  $\text{C}_{18}\text{H}_{22}\text{N}_6\text{O}_5$ , and 6,7-dimethyl-9-(d-1'-ribityl)isalloxazine (lactoflavin), m.  $280^\circ$ . W. F. Bruce

$\gamma$ -Triazines. XXIX. The passage from some alkyl, aryl and aralkylaminothiotriazines to derivatives of the corresponding aminohydroxytriazines. General considerations and a description of two new alkyl homologs ethylaminohydroxytriazine and propylaminohydroxytriazine. Adnarno Ostrogovich and Virgil Galea. *Gazz. chim. ital.* 65, 349-56(1935); cf. C. A. 29, 5449<sup>1</sup>.—Method of prepn.—Add to the alk. aminothiotriazine (I g in 25 cc of 10% NaOH or KOH) water (5 cc) and perhydrol (5 cc) dropwise (in some cases the solns become intensely yellow and then colorless), let stand overnight, acidify with dil.  $\text{AcOH}$ , let stand, and wash the ppt with cold water. Ethylaminohydroxytriazine,  $\text{C}_6\text{H}_8\text{ON}_3$ , 311<sup>0</sup> (II), m.  $277-8^\circ$  (all m. ps uncor.) (decompn.), loses water of crystn at room temp with formation of  $\text{C}_6\text{H}_7\text{ON}_3$ , 2H<sub>2</sub>O, sol in mineral acids. I and concd  $\text{HCl}$  yield on evapn the  $\text{HCl}$  salt,  $\text{C}_6\text{H}_8\text{ON}_3 \cdot \text{HCl}$ , m.  $214-16^\circ$  (decompn.), hydrolyzes immediately in water. Picrate,  $\text{C}_{12}\text{H}_8\text{ON}_3 \cdot \text{C}_6\text{H}_3\text{O}_7\text{N}_3$ , H<sub>2</sub>O, of I, dark yellow, loses its H<sub>2</sub>O of crystn *in vacuo* at  $100^\circ$ , with formation of the anhyd salt, which is paler yellow, and m. around  $191-2^\circ$  (decompn.). In hot dil.  $\text{NH}_4\text{OH}$ , I ppts with excess  $\text{AgNO}_3$  the Ag salt,  $\text{C}_6\text{H}_7\text{ON}_3 \cdot \text{Ag}$ , H<sub>2</sub>O, turns brown around  $240^\circ$ , chars at  $265-70^\circ$ , sensitive to light, sol. in  $\text{HNO}_3$ . The H<sub>2</sub>O of crystn is eliminated *in vacuo* at  $135-40^\circ$ . Propylaminohydroxytriazine (II), m.  $274-5^\circ$ , sol in mineral acids, with formation of the resp salts, which hydrolyze easily, sol in alk. hydroxides and  $\text{NH}_4\text{OH}$  (reppd by  $\text{CO}_2$ ). Picrate,  $\text{C}_{12}\text{H}_8\text{ON}_3 \cdot \text{C}_6\text{H}_3\text{O}_7\text{N}_3$ , H<sub>2</sub>O, of II, golden yellow, loses its H<sub>2</sub>O of crystn, *in vacuo* at  $100^\circ$ , with formation of the much lighter yellow anhyd salt, m.  $195-6^\circ$  (decompn.). Like other aminohydroxytriazines, I and II are amphoteric, and all members of

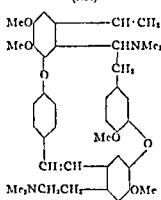
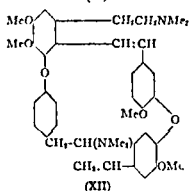
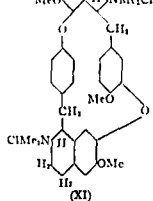
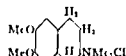
the group can be represented by the formulas:  $\text{N} \cdot \text{CR} \cdot \text{N} \cdot \text{C}(\text{OH}) \cdot \text{N} \cdot \text{CNH}_2$  (amino-enolic form) and  $\text{N} \cdot \text{CR} \cdot$

$\text{NH} \cdot \text{CO} \cdot \text{NH} \cdot \text{C} \cdot \text{NH}$  (imino-carbonyl form), or by one of the 2 intermediate aminocarbonyl or imino-enolic forms. The imino-carbonyl form is the most probable in all cases. XXX. Some arylaminohydroxytriazines. Ibid. 357-65. Three new arylaminohydroxytriazines were prep. in

almost 100% yields by oxidation of the corresponding arylaminobenzotriazines. Since phenylaminobenzotriazine and phenylaminohydroxytriazine (I) are easily prepd. in high yield (cf. *C. A.* 25, 1531), I was studied further and various salts were prepd. Hot concd. HCl and I form the *mono-HCl salt*,  $C_{10}H_{10}ON_4 \cdot HCl \cdot 2H_2O$ , of I, m. 295-6° (decompn.), hydrolyzes in water, is stable in hot EtOH, loses HCl slowly on standing in air, loses its  $H_2O$  of crystn. *in vacuo* at 105-10°. Hot 2 *N*  $H_2SO_4$  and I form the *monosulfate*,  $(C_{10}H_{10}ON_4)_2 H_2SO_4 \cdot 4H_2O$ , of I, m. 238-40° (decompn.), loses its  $H_2O$  of crystn. *in vacuo* at 100°, hydrolyzes in water and in EtOH. The picrate of I, already prepd. (cf. *O. C. A.* 24, 5752), m. 295-7° (decompn.), or 300-7° (cor.), has the compn.  $C_{10}H_{10}ON_4 \cdot C_{10}H_6O_4N_4 \cdot 2H_2O$ , loses its  $H_2O$  of crystn. *in vacuo* at 170°, sol. in cold aq. alk. hydroxides and  $NH_4OH$  (reptd by hot dil. AcOH or by satg. with  $CO_2$ ). The alkali salts absorb atm.  $CO_2$  and they were not analyzed. With hot aq.  $NH_4AgNO_3$ , I ppt. the *Ag salt*,  $C_{10}H_{10}ON_4Ag \cdot H_2O$ , does not fuse, but carbonizes at an elevated temp. (residue of Ag), more stable to light in a wet than in a dry state. Prepd. like I, almost a 100% yield of *o*-tolylaminohydroxytriazine (II) was obtained, m. 212-3° to a viscous orange-brown liquid, sol. in hot glacial AcOH with formation of its *acetate*, which on standing or in contact with water loses AcOH, sol. in mineral acids with formation of the corresponding *hydrohalide salts*, which are easily hydrolyzable. With aq. alkali salts, it forms the corresponding *alkali salts*, which absorb atm.  $CO_2$ . *Ag salt* *Picrate*,  $C_{10}H_{10}ON_4 \cdot C_{10}H_6O_4N_4 \cdot H_2O$ , S-yellow, m. 275-6° (decompn.), loses its  $H_2O$  of crystn. *in vacuo* at 140°. Prepd. like I and II, the yield of *m*-tolylaminohydroxytriazine is almost 100%, m. 217-10° (decompn.), in boiling glacial AcOH it forms an easily hydrolyzable *acetate*. *Alkali salts*. *Ag salt*. With aq. mineral acids it forms the corresponding *hydrohalide salts* *Picrate*,  $C_{10}H_{10}ON_4 \cdot C_{10}H_6O_4N_4$ , lemon-yellow, m. 297-8° (decompn.). The yield of *p*-tolylaminohydroxytriazine,  $C_{10}H_{10}ON_4 \cdot H_2O$  (III), is almost 100%, m. 333-4° (decompn.); in boiling glacial AcOH it forms the *acetate*. In aq. alk. hydroxides III forms the corresponding *alkali salts*, which are decomposed by  $CO_2$  and it is sol. in hot  $NH_4OH$ . In aq. HCl (2:1) III forms the *HCl salt*,  $C_{10}H_{10}ON_4 \cdot HCl \cdot H_2O$ , m. 291-6°, hydrolyzes in water and in EtOH, loses its  $H_2O$  of crystn. *in vacuo* at 140-6°. *Picrate*,  $C_{10}H_{10}ON_4 \cdot C_{10}H_6O_4N_4$ , light greenish yellow, m. 301-5°. *Ag salt*,  $C_{10}H_{10}ON_4Ag \cdot H_2O$ , sol. in  $NH_4OH$  and more so in  $HNO_3$ , its  $H_2O$  of crystn. cannot be eliminated without further decompn. *Amidylaminohydroxytriazine*,  $C_{10}H_{10}ON_4$  (IV), m. 327-8°, is amphoteric, and forms salts with bases and with acids. It crystallizes in 2 monoclinic forms, an acicular prismatic form and a tabular prismatic form. *HCl salt*,  $C_{10}H_{10}ON_4 \cdot HCl$ , m. 290-1°, hydrolyzed by water and by concd. EtOH. *Picrate*,  $C_{10}H_{10}ON_4 \cdot C_{10}H_6O_4N_4$ , dark yellow, m. 304-5°. *Alkali salts* of IV absorb in water atm.  $CO_2$ . *Ag salt*,  $C_{10}H_{10}ON_4 \cdot H_2O$ , very sensitive to light, decomposes before its  $H_2O$  of crystn. is eliminated. XXI. Benzylaminohydroxytriazine and styrylaminohydroxytriazine. *Ibid.* 367-70.—By the same method used for the previous amidinohydroxytriazines, benzylaminobenzotriazine (I) yields benzylaminohydroxytriazine (II), *reptd.* otherwise as phenylacetoguanidine by Flaznowski (*Thesis*, Freiburg, Switzerland, 1898), m. 277-8° (decompn.) (cf. "above 300°" of Flaznowski). Evapd. with concd. HCl, II gives the *HCl salt*,  $C_{10}H_{10}ON_4 \cdot HCl$ , hydrolyzes rapidly in water. *Picrate*,  $C_{10}H_{10}ON_4 \cdot C_{10}H_6O_4N_4 \cdot H_2O$ , yellow, m. 208-10° (decompn.), loses its  $H_2O$  of crystn. *in vacuo* at 110°. In hot dil.  $NH_4OH$  with  $AgNO_3$ , II forms a *Ag salt*,  $C_{10}H_{10}ON_4Ag$ , slightly sensitive to light, especially when dry, m. 270-60° (decompn.), sol. in  $NH_4OH$  and  $HNO_3$ . Prepd. similarly from styrylaminobenzotriazine (III), styrylaminohydroxytriazine,  $(C_{10}H_{10}ON_4) \cdot CH_2CH=CH_2 \cdot H_2O$  (IV), m. 304-5° (decompn.), decolorizes rapidly Br-water and aq.  $KMnO_4$ , sol. in hot  $NH_4OH$  and in alk. hydroxides with formation of *alkali salts*, which are reprecip. by  $CO_2$  or by AcOH. *Picrate*,  $C_{10}H_{10}ON_4 \cdot C_{10}H_6O_4N_4 \cdot H_2O$ , of IV, S-yellow, m. 271-3° (decompn.), loses its  $H_2O$  of crystn. *in vacuo*

at 100-5°. The formation of IV and II from III and I, resp., would with the very high yields appear to be an excellent method for prepn., but the prepn. of large quantities of I and of III offers great difficulties because of the instability of phenylthiolacetic acid and especially of thiocinnamic acid. This prevented a more extensive study of the properties and behavior of II and IV. It should be preferable to treat guanyllurea acetate with phenacetyl chloride or cinnamyl chloride, then to pass to the aminohydroxytriazine by treatment with  $NH_3$  or other aq. base. C. C. Davis

Curare alkaloids I Tubocurarine. Harold King. *J. Chem. Soc.* 1935, 1381-9.—Details are given of the isolation of *d*-tubocurarine chloride (I) from tubocurarine (II), a South American arrow poison II (25 g.) in 625 cc. 1% warm aq. tartaric acid, made alk. with 250 cc. cold aq.  $NaHCO_3$  and extd. with EtO and  $CHCl_3$ , gives about 4 g. curine (III); the aq. soln. was made



(XIII)

acid with 2 *N*  $H_2SO_4$ , basic  $Pb(OAc)_2$  added, the  $Pb$  removed from the filtrate and ppt., and I pptd. by phos-



photungstic acid and  $\text{HgCl}_2$ . I,  $\text{C}_{11}\text{H}_{14}\text{O}_2\text{N}_2\text{Cl}_2 \cdot 5\text{H}_2\text{O}$ , m.  $274-5^\circ$  (decompn.),  $[\alpha]_D^{25}$ ,  $204.8^\circ$  ( $\text{H}_2\text{O}$ ,  $c$  0.97) (anhyd. salt).  $\text{FeCl}_3$  gives a weak green color; I reduces  $\text{Ni}(\text{OH})_2\text{Ag}_2\text{O}$  on warming and gives amorphous ppts with Au and Pt compds., KI, KBr,  $\text{KCNO}$ ,  $\text{HgCl}_2$ ,  $\text{NH}_4\text{NO}_3$ , and  $\text{HClO}_4$ . I with MeI and  $\text{MeOH-KOH}$  yields *O*-methylbucurarine iodide (IV), with 3 mols  $\text{H}_2\text{O}$ , m.  $267^\circ$  (decompn.),  $[\alpha]_{\text{MeI}}^{202.1^\circ}$  ( $\text{H}_2\text{O}$ ,  $c$  1.01, anhyd. salt). I and I-III MeCl are isomeric but not enantiomorphous. Hofmann degradation of the chloride from IV gives methine bases, characterized as their MeI derivs. *O*-Methylbucurarinemethine methiodide B (V), with 2.5 mols  $\text{H}_2\text{O}$ , m.  $230^\circ$ , the *A*-isomer (VI), with 2.5 mols  $\text{H}_2\text{O}$ , m.  $234^\circ$ , *d*-Methylbucurarinemethine-MeI (VII), with 5 mols  $\text{H}_2\text{O}$ , m.  $190^\circ$ ,  $[\alpha]_{\text{MeI}}^{105^\circ}$  ( $\text{MeOH}$ ,  $c$  0.22); the *I* isomer (VIII), with 4 mols  $\text{H}_2\text{O}$ , needles, m.  $171-2^\circ$  or plates, m.  $178-80^\circ$ ,  $[\alpha]_{\text{MeI}}^{56.9^\circ}$  ( $\text{MeOH}$ ). Degradation of the methylation product of *d*-beberine (identical with I III, except for rotation), gives V, VI and VII, VIII was not found. At the 2nd stage of the Hofmann degradation of the mixed *O*-methylbucurarinemethine-MeCl there results  $\text{Me}_2\text{N}$  and *O*-methylbeberine, m.  $198-9^\circ$ , identical with that obtained from *O*-methylbeberine-MeCl (IX). The chloride (X) from IV and IX are both represented by formula XI, with 2 asym. C atoms adjacent to the N atoms. The assumption is made that in IX each center of asymmetry is *d*-rotatory, whereas in X 1 center is *d*- and the other *l*-rotatory. Since the same *d*-methine-MeI, m.  $190^\circ$ , is formed from both alkaloids, VII can only have retained the common *d*-center of asymmetry and should have the structure of a dimethiodide based on XII or XIII; VIII is obtained only from X and is therefore probably represented by XIII or XII, in which only the *l*-center of asymmetry has survived. Although IX and X are diastereoisomers, it is probable that the parent phenolic substances, although isomeric, differ in the orientation of  $\text{H}$  and  $\text{MeO}$  groups. I produces complete "curare" paralysis of the frog in doses of 0.5 mg. per kg. The isomeric *d*-beberine-MeCl has about  $1/10$  of the activity.

C. J. West

The alkaloids of the Chinese drug, mu fang-chi T. Q. Chou *Chinese J. Physiol.* 9, 267-74 (1935).—Two alkaloids, menisidine and menisidine, have been isolated. Menisidine crystallizes with 1 mol of  $\text{H}_2\text{O}$  in fine needles, m.  $127^\circ$ . The anhyd. compd has the formula  $\text{C}_{19}\text{H}_{21}\text{NO}_4$  (?), m.  $152^\circ$ ,  $[\alpha]_D^{20}$ ,  $290^\circ$ . It is a tertiary base, contains 2 MeO groups and forms the following salts: hydrochloride, amorphous powder, m. about  $260^\circ$ ; acid phosphate, rhombic prisms, m.  $280^\circ$ ; methiodide, rhombic prisms, m.  $263^\circ$  (decompn.). Menisidine crystallizes from alc. in prismatic needles, m.  $176^\circ$ . It has the formula,  $\text{C}_{20}\text{H}_{23}\text{NO}_4$  (?),  $[\alpha]_D^{20}$ ,  $260^\circ$ , and contains 3 MeO groups. With acetone it forms a diacetone compd. It is also a tertiary base, and forms the following salts: hydrochloride, a cryst. powder, m. about  $265^\circ$  when anhyd., acid phosphate, minute rhombic prisms, m. about  $290^\circ$ , softening at about  $210^\circ$ ; dimethiodide, amorphous powder with no definite m. p.

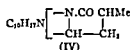
L. A. Maynard

Constitution of matrine XVII Hensaburo Kondo, Eiju Ochias and Kyosuke Tsuda. *Ber.* 68B, 1899-1904 (1935), cf. *C. A.* 29, 4015.—It had been established that 11 of the C atoms and the 2 N atoms of matrine (I) are present in the form of the skeleton II; it was presumed that the other 4 C atoms constitute a lactam ring which, by further condensation with the norpiperidine nucleus, forms a 3rd heterogeneous ring. When I is hydrolyzed with alc. KOH the lactam ring is ruptured, with formation of matricinic acid, which is methylated to Me methylmatrine. This ester is degraded by the Hofmann method to decarbonylmethylmatrinyl alc. and its anhydride, decarbonylmethylmatrine, P and HI reduce the anhydride to a secondary tertiary amine  $\text{C}_{11}\text{H}_{15}\text{N}$  (decarboxylated matricinic acid) which is dehydrogenated by heating with Pd asbestos to the same base,  $\text{C}_{11}\text{H}_{15}\text{N}$  (III), as had been obtained by catalytic dehydrogenation of I. The methoxyhydroxide of III gives  $\text{PrCO}_2\text{H}$  with  $\text{KMnO}_4$ , and hence contains a Pr side chain, as I MeOH gives no  $\text{PrCO}_2\text{H}$

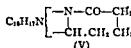
under the same conditions, the Pr side chain must be formed by degradation of the lactam ring. Of the 2 possible formulas (IV and V) for the lactam ring, only IV can account for the formation of the  $\text{C}_{11}$  base (VI) from K matrine with loss of 3 C atoms. Moreover, since the oxidation of the compd.  $\text{C}_{11}\text{H}_{15}\text{N}$ , obtained by BrCN degradation of dihydro- $\alpha$ -matrinidine (VII) and subsequent dehydrogenation gives only quinolinic and no  $\alpha$ -picolinic or pyridimetricarboxylic acid, the structure of VII is best represented by the formula VIII or IX. Nucleus C in matrinidine (X) would then be a  $\Delta^4$ -tetrahydro- $\alpha$ -picoline, which agrees with the chem. properties of the base. X is quite unstable in the air; on acetylation it simultaneously adds 1 mol.  $\text{H}_2\text{O}$  more and on dehydrogenation changes, with loss of 2 mols.  $\text{H}_2$ , to the more stable dehydro- $\alpha$ -matrinidine (XI). According to these formulas XI has an  $\alpha$ -methylpyridine nucleus which should condense with aldehydes, and as a matter of fact a benzal deriv. (XII) was obtained from XI. If there is no ring shifting in the formation of X, the structure of I may be represented by the formula XIII or XIV. Further studies will be made to decide, if possible, between these 2 structures and also to explain earlier observations which are apparently not in harmony with them. The base  $\text{C}_{11}\text{H}_{15}\text{N}$ , b,  $130^\circ$  (methiodide, m.  $213^\circ$ ); 1 g. with 1 g. of 42%



(II)



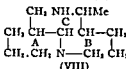
(IV)



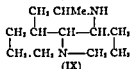
(V)



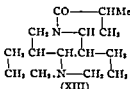
(VI)



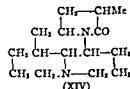
(VIII)



(IX)



(XIII)



(XIV)

Pd-asbestos at  $270-80^\circ$  gives 0.6 g. III, b,  $195-205^\circ$  (HCl salt, m.  $208-9^\circ$ ,  $[\alpha]_D^{20}$  0.81° (alc.); the m. p. of the methiodide, previously given as  $128-9^\circ$ , is raised to  $139-40^\circ$  by repeated crystn. from acetone). With Pt oxide in AcOH and H under 35 lbs., the base  $\text{C}_{11}\text{H}_{15}\text{N}$  yields the base  $\text{C}_{11}\text{H}_{15}\text{N}$ , m.  $189-90^\circ$ , XI, b,  $176^\circ$  (HCl salt, decompn.  $282^\circ$ ,  $[\alpha]_D^{20}$  0.57° (alc.); XII, m.  $109-74^\circ$ , is reduced by Pt oxide in dil. HCl and H to a base isolated as the chloroplatinate,  $(\text{C}_{11}\text{H}_{15}\text{N})_2(\text{HCl})_2\text{PtCl}_4$ , decompn.  $190^\circ$ . I and lupanine (XV) are not stereoisomers, as shown by the nonformation of octadehydromatrine in the dehydrogenation of XV with Pd-asbestos at  $320-30^\circ$  and by the fact that XV MeOH gives glutaric acid with  $\text{KMnO}_4$ , while I MeOH does not. The glutaric acid is certainly not formed from the norpiperidine but from the  $\alpha$ -piperidine nucleus, for it is not obtained in the analogous oxidation of sparteine.

C. A. R.

Cotarnine series. II. The reactivity of the aldehyde group in cotarnine and benzoylcotarnines. D. B. Drey and P. Lakshmi Kantam. *J. Indian Chem. Soc.* 12, 421-9 (1935), cf. *C. A.* 29, 2981.—To a suspension of 2 g. of cotarnine (I) in 20 cc. of  $\text{H}_2\text{O}$  were added 1 g. of  $\text{H}_2\text{NOH} \cdot \text{HCl}$  and 2 g. of AcONa and the mixt. was warmed to a clear soln. which was concd. and made alk. with  $\text{Na}_2\text{CO}_3$ . The almost quant. yield of crystals which sepd. was

recrystd. from alc. and gave cotarnine oxime (II), *m.* 157°; di-Ac deriv.,  $C_{12}H_{11}N_3O_4$ , *m.* 113°; di-Bz deriv. (III),  $C_{22}H_{19}N_3O_4$ , *m.* 158°. II reacted smoothly with 2 mols. of PhNCO to produce cotarnomethylphenylurea oxime phenylurethane, *m.* 161°, identical with the compd. obtained by the action of PhNCO on cotarnomethylphenylurea oxime. PhNCS reacted with II in  $C_6H_6$  to give cotarnomethylphenylthiourea oxime, *m.* 142°, previously prepd. by the oxidation of cotarnomethylphenylthiourea. As previously observed, PhNCS does not react with the  $NOH$  group. The  $\alpha$  reactions clearly indicate the simultaneous presence of the  $NOH$  group and of the rec. imino-II atom in II and rule out the carbinal form. Reduction of II with Na and abs. alc. gave hydrocotarnine with evolution of  $NH_3$ , because of the reduction of  $NOH$  to  $NH_2$  and elimination of  $NH_3$  consequent on the closure of the isoquinoline ring. I condenses readily with aromatic amines to form unstable salts which are decomposed quickly by alkalis but dissolve unchanged in cold acids. Attempts to prep. the Bz deriv. of cotarno-*p*-toluidid gave benzotoluidid, *m.* 158°. The trituration of 2 g. of I with 1 cc. of BzCl and 20 cc. of 10 N NaOH for 15 min. gave a quant. yield of benzoylcotarnine (IV), *m.* 121°, oxime, *m.* 165°, benzoylated to III, Ac deriv.,  $C_{12}H_{11}N_3O_4$ , *m.* 162°; azine,  $C_{12}H_{11}N_3O_4$ , *m.* 220°, anil., *m.* 161°, *o*-, *m*- and *p*-toluidids, *m.* 160°, 113° and 111°, resp. *p*-phenetidid, *m.* 135°. IV was oxidized by alk.  $KMnO_4$  to benzoylcotarnic acid, *m.* 160°, which formed an analyzable Ag salt. The condensation of I with *o*- $O_2NC_6H_4COCl$  gave excellent yields of *o*-nitrobenzoylcotarnine (V), *m.* 138°; oxime, *m.* 193-4°; anil., *m.* 164°. These benzoylated products are insol. in dil. acids, form cryst. mono-oximes sol. in cold alkali which, in turn, give mono-Ac and Bz derivs. insol. in alkali, are oxidized by alk.  $KMnO_4$  and condense easily with aromatic amines to form stable anils. The stability of these compds. as well as of the anils and toluidids derived from cotarnomethylphenylurea is in striking contrast with the instability of the corresponding derivs. of I and is attributed to the presence of the mobile imino II atom. The elimination of this active II atom seems to activate the otherwise bound  $CHO$  group. Products of condensation of I with compds. contg. an active  $CH_2$  group should be represented either as derivs. of 1- $\beta$ -N-methylaminoethyl-2-benzaldehyde or as substituted *N*-methyltetrahydroisoquinoline derivs. according as to whether I reacts in the aldehyde or carbinal form. Such condensations are usually carried out in the presence of alc., which has been shown to favor the shift from the aldehyde to the carbinal modification. Condensation of 1 g. of I with 0.6 g. of *p*- $H_2NC_6H_4Ac$  in 12 cc. of alc. at 40° for 15 mins. gave 1 g. of anhydrotarnine-*p*-aminoacetophenone (VI),  $C_{17}H_{17}N_3O_4$ , *m.* 100-1°; mono-Ac deriv., *m.* 105°, sol. in cold dil. acids, indicating that VI should be regarded as a tetrahydroisoquinoline deriv. formed by the reaction of I in the carbinal form. Condensation of I with  $(NH_4)_2CO_3$  oxalide, resorcinol mono-Ac ether and 2-nitroresorcinol gave the corresponding anhydrotarnineoxalamide, *m.* 182°, phthalimidine, *m.* 147-4°, resorcinol *Me ether*, *m.* 221-2°, and 2-nitroresorcinol, *m.* 201°, as a result of condensation in the carbinal form. Similar condensations with *o*-, *m*- and *p*-cresols and  $\alpha$ - and  $\beta$ -naphthols were tried without success. III. Isomeric biscotarninoacetones. *Ibid.* 430-5.—Reinvestigation of the condensation of cotarnine (I) with  $Me_2CO$  has made possible the isolation of both isomeric forms of the product. The condensation of I with  $Me_2CO$  contg.  $Na_2CO_3$  gave biscotarninyldiacetone, *m.* 83° (identical with the anhydrotarninoacetone of Liebermann and Kropf (*Ber.* 37, 211 (1904)));  $HCl$  salt, *m.* 171°; Bz deriv., *m.* 124°;  $MeI$  deriv. (II), *m.* 168° (L. and K. designated this deriv. as anhydrotarninecotarninoacetone methylate and found the *m.* 144°). Decompos. of II with boiling alkali gave  $NMe_2$  and biscotarninodienacetone. Condensation of II with  $Me_2CO$  in the presence of alc., with  $Na_2CO_3$  as condensing agent yields an isomeric product, biscotarninoacetone (III),  $C_{17}H_{17}N_3O_4$ , *m.* 150-1°;  $HCl$  salt, *m.* 167° (decompn.);  $MeI$  deriv., *m.* 210°, which is decomposed with alkali to *N*-methylbiscotarninyldiacetone

(*HCl* salt, *m.* 181-2° (decompn.));  $MeI$  deriv., *m.* 168°, identical with II. A cyclic structure is assigned to III and the view that the change from the aldehyde to the carbinal form is brought about by the effect of the solvent is supported by the quant. conversion of the unsatd. compds. *m.* 83°, into III by crystn. from hot alc. This transformation is also brought about in attempts at acetylation and the reaction is explained by the assumption of the formation of an intermediate Ac compd., its hydrolysis and the closure of the isoquinoline ring by migration of the imino-II atom. The proposed mechanism renders intelligible the condensation of I with compds. capable of ketonal tautomerism and others contg. reactive  $CH_2$  groups. In the presence of non-ionizing solvents the condensation takes place with the  $CHO$  group but in most cases a shift takes place which produces tetrahydroisoquinoline derivs. Only with  $Me_2CO$  is the unsatd. compd. first formed sufficiently stable to permit of its isolation and study. IV. 5-Bromocotarnine, 5-bromocotarnine, 5-bromohydrocotarnine and 5-bromocotarnine and their derivatives. B. B. Dey and T. K. Srinivasan. *Ibid.* 626-30.—A soln. of 5 g. of nitrocinne (I) in 20 cc. of  $H_2O$  contg. 2 cc. of 48%  $HBr$  was cooled and treated with acid  $Br$  soln. until the yellowish-white ppt. became permanent. The turbid soln. was acid with  $H_2SO_4$  and the filtered soln. was made basic with  $NH_4OH$ , producing 5.6 g. of 5-bromocotarnine (II),  $C_{12}H_{11}BrN_3O_4$ , *m.* 176°,  $[\alpha]_D^{25} -95^\circ$ ,  $HCl$  salt, *m.* 120°,  $HBr$  salt, *m.* 168°, picrate, *m.* 107°;  $MeI$  deriv. (III), *m.* 220°, chloroplatinate. Bromination of I in  $HBr$  (0.5 g.) gave 0.7 g. of 5-bromocotarnine perbromide- $HBr$  (IV),  $C_{12}H_{11}Br_3N_3O_4$ ,  $HBr$ , *m.* 220° (decompn.), which was reduced completely by  $H_2S$  to II. The oxidation of II with  $HNO_3$  gave 5-bromocotarnine (V),  $C_{12}H_{11}BrN_3O_4$ , *m.* 175°,  $HCl$  salt, *m.* 170° (decompn.), picrate, *m.* 170°, chloroplatinate. This prepn. is preferable to the direct bromination of cotarnine to 5-bromocotarnine perbromide- $HBr$ ,  $C_{12}H_{11}Br_3N_3O_4$ ,  $HBr$ , and subsequent reduction with  $H_2S$ . On heating under pressure with  $MeI$ , V gave 5-bromocotarnomethine methiodide (VI),  $C_{12}H_{11}BrN_3O_4$ , *m.* 179°. V forms a Bz deriv.,  $C_{12}H_{11}BrN_3O_4$ , *m.* 101°, and on treatment with  $HONH_2$ ,  $HCl$  and  $AcONa$  yielded the oxime- $HCl$ , *m.* 169°, converted by  $NH_4OH$  into the free oxime, *m.* 178°. Steam distn. of 2 g. of VI in 20 cc. of 10% NaOH produced 5-bromocotarnone, *m.* 101°, oxidized by  $KMnO_4$  in alk.  $Me_2CO$  to 5-bromocotarnolactone,  $C_{12}H_{11}BrO_4$ , *m.* 172°, further oxidized by alk.  $KMnO_4$  to 5-bromocotarnic acid,  $C_{12}H_{11}BrN_3O_4$ , *m.* 185°; anhydride, *m.* 225°. Treatment of V with PhNCO,  $H_2NOH$ ,  $ONMe$  and  $Ac_2O$  gave 5-bromocotarnomethylphenylurea,  $C_{12}H_{11}BrN_3O_4$ , *m.* 140°; oxime, *m.* 168°; anhydrotarnocotarninonitromethane,  $C_{12}H_{11}BrN_3O_4$ , *m.* 118° ( $HCl$  salt, *m.* 166°); *N*-acetyl-5-bromocotarnindiacetic acid,  $C_{12}H_{11}BrN_3O_4$ , *m.* 211°, resp. The reduction of V with 4%  $Na-Hg$  in  $N$   $HCl$  yielded 5-bromohydrocotarnine,  $C_{12}H_{11}BrN_3O_4$  (VII), *m.* 80°,  $HBr$  salt, *m.* 212°; perbromide- $HBr$ , *m.* 168° (decompn.), which could not be reduced to V but invariably gave unchanged VII on treatment with  $H_2S$ . VII was also obtained in good yields by brominating hydrocotarnine in acid soln. by Wright's method (*J. Chem. Soc.* 32, 626 (1877)). Steam distn. of a mixt. of 5 g. of II with 4 g. of *p*- $MeC_6H_4SO_3Me$  and NaOH (*C. A.* 20, 1795) produced 2.7 g. of soft colorless needles of 5-bromocotarnine,  $C_{12}H_{11}BrN_3O_4$ , *m.* 193°,  $HCl$  salt, *m.* 211°;  $HBr$  salt, *m.* 225°; picrate, *m.* 151°; chloroplatinate, *m.* 198° (decompn.);  $Me$  ester, *m.* 111°. The position of the  $Br$  atom in all these derivs. follows from the conversion of V to 2-methoxy-3,4-methylenedioxy-5-bromophthalic acid. The observation that neither V nor II condenses with  $HCHO$ , which is known to attack only the free  $\beta$ -position in these bases, supports the argument for the assigning of this position. V. Condensation of cotarnine with aromatic nitro aldehydes. B. B. Dey and P. Kakshini Kantam. *Ibid.* 601-7.—The reaction of cotarnine (I) with 2 mols. of aromatic nitroaldehydes (*C. A.* 18, 2161) has been found to be general. A soln. of 1 g. of I and 1.28 g. of *o*- $O_2NC_6H_4CHO$  in 12 cc. of alc. was warmed to 60° and exposed to direct sunlight for 7-8 hrs., yielding 0.8 g. of cryst. 5-*o*-nitrobenzaldehydyl-1-*o*-nitrobenzoylhydrocotarnine (II),

$C_{10}H_{11}N_7O_3$ , m. 153°; HCl salt, m. 172° (decompn.); HBr salt, m. 177° (decompn.); nitrate, m. 181° (decompn.); sulfate, m. 191-2° (decompn.); picrate, m. 175-6°. II was reduced by  $SnCl_4$  in HCl to the corresponding amino compd., m. 122° (di-Ac deriv., m. 126°), which, on diazotizing, coupled with  $\beta$ - $C_{10}H_7O_2H$  to a deep-red dyestuff. The m-nitro deriv. corresponding to II was similarly prepd., yielding a cryst. material, m. 146° (HCl salt, m. 188°), reduced to the m-amino compd., m. 113°. I condensed with nitrovanillin Me ether to give yellow crystals of 5-(6-nitro-3,4-dimethoxybenzaldehyde)-1-(6-nitro-7,8-dimethoxybenzoyl)hydrocotarnine,  $C_{20}H_{19}N_5O_8$ , m. 168°; HCl salt, m. 193° (decompn.). Condensation with 6-nitropiperonal similarly gave 5-(6-nitropiperonyl)-1-(6-nitropiperonyl)hydrocotarnine, m. 168°; HBr salt, m. 180° (decompn.). The observation has been made that if position 5 in the cotarnine mol. be blocked, the reaction occurs only with 1 mol. of the nitro aldehyde. Thus 5-bromocotarnine condensed with  $o$ - $C_6H_4CH_2CHO$  and 6 nitropiperonal to give 1-( $o$ -nitrobenzoyl)-3-bromohydrocotarnine,  $C_{15}H_{13}BrN_2O_4$ , m. 120° (HBr salt, m. 162° (decompn.)), and 1-(6-nitropiperonyl)-5-bromohydrocotarnine,  $C_{15}H_{13}BrN_2O_4$ , m. 125° (HBr salt, m. 172°). On attempting the condensation of  $o$ -nitro aldehydes with hydrocotarnine and narcotine in alc. media in exactly the same way, only unchanged materials were recovered.

C. R. Addinall

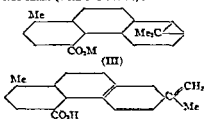
Phenobarbiturate of papaverine (pavamal). Antonio Mossini and Giovanni Recordati. *Boll. chim. farm.* 74, 638-9 (1935).—Alc. solns of 0.1 M phenobarbital and 0.1 M papaverine, heated on a water bath for several min. and cooled, give crystals composed of 1 mol. of each substance. This compd. (pavamal) m. 145-6° and is insol. in cold  $H_2O$  and ether; it is sol. in hot alc., benzene, petr. ether,  $AcOEt$  and in the cold in  $CHCl_3$ , acetone, and is slightly sol. in hot glycerol, vaseline oil and olive oil.

Helen Lee Gruel

Condensation of cotarnine and  $o$ -nitrobenzaldehyde. K. N. Kaul and G. S. Ahluwalia. *J. Indian Chem. Soc.* 12, 610 (1935); cf. C. A. 27, 4804.—K. and A. desired to correct their formulation of the condensation product and bring it in line with the later formula of Robinson and Robinson (C. A. 18, 2161).

C. R. Addinall

Resin acid in the oleoresin of *Pinus sylvestris*. I. P. S. Pishchumka. *J. Gen. Chem. (U. S. S. R.)* 5, 738-51 (1935). II. Product of nitration of resin acid from *Pinus sylvestris*. P. S. Pishchumka. *Ibid.* 752-63; cf. C. A. 29, 6038.—Solid or solvated resin acid (I) on heating is isomerized, changing gradually from a strongly l-rotatory to d-rotatory compd. After reaching a max. of  $[\alpha]_D^{25}$  62° the rotation begins to decrease and becomes stable at  $[\alpha]_D^{25}$  8-9°. The isomerizing action of atm.  $O_2$ , if any, is considered purely catalytic. The following reactions of I (and of abietic acid (II)) do not accord with the structural formula of II proposed by Ruzicka (C. A. 25, 3657), an easy oxidation by atm.  $O_2$  and resistance to the action of oxidizing agents; the cleavage of 1 C atom as  $HCO-H$  by oxidation of I with atm.  $O_2$  as  $CH_3$  on heating with Ni-pumice, and as  $MeSH$  on heating with S; the existence of the tricozonic and hexaozodic of II. The formula III explains satisfactorily all these reactions. An acid of this structure when polymerized (by the action of  $O_3$ , atm.  $O_2$  and probably some other agents) is capable of the conversion into the phenanthrene system with 1 unsatd. side chain (with 3 C atoms):



This formulation does not disagree with the formation of

retene and octahydroretene from II, because these reactions are accompanied by reducing processes which result in the conversion of the unsatd. side chain into the satd. one (iso-Pr). On the basis of this assumption the empirical formula of I (and II) is  $C_{19}H_{26}O_2$  and not  $C_{19}H_{24}O_2$ . The nitration of I, with and without the use of a solvent (ligroin), results in the addn. of the elements of  $HNO_3$  at 1 of the 2 bonds with the formation of hydroxynitrosous acid (hydroxynitrosobietic acid),  $C_{19}H_{25}N_2O_5$  ( $C_{19}H_{24}O_2 + NO_2 + NO_2 + O_2$ ), m. 125°, which in the presence of an excess of concd.  $HNO_3$  forms N ethers of the nitro acids. The nitro ether absorbs from air 1 mol. of  $O_2$  (probably at the other double bond, since the 3rd one is generally considered as a passive one), giving an amorphous peroxide compd. (I with  $HNO_3$  is probably first converted into II). These peroxides act as dibasic acids, giving stable,  $H_2O$ -sol. red salts of  $NH_4$ , alkalis and all-earth metals. Evidently the nitrate ether of the nitro acid is isomerized by the action of inorg. bases into a dibasic pseudonitro acid (the acid groups  $CO_2H$  and  $NO-H$ ). The nitro product adsorbs energetically some gases and vapors ( $HCl$ ,  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ , etc.), which can be explained by its amorphous state.

Chas. Blanc

The original acids in American pine resins. Kurt Kraft. *Ann.* 520, 133-43 (1935).—Sapinic acid obtained from the resin of *Pinus polastris* which was collected during the summer has  $[\alpha]_D^{25}$  -70°, while the common acid has  $[\alpha]_D^{25}$  -123°. Hydrogenation of the former yields 20% of dehydro-d-pimelic acid. Rearrangement of the sapinic acid by boiling with  $AcOH$  gives 80% of abietic acid, the remainder being d-pimelic acid. Ozonization expts. confirmed these results. The sapinic acid mixt. with  $[\alpha]_D^{25}$  -110° to -130° obtained from resin collected during the spring could be sep'd. by repeated crys. on a l-pimelic acid,  $[\alpha]_D^{25}$  -280.4°, and a d-acid,  $[\alpha]_D^{25}$  52.9°.

C. J. West

Constitution of mataresinol. L. H. Briggs, D. A. Peak and J. L. D. Woolloxall. *J. Proc. Roy. Soc. N. S. Wales* 69, 61-7 (reprint) (1935).—Mataresinol,  $C_{19}H_{26}O_2$  (I), occurs in the heart shakes of the wood of *Podocarpus spicatus* as a resinous or cryst. deposit. Recryst. from  $EtOH$  yields a product contg. alc. of crysln, m. 78°. Recryst. from 60%  $AcOH$  yields large, anhyd rhombic crystals of I, m. 119°. I in  $AcOH$  in the presence of Pd-norite did not absorb H. I dissolved in just enough 5%  $NaOH$  soln. and then treated with 1.3  $AcOH$  until faintly acid, yielded hexagonal plates of mataresinic acid,  $C_{19}H_{24}O_4$ , m. 78-81°. Dibenzoylmataresinol, prep'd. by shaking together 5 g. I, 7 mols.  $NaOH$  and 5 mols.  $BzCl$ , m. 134.5°, and was ppt'd. from alk. soln. with the lactone group intact. Dimethylmataresinol (II) was prep'd. by shaking together 1 g. I dissolved in 25 cc. 10%  $NaOH$  and 5.3 g.  $Me_2SO$ , and finally heating the mixt. for 5 mins. at 100°. Acidification of the cooled mixt. with  $HCl$  produced a sticky ppt., m. 126.5-7°, after 3 recrystns. from  $EtOH$ . The product was a lactone, neutral to litmus, and produced no color with  $FeCl_3$  or concd.  $H_2SO_4$ . Neutralization of a soln. of II in excess  $NaOH$  yielded a colorless, cryst. ppt. of dimethylmataresinic acid, m. 80-4° due to lactone formation. The ester could not be prep'd. The slow addn. of 120 cc. 3%  $KMnO_4$  to a soln. contg. 1 g. II in 20 cc. 1%  $NaOH$  yielded, after satn. of the mixt. with  $SO_2$ , 0.51 g. (over 50%) of clusters of homogeneous needles of veratric acid, m. 173.5-4.5°. No trace of veratroylformic acid was detected. Gradual addn. of 1.77 g. Br in 35 cc.  $CHCl_3$  to 2 g. of I in 20 cc.  $CHCl_3$  produced, after 36 hrs., a nearly theoretical yield of tetra bromomataresinol, m. 169-9.5° (from  $CHCl_3$ ) or 171° (from  $EtOH$ ). The foregoing chem. evidence supported by phytochem. considerations indicates that I may be formulated as  $RCH_2CH(CH_2R)CH_2CO_2CO$  ( $R =$

4,3-HO( $MeO$ ) $C_6H_3$ ). The C skeleton of I is the same as that of guaretic acid, olivil, podophyllotoxin, and probably pinoresinol.

W. A. LaLande, Jr.

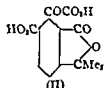
Synthesis of tetrahydroharman (4-carbolone) derivatives under physiological conditions. II. G. Hahn, L. Borkwardt, O. Schales and H. Werner. *Ann.* 520, 107-2

(1935); cf. *C. A.* 29, 1820'.—Tryptamine-HCl (I) (0.190 g.) and 1.2 g. homopiperonal in 8 cc.  $H_2O$  and 4 cc. acetate buffer (pH 5.2) at 23° give, after 8 days, 5% (15% with a phosphate buffer of pH 6.2) of 3-(3,4-methylenedioxybenzyl)-3,4,5,6-tetrahydro-4-carboline (II), m. 107°, *HCl* salt, decomp. 273°. I and 3,4,5-(MeO) $_3$ C $_6$ H $_2$ CHO at pH 6.2 for 10 days at 25° give 16% of the 3,4,5-trimethoxybenzyl analog, m. 84°; the yield is 20% at pH 3.8 and 6% at pH 4.0. I and BuH at pH 5.2 give after 3 weeks at 25° 48% of the 3-Ph analog, m. 168-69° (*HCl* salt, m. 253° (decompn.)). *o*- and *p*-HO-C $_6$ H $_4$ CHO, piperonal, vanillin and citral do not react; AcCHO is oxidized to the acid, AcCO $_2$ H; furfural gives 10% of a compd., m. 122°. I and AcCO $_2$ H at pH 3.4, 4.0, 5.2 and 6.2 give, resp., 47, 50, 67 and 93% yields (in 2 days) of 3-methyl-3,4,5,6-tetrahydro-4-carboline-3-carboxylic acid (III), decomp. 220°; Me ester, m. 138° (*HCl* salt, decomp. 268°). PhCl $_3$ /COCO $_2$ H and I at 25° and pH 3.8, 4.6, 5.2 and 6.2 give, resp., 74, 32, 18.5 and 0.6% (after 13 days) (50, 19, 11 and 49.5% after 7 days) of the 3-benzyl analog, decomp. 253° (CO $_2$  evolution), attempts to prep the Me ester with MeOH and HCl resulted in the splitting off of CO $_2$ . 3-(3,4-Methylenedioxybenzyl) analog, decomp. 250°, results in 59, 61 and 63.6% yields (after 7 days) at pH 4.0, 5.3 and 6.2. MeOH-HCl gives II 3-(3-Alethoxy-4-hydroxybenzyl) analog of III, decomp. 230-40°, results in 49.3, 39.9 and 58.8% yield (after 15 days) at pH 4.2, 4.9 and 6.2. MeOH-HCl gives 65.9% of the 3-(3-methoxy-4-hydroxybenzyl) analog of II, as the *HCl* salt, decomp. 252-4°. 3-(3,4-Dimethoxybenzyl) analog of III, decomp. 230-2°, yield after 15 days, 49.4, 32.5 and 44.4% at pH 4.2, 4.9 and 6.2. The 3-(3,4-dimethoxybenzyl) analog of II, as the *HCl* salt, decomp. 230° (about 50% yield) 3-(3,4,5-Trimethoxybenzyl) analog of III, decomp. 191°, yields after 11 days at pH 4.4, 5.3 and 6.2 are 32.2, 43.1 and 35.5%. The 3-(3,4,5-trimethoxybenzyl) analog of II, m. 85° (73% yield). The azlactone from vanillin and hippuric acid, refluxed with 20% NaOH for 6 hrs., give 43% of 3-methoxy-4-hydroxyphenylpyruvic acid, m. 161°. III Syntheses of yohimbine skeleton: Georg Hahn and Heinrich Werner. *Ibid.* 123-33.—*m*-Hydroxyphenylpyruvic acid, decomp. 165°, results in 33.7% yield from the azlactone of *m*-HO-C $_6$ H $_4$ CHO and hippuric acid. With tryptamine-HCl this gives in 10 days at pH 4.2 and 25° 87.4% of 3-(*m*-hydroxybenzyl)-3,4,5,6-tetrahydro-4-carboline-3-carboxylic acid (I), m. 225-7° (decompn.); MeOH-HCl gives 87.6% of 3-(*m*-hydroxybenzyl)-3,4,5,6-tetrahydro-4-carboline (II), whose *HCl* salt decomposes 245-5°; *HCl*HO at pH 4.4 gives 87.8% of the 3-hydroxy-6-hydroxyphenylbenzyl analog, whose *HCl* salt decomposes 283-5°; pptn. of the hot aq. soln. of this salt with Na $_2$ CO $_3$  gives 88.3% of a hexahydroxyhombol, C $_{11}$ H $_{11}$ N $_2$ O $_6$ , decomp. 267-8°; this also results from the cold soln. with NH $_4$ OH or with a buffered soln. of pH 8. The *p*-HO isomer of I decomposes 240-1°; in the light the yield is 74% after 10 days at 25° and pH 4.2; the yield is decreased in diffused daylight. The *p*-HO isomer of II (*HCl* salt) decomposes 246-7° (63.5% yield).

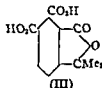
C. J. West

Constitution of aromatic transformation products of picrotoxin. Karl Tettwiler and Irmgard Drilhaus. *Ann.* 520, 163-84 (1935); cf. Mercer, Robertson and Cahn, *C. A.* 29, 6003'.—The lactone acid (I) from picrotoxin with alk. KMnO $_4$  gives the acid (II), m. 194° (decompn.); the KOH melt yields Me $_2$ CO, (CO $_2$ H), and *m*-Cl $_2$ (CO $_2$ H) $_2$ . Acid KMnO $_4$  with I gives the acid (III), m. 284°, which with KOH yields 1,2,3-C $_6$ H $_3$ (CO $_2$ H) $_3$  and on heating with Naturkupper C in quinoline for 1 hr. yields dimethylphthalide. I and KOH give 2,6-(HO-C $_6$ H $_4$ )(CH $_3$ ) $_2$ CO $_2$ H, oxidized by alk. KMnO $_4$  to 2,6-(HO-C $_6$ H $_4$ ) $_2$ C $_6$ H $_2$ (CO $_2$ H) $_2$ , m. 235° (decompn.). Distn. of I with Ac $_2$ O gives IV, m. 154° (semicarbazone, m. 315° (decompn.)). The distn. of 2,6-Me(HO-C $_6$ H $_4$ )(CH $_3$ ) $_2$ CO $_2$ H (V) with Ac $_2$ O yields 5-keto-1-methyl-5,6,7,8-tetrahydronaphthalene, m. 63° (semicarbazone, m. 242° (decompn.)); *p*-nitrophenylhydrazide, red, m. 203° (decompn.); the same compd. results by the dry distn. of the Ca salt of V. The isomeric deriv. of picrotoxin ketone is rearranged by SOCl $_2$  to the nitride (VI), m. 208°,

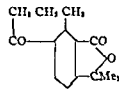
which is hydrolyzed to the acid (VII), m. 149°, and in the KOH melt yields *o*-MeC $_6$ H $_4$ (CO $_2$ H) $_2$ . VII with KMnO $_4$  yields III. Picrotin ketol (VIII) with HCl in AcOH yields an  $\alpha$ -Cl ketone (IX), also formed from picrotoxin ketone and HOCl. The action of KOH upon VIII or IX yields the same di-Me deriv. of dimethylphthalide, oxidized by KMnO $_4$  to III.



(II)



(III)



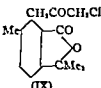
(IV)



(VI)



(VII)



(IX)

C. J. West

A new reaction of lignin and vanillin. Pierre Fourment and Henry Roques. *Bull. sci. pharm.* 41, 449-51 (1935).—Vanillin gives with diphenylenediamine in AcOH a red color which disappears after a few sec. The color remains stable when the vanillin is covered with liquid petrolatum and shaken with diphenylenediamine acetate. It is supposed that the color produced by the latter substance with lignin is caused by the presence of a vanillin nucleus in wood.

A. E. Meyer

Chemistry of jute lignin. VII. Behavior of organic compounds toward ClO $_2$  and its significance on the constitution of lignin. Pulin Behari Sarkar. *J. Indian Chem. Soc.* 12, 470-5 (1933); cf. *C. A.* 29, 5852'.—ClO $_2$  (I) was prep. by warming an intimate mixt. of (CO $_2$ H) $_2$  (20 g) and KClO $_3$  (25 g.) with 80 cc. of dil. H $_2$ SO $_4$  (100 cc. H $_2$ SO $_4$ , 225 cc. H $_2$ O) in an all-glass app. and absorbing the gas in H $_2$ O. The soln. was dild. to 0.25 N and was preserved in a dark bottle with a gas-tight ground-glass stopper. By shaking 10 cc. of I with 0.5 g. of the finely divided material and titrating after 12-48 hrs., the action of I on a large no. of org. compds. has been tried. All phenolic compds. are readily oxidized and, although not resistant, MeO and AcO groups are more stable. Since lignin contains more than 1 MeO group attached to the benzene ring, its reactivity with I does not prove the presence of a free phenolic OH group in the mol. Cl-, Br- and NO $_2$ -substituted phenols are more resistant to the attack of I and it is inferred that these groups enter the benzene ring in lignin since chloro-, bromo- and nitro-lignin behave similarly. The methylenedioxy group is stable toward I, as are also CO $_2$ H groups attached directly to the ring. I oxidizes side chains to CO $_2$ H groups. From the behavior of methylated or acetylated aliphatic compds. and that of delignified jute it is concluded that jute-pectin is aliphatic in nature. Small amts of chlorolignin (II) are formed by the action of I on lignin and II is evidently responsible for the slight yellow color of the delignified fiber. II prep. by the action of I is identical with that obtained in the usual way by the use of Cl $_2$ . VIII. Methylation of lignin. Pulin Behari Sarkar. *Ibid.* 542-6.—Jute-lignin (I), sep. in the ordinary way, was methylated with Me $_2$ SO and KOH by Urban's method (*C. A.* 20, 3080) at 20-25°, the amt. of KOH being increased 3-fold to facilitate stirring. The highest MeO value that could be reached (35-6%) was not in any simple ratio with its

original MeO content, reckoning 830 as the mol. wt. of I. Lignin (II), isolated at low temps. (C. A. 28, 75219), gave a const. final MeO value (34.51%), an increase of 5 added OMe groups over the 5 groups actually found in II. As the reducing lignin no longer reduces Fehling soln. after methylation it is concluded that I contains no CHO group. This corroborates the finding that the reducing action is due to 2 OH groups in the *o*-position in the benzene ring, formed as a result of the cleavage of the  $-OCH_2O-$  group and the splitting-out of HCHO. From the temp. at which MeI first appears in the Zeisel esters, it is inferred that the OMe groups are linked to the benzene ring in natural lignin. All the MeO groups in I are of the ether type as are also those entering the mol. on methylation. Methylated II was oxidized with 5 N HNO<sub>3</sub> (C. A. 27, 4779) but no asinic acid could be detected in the product, which contained a small amt. of (CO<sub>2</sub>H). The tarry mass obtained after methylation could not be purified without recourse to boiling with alkali and thus no information regarding the CO<sub>2</sub>H group could be obtained. IX. Acetylation of lignin. Pulin Behari Sarkar. *Ibid.* 547-51. —To confirm the results obtained by the methylation of jute lignin (I) samples prepd. in the usual way and by the modified method have been acetylated by heating 2 g. of very finely powd. I (dried at 105°) on a water bath for 2-3 hrs. with 25 cc. of a mixt. of Ac<sub>2</sub>O (12 cc.) and pyridine (88 cc.) and, after washing and drying the product at 105°, repeating the acetylation process. The highest AcOH content found was 26.45% (calcd. 28.83%), corresponding to 5 OH groups, a result in good agreement with methylation data. As the acetylated lignin (II) has no longer a reducing action and does not absorb II in the presence of metallic Pd, it has no CHO group or ethylene linkage. The slight absorption of IBr or ICl by I does not prove the presence of a double bond in the side chain. After the expulsion of HCHO from I the black product was treated with Me<sub>2</sub>CO in the presence of P<sub>2</sub>O<sub>5</sub> below 10°. The light gray material was no longer sol. in dil. NaOH and is probably a cyclic compd. (C. A. 27, 3457). The formation of this Me<sub>2</sub>CO addn. compd. proves the presence of 2 OH groups in the *o*-position formed by the cleavage of the  $-OCH_2O-$  group present in lignin. C. R. Addinall

Aldol condensations III. Synthesis of carotenoid chains by condensation with 3-methyl 2-buten-1-ol F. G. Fischer and K. Hultsch. *Ber.* 68B, 1726-34 (1935); cf. C. A. 29, 1386. —Any attempt at synthesis of the typical carotenoid chain must provide for the branched Me group and the necessary conjugated linkages. It seemed that Me<sub>2</sub>C=CHCHO (I) was adapted to this type of synthesis through a linear condensation. Under the influence of weak alkali in aq. soln. the Me group of I mol. reacts with the CO group of a 2nd mol. to give dehydrocitra (II), b. 81-84°, which could only be recovered from the reaction mixt. by conversion into tetrahydrocitra and its semicarbazone, m. 223°. In the presence of NaNH<sub>2</sub>, Et<sub>2</sub>O soln. of I undergo a diene synthesis and yield 2,2,4-trimethyl-4,6-cyclohexadienal, b. 85-87°; semicarbazone, m. 213°. Under definite conditions (to be published) the linear condensation of I takes place smoothly with satisfactory yields of II and 3,7,11-trimethyl-2,4,6,8,10-dodecapental (III), formed from 3 mols. of I. The direct distn. in the absence of air of the reaction product from 70 g. of I gave 23 g. of distillable condensate which, in turn, was fractionally distd. to give 5 fractions. Fraction 1 consisted mainly of the above-mentioned cyclic aldehyde. The repeated distn. of fractions 2 and 3 yielded II, C<sub>10</sub>H<sub>16</sub>O, m. 40.5-41°; oxime, m. 123°; phenylhydrazone, m. 141-2°; *p*-nitrophenylhydrazone, m. 103-4°. II was oxidized by Ag<sub>2</sub>O to dehydrogeranic acid, m. 187°. The *O*-sensitive II contained in fraction 3 was reduced with (iso-PrO)<sub>2</sub>Al and iso-PrOH, according to the procedure of Ponnordf (C. A. 20, 1611), to the corresponding unsatd. alc., 3,7-dimethyl 2,4,6-octatrienol, C<sub>10</sub>H<sub>18</sub>O, m. 46-7°. Fraction 4 contained a monocyclic C<sub>10</sub>-aldehyde, b. 130° in *vacuo*, which could not be crystd. Fraction 5 yielded golden yellow prismatic needles of III (larnesinal), C<sub>15</sub>H<sub>20</sub>O, m. 120-1°; oxime, m. 192° (de-

compn.); semicarbazone, m. 236° (decompn.). A Ponnordf reduction of III yielded farnesinol, 3,7,11-trimethyl-2,4,6,8,10-dodecapentenol, C<sub>15</sub>H<sub>26</sub>O, m. 136-7°, whose reaction with SbCl<sub>5</sub> in CHCl<sub>3</sub> is extremely sensitive, yielding an intense blue color, in shade very like that of vitamin A. C. R. Addinall

Coloring matters of Acanthodiptomus yamanacensis Brehm. Harusada Sugimoto, Kiyoshi Ueno and Sigeto Watanabe. *J. Chem. Soc. Japan* 56, 1199-1204 (1935). —Atsican (I),  $\beta$ -carotene (II) and a new pigment (III) were isolated. I was also found in *Combroses japonicus* Haan II and III were found in free or in combination with wax. K. Kitsuta

Chemical interrelationships in the cholane group. H. Jensen. *J. Chem. Education* 12, 559-64 (1935). —A review. E. H.

Bromination of 3-ketobisnorcholelolic acid and 3-ketobisnorcholelolic acid. 3-ketobisnor-4-cholelolic acid and 3-ketobisnorallo-1-cholelolic acid Adolf Butenandt and Luigi Mamoli. *Ber.* 68B, 1854-9 (1935); cf. C. A. 29, 1469. —Bromination of pregnenedione takes place at C atom 4 and the Br atom is easily removed as HBr whereas the Br atom which enters at C atom 2 in allopregnenedione is eliminated with difficulty. The behavior of other members of the normal (cis) and allo (trans) series of 3-hydroxysterol derivatives is being investigated to det. whether a general fundamental law governs substitution in these structural isomers, and the bromination of 3-hydroxybisnorcholelolic acid (I) and 3-hydroxybisnorcholelolic acid (II) is described. A suspension of 5 g. of 3-hydroxybisnorcholelolic acid, prepd. from stigmasterol (C. A. 29, 1831), in 50 cc. AcOH was treated with 25 cc. of Br in AcOH (contg. 1 mol. Br calcd. on the cholelolic acid) and the clear soln. of the dibromide was oxidized with CrO<sub>3</sub> in AcOH over a period of 16 hrs. at 20°. The clear reaction mixt. was extd. with Et<sub>2</sub>O, the ext. was taken up in 100 cc. AcOH, heated for 10 min. with 5 g. of Zn dust, pptd. with H<sub>2</sub>O and shaken out with Et<sub>2</sub>O. The evapd. residue was crystd. from AcOH and gave large prisms of 3-ketobisnor-4-cholelolic acid (III), C<sub>27</sub>H<sub>44</sub>O<sub>2</sub>, m. 268° (decompn.), [α]<sub>D</sub><sup>20</sup> 60° (in CHCl<sub>3</sub>), showing an absorption max. at 240 mμ, and reduced catalytically in the presence of Pd black to 3-ketobisnorcholelolic acid (IV), C<sub>27</sub>H<sub>46</sub>O<sub>2</sub>, m. 184°, [α]<sub>D</sub><sup>20</sup> 4.55° (in CHCl<sub>3</sub>), and 3-ketobisnorallocholelolic acid (V), m. 244° (C. A. 29, 1831). The bromination of 500 mg. of IV in 10 cc. AcOH with 10 drops of HBr in AcOH and 0.75 cc. of Br in AcOH (0.9 mol. Br) gave 487 mg. of crude cryst. material from which 125 mg. of pure 4-bromo-3-ketobisnorcholelolic acid, m. 221°, was recovered. Cleavage of 110 mg. of VI by heating with anhyd. pyridine for 13 hrs. gave 38 mg. of III. The bromination of V yielded needles of 2-bromo-3-ketobisnorallocholelolic acid, m. 230° (decompn.), which, on heating with a 21% soln. of AcOK in AcOH for 5 hrs. at 175-83°, cleaved with difficulty to give a poor yield of 3-oxobisnorcholelolic acid, m. 235° (decompn.), with an absorption max. at 240 mμ. Although III is so similar to progesterone it shows no physiol. activity either in the Clauberg test (25 mg.) or in the capon-comb test (6 mg.), a result which again demonstrates the highly specific nature of the corpus luteum effect. The behavior of IV and its allo isomer V toward bromination and cleavage of the resulting products is identical with that noted with pregnenedione and its allomer. Similar results have been previously noted, particularly in the bromination of dehydrocholelolic acid (C. A. 29, 1411) and the chlorination of *trans*- $\beta$ -decalone (C. A. 25, 2151). A parallel to this behavior of stereoisomers is found in the oxidative cleavage of the ring of the cis-steroids between the 3- and 4-positions whereas the *trans*-dehydrocholesterols rupture between the 2- and 3-C atoms. C. R. Addinall

Allo-1-pregnene-3,20-dione, a further contribution to the specificity of corpus luteum activity. Adolf Butenandt and Luigi Mamoli. *Ber.* 68B, 1850-4 (1935); cf. C. A. 29, 1469. —A soln. of 850 mg. of allopregnenedione (I) (C. A. 28, 6893) in 32 cc. of AcOH was treated, at 29°, with 10 drops of AcOH-HBr and 1.27 cc. of a Br-AcOH soln. contg. 1 mol. of Br (reckoned on I). The ppt.

formed on the addn. of  $H_2O$  crystd. from alc. and dil.  $Me_2CO$  to give 660 mg. of needles of a brominated compd (II),  $C_{27}H_{44}BrO_2$ , m. 199° (decompn.), which was stable to heating for 3 hrs. with  $AgOAc$  in  $AcOH$  and to the action of 10%  $NaOH$  on its benzene soln. at 20°. By heating with  $BzOK$  in  $BzOH$  at 205°, II was converted into 2-hydroxy-3-pregnene-3,20-dione benzoate, m. 235°. On heating 150 mg. of II with 5 cc. of 21%  $AcOK$  in  $AcOH$  for 5 hrs. at 175–85°, treating the product with  $H_2O$  and shaking out with  $Et_2O$ , a neutral product was obtained. Sublimation at 0.001 mm. Hg and 80° gave 13 mg. of *allo-1-pregnene-3,20-dione* (III),  $C_{27}H_{44}O_2$ , m. 140°,  $[\alpha]_D^{25}$  68° (in alc.). III shows the absorption in the ultraviolet at 235 m $\mu$  characteristic for  $\alpha,\beta$ -unsatd ketones. The behavior of II shows that, contrary to the course of the bromination of pregnenedione (C. A. 29, 1754), the Br atom has, in all probability, entered the 2-position. III is not identical with progesterone but is an isomer, differing only in the position of a double bond in conjugation with the CO group. It is physiologically inactive, a further proof of the extreme specificity of the action of the corpus luteum. C. R. Addinall

An isomeric *allopregnan-3-ol-20-one*. Adolf Butenandt and Luigi Mamoli. *Ber* 68B, 1847–50 (1935); cf. C. A. 29, 1469°.—Corpus luteum exts. contain progesterone and its tetrahydro deriv., *allopregnan-3-ol-20-one* (I),  $C_{27}H_{44}O_2$ , m. 194°,  $[\alpha]_D^{25}$  90.8°, acetate (II), m. 144.5°. The addn. of  $H_2O$  to a product formed by heating 3.6 g. of I with 5%  $KOH$  in  $MeOH$  for 1 hr. gave 2 g. of I and 1 g. of crude material which on fractional sublimation at 0.001 mm. Hg yielded 0.5 g. of cryst. *isoolopregnanolone* (III), m. 148°,  $[\alpha]_D^{25}$  6.05 (in alc.). Similarly, by heating 245 mg. of I with 5%  $KOH$  in  $MeOH$  for 2 hrs. and acidifying the product, 163 mg. of I and 63 mg. of III were isolated. III forms an acetate (IV), m. 101°, which on sapon. as above liberates 50% of I and 27% of III. The  $CrO_3$  oxidation of I produced *allopregnanedione* (V) (C. A. 28, 6803), m. 200.5°,  $[\alpha]_D^{25}$  126.9°, whereas that of III under the same conditions gave *isoolopregnanedione* (VI), m. 134–5°,  $[\alpha]_D^{25}$  –14.56°, together with a small amt. of the normal dione. Both I and III gave immediately difficulty sol. addn. compds. with digitonin. In the presence of alkali there seems to be an equil. between the forms I and III which favors the former. The isomerism can only reside in the stereochem. arrangement around the 17-C atom at which a tertiary H atom is adjacent to a CO group. A similar isomerism must be present in pregnenedione and progesterone, the other known types of the progesterone series. I and III correspond to cholesterol and stigmasterol in the location of a OH group on the 3-C atom. Thus the formation of digitonin addn. compds. was predictable. However, II and V (but not IV and VI) also gave addn. compds. on standing for 2–4 hrs. after the addn. of the reagent. Consequently the generally valid conception that the presence of a free OH group in the 3-C position presupposes the formation of a difficulty sol. digitonide must be modified within the above limitation. C. R. Addinall

Systematic degradation of 3-hydroxyallocholic acid to androsterone. O. Dalmer, F. v. Werder, H. Honigsmann and K. Heyns. *Ber* 68B, 1814–25 (1935).—A systematic degradation of the side-chain of 3-hydroxycholehanic acid (I) has been successfully carried out and, since I can be obtained from hydoxycholeic acid, this method is the first devised for the transformation of a natural bile acid into androsterone (II). A soln. of 3.8 g. of the *Me* ester of I (C. A. 19, 2818) in 160 cc. of abs.  $Et_2O$  was run into a Grignard reagent consisting of 3 g. Mg and 23 g.  $PhBr$  in 70 cc. abs.  $Et_2O$ . After 3 hrs. refluxing, the mixt. was evapd. and the residue was heated for 2 hrs. at 100°. The product was decompd. with cold dil.  $H_2SO_4$  and freed from  $Ph$  by steam distn. The unchanged ester was sapond. and the carbinol was extd. with  $Et_2O$ . The ext. was worked up and yielded 5 g. of crude material which on recrystn. from cyclohexane gave (3-hydroxyornallocholic)-diphenyl carbinol (III),  $C_{27}H_{44}O_2$ , m. 150°,  $[\alpha]_D^{25}$  20° (in abs. alc.). To avoid ring cleavage in the process of shortening the chain, the 3-OH group was blocked by

acetylation. A mixt. of 50 g. of III in 15 cc. of  $Ac_2O$  was heated for 30 min. and the residue after vacuum distn. was dissolved in 100 cc. of  $AcOH$ . The soln. was heated to 100° and a soln. of 5 g. of  $CrO_3$  in 5 cc.  $H_2O$  and 50 cc.  $AcOH$  was introduced, with continuous stirring over a period of 1.25 hrs. The soln. was stirred for 2 more hrs. and after distn. was shaken out with  $H_2O$  and  $Et_2O$ . The washed  $Et_2O$  layer was shaken out with 10%  $KOH$  and the insol salt formed was centrifuged. The salt was decompd. with  $H_2SO_4$  in the presence of  $Et_2O$  and the evapd. ext. gave 2.2 g. of 3-acetoxyornallocholic acid (IV),  $C_{27}H_{44}O_4$ , m. 235°,  $[\alpha]_D^{25}$  26° (in  $CHCl_3$ ); *Me* ester (V), m. 189–90°,  $[\alpha]_D^{25}$  26°. Treatment of IV with alc.  $KOH$  produced 3-hydroxyornallocholic acid (VI), m. 205–7°,  $[\alpha]_D^{25}$  21° (in abs. alc.), *Me* ester, m. 169–70°,  $[\alpha]_D^{25}$  21° (in  $CHCl_3$ ). A soln. of 18 g. of V in 350 cc. abs.  $Et_2O$  and 150 cc. benzene was poured into a Grignard reagent prepd. from 15.9 g. Mg and 103 g.  $PhBr$  in 300 cc. abs.  $Et_2O$ . A processing analogous to that for III gave 1 g. of VI (after 8 hrs. sapon.) and 27 g. of a yellow resinous (3-hydroxyornallocholic)-diphenyl carbinol, which was acetylated. The product was dissolved in 500 cc.  $AcOH$ , and, after the introduction of 30 g. of  $CrO_3$  in 200 cc.  $H_2O$  and 150 cc.  $AcOH$  at 100–105° with stirring over a period of 3 hrs., and a further 5 hrs. stirring of the reaction mixt., gave, on working up through the K salt, 7.8 g. of 3-acetoxyornallocholic acid (VII),  $C_{27}H_{44}O_4$ , m. 225–7°,  $[\alpha]_D^{25}$  2° (in  $CHCl_3$ ); *Me* ester (VIII), m. 128–9°,  $[\alpha]_D^{25}$  10°; 3-hydroxyornallocholic acid, m. 220°,  $[\alpha]_D^{25}$  17°, *Me* ester, m. 162–3°,  $[\alpha]_D^{25}$  12°. By the action of a Grignard reagent contg. 8.5 g. Mg and 55 g. of  $PhBr$  in 200 cc. abs.  $Et_2O$  on a soln. of 8.6 g. of VIII in 150 cc. abs.  $Et_2O$  and 150 cc. benzene, a yield of 12.2 g. of (3-hydroxyornallocholic)-diphenyl carbinol was obtained. The acetate was oxidized with  $CrO_3$  in  $AcOH$  to 1.5 g. (19.5%) of 3-acetoxyisovallocholic acid, converted by  $CH_3MgI$  in  $MeOH$  to the *Me* ester (IX), m. 201–2°,  $[\alpha]_D^{25}$  56° (in  $CHCl_3$ ). Treatment of IX with  $PhMgBr$  gave 1.8 g. of carbinol which was heated in an evacuated retort at 1 mm. to 150° until  $H_2O$  ceased to be evolved. The temp. was then slowly raised to 200°. Distn. at 0.0001 mm. and 260° gave 1.1 g. of distillate which was acetylated. The evapd. product was dissolved in  $CHCl_3$  and satd. with  $O_2$  for 25 min. at 0°. The  $CHCl_3$ -free product was heated with 50 cc.  $AcOH$  for 1 hr. at 95° and, after removal of the  $AcOH$  in *vacuo*, was taken up in  $Et_2O$  and freed from acid substances by a 5-fold shaking with 2 N  $KOH$ . The evapd. ext. gave 1 g. of neutral material which on heating with semicarbazide acetate for 8 hrs. yielded 3-epiacetoxy-*isovallocholan-17-one* semicarbazone (X),  $C_{27}H_{44}N_2O_4$ , m. 272–3° (decompn.), which on hydrolysis with alc.  $H_2SO_4$  and recrystn. of the product from cyclohexane gave androsterone, m. 179–80°,  $[\alpha]_D^{25}$  94°, showing in 150–200 daily doses the cock's comb reaction of Butenandt. X was also recovered from the collected neutral oxidation products of the degradation. The hydrogenation of 83 g. of sitostanone (C. A. 13, 130) in 2 l. of  $AcOH$  in the presence of 30 cc. of 48%  $HBr$  and 4 g. of  $PtO_2$  at 60° gave, on sapon., 58 g. of the previously unknown satd. sterol, *epistostanol*,  $C_{27}H_{48}O$ , m. 203°,  $[\alpha]_D^{25}$  26° (in  $CHCl_3$ ); acetate (XI), m. 88°,  $[\alpha]_D^{25}$  28°. The oxidation of XI with  $CrO_3$  in  $AcOH$  and *conca* in *vacuo* gave a product which was taken up in  $Et_2O$ . The ext. was shaken out with 2 N  $NaOH$  and the insol salt was centrifuged from the  $Et_2O$  soln. of the neutral oxidation product (XII). The salt was decompd. with dil.  $H_2SO_4$  and the free acid was converted into V. The evapd. residue from XII was taken up in  $MeOH$  and after recrystn. of 11.8 g. of XI and filtering, the soln. was evapd., steam-distd., extd. with  $Et_2O$ , evapd., and taken up in alc. Refluxing with semicarbazide acetate gave X, hydrolyzed to androsterone.  $CrO_3$  oxidation of 35 g. of stigmasterol at 55° gave colorless leaflets of *stigmasterone hydrate* (XIII),  $C_{27}H_{46}O.H_2O$ , m. 81–2°,  $[\alpha]_D^{25}$  23° (in  $CHCl_3$ ) (*oxime*, m. 219°,  $[\alpha]_D^{25}$  30°), converted by treatment with  $BzCl$  in pyridine into the  $H_2O$ -free ketone (XIV), m. 157°,  $[\alpha]_D^{25}$  42°. The catalytic hydrogenation of XIV in a manner similar to that of sitostanone gave *epistigmasterol*, m. 200°,  $[\alpha]_D^{25}$  25°; acetate, m. 85°,  $[\alpha]_D^{25}$

27°, oxidized to a free acid from which V was isolated. The neutral oxidation product yielded, on treatment with semicarbazide acetate, X, m. 273°. The oxidation of 120 g. of epicholesterol acetate (C. A. 29, 810<sup>1</sup>) gave 12 g. of I, m. 209°, [α]<sub>D</sub><sup>25</sup> 29°; Me ester, m. 164-5° (acetate, m. 147-8°, [α]<sub>D</sub><sup>25</sup> 21°).

C. R. Addinall

A comparison between sitostanol and stigmasterol. B. E. Bengtsson. *Z. physiol. Chem.* 237, 46-51 (1935) — On the basis of m. p., mixed m. p. and optical rotation of their various derivs., sitostanol and stigmasterol are believed to be identical. Close agreement is shown in the acetates, benzoates and *m*-dimethylbenzoates of the 2 hydrogenated sterols, in the ketones sitostanone and stigmasterone, and in the hydrocarbons sitostane and stigmasterane. The slight differences decrease in the order alc., ketone, hydrocarbon, due to progressive removal of minimal impurities. Corresponding mixts with cholesterol and cholestane, on the contrary, showed decrease of lowering of m. p.

A. W. Dox

Actinasterol, a new sterol of the animal kingdom. E. Klenk and W. Diebold. *Z. physiol. Chem.* 236, 141-4 (1935) — Extn. of the sea anemone (*Anemone sulcata*), collected in the Bay of Naples, with MeAc and Et<sub>2</sub>O yielded an oil which on cooling to 0° deposited crystals of a new sterol, actinasterol, C<sub>27</sub>H<sub>46</sub>O, m. 145-6°, [α]<sub>D</sub><sup>25</sup> -42.5°. The substance has the same m. p. as cholesterol but its acetate m. 12° higher than cholesterol acetate, and it contains an addnl double bond. Actinasteryl acetate, m. 126-6.5°, was obtained by esterification with Ac<sub>2</sub>O at 100°. Hydrogenation of this with PtO<sub>2</sub> yielded tetrahydroactinasteryl acetate, m. 120°, from which tetrahydroactinasterol, m. 139-40°, was obtained by sapon.

A. W. Dox

Sex hormones VII. Synthetic preparation of the testicular hormone, testosterone (androsten-3-on-17-ol). L. Ruzicka and A. Wettstein. *Helv. Chim. Acta* 18, 1264-75 (1935), cf. C. A. 29, 6988<sup>1</sup>. —trans-Dehydroandrosterone, reduced with Na and PrOH or H and Ni in EtOH, gave Δ<sup>4</sup>-trans-androstene-3,17-diol (II), m. 182-3°. Δ<sup>4</sup>-Acetoxyandrosten-17-one, hydrogenated over Ni, gave Δ<sup>4</sup>-3-acetoxyandrosten-17-ol, m. 147-8°. I, heated with Ac<sub>2</sub>O, gave Δ<sup>4</sup>-trans-androstene-3,17-diol diacetate (III), m. 165-6°. II on sapon, with KOH in MeOH gave Δ<sup>4</sup>-17-acetoxyandrosten-3-ol (III), m. 148.5-8.5°. III, brominated, oxidized and then debrominated, gave Δ<sup>4</sup>-17-acetoxyandrosten-3-one (IV), m. 139-41°. IV on sapon gave Δ<sup>4</sup>-17-hydroxyandrosten-3-one, m. 154.5-5.5°; oxime, m. 222-3°. III has a much greater physiol. activity than androstene-3,17-dione (V), androsterone (VI) and trans-dehydroandrosterone (VII). 10 γ of III had a greater activity than 50, 200 and 500 γ of V, VI and VII, resp.

Julius White

Sex hormones and related substances V. The preparation of epidihydrocholesterol and its oxidation to 3-epidihydrocholesterol-17-one (androsterone). Wilhelm Dürschel. *Z. physiol. Chem.* 237, 52-6 (1935); cf. C. A. 29, 8079<sup>1</sup>. —Dihydrocholesterol (I), m. 145-6°, [α]<sub>D</sub><sup>25</sup> 22° (in CHCl<sub>3</sub>), was prep. by sapon of its acetate, and oxidized in AcOH with CrO<sub>3</sub> to dihydrocholesterol, m. 163°, [α]<sub>D</sub><sup>25</sup> 42° (in CHCl<sub>3</sub>). The latter was hydrogenated at 65-70° in Bu<sub>2</sub>O contg. HBr with PtO<sub>2</sub> catalyst to epidihydrocholesterol (II), m. 206°, [α]<sub>D</sub><sup>25</sup> 26°. II was obtained also in 20% yield by rearrangement of I by heating 15 hrs at 215° with NaOEt and removal of unchanged I by digestion pptn. It was converted into the *Ac* deriv., m. 90°, [α]<sub>D</sub><sup>25</sup> 28.4°, and the latter oxidized by CrO<sub>3</sub> to androsterone, m. 182-3°, [α]<sub>D</sub><sup>25</sup> 94.4°, which was purified by decomp. with semicarbazone, m. 284-5°, with C<sub>2</sub>H<sub>5</sub>O, and extg. with CCl<sub>4</sub>. The androsterone thus obtained showed no lowering of m. p. when mixed with androsterone from epidihydrocholesterol. In the capon comb. test its activity was 1 unit per 1-2 γ.

A. W. Dox

C<sub>2</sub>O<sub>2</sub> (Vol'kenshtein) 6 Constitution of nucleic acid (Makino) 11A Org. mineral substances (Treibs) 8 Magnetochem. investigations of org. substances (Muller) 2 Titration curves and dissociation constants of L-ascorbic

1 acid and diethyl dihydroxymaleate (Kumler, Daniels) 2. Photolytic and thermal decomposition products of azomethane (Heidt, Forbes) 2 Oxidation of sugars by air in the presence of Ce(OH)<sub>3</sub>, sol and Ce(OH)<sub>3</sub> gel (Ghosh, Ralshit) 2 App. for effecting exothermic or endothermic reactions between fluids [manuf. of acetone] (Ger. pat. 619,295) 1 Effecting reactions [MeOH synthesis] (Brit. pat. 432,574) 13.

2 Separating liquid hydrocarbons. I. G. Farbenind. A.-G. Fr. 785,974, Aug. 23, 1935 Mixts of liquid hydrocarbons are sep'd into fractions of different compns by dissolving I under pressure in the mixt. at a temp. below that of decomn and preferably with the addn of a solvent such as liquefied propane. The layers which form are sep'd.

3 Separating aromatic mono- and polychloro hydrocarbons. Chemische Fabrik von Heyden A.-G. (Kurt Buchheim, inventor). Ger. 616,596, Aug. 1, 1935 (Cl. 12a 2 01). The hydrocarbons are sep'd by azeotropic distn., water being used as the auxiliary substance. Thus, a crude mixt. obtained by chlorinating C<sub>6</sub>H<sub>6</sub>, contg. 15 parts of polychloro compds and the rest C<sub>6</sub>H<sub>6</sub>Cl is mixed with water to give an azeotropic b. p. of 91°. The mixt. is dist'd, the C<sub>6</sub>H<sub>6</sub>Cl going over and the polychloro compds. remaining. Other examples are given.

4 Catalytic dehydrogenation of hydrogenated heterocyclic compounds. Carl Wulff and Wilhelm Breuers (to I. G. Farbenind. A.-G.). U. S. 2,019,833, Nov. 5. A compd., such as piperidine from which pyridine is to be formed, is brought into contact (suitably at a temp. of about 410°) with a catalyst essentially comprising a heavy metal of the left subgroup of the 6th group of the periodic system or a heat-resistant compd. of such a metal such as WO<sub>3</sub>. Several examples are given.

5 Oxidation of hydrocarbons. I. G. Farbenind. A.-G. (Max Harder and Wilhelm Dietrich, inventors). Ger. 619,113, Sept. 21, 1935 (Cl. 12a 11). The manuf. of fatty acids and alcs. by treating higher paraffin hydrocarbons with air is improved by first refining the hydrocarbons with H<sub>2</sub>SO<sub>4</sub> in the presence of a small proportion of an oxide of N. The conditions during the refining treatment must be sufficiently mild to avoid reaction of the hydrocarbon. Sp. processes are described.

6 Oxygenated compounds from normally gaseous paraffinic hydrocarbons. Henry Dreyfus U. S. 2,020,671, Nov. 12. Hydrocarbons such as CH<sub>4</sub> and C<sub>2</sub>H<sub>6</sub> are caused to react with steam under a pressure of at least 50 atm. and at a temp. below 700° (suitably about 200-500°) in the presence of an oxide-free catalyst mass such as the chromate of Mg, Zn or Al, forming products such as alcs., acids, aldehydes and ketones. Cf. C. A. 29, 3349<sup>1</sup>.

7 Hydrocarbons and alcohols. Jean J. Pung and Rafael Sunen-Benedict. Fr. 784,742, July 23, 1935. An alkali, alk., earth or earth carbonate or bicarbonate is partially reduced by a metal or carbonaceous reducing agent to obtain a gas rich in CO, which is caused to react with H<sub>2</sub> in the presence of catalysts to produce hydrocarbons or alcs. as desired. The H<sub>2</sub> may be obtained by reduction of steam with a part of the CO formed.

8 Olefins. E. I. du Pont de Nemours & Co. Brit. 432,430, July 26, 1935. Olefins contg. less than 10 C atoms, e. g., C<sub>2</sub>H<sub>4</sub>, C<sub>3</sub>H<sub>6</sub>, industrial gas mixts. rich in C<sub>2</sub>H<sub>4</sub> and C<sub>3</sub>H<sub>6</sub>, are converted into higher olefins, e. g., C<sub>4</sub>H<sub>8</sub>, C<sub>5</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>12</sub>, by heating to 400-700°, preferably less than 2 sec., in presence of a small amt., e. g., 0.5-1% and not more than 5%, of O.

9 Converting normally gaseous olefins into hydrocarbon liquids. Vladimir Ipatieff (to Universal Oil Products Co.). U. S. 2,020,649, Nov. 12. Olefins such as C<sub>2</sub>H<sub>4</sub>, C<sub>3</sub>H<sub>6</sub>, etc., are polymerized (suitably at a temp. of about 100-250°) in the presence of a solid mixt. of H<sub>2</sub>O<sub>2</sub> and ZnO which serves as a catalyst and with which ZnCl<sub>2</sub>, fuller's earth, etc., also may be used.

Hydrating olefins. Henry Dreyfus. U. S. 2,020,672, Nov. 12. A molten salt (such as Ba pyrosulfate or ZnCl<sub>2</sub>) which is a hydration catalyst is used in reactions, such as

the hydration of  $C_2H_4$  at 300° under 20 atm. pressure to produce  $EtOH$ . Cf. *C. A.* 29, 7342.

**Hydration products of olefins.** Henry Dreyfus. *U. S.* 2,020,674, Nov. 12. An olefin such as  $C_2H_4$  is treated with water vapor at an elevated temp. (suitably about 300°) in the presence of a metal metaphosphate such as that of Na which serves as a catalyst in the formation of products such as  $EtOH$ .

**Alcohols.** Imperial Chemical Industries Ltd and Stanley J. Green. Brit. 433,519, Aug. 16, 1935. Fatty oils or wax esters, including sperm and like oils, or the corresponding fatty acids or other esters thereof, are reduced to the corresponding alcs. by treatment with H and a hydrogenation catalyst, e. g., Ni, Co, Cu, Fe, Pt, Pd, or oxides thereof, or  $ZnO$ , with or without an acidic oxide, e. g.,  $SiO_2$ ,  $Al_2O_3$  or an oxide of W, Cr, Mo or V, the reaction mixt. being vigorously agitated and the H passed through rapidly to carry over the alcs. Pressure may be employed. App. is described.

**Alcohols from olefins.** Floyd J. Metzger (to Air Reduction Co.) *U. S.* 2,021,564, Nov. 19. In a continuous process of producing an alc. such as  $EtOH$ , water and an olefin such as  $C_2H_4$  are supplied to a combined boiler and heat interchanger and heated in it to convert the water into steam, the resulting mixt. is brought into contact with an acid catalyst such as dil.  $H_2SO_4$  (suitably at a temp. of about 220–300°) and resulting hot alc. contg. gases are passed through the heat interchanger to heat the incoming mixt. (various operative details being described).

**Alcohols from olefins.** N. V. de Bataafsche Petroleum Maatschappij. Ger. 618,515, July 30, 1935 (Cl. 12a, 5 04). Olefins, especially those obtained by "cracking" mineral oils are absorbed in  $H_2SO_4$ . The  $H_2SO_4$  is diluted with water to hydrolyze the alkyl sulfate. The hydrolyzed product is repeatedly extd. to remove polymers. The extn. residue is subjected to azeotropic distn. to obtain the olefin. The same low-boiling liquid is used for the extn. as for the distn. Examples are given.

Bubble still suitable for distillation of alcohol, etc. August A. Zentler. *U. S.* 2,020,009, Nov. 5. Various structural and operative details.

**Drying alcohols or ketones.** Standard Oil Co. of California. Fr. 785,214, Aug. 7, 1935. See *U. S.* 2,000,043 (*C. A.* 29, 4021).

**Tertiary alcohols.** Richard Z. Moravec and Wm. Eng (to Shell Development Co.). *U. S.* 2,019,762, Nov. 5. For the manuf. of tertiary alcs. such as *tert*-butyl alc. from acid liquor obtained by the absorption of tertiary olefins such as *tert*-butylene in an aq. soln. of a "polybasic" strong acid such as  $H_2SO_4$ , sufficient basic material such as  $NH_3$  is added to the acid liquor to form 2 phases, the alc. phase is sep'd. from the nonalc. phase, and the former is independently treated, as by distn., to recover tertiary alc. from it.

**Hydrobetyl alcohols.** Clyde O. Henke and Milton A. Frahl (to E. I. du Pont de Nemours & Co.). *U. S.* 2,021,100, Nov. 12. See Brit. 430,578 (*C. A.* 29, 80009).

**Esters of unsaturated acids** such as those of methacrylic acid. Emmette F. Izard (to E. I. du Pont de Nemours & Co.). *U. S.* 2,020,685, Nov. 12. In forming an ester such as methyl methacrylate, the ester of a halogenated formic acid such as methyl chloroformate is caused to react with an olefin hydrocarbon such as  $C_2H_4$  and the resulting ester of a halogenated satd. aliphatic monocarboxylic acid is dehydrohalogenated to form an unsatd. ester.

**Esters of glycols.** Gerald H. Coleman and Garnett V. Moore (to Dow Chemical Co.). *U. S.* 2,021,852, Nov. 19. A glycol diester such as the diacetate ester of ethylene glycol is made by the reaction of an alkylene dichloride such as ethylene dichloride with an alkali metal salt of a lower aliphatic acid such as  $NaOAc$  in the presence of a catalyst including an alkylamine, an alkanolamine, an alicyclic amine, a pyridine base such as pyridine itself or a salt of such an amine or base.

**Isopropyl esters of aliphatic acids.** Gerald H. Coleman (to Dow Chemical Co.). *U. S.* 2,021,851, Nov. 19. In forming an ester such as isopropyl acetate, propylene is

caused to react with a substantially anhyd. satd. aliphatic monocarboxylic acid such as glacial  $H_2OAc$  under superatm. pressure and in the presence of  $H_2SO_4$  as a catalyst. A temp. of about 125° is suitable for the production of isopropyl acetate. Various examples are given.

**Vinyl esters.** Granville A. Perkins (to Carbide and Carbon Chemicals Corp.). *U. S.* 2,021,098, Nov. 19. See Brit. 418,943 (*C. A.* 29, 2176').

**Vinyl esters.** Otto Nicodemus and Walter Weisbehn (to I. G. Farbenind. A.-G.). *U. S.* 2,021,873, Nov. 19. See Ger. 604,640 (*C. A.* 29, 813').

**Dicarboxylic acid esters.** Charles W. Hawley (to E. I. du Pont de Nemours & Co.). *U. S.* 2,020,356, Nov. 12. In forming an ester, a polycarboxylic acid capable of forming an anhydride or an anhydride of a polycarboxylic acid such as phthalic anhydride is treated with an alc. in an amt. less than required for complete esterification of the acid or anhydride and the temp. of the reaction mixt. is maintained above the b. p. of the alc. while a stream of alc. or deriv. is passed through the reaction mixt.

**Nitric esters.** Frederick A. F. Crawford, Wm. A. P. Challener and Imperial Chemical Industries Ltd. Brit. 432,488, July 22, 1935. Nitroglycerin, nitroglycol and similar liquid nitric esters, or mixts thereof, are obtained by a continuous process in a series of reaction vessels, a proportion of the polyhydric alc. or mixt. of such, being added to each vessel and the whole of a suitable quantity of nitrating acid being added to the 1st vessel only. The reaction products are passed from each vessel to the next and the proportion of the alc. added to the 1st vessel is insufficient to produce an acid compn that has a deleterious effect on the nitric ester in contact therewith. Intensive cooling is used in the 2nd and succeeding vessels to minimize the effect of waste acid on the product.

**Enolic sulfuric esters of reduced indantrones.** David A. W. Fairweather and John Thomas (to Scottish Dyes Ltd.). *U. S.* 2,019,530, Nov. 5. In prep. sulfuric acid esters of the indantrone series by a process in which a tetrasulfuric acid ester of a reduced indantrone is hydrolyzed in an aq. medium, the tetrasulfuric acid ester is heated in an aq. medium contg.  $NaOH$  and  $Na$  hyposulfite to a temp. of about 60°, the heating being discontinued when one of the sulfuric acid groups has been removed and the resulting trisulfuric acid ester being subsequently heated in an aq. soln. contg. an alk. oxidizing agent and the reaction stopped when a substantial quantity of a disulfuric acid ester is formed. Various examples with details and modifications of procedure are given and various products are obtained which have good affinity for fibers and are converted into dyes of different shades by the action of very dil. acid. General claim is made to trisulfuric enolic esters of reduced indantrones as being new compds.

**Methylene ethers.** Tom Birchall and Samuel Coffey (to Imperial Chemical Industries Ltd.). *U. S.* 2,021,690, Nov. 19. A methylene ether such as methylene diethyl ether is prep'd. by interacting  $(CH_3)_2N_2$  with an alc. such as  $EtOH$  in the presence of  $HCl$  or  $H_2SO_4$  in the liquid phase (the acid being in quantity sufficient to decompose the  $(CH_3)_2N_2$ ). Several examples are given.

**Vinyl ethers.** Walter Reppe and Werner Wolff (to I. G. Farbenind. A.-G.). *U. S.* 2,021,869, Nov. 19. See Brit. 430,764 (*C. A.* 29, 8001').

**Amino alkyl amines.** I. G. Farbenindustrie A.-G. Brit. 433,625, Aug. 19, 1935. See Ger. 602,049 (*C. A.* 29, 474').

**Alkylene diamines.** Werner M. Lauter (to Wingfoot Corp.). *U. S.* 2,020,690, Nov. 12. An alkylene diamine such as ethylenediamine is formed by the reaction of an alkylene dihalide such as ethylene dichloride under the influence of heat and pressure (suitably a pressure of about 150–300 lb. per sq. in. and a temp. of about 95°) in the presence of a metal compd. such as cuprous chloride which forms a complex salt with the resulting alkylene diamine.

**Primary aromatic amines.** Ford H. McBerty and Kenneth C. Simon (to E. I. du Pont de Nemours & Co.). *U. S.* 2,020,368, Nov. 12. An amine such as  $PhNH_2$  is



produced from the corresponding aromatic nitro compd such as  $\text{PhNO}_2$  by the use of comminuted sheet ferrous material the surface of which is partly covered by Sn, in the presence of water and an Fe reduction catalyst

Amines of high molecular weight. H. Th. Böhm A-G. Fr. 785,004, July 31, 1935.  $\alpha$ -Amino fat acids derived from aliphatic, aromatic, cycloaliphatic, heterocyclic or mixed amines are heated in an inert solvent of sufficiently high b.p. until the sepn. of  $\text{CO}_2$  ceases. Thus, heptadecylamine is prepd. by heating  $\alpha$ -aminostearic acid in paraffin oil.

Alkylenes. H. Th. Böhm A-G. Fr. 785,005, July 31, 1935. Aliphatic alkylenes of high mol. wt. having more than 8 C atoms in the chain are prepd. by causing compds. which give salts or complex compds. with bases to react with secondary amines having at least one aliphatic chain of more than 8 C atoms, and afterward heating to dissolve the amine. Thus heptadecylene is prepd. by heating heptadecylphenylamine in a current of HCl, and hendecylene from hendecylphenylamine and  $\text{ZnCl}_2$ .

Acetals. I. G. Farbenind. A-G. Fr. 785,791, Aug. 19, 1935. Acetals are prepd. from polyvinyl alc. or its esters under pressure in the presence of a substance which acts as a solvent and is liquid at the pressure of the reaction but gaseous at normal pressure, e.g.,  $\text{SO}_2$ ,  $\text{MeCl}$ ,  $\text{EtCl}$ ,  $\text{Me-O}$  and ethylene oxide.

Diketene. George H. Law (to Carbide and Carbon Chemicals Corp.). U. S. 2,019,933, Nov. 5. See Can. 352,920 (C. A. 29, 80089).

Oxygen-containing addition products of ethylene. Henry Dreyfus U. S. 2,020,673, Nov. 12. Ethylene is treated with a lower aliphatic carboxylic acid such as  $\text{HOAc}$  and with water at a temp. of at least  $350^\circ$  and under a pressure of at least 5 atm. to form products such as  $\text{EtOAc}$ .

Aralkyl trithiocarbonates. Raymond W. Hess (to National Aniline & Chemical Co.) U. S. 2,021,726, Nov. 19. Compds. (suitable for use as rubber-rulization accelerators, ore-floation agents or intermediates in *org. synthesis*) such as Na benzyltrithiocarbonate, etc., are produced by treating an alkali metal salt of an aralkyl mercaptan such as benzyl mercaptan with  $\text{CS}_2$  (suitably with stirring and cooling) and the free aralkyl thiocarbonate acid may be obtained by treating the salts with dil.  $\text{H}_2\text{SO}_4$  or HCl. Several examples with details of procedure are given.

Acenaphthene derivatives. Imperial Chemical Industries Ltd., Max Wyler and Arnold Kershaw. Brit. 432,885, Aug. 6, 1935. The derivs. are manifold by condensing acenaphthene (I) or 1-5-carboxylic alkylamide with an alkylphenylcarbamyl chloride (which may have a Me or chloro substituent in the Ph nucleus) in the presence of an Al halide condensing agent and, if desired, hydrolyzing the resulting 1-5-carboxylic or -5,6-dicarboxylic alkylamide to yield 1-5-carboxylic or -5,6-dicarboxylic acid, which are dye intermediates, or their anhydrides. Among examples, (1) I and phenylethylcarbamyl chloride are heated in light petroleum with  $\text{AlCl}_3$  to produce 1-5-carboxylic ethylamide, and (2) the product of (1) is hydrolyzed by alc. KOH.

Benzimidazole derivatives. Soc. pour l'ind. chim. A. Bile. Fr. 784,869, July 27, 1935. See Swiss 175,020 (C. A. 30, 3041).

Imidazole derivatives. Adolf Sonn. Ger. 615,227, Oct. 17, 1935 (Cl. 12 p. 9). Imidazoles (4,5-dihydroimidazoles) substituted in the 2 position are prepd. by the action of aliphatic 1,2-diamines on imino ethers derived from aryl, arylkory or carboxyalkyl substitution products of  $\text{HCOOH}$ ,  $\text{AcOH}$ ,  $\text{EtCOOH}$  or  $\text{PrCOOH}$ . Thus,  $\text{PhCH}_2\text{C}(\text{NH})\text{OMe} \cdot \text{HCl}$ , warmed with  $(\text{CH}_3\text{NH}_2)_2$ , yields  $\text{Ph}$ -

$\text{CH}_2\text{C}_6\text{H}_4\text{NCH}_2\text{CH}_2\text{NH}$ , b.  $147^\circ$ , HCl salt m.  $168-70^\circ$ . Examples are given also of the prepn. of imidazoles from  $(\text{CH}_3\text{NH})_2$  and (1) an imino ether prepd. from  $\text{CNCH}_2\text{COOEt}$  (imidazoline m.  $110-12^\circ$ ), (2) salicylic acid imino ethyl ether (imidazoline m.  $209^\circ$ ), (3) an imino ether prepd. from the condensation product of  $\alpha$ -allylguaiacol and bromobutyronitrile (imidazoline b.,

$200-220^\circ$ , picrate m.  $189-90^\circ$ ) and (4) an imino ether prepd. from the condensation product of 8-hydroxyquinoline and  $\text{CICH}_2\text{CN}$ . A product has also been prepd. from propylenediamine and benzimidazole ethyl ether.

Amide derivatives. Henkel & Cie. G. m. b. H. Ger. 616,785, Aug. 5, 1935 (Cl. 12a 16). See Fr. 770,539 (C. A. 29, 4759).

Naphthalene derivatives. Imperial Chemical Industries Ltd. Fr. 785,586, Aug. 13, 1935. See Ger. 609,928 (C. A. 29, 51261).

Phthalic acid derivatives. Chemische Fabrik Grünau Landshoff & Meyer, A.-G. Fr. 785,423, Aug. 9, 1935.  $N,N,N',N'$ -Tetraalkylphthalimides are prepd. by treating phthalyl halides, particularly phthalyl chloride, with dialkyl amines. An example is given of the prepn. of  $N,N,N',N'$ -tetraethylphthalimide, b.  $175-80^\circ$ , m.  $39^\circ$ .

Pyridine derivative. Chemische Fabrik von Heyden A.-G. (Erich Haack, inventor). Ger. 613,402, May 18, 1935 (Cl. 12 p. 101). Addn. to 598,579 (C. A. 28, 7264). The deriv. 4-pyridylpyridinium dichloride is prepd. by treating 2 mols. of pyridine with 1 mol. of Cl in the presence of an indifferent solvent or diluent, and heating the resulting pyridine-Cl compd. with excess of pyridine to  $50-150^\circ$  till the above deriv. is formed. A small amt. of Br may be present in the Cl. Examples are given.

Derivatives of tetrahydro-3-hydroxyquinoline. I. G. Farbenind. A.-G. Fr. 785,541, Aug. 12, 1935. These derivs. are prepd. by treating with epichlorohydrin at above  $130^\circ$ , with or without pressure and in the presence or absence of solvents, alkyl or aralkyl amines of the  $\text{C}_6\text{H}_5$  series in which the position of a substituent in the ring does not prevent closing of the quinoline ring. Examples are given of the prepn. of 1-butyl-3-hydroxy-(b.  $157-9^\circ$ ), 1-ethyl-3-hydroxy-7-methyl-(b.  $171-2^\circ$ ), 1-butyl-3-hydroxy-7-chloro-(b.  $202-5^\circ$ ), 1-benzyl-3-hydroxy-(b.  $227^\circ$ ), 1-butyl-3-hydroxy-8-methyl-(b.  $175-6^\circ$ ) and 1-methyl-3-hydroxy-tetrahydroquinoline, b.  $165-6^\circ$ .

Derivatives of 2-phenylquinoline-4-carboxylic acid. Hauns John. Brit. 433,045, Aug. 6, 1935. Esters of 2-phenylquinoline-4-carboxylic acid (I), its homologs and substitution products are prepd. by esterifying the acids with glycerol (II) or halohydrins of II. When the products contain halogen, they may be caused to react with further quantities of the acids or their salts. The products may be used therapeutically. Among examples, (1) the  $\alpha$ -monoglycerol ester of I is prepd. by heating I with II in the presence of  $\text{H}_2\text{SO}_4$ , or by treating the acid chloride with II or the K salt with  $\alpha$ -monochlorohydrin, and (2)  $\alpha,\beta,\gamma$ -tris (2-phenylquinoline-4-carboxylic acid) glycerol ester is formed when K 2-phenylquinoline-4-carboxylate is heated with  $\beta,\beta'$ -dichloroisopropyl ester of I.

Ester-like compounds. Henkel & Cie. G. m. b. H. (Alfred Kirstahler and Wilhelm J. Kaiser, inventors). Ger. 616,847, Aug. 13, 1935 (Cl. 12a 11). High mol. ester-like compds. are obtained by treating esters obtained from unsubstituted or substituted, sated or unsated, hydroxylated aliphatic, hydroaromatic or aliphatic-aromatic compds. congt. at least 6 C atoms, and aliphatic carboxylic acids (or substitution products) congt. reactive halogen atoms, with *org. hydroxy compds.* in an alk. medium. Thus, the dodecyl ester of chloroacetic acid is treated with  $\text{PhONa}$  and  $\text{PhOH}$  to obtain the dodecylester of phenoxyacetic acid, m.  $33-34^\circ$ . Other examples are given. The compds. are used as soaps and active O-yielding agents.

Condensation products. I. G. Farbenind. A.-G. Fr. 785,458, Aug. 10, 1935. Allophanyl chloride is condensed with aromatic hydrocarbons or their substitution products in the presence of acid-condensing agents. The products may be further treated with acid or alk. agents. Arylureas, arylbiurets, 2-aryl-4,6-diketo-1,3,5-triazines and arylcarboxylic acids are obtained in this way. Examples are given of the prepn. of 2-phenyl-4,6-diketo-, 2-phenyl-4,6-dihydroxy-, 2-tolyl-4,6-diketo- and 2-tolyl-4,6-dichloro-1,3,5-triazine, anisoylurea and 4-methoxybenzenecarboxylic acid.

**Polymerization products.** I. G. Farberind, A.-G. Fr. 785,010, Aug. 22, 1915. Org. compds. contg. a carbon double bond, such as vinyl ether, vinyl ester, acrylic ester, acrylic acid, acrylic nitrile, styrene and isopropenyl methyl ketone, are polymerized in the presence of a small amt. of a polymerizable 1,3-diene, e. g., 2-chloro- or 2-methyl-1,3-butadiene.

**Carbohydrate polymers.** Hatziz (Halyse A.-G. (Edward Jarber, inventor). Ger. 619,215, Sept. 20, 1913 (Cl. 120. 6). Addn. to 618,161 (C. A. 30, 1109). Alkoxes are heated to 160–170° with substantially less than 0.1% of mineral acid until decomposition begins. This may occur after about 15 mins. Water-sol. polymerization products less hygroscopic than those of Ger. 618,161, are obtained. The hygroscopicity of the products may be further reduced by heating them to 100–130° for one or a few hrs., with or without preliminary neutralization of the acid.

**Phenols.** I. G. Farberind, A.-G. Fr. 785,252, Aug. 6, 1915. Phenols are recovered from aq. soln. by a rectifying distn. of the soln., in the presence of CO<sub>2</sub> if NH<sub>3</sub> is also present. App. is described.

**Cyclohexylphenols.** I. G. Farberind, A.-G. (Karl Broderick, Hermann Helnicke and Ernst Korten, inventors) Ger. 616,515, July 31, 1913 (Cl. 120. 25). See 1r. 705,024 (C. A. 28, 67319).

**Halogenated di- and tri phenylmethanes.** The British Thomson-Houston Co. Ltd. Brit. 431,072, Aug. 8, 1935. These are obtained by the Friedel-Craft process by condensing a halogenated benzyl or benzil chloride with a halogenated or unsubstituted C<sub>6</sub>H<sub>5</sub> in the presence of AlCl<sub>3</sub>. In examples, monochlorobenzyl chloride and C<sub>6</sub>H<sub>5</sub>Cl (I) are condensed to give a dichlorodiphenylmethane; dichlorobenzyl chloride (II) and I, and trichlorobenzyl chloride (III) and C<sub>6</sub>H<sub>5</sub>Cl, are condensed to give trichlorodiphenylmethanes, III and I, and II and C<sub>6</sub>H<sub>5</sub>Cl (IV), are condensed to give tetrachlorodiphenylmethanes; III and IV are condensed to give a pentachlorodiphenylmethane; and III and C<sub>6</sub>H<sub>5</sub>Cl are condensed to give a hexachlorodiphenylmethane. The products are noncryst. and consist of mixts. of isomers. The main constituents being those mentioned in the examples, and they are used as dielec., insulating and cooling media in elec. app.

**Alkylenediguanidine salts.** I. Rich Prochnow (to Anticomman G. m. b. H.). U. S. 2,019,872, Nov. 5. In the reaction of an alkylenediamine such as decamethylenediamine with guanidine sulfoxyanate, the materials are boiled together in an aq. soln. in the presence of NH<sub>3</sub> and the alkylenguanidine formed is taken up in solvents such as alc. and the resulting soln. is treated with a hot K<sub>2</sub>CO<sub>3</sub> soln. or the like. Several examples are given. Cf. C. A. 29, 29033.

**Catalysts for hydrogenation.** Anthony M. Muckenfuss (to E. I. du Pont de Nemours & Co.). U. S. 2,021,607, Nov. 19. An aromatic hydrocarbon such as naphthalene, anthracene or acenaphthene, or a lubricating mineral oil fraction from mid-continent crude oil is heated with Na and H under a pressure of 250–500 lb. per sq. in. at 255–450° (for 8–10 hrs. in examples given) to produce catalysts suitable for use in hydrogenation of hydrocarbons such as those from which the catalysts are derived or in hydrogenating other org. compds.

**Mercaptans and thio ethers.** Wm. M. Lee. U. S. 2,020,421, Nov. 12. A secondary or tertiary alkyl ester (such as may be obtained by treating olefin material contg. over 2 C atoms with an O acid such as H<sub>2</sub>SO<sub>4</sub>) is treated with a mercaptan or with H<sub>2</sub>S to produce an alkyl substituted deriv. of the sulphydryl radical contg. compd. corresponding in the C content of the added alkyl substituent to the original olefin. Various examples with details of procedure are given, including the production of *tert*-butyl mercaptan and *tert*-amyl ethyl sulfide.

**Quaternary ammonium salts.** Deutsche Hydrierwerke A.-G. Brit. 437,350, Aug. 13, 1935. Saturated heterocyclic bases are caused to react with alkyl chlorides contg. at least 8 C atoms and the N-alkyl derivs. obtained are converted into quaternary NH<sub>4</sub> salts by reaction with an alkylating agent that introduces an alkyl radical of not more than 6 C atoms. In an example, piperidine is heated with

cetyl chloride and the product is treated with Me<sub>2</sub>SO; the cetyl methylpiperidinium methylsulfate so obtained is useful in making aq. emulsions of paraffin, cetyl alc., olive oil, etc., and the emulsions may be used, as such or after addn. of Na<sub>2</sub>CO<sub>3</sub>, for the treatment of fibrous materials.

**Copper ammonium salt of diazoaminotetrazole.** Willi Brün (to Remington Arms Co.). U. S. 2,021,178, Nov. 19. A soln. of the Na salt of diazoaminotetrazole is mixed with a cupric salt and NH<sub>3</sub> in the presence of HIOAc and in such sequence that no intermediate reaction forms a ppt. The product may form plate crystals or rhombic crystals. Cf. C. A. 29, 43701.

**Selenium-containing anthraquinone compounds.** Ivan Gulchmann and Wm. L. Rinselman (to E. I. du Pont de Nemours & Co.). U. S. 2,019,850, Nov. 5. In the prepn. of a C-dialkylanthraquinonedisulfoxide such as C-diphenyl-1,2,8,7-anthraquinonedisulfoxide, a compd. such as an  $\alpha,\alpha$ -dichloro- $\beta,\beta$ -diminoanthraquinone, in which the amino groups are in opposite rings of the anthraquinone nucleus and the Cl atoms are in ortho position to them, is caused to react with an alkali metal selenide and the product is condensed with an aroyl chloride such as benzoyl chloride (various examples and modifications of procedure being described).

**Addition products of sodium with naphthalene, etc.** Norman D. Scott (to E. I. du Pont de Nemours & Co.). U. S. 2,019,812, Nov. 5. By the reaction of Na with naphthalene (or an alkyl naphthalene in the presence of a suitable reaction medium such as dimethyl ether, methyl isopropyl ether or a mixt. of xylene and dimethyl ether, derivs. are formed such as 1,2- or 1,1-disodium naphthalene, which can be hydrolyzed to dihydronaphthalene or treated with CO<sub>2</sub> and converted into Na salts of dihydronaphthalenedicarboxylic acids. Various examples with details of procedure are given.

**Organic silver compounds.** Karl Roth. Brit. 432,814, Aug. 2, 1935. A H<sub>2</sub>O-sol. complex compd. of Ag acetyl-salicylate with (CH<sub>3</sub>)<sub>3</sub>N, is prepd. by causing acetyl-salicylic acid, or a salt thereof, to react with a Ag salt and treating the pptd. Ag salt with (CH<sub>3</sub>)<sub>3</sub>N. In an example, Na acetyl-salicylate is treated with AgNO<sub>3</sub> and (CH<sub>3</sub>)<sub>3</sub>N, in soln.

**Basic lead trinitroresorcinol.** Hans Tauson (to Winchester Repeating Arms Co.). U. S. 2,020,665, Nov. 12. NH<sub>4</sub>OH is added to a soln. of trinitroresorcinol until the soln. is slightly alk. and a soln. of Pb nitrate is then added.

**Organic acid chlorides.** Wilhelm Stelmann. Fr. 785,075, Aug. 1, 1915. The corresponding anhydrides are treated with COCl<sub>2</sub>, if desired in the presence of metal chlorides or acetates as catalysts.

**Distilling carbon tetrachloride.** Joseph D. Kreis (to I. L. Irazz, trustee for "Dry Cleaning Syndicate"). U. S. 2,020,014, Nov. 12. Various details of app. and a method in which a mist is produced by substantially isothermal compression of vapors of a substance at a temp. below the normal b. p. of the substance are described, consequence of the minute globules of the mist being produced by addn. of satd. vapors of the substance at substantially the normal b. p.

**Aliphatic organic acids.** Gilbert B. Carpenter (to E. I. du Pont de Nemours & Co.). U. S. 2,021,127, Nov. 19. An acid such as HIOAc is formed from CO and an aliphatic monohydroxy alc. such as MeOH or alkoxyl compd. such as MeOAc (usually at a reaction temp. of about 300° and under 700 atm. pressure) in the presence of a catalyst contg. an acid of a metal of group 6-A of the periodic table such as chromic acid or tungstic acid. Cf. C. A. 29, 40277.

**Apparatus for distilling fatty acids.** Clemens Beggell. Ger. 601,800, July 4, 1935 (Cl. 234. 1).

**Synthesis of acetic acid and other organic compounds.** John C. Woodhouse (to E. I. du Pont de Nemours & Co.). U. S. 2,019,751, Nov. 5. HIOAc is produced by bringing MeOH and CO into contact with a metal oxide catalyst such as oxides of Si, Mg, Al, Ti, Zr and W or their mixts., which are adsorbents for gases, and at the same time some

other compds. such as Me formate and Me acetate are formed. A reaction pressure of 250-700 atm is preferred and the temp. may be 150-300°. Cf. C. A. 29, 179°.

Synthesis of aliphatic carboxylic acids such as propionic, butyric and valeric acids. Alfred T. Larson (to E. I. du Pont de Nemours & Co.). U. S. 2,020,589, Nov. 12. An acid is produced from steam, CO and an olefinic hydrocarbon such as  $C_3H_6$ ,  $C_4H_6$  or  $C_5H_6$  in the presence of  $CCl_4$  and active carbon (suitably at a temp. of about 325° under 700 atm pressure).

Reduction of fat acids and their esters. Imperial Chemical Industries Ltd. Fr. 785,718, Aug. 17, 1935. Fat acids and their esters are reduced to the corresponding alcs. by passing a rapid current of H through a strongly agitated mixt. of catalyst and acid or ester, so that the alcs. are removed as they are formed. App. is described in which the H passes in a closed cycle.

1,4-Dihaloanthracene-2-carboxylic acids. Berthold Benert and Robert Held (to General Aniline Works). U. S. 2,019,840, Nov. 5. Ring-closing agents such as  $H_2SO_4$  are caused to react upon a 2',5'-dihaloanthracene-2,4'-dicarboxylic acid which has the 6'-position unsubstituted, at an elevated temp. (suitably about 120-220°).

Adenosinephosphoric acids. Georg Henning, Chem. pharm. Werke G. m. b. H. Ger. 619,455, Oct. 1, 1935 (Cl. 12p. 7.01). In recovering adenosine-phosphoric and -polyphosphoric acids by grinding and extg. horse-flesh, muscle and other suitable animal materials, better yields are obtained by adding an alkali or alk. earth phosphate to the material during grinding. The added phosphate may afterward be converted into adenosine-phosphoric acid by adding acetylenic acid or a salt thereof to the ground material. Sp. processes are described.

Alkyl sulfonic acids. Imperial Chemical Industries Ltd. and Alfred Davidson. Brit. 433,312, Aug. 13, 1935. The conversion of alkyl inorg. esters having 10-18 C atoms into the corresponding sulfonates by means of an aq. soln. of sulfites is effected in a closed vessel at 120-150° in the presence of diarsinodiphosphoric acid (II) or a substituted I or a mixt. of a phosphate and an aromatic base. In examples, cetyl bromide, cetyl Na sulfate, dodecyl bromide and dodecyl Na sulfate are treated with  $Na_2SO_3$  in the presence of a mixt. of  $PhNH_2$  and Na phosphate or of I.

Colloidal dispersions of higher aliphatic anhydrides. Hans von Recklinghausen. Ger. 619,342, Sept. 28, 1935 (Cl. 23c. 2). A higher aliphatic anhydride is dissolved in a restricted amt. of a water-sol. org. solvent at a temp. above 40°, and the warm soln. is sprayed into water which contains a little  $NH_3$  or other base, or to which  $NH_3$  or other base is added after the anhydride soln. has been introduced. The dispersions are intended for the treatment of textiles.

Fat acid nitriles. I. G. Farbenind. A.-G. Fr. 785,622, Aug. 14, 1935. Nitriles are formed by passing vapors of higher fat acids free from OH groups and capable of forming soaps, e. g., oleic acid, coco- or palm-oil fat acid or functional derivs. of these fat acids, at the same time as  $NH_3$ , over a catalyst capable of eliminating water and at a temp. of 320-420°.

Benzoates from phthalates. Alphons O. Jaeger (to American Cyanamid & Chemical Corp.). U. S. 2,020,595, Nov. 12. In transforming a salt of a polycarboxylic acid to that of the corresponding monocarboxylic acid as in the production of Ca benzoate from Ca phthalate, the initial material is heated with a strong inorg. base such as  $Ca(OH)_2$  and  $CaCO_3$  to a reaction temp. (which may be about 420-450°) in a rotary kiln at least partially filled with balls of relatively heavy, heat-conductive material such as steel balls. App. is described.

Benzoates from phthalates, etc. Joseph E. Jewett (to American Cyanamid & Chemical Corp.). U. S. 2,020,596, Nov. 12. A conversion such as the production of Ca benzoate from Ca phthalate is caused by heating the material (suitably to a temp. of about 400-450°)

in the presence of an alc. earth hydroxide such as  $Ca(OH)_2$  and  $CaCO_3$  and while passing through a mass of inert heavy, heat-conductive bodies such as chrome steel balls which are being tumbled without advancement as a mass in a direction transverse to a sinuous path of advance of the material under treatment. App. is described.

Methanol. Deutsche Hydrierwerke A.-G. (Walther Schraut, inventor). Ger. 619,438, Oct. 17, 1935 (Cl. 12o. 5.01). Crude wood spirit is treated in the liquid phase with H or a gas contg. H at a raised pressure, e. g., 60 atm, and at a temp. below 300°, e. g., 100-150°, in the presence of a hydrogenation catalyst. MeOH is then recovered by distn.

Apparatus for making ethylene glycol from ethylene, oxygen and water. Karl E. Skarblom. Ger. 619,195, Sept. 25, 1935 (Cl. 12o. 5.03). Addn. to 561,049 (C. A. 27, 1012).

3. Formaldehyde. Gutehoffnungshütte Oberhausen A.-G. Brit. 433,559, Aug. 16, 1935. This corresponds to Fr. 770,179 (C. A. 29, 480°).

Propylene oxide. Soc. française de catalyse généralisée. Fr. 785,149, Aug. 2, 1935. A mixt. of propylene, O and an inert gas such as N or  $CO_2$  (propylene, air and  $CO_2$ ) are heated to 300-375° in the presence of a catalyst such as Ag, Ag and Au, or Ag, Au and Cu.

4. Drivnyl ether. Randolph T. Major and Wm. L. Rough (to Merck & Co.). U. S. 2,021,872, Nov. 19. Substantially pure drivnyl ether is claimed as a new product,  $b_m$  about 28.3°. It is made by the reaction of fused KOH upon  $\beta,\beta'$ -dichlorodimethyl ether with const. stirring, refluxing in a continuous system, distg. through a condenser trap and maintaining a flow of  $NH_3$  throughout the entire system. App. is described.

5. Diphenyl ether. I. G. Farbenind. A.-G. (Karl Marx and Hans Wesche, inventors). Ger. 616,825, Aug. 5, 1935 (Cl. 12g. 14.04). See U. S. 2,008,987 (C. A. 29, 585°).

Glycol monochlorohydrin. Compagnie de produits chimiques et électrometallurgiques Alais, Froges & Camargue. Fr. 785,170, Aug. 3, 1935. Cl is caused to react on  $CaH_2$  in aq. medium, the formation of insol. Cl products being reduced by eliminating them from the reaction vessel as they are formed.

6. Biacetyl. Lonza-Werke elektrochem. Fab. G. m. b. H. (J. Pritzker, inventor). Ger. 619,348, Sept. 28, 1935 (Cl. 12o. 10). Biacetyl is recovered from mixts. contg. it, particularly from pyrolytic mixts., by treating the mixts. with a soln. of the Ni compd. of  $NH_4OH$ , whereby a ppt. of the known Ni compd. of biacetyl dioxime is obtained. The ppt. is thoroughly washed, dried at about 100°, and treated with dil. acid, and biacetyl is then extd., e. g., with ether. Preferably, the starting material is first neutralized and distd., and biacetyl then recovered from the distillate. The yield may be increased by treating the neutralized starting material or its distillate with a weak oxidizing agent, e. g.,  $FeCl_3$ , whereby biacetyl is produced from acetyl methylcarbinol or other conversion product of biacetyl which may be present in the material. Sp. processes are described.

8. Bornyl oxalates. Lucienne Darrasse and Lucien Dupont (one-half to Leon Darrasse and Egon Elod). U. S. 2,020,769, Nov. 12. In the manuf. of bornyl oxalates by the action of anhyd. oxalic acid on essence of turpentine without the presence of a solvent or condensing agent, the reaction is conducted under continuous agitation at temps. of about 110-125° and the reaction water is eliminated from the liberated vapors by carrying out the operation in the absence of O in an atm. of inert gas such as  $CO_2$  having no action on the pinene.

9. Isochroman and its derivatives. I. G. Farbenind. A.-G. (Hans Buschmann and Richard Michel, inventors). Ger. 617,549, Aug. 23, 1935 (Cl. 12g. 24). Addn. to 614,461 (C. A. 29, 596°).  $\beta$ -Phenylethyl alc. (I), treated with paraformaldehyde and gaseous HCl in the absence of water, yields I chloromethyl ether, which is converted into isochroman (II) by treating it with aq. acids, preferably at about 100°. Sp. processes are described. Sub-

stitution products of I having at least one free  $\alpha$ -position react similarly. Thus, a methyl-II,  $m$ , 128-35°, and a nitro-II,  $m$ , 97-0°, have been prepared from a  $\beta$ -tolylethyl alc. and a nitro-I, resp.

**Quinone.** I. G. Farlenund, A.-G. (Curt Schuster and Franz H. Broch, inventors). *Ger.* 616,544, July 30, 1935 (Cl. 120 10). See *Int.* 779,214 (C. A. 29, 4774).

**Urethans.** I. G. Farlenund, A.-G. (Heinrich Ulrich and Paul Körding, inventors). *Ger.* 619,550, Oct. 11, 1935 (Cl. 120, 17 01). Carbonic or chlorocarbonic esters of higher aliphatic alcs. are treated with mono- or dialkyl, cycloalkylalkyl, or arylalkyl amines containing at least one OH group linked to an alkyl residue. Thus, the carbonic or chlorocarbonic ester of lauryl, cetyl, octadecyl or oleyl alc. may be treated with  $\text{HOC}_2\text{H}_4\text{NH}_2$ ,  $(\text{HOC}_2\text{H}_4)_2\text{NH}$ ,  $\text{E}_2\text{NH}(\text{CH}_2)_2\text{OH}$ ,  $\text{HOC}_2\text{H}_4\text{NHC}_2\text{H}_4\text{OH}$ , or  $\text{PhNHCO}_2\text{H}_2\text{OH}$ . The reaction may be effected at 0-70° in aq. alk. soln. in the presence or absence of an org. solvent. The urethan derivs. so obtained are useful as dispersing agents for oils, fats and waxes and as softening or sizing agents for textiles. Products of similar properties, but more sol. in water, are obtainable by treating the urethan derivs. with an alkylene oxide.

1-Phenyl-2,3-dimethyl-5-pyrazolone Baptist Reuter. *Ger.* 534,908, Aug. 7, 1935 (Cl. 12p 8 01). See U. S. 2,005,605 (C. A. 29, 5130).

Benzoylaminochloroanthraquinones Alexander J.

Wuertz and Wm. Dettwyler (to E. I. du Pont de Nemours & Co.). U. S. 2,019,837, Nov. 5. 1-Benzoylamino-8-chloroanthraquinone and 1,8-dibenzoylaminoanthraquinone are chlorinated in  $\text{PhNO}_2$  or other inert high-boiling solvent and HOAc and in the presence of an acid-binding agent such as NaOAc or KOAc and the chlorination may be controlled to give a 1,8-dibenzoylamino-4-chloro- or 4,5-dichloroanthraquinone in exceptionally pure form and also in the chlorination of 1-benzoylamino-8-chloroanthraquinone to produce either 1-benzoylamino-4,8-dichloro- or 1-benzoylamino-4,5,8-trichloroanthraquinones in substantially pure form and in good yields. By this procedure the introduction of 1 or 2 atoms of Cl into the starting material is readily controlled. The first atom introduced into the 1-benzoylamino-8-chloroanthraquinone enters the 4-position, giving substantially a pure 1-benzoylamino-4,8-dichloroanthraquinone, substantially free from the 4,5,8-trichloro body. Further chlorination gives the 4,5,8-trichloroanthraquinone. So also in the chlorination of 1,8-dibenzoylaminoanthraquinone, a substantially pure mono- $\alpha$ -chloro-1,8-dibenzoylaminoanthraquinone is obtained before any substantial amt. of dichlorination takes place. It is also possible to start with the diamino- or aminochloroanthraquinone which can be first benzoylated in the inert high-boiling org. solvent, and then chlorinated without isolation. Several examples are given.

## 11—BIOLOGICAL CHEMISTRY

PAUL E. HOWE

### A—GENERAL

ARTHUR W. DOX

**Salt activation.** II. Influence of salts on the stability of amylase. K. Venkata Giri. *J. Indian Chem. Soc.* 12, 567-74 (1935); cf. C. A. 29, 6611<sup>2</sup>.—Purified sweet-potato amylase was kept 30 min. at 50° in 0.05 N acetate buffers from  $pH$  4 to 6.5. It was found that solns. containing 0.05 N NaCl also had their activity protected in the lower  $pH$  range but not in the higher. At  $pH$  4 fluoride, chloride, sulfate and nitrate were diminishingly effective in that order (all as Na salts). All but the first showed a max. protective action at about 0.05 N, but fluoride was most effective in the strongest soln. used, 0.2 N  $\text{Li}^+$ ,  $\text{Na}^+$  and  $\text{K}^+$  showed very little difference in their behavior, but  $\text{Ca}^{++}$  was much more efficient in dil. and less in concd. solns.  $\text{Ca}^{++}$  behaved similarly in its effect on the enzyme activity also. The stability of amylase was also reduced by high concns. of salt at higher  $pH$ . The effect of the salts studied on the stability of amylase is thus entirely paralleled by their effect on the activity. R. Baltzy.

**Enzymic proteolysis.** V. The structure of the prothromases. 2. Elucidation of the structure of clupein. Ernst Waldschmidt-Leitz and Ernst Kofranyi. *Z. physiol. Chem.* 236, 181-91 (1935); cf. C. A. 29, 3673<sup>2</sup>.—The recent isolation of protaminase, the enzyme specific for the splitting off of terminal arginine from the carboxyl end of protamine chains furnishes a means of detg. the order of amino acid linkages in the mol. It liberates  $1/4$  of the total arginine from clupein. Since 2 arginines are present for each monoamino acid, there must be at least 15 amino acids in the chain. After protaminase has split off the terminal arginine from clupein, the remaining clupein is further broken down by activated trypsin with rupture of 4 peptide linkages. The products may then be sepd. into 2 fractions: one representing 73% of the total and having a N:NH<sub>2</sub> ratio of 9, the other constituting 27% and having a ratio of 5. The larger fraction is a mixt. of tripeptides, each consisting of 2 arginine and 1 monoamino acid. The smaller fraction consists of 2 dipeptides, each contg. 1 arginine. One proline is present in the tripeptide mixt., none in the dipeptide mixt. The tripeptide fraction is not attacked by aminopolypeptidase, signifying that no monoamino acid with terminal NH<sub>2</sub> is present. One arginine is split off by carboxypolypeptidase; hence

the tripeptides consist of 1 monoamino acid between 2 arginines. From the resulting mixt. only  $1/4$  of the peptide linkages are split by dipeptidase, hence 1 of the 3 must represent tertiary N as in arginylproline. The dipeptide fraction is not attacked by arginase and therefore contains no free arginine. Dipeptidase breaks all of the peptide linkages. Of the 4 possible combinations, arginylarginine and the dipeptide of monoamino acid are ruled out because carboxypolypeptidase splits half of the total peptide linkages. The dipeptides are therefore arginylmonoamino acid and aminoacylarginine and contain no proline. On the basis of these cleavage products the amino acid sequence in clupein is (MA) (AMA) (AMA) (APA) (AM), where A denotes arginine, M monoamino acid and P proline. Activated trypsin therefore splits the peptide linkage between each pair of contiguous arginines in the interior of the polypeptide chain. Linderström-Lang's theoretical deductions are thus corroborated. The specificity of a true protease, like that of peptidases, is detd. solely by the nature and arrangement of the amino acid units, and the assumption of a cyclic structure for proteins becomes superfluous. It is improbable that one and the same enzyme is active toward peptide linkages in both open peptides and diketopiperazines. Adding the 2 arginines split off by protaminase, the amino acid sequence in clupein becomes (MA) (AMA) (AMA) (APA) (AM) (A) (A), the only uncertainty being the exact location of the proline. Clupein, with mol. wt. 2021, cannot be a multiple of this formula. The double mol. wt. obtained by Van Slyke NH<sub>2</sub> titration may be due to salt formation between 2 clupein mols. A. W. Dox.

**The constitution of nucleic acid.** Katashi Makino. *Z. physiol. Chem.* 236, 201-7 (1935); cf. C. A. 29, 3695<sup>2</sup>.—Titration of yeast nucleic acid with NaOH and phenolphthalein shows it to be a tetrabasic acid. This is taken as evidence of the cyclic structure of the mol., in which the ribose portion of each of the 4 nucleotides is esterified to 2  $\text{H}_2\text{PO}_4$  mols. and each  $\text{H}_2\text{PO}_4$  to 2 ribose groupings, leaving an acidic OH on each of the 4 phosphoric acid groups. Depolymerization is due to the rupture of these ester linkages. The resulting increase in acidity is then in close agreement with that calcd. on the basis of the cyclic structure. A. W. Dox.

**Lactic acid dehydrogenase.** Th. Wagner-Jauregg and E. F. Möller. *Z. physiol. Chem.* 236, 216-21 (1935).—Al-

though the dehydrogenation of other hydroxy acids, malic and citric, requires in addition to the specific dehydrogenase and coenzyme the yellow flavin enzyme as H-carrier, this is not true of lactic acid. A lipoamide-free dehydrogenase prep. obtained from *Es. coli* readily dehydrogenated lactic acid in the presence of methylene blue as O-donor and in the absence of coenzyme. The addition of yellow enzyme shortened the decolorization time only very slightly. This slight effect is attributed to coenzyme in the flavin enzyme soln. Neither crystal lactoflavin nor lactoflavophosphoric acid had any such effect. The coenzyme responsible could not have been coenzyme as the boiled soln. inhibited fermentation. Addition of flavin enzyme to dehydrogenase preps. from *Lactobacillus* likewise failed to accelerate the dehydrogenation of lactic acid. Evidently the flavin enzyme of yeast does not participate in the dehydrogenation of lactic to pyruvic acid.

A. W. Dox

Activation of the enzymic dehydrogenation of alcohol by glutathione. Th. Wagner-Jauregg and E. F. Möller. *Z. physiol. Chem.* 236, 222-7 (1935).—Dehydrogenation of EtOH by dehydrogenase preps. from frog muscle and from yeast is activated, as measured by methylene blue decolorization, by SH-glutathione or KCN. The effect is probably a suppression of heavy metal inhibitors. Other substances which bind heavy metals have a similar effect, viz., 0.01 M Na 1-amino-8-naphthol-4-sulfonate, HSC<sub>2</sub>H<sub>4</sub> and Na<sub>2</sub>S. Na<sub>2</sub>P<sub>2</sub>O<sub>7</sub> is without effect, and cysteine inhibits the SH-glutathione activation. The activation is less pronounced when contamination with heavy metals has been avoided in the prep. of enzyme. A function of glutathione, in addition to its activation of certain hydrolytic and dismutative enzymes (papain, cathepsin, arginase, methylglyoxalase) is, in conjunction with heavy metals, the regulation of dehydrogenation processes. A. W. D.

The behavior of urobilinogen in the liver. K. Felix and H. Moebus. *Z. physiol. Chem.* 235, 230-6 (1935).—When urobilinogen is mixed with a pulp of fresh hog liver and the mixt. aerated with air or O<sub>2</sub>, an alteration occurs whereby the substance no longer gives the Ehrlich aldehyde reaction. The optimum pH for the reaction is at 7.1, and the presence of O<sub>2</sub> is essential, since no change occurs in an atm. of N<sub>2</sub>. The diazo and Gmelin reactions are neg.; hence no bilirubin is formed in the oxidation. The process is enzymic and does not occur if the liver has been heated. Probably the point of attack is the CH<sub>2</sub> bridge between the pyrrole rings, since this has been shown by Fisher to be the essential grouping for the aldehyde reaction.

A. W. Dox

Pigments of human fat. L. Zechmeister and P. Tuzson. *Bull. soc. chim. biol.* 17, 1110-18 (1935); cf. C. A. 29, 6088<sup>1</sup>.—Carotene, lycopene, capsanthin and xanthophylls of dietary origin, were isolated from human fat.

L. E. Gilson

Comparative rates of hydrolysis of  $\alpha$ - and  $\beta$ -glycerophosphoric acids by various plant phosphatases II. Takadiastase. Jean Courtois. *Bull. soc. chim. biol.* 17, 1318-39 (1935).—See C. A. 28, 5836<sup>1</sup>. L. E. Gilson

Fixative power of takadiastase toward glycerophosphates. Jean Courtois. *Bull. soc. chim. biol.* 17, 1340-5 (1935).—"Takadiastase, which has different affinities for  $\alpha$ - and  $\beta$ -glycerophosphates, has the same fixative power for the 2 isomers; affinity is a qual. test of an enzyme; fixative power, on the other hand, is a quant. phenomenon."

L. E. Gilson

[Inhibiting] action of reduced glutathione and ascorbic acid on the destruction of adrenaline by the oxidizing enzymes of potato juice. J. Toscano Roco and A. Malafaya Baptista. *Compt. rend. soc. biol.* 120, 45-8 (1935).

L. E. Gilson

Action of glutathione on cytochrome C in vitro. E. J. Bigwood and J. Thomas. *Compt. rend. soc. biol.* 120, 69-72 (1935).—Oxidized glutathione is able to take up the H. mobilized by certain dehydrogenases. The reduced glutathione formed gives up H to oxytocyochrome C, thereby reducing the latter.

L. E. Gilson

Action of hydrogen cyanide on the oxidase of *Althaea rosea*. Vittorio Zanotti. *Boll. chim. farm.* 74, 669-70

(1935).—Leaves of *Althaea rosea* were subjected to HCN for 48 hrs., then dried, macerated and extd. with H<sub>2</sub>O. The action of the oxidase in this ext. on 30% tincture of liguim vitae was compared with that of a similar ext. of noncyanated leaves. HCN inhibited the oxidase without destroying it.

Helen Lee Gruhl

Effect of Röntgen rays on the lipides of the epidermis. U. J. Wile, O. J. Cameron and H. C. Eckstein. *Arch. Dermatol. Syphilol.* 32, 69-72 (1935).—Samples of epidermis taken from 9 cadavers just after death were irradiated and analyzed for change in lipid content. Cholesterol was detd. by the Liebermann-Burchard method, and phospholipids were detd. by Whitehorn's method (C. A. 19, 663). Results showed a loss in cholesterol, in phospholipid and in total lipids.

O. Hartley

Insulin and glycolytic systems isolated from the myocardium of the rabbit and the cat. B. Tamm. *Biochim. therap. exp.* 22, 451-9 (1935).—A glycolytic enzyme was obtained from the myocardium of the rabbit and the cat. The former has an influence on glycogen and to a lesser degree on glucose, the latter acts on glycogen only. Insulin has no influence on the enzymic action.

A. E. Meyer

Chemical structure and biological action. E. C. Dadds. *Cambridge Univ. Med. Soc. Mag.* 12, No. 3, 112-14 (1935).—Thyroid, sex hormones and plant auxins are discussed in a lecture, developing the relationship between chem. constitution and biol. action. J. C. M.

Physiology of muscle protein. E. C. Smith. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 10-22 (1935); cf. C. A. 29, 5191<sup>1</sup>.—From previously published data (C. A. 28, 5087<sup>1</sup>) it would be expected that the protein of the living muscle plasma (intracellular protein) would be readily and completely extd. by salt solns within a wide range of concn. of salt; contrary to expectation, exhaustive extn. with any given salt soln yields only part of the total intracellular protein (C. A. 28, 1119<sup>1</sup>). Dil. HCl exts. the whole of the intracellular protein, including any denatured protein that may be present, leaving behind only the connective tissue. Since the intracellular proteins are completely sol. in all the extractant solns. tested (NH<sub>4</sub>Cl, LiCl, NH<sub>4</sub>NO<sub>3</sub>, NaClO<sub>4</sub>, MgSO<sub>4</sub>, KCl, NaCl), the fact that part is left undissolved

is best explained by assuming that fragments of muscle have escaped grinding, and solvents vary in their power of opening up the connective tissue which surrounds these fragments. The residual protein may be unaltered sol. protein identical with that already extd., or it may consist partly or entirely of protein which has been rendered insol. by denaturation during extn. When muscle is extd. with 0.01 N HCl a considerable proportion of the sol. protein is denatured; denatured myogen is co-pptd. with myosin between pH 4.8 and 8.0, but it remains undissolved when the joint ppt. is treated with buffered neutral salt and can therefore be detd. During the actual process of stiffening during rigor mortis, no change could be detected in the properties of myosin, nor was there any appreciable decrease in the salt-extractable fraction of the proteins. This contradicts the results of previous workers, and the difference is attributed to either the strict maintenance of a low temp. during storage or the absence of antiseptics in the present tests. Tests carried out with the sclerometer failed to reveal any measurable difference in the rate of onset of rigor mortis between 0° and 25°. The absence of a temp. coeff. seems to imply that the process of stiffening is due to a purely phys. change. It would appear that the colloidal system of the plasma is rendered unstable soon after death by the removal of one of the components of the system (possibly phosphagen), after which the system changes at a rate independent of temp. It remains an open question whether this change is of the nature of a sol-gel transformation.

A. Papineau-Couture

The action of x-rays upon the lactate, glucose, citrate and succinate dehydrogenases. R. E. Harward. *Brit. J. Radiol.* 8, 787-92 (1935).—Lactate, glucose, citrate and succinate dehydrogenases were prepd. from yeast, fresh minced liver, acetone-extd. liver and muscle, resp.

by standard methods. The preps. were irradiated with large doses of x-rays generated at 200 kv. and filtered by 0.5 mm. Al. Twenty thousand röntgens of such radiation had no effect on any of the first 3, but produced slight inactivation of the fourth. A similar dose of  $\gamma$ -rays produced the same effect on the succinate dehydrogenase.

**Fifth H. Quimby**  
Present status of the question of the autolysis of living tissue. I. A. Smorodintzev. *Uspekhi Khim.* 4, 632-55 (1935).—A review on the concept of autolysis, the action of enzymes and their specificity, especially protease, and of phys. and chem. factors. The physiol. effects and the products formed by synthesis are discussed. One hundred and fifty-one references.

**The annual organic production and nutrient phosphorus requirement in the tropical western North Atlantic**  
H. R. Seiwel. *J. conseil intern. exploration mer.* 10, 20-31 (1935).—Based on the annual dissolved O consumption of 62 cc. per sq. cm. of surface (H. R. Seiwel, *Papers in Phys. Oceanogr. and Meteor.* 3, 86 (1934)), the annual production of carbohydrate in the western N. Atlantic was calculated to be 0.0035 g. of glucose or 0.056 g. moisture and salt-free plankton per sq. cm. of surface. Analysis of 9 oceanic plankton samples gave an av. value of 49.43% C and 1.35% P with a ratio of 1 part C to  $2.73 \times 10^{-3}$  part P. Based on this ratio the annual dissolved P requirement for photosynthesis was estd. to be  $75.6 \times 10^{-3}$  g. per sq. cm. of surface. In the Barents Sea and English Channel only one tenth to one half as much phosphate is consumed annually. Eddy currents supply much of the consumed phosphate to the photosynthetic zone from the phosphate-rich mid-stratum ocean water.

**The chemistry of chromosomes**  
H. H. Hellström, Knut Brandt and H. v. Euler. *Svensk Kem. Tids.* 47, 219-50 (1935) (in German).—Ultraviolet photomicrographs of *Drosophila* chromosomes are shown in different fluid reagents.

**Researches on the blood serum proteins of some migratory fishes**  
O. Gomez Ibañez and M. Fontaine. *Bull. inst. océanogr.* No. 679, 5 pp. (1935).—Osmotic changes in the blood concomitant with a transfer from sea water to fresh water perhaps does not alter the serum to globulin ratio of the blood.

**The nature of snake poisons**  
F. Michael and K. Kraft. *Nachr. Ges. Wiss. Göttingen, Math.-physik. Klasse III*, [N. S.], 1, 85-93 (1935).—It is believed that snake poisons are not single entities, protein in nature, but consist of prosthetic groups requiring a colloidal carrier. When native snake poison is treated with adsorption agents the prosthetic group is removed. After elution the soln. again shows activity if a protein soln. is added. An ultrafilter through which only a small amt. of poison will pass will permit about 85% of it to pass through if the poison is first partially hydrolyzed with acid. If such hydrolysis is complete enough to destroy most of the toxicity the latter can be restored by adding protein soln. to the hydrolyzate.

**The action of the two amylases of barley**  
Charles S. Haney. *Can. J. Research* 13B, 185-209 (1935).—Expts. confirmed the view that  $\beta$ -malt amylase selectively hydrolyzes one portion of the starch substance, this fraction (approx. 60% of the starch substance) is transformed into maltose and a residual nonreducing fraction remaining (erythrogranulose) which retains the property of chlorination by I. With regard to  $\alpha$ -malt amylase, several lines of evidence indicate that this enzyme does not exert a selective action upon the erythrogranulose fraction, as is suggested in a current theory of starch constitution, but that it induces the breakdown of both fractions. In this case the reducing products do not consist exclusively of maltose.

**Serum proteins in parathyroidectomized animals**  
Andor Illényi. *Magyar Orvosi Arch.* 36, 257-69 (1935).—The protein fractions of serum remain unchanged in animals in which the parathyroid has been extirpated.

**The effect of ultraviolet radiation on lens protein in the presence of salts and the relation of radiation to industrial**

and senile cataract. Janet H. Clark. *Am. J. Physiol.* 113, 638-47 (1935).—Fts. of lens protein (from ox lenses) were made in solns. of NaCl, KCl and CaCl<sub>2</sub>. They were exposed to ultraviolet radiation at  $\mu$  1-7.8 and the development of opacity was followed by the intensity of the Tyndall beam from the opalescent soln. At  $\mu$  7.2 moderate heat alone produced no opacity and ultraviolet radiation at 4', although denaturing the protein, produced no opacity except to a slight degree in the presence of Ca. If the radiated exts. were heated to 40° for 2 hrs. the opacity was very marked in all the exts. contg. Ca. The effect was diminished by the presence of Na but not by the presence of K. It is probable that there is always some denatured protein present as a result of exposure to sunlight. This does not ppt. in the presence of K but a Ca concn. as low as 0.03% in the lens is sufficient to cause pptn. at body temp. An accumulation of denatured protein in the lens in old age combined with a higher blood Ca may therefore be responsible for senile cataract. The higher incidence of cataract in industrial workers is probably caused by exposure to large sources of radiant heat when low concns. of Ca or other substances producing a similar effect are present.

**The effect of light and of darkness on the thyroid gland of the rat**  
H. S. Maycrson. *Am. J. Physiol.* 113, 659-62 (1935).—Rats kept in the dark, whether born and reared or placed there at weaning, when fed a well-balanced diet, show no differences in rate of growth, body wt. or in the microscopic structure of the thyroid gland in comparison with rats kept in diffuse roomlight or exposed to direct sunlight. C or quartz Hg vapor arc radiation. Darkness does not enhance, and radiation does not perceptibly modify the hyperplasia produced by feeding goitrogenic diets.

**Physicochem. studies of the colloidal state of cholesterol, cholesterol esters and lecithin**  
(Remezov, Zepalova) 2

## B—METHODS AND APPARATUS

STANLEY R. DUNFORD

**Two types of modified Petri dishes**  
C. G. Dobbs. *Ann. Botany* 49, 852-4 (1935).—Edge infection can be prevented by a double wall so constructed as to form a "moat" around the dish. Type B is a rectangular dish suitable for use on the stage of a microscope.

**Comparative investigations on colorimetric and lodometric determination of glutathione**  
Kurt Uhlenbrock. *Z. physiol. Chem.* 236, 192-6 (1935).—The colorimetric method is essentially the Na<sub>2</sub>Fe(CN)<sub>5</sub>NO reaction used by Blerich and Rosenbohm (*C. A.* 27, 2165) except that sulfosalicylic instead of tungstic acid is used for removal of potassium, the soln. is made alk. with Na<sub>2</sub>CO<sub>3</sub> instead of KCN, and a photoelec. colorimeter is used because of its greater rapidity in measuring the fugitive color. Dets. of glutathione in numerous tissue exts. showed in most cases good agreement with the lodometric method.

**Phosphatase determination in small quantities of serum**  
Ernst Möller. *Z. physiol. Chem.* 237, 35-9 (1935).—The superiority of serum phosphatase detn. over serum P and Ca dets. for the diagnosis of rickets has led to the development of this micromethod. One-half cc. of serum is mixed with 0.5 cc. of 0.015% Na  $\beta$ -glycerophosphate. For the blank detn. a 4-cc. aliquot is treated with 1 cc. of 20% CCl<sub>3</sub>CO<sub>2</sub>H and centrifuged, then 2 cc. of the supernatant liquid transferred to a 4-cc. volumetric flask and treated successively with 1 cc. standard KH<sub>2</sub>PO<sub>4</sub> (1 mg. %), 0.5 cc. (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> soln. (25 g. (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> and 300 cc. N H<sub>2</sub>SO<sub>4</sub> per l.), 0.4 cc. of 2,4-(H<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH (0.5 g. concn. amolol in 100 cc. 5% Na<sub>2</sub>SO<sub>3</sub>) and dild. to the mark. After 10 min. the photometer reading is taken. For the phosphatase detn. 2 cc. of the glycerophosphate mixt. is treated with 1 cc. of barbitol buffer ( $\mu$  7.8), the flask is stoppered and placed in a thermostat 48 hrs. at 38°. The soln. is pptd. with 2 cc. of 20% CCl<sub>3</sub>CO<sub>2</sub>H and centrifuged or filtered. Two cc. of the clear filtrate (0.04 cc. serum) is treated with standard phosphate, (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> and amolol, exactly as in the blank, and the photometer reading taken. The P liberated is calcd. from the equa-

1936.  $C_2 = K_2 (A_1/100)$ , where  $C_2$  is mg. P in the unknown soln.,  $A_1$  the extinction coeff. of the unknown soln., and  $A_2$  the  $A$  value of the standard phosphate soln.

A photometric method for the determination of  $Cr_2O_3$  in digestibility experiments by the so-called quantitative indicator method. Lauri Paloheimo and Irja Paloheimo. *Biedermanns Zeits. B. Tierernähr.* 7, 317-24 (1935).—See C. 1 29, 645 P.

Ondase test in avian pathology. Charles S. Gibbs and L. W. Chapman. *Mass Agr. Expt. Sta. Bull.* 315 (Ann. Rept. 1934), 78 (1935).—The oxidase test is of value in differentiating leucemic from nonleucemic avian blood. The method of Sato and Sekiya (C. A. 20, 2515) was superior to other methods from the standpoint of keeping quality of the stain and the rapidity of staining.

Micromethod for the determination of urea in blood or urine. Levinson. *Bull. soc. chim. biol.* 17, 1157-62 (1935).—Blood is deproteinized with  $ZnSO_4$  and  $NaOH$ . A known quantity of  $NaOBr$  is added to the blood filtrate or urine and the excess remaining after reaction with the urea detd. iodometrically.

The determination of the carotenoid contents of blood, tissues and fluids in daily clinical practice. P. Ratchevsky. *Bull. soc. chim. biol.* 17, 1187-93 (1935).—For blood, absorb 0.1-0.4 cc. on cotton, weigh and treat with 2 cc. 95%  $EtOH$  for each 100 mg. blood. Add 2 cc. petr. ether, shake well and add enough water to cause sharp sepn. of the petr. ether layer. Contg. the carotenoids. 1 vial successive drops of the petr. ether soln. on the same spot in a porcelain dish until a barely visible yellow ring appears and note the vol. required. This is equiv. to 0.03-0.05  $\gamma$  carotene or about twice as much xanthophyll. Bile, urine and spinal fluid are concd. by evapn. and treated as blood. Tissues are ground with a little sand and  $KOH$  soln. and then extd. with  $EtOH$  as above.

Physicochemical study of the dispersion of readings in colorimetry. R. Dolique. *Bull. soc. chim. biol.* 17, 1304-17 (1935).—Errors in colorimeter reading are discussed.

Colorimetric determination of polypeptides by the triketohydrindene hydrate reaction. Michel Polonovski. *Compt. rend. soc. biol.* 120, 28, 94 (1935); cf. C. A. 29, 6172. —The first step toward the formation of the blue color is the oxidation of  $RCH(NH_2)CO_2H$  to  $RCHO$ ,  $NH_2$  and  $CO_2$  by the ninhydrin. The hydrated ketone group of the latter is reduced to a carbinol group. When the  $H$  in the  $\alpha$ -position is replaced by an alkyl group as in synthetic  $\alpha$ -aminoisobutyric and  $\alpha$ -amino- $\alpha$ -ethylpropionic acids the reaction is impossible and these compounds do not give the blue color. In the method described by Cherbitzer and Herzenstein (C. A. 29, 1449, 7369) where the reaction is carried out in a soln. satd. with  $(NH_4)_2SO_4$ , glycylglycine and glycylleucine give the blue color while alanylalanine, leucylglycine and leucylglycylglycine do not. From this it is inferred that only peptides contg. a glycid radical with a free amino group will give the reaction under such conditions.

Colorimetric determination of urea. Juan A. Sánchez. *Rev. centro estd. farm. bioquím.* 25, 304-72 (1935).—See C. A. 29, 8037.

Determination of phosphorus in feces. Corazza Maria. *Arch. farmacol. sper.* 60, 386-8 (1935).—The org. matter is destroyed by the Fringsheim and Liebig methods and P detd. volumetrically by the Pincus method. Both methods give comparable results.

Determination of mercury in viscera. Clive Newcomb, S. Rajagopal Naidu and K. S. Varadachari. *Analyst* 60, 732-5 (1935).—The viscera are digested with portions of  $HNO_3$  until finally a uniform yellow soln. is obtained.  $H_2SO_4$  is then added and the soln. heated until white fumes are evolved. During this heating, air is passed through the soln. and the distillate is passed through water in receivers. In this way about  $1/2$  of the  $Hg$  is distd. off. The heating is stopped and the contents of the receivers are treated with  $KMnO_4$  to oxidize  $HNO_3$  and

then decolorized with oxalic acid. The  $Hg$  is then pptd. as  $HgS$ . A fresh supply of water is placed in the 3 receivers and the heating repeated but this time the back end of the distg. flask is connected with a flask contg. concd.  $HCl$  so that when suction is applied at the front end, a stream of  $HCl$  gas is made to pass through the  $H_2SO_4$  soln. contg.  $Hg$ , which is heated nearly to the b. p. of the acid. In this way the remaining  $Hg$  is distd. off as  $HgCl_2$ . The aq. soln. contg.  $HgCl_2$  is treated with  $H_2S$  and the remainder of the  $Hg$  pptd. The entire  $HgS$  ppt. is dissolved in  $H_2O$ , water, to remove excess  $S$ , and the  $Hg$  is pptd. a second time as  $HgS$  which is filtered off, dried and weighed. In this way the recovery of the  $Hg$  in synthetic samples was nearly perfect. Arsenic does no harm because it is oxidized to the highest state and no  $AsCl_3$  is formed.

Determination of the calcium and phosphate contents of bones. C. K. Deisher and W. M. McNabb. *Analyst* 60, 750-1 (1935).—In the study of bone obtained from excavations a check-up was made of the accuracy of the volumetric detn. of both Ca and phosphate. The basic acetate sepn. prior to the Ca detn. was compared with the direct detn. without removal of phosphate by means of an excess of oxalic acid and  $NH_4$  oxalate. The former method gave low results when only one basic acetate sepn. was used but the direct oxalate pptn. gave good results, which were, however, high when 6.5% of  $Mg$  was present. The alkalimetric method for titrating the  $NH_4$  molybdate ppt. gave high results if the titration was carried out in a hot soln. but was more satisfactory in the cold.

Clinical spectroscopy. Advantages and physical principles of the spectrograph and technique in taking specimens for biospectrometric analysis. L. Edward Gaul and A. H. Staud. *Arch. Dermatol. Syphilol.* 32, 385-93 (1935), cf. C. A. 29, 223.

State and localization of inorganic salts in the skin as revealed by extraction and microincineration. D. J. Koovman. *Arch. Dermatol. Syphilol.* 32, 394-403 (1935).—Differences in microscopic appearance occur when ash is extd. with org. solvents or water before incineration, as compared with unextd. material. Their significance is discussed.

The determination of alanine in biological materials. E. W. McChesney. *J. Elisha Mitchell Sci. Soc.* 51, 147-50 (1935).—The method of Kendall and Friedemann, cf. C. A. 24, 5777, for the detn. of alanine is unsatisfactory. The conversion of alanine to  $AcH$  either by their procedure or any of a no. of modifications reaches an equl. at about 91% of completion. If no method can be found for the quant. conversion of alanine into lactic acid, a 9% correction must be added to the results obtained by the K and F method. The presence of certain other amino acids, such as valine and leucine, interfere with the detn.

A. L. Mehning. The determination of total carbon in urine. Modifications of Dumas's method. Norberto E. Insua. *Rev. sudamericana endocrinol. inmunol. quimioterap.* 18, 609-17 (1935).—Drying *in vacuo* over  $H_2SO_4$  causes a loss of C of about 10%, at 37° of 16%. Drying at 57-60° causes evapn. of the total  $Me_2CO$  but because of the short time required (2-4 hrs.) no other loss occurs. Detn. of  $Me_2CO$  in the original sample and of the C in the dried residue gives accurate results.

A. E. Meyer. The reliability of clearance tests for renal efficiency. Cuthbert Leslie Cope. *Clin. Sci.* 2, No. 1, 35-42 (1935).—In tests on nephritics, samples of urine and blood were analyzed at hourly intervals after the oral administration of a soln. of either 15 g. of urea, 3 g. of creatinine or 30-50 g. of xylene. Good agreement was obtained by the 3 test methods.

James C. Munch. A proposed device which is capable of continuously indicating the approximate percentage of carbon dioxide in a stream of flowing gases. Wm. B. Draper and Bernard B. Longwell. *Colorado Med.* 32, No. 11, 879-80 (1935).—Anesthetic gases are bubbled through aq. soln. of bromocresol purple or methyl red. The  $CO_2$  content is estd. from the color change. Concn. of 0.3% produces a

yellow color with bromocresol purple. With methyl red a 1%  $\text{CO}_2$  concn is a pale pink color, 3% light red and 5% deep red, suggesting the need for replacement of soda lime. Soda lime dust is very irritant and should be removed from the anesthetic gas. J. C. M.

A portable thermoelectric couple for measuring skin temperature. R. T. Grant. *Gow's Hospital Rep.* 85, No. 2, 209-11 (1935).—A Cu-constantan thermocouple is described. James C. Munch

Yellow phosphorus from bones. G. H. Locket. *Schweizer. Z. Naturf.* 17, 541-7 (1935).—Student lab. prep. O. Keimnuth

A simple respiratory apparatus. Josef Gyurik. *Magyar Orvosi Arch.* 36, 240-2 (1935). Henry Tauber

## C—BACTERIOLOGY

LAWRENCE H. JAMES

Infection by, and resistance to, the Preiss-Nocard bacillus. IV. Notes on the toxin, the pyogenic action, and the lipid content of the bacillus. I. B. Bull and C. G. Dickinson. *Australian Vet. J.* 11, 129-38 (1935).—The most potent toxin was obtained when the organisms were grown as a pellicle in 250-ml. Erlenmeyer flasks containing 125 ml. fermented hormone broth plus 0.2% glucose, at  $\text{pH}$  7.7, and in an atm. concn 18% of added  $\text{CO}_2$ . Incubation was at 37° for 48 hrs. The addition of reducing substances, notably  $\text{NaHSO}_4$ , to the toxin increased its stability. The addition of 0.4% of colloidal Fe to the toxic broth filtrate completely precipitated the toxin. Potassium failed to do so. No evidence was found of the presence of chitin in the organism. Successive extractions with acetone, ether, alc., boiling alc. and petroleum ether removed lipid equal to 15.0% of the dry wt. When the residue was submitted to alk. hydrolysis, a further 15.51% of the extractable lipid was removed. K. C. Beeson

The kinetics of the decomposition of glucose and lactic acid by small bacterial masses. The idea of minimum active mass. Paulette Chail. *Compt. rend.* 201, 626-8 (1935).—In experiments with 3 species of propionic bacteria there was a min. mass of the bacteria which could attack glucose and lactic acid, the speed of the reaction was proportional to the mass and lactic acid was attacked by smaller masses than glucose. Rachel Brown

The influence of *Phytomonas tumefaciens* and *Phytomonas rhizogenes* on the actual activity of certain liquid and agar substrata. A. R. Wilson. *Phytopathology* 25, 634-63 (1935).—The changes in  $\text{pH}$  value induced by the hairy-root and by both pathogenic and nonpathogenic crown-gall bacteria on agar media were more rapid than those found in corresponding liquid media. E. C. B.

Artificial enhancing of the virulence of BCG vaccine. Franz Gerlach, Joseph Brisch and Mikhael Kaplan. *Z. Tuberk.* 72, 21-7 (1935).—All repetition of previous recorded expts. revealed the stability of the virulence of BCG vaccine. Cultivation on hematin-egg mediums of  $\text{pH}$  7.1, 6.8 and 6.6, and a hematin content of 3-5% revealed no virulence change even after 7 successive passages on these mediums. The addition of alurate to the suspensions and intraperitoneal injection showed no change to increased pathogenicity. The infectious of organs suspended in salt soln. were without avail. The use of 5%  $\text{H}_2\text{SO}_4$  for 3, 10 and 20 min. before inoculation did not alter virulence. The bacilli remained localized and benign. Deep cultures on Resredka bouillon treated with  $\text{H}_2\text{SO}_4$  before and after 4 months' incubation and likewise after 16 months showed no increase in virulence. Even 10 and 25% beef and guinea-pig hematin added to Resredka bouillon caused no increased virulence in BCG cultures over 5 to 6 months' culturing. H. J. C.

The chemistry of the tubercle bacilli. XL. The occurrence of sterols in tubercle bacilli. R. J. Anderson, R. Schoenheimer, J. A. Crowder and F. H. Stodola. *Z. physiol. Chem.* 237, 40-5 (1935).—In a search for sterols in the lipid fractions of tubercle bacteria only minimal traces were found, and these occurred in the  $\text{Me}_2\text{CO}$ -sol. fat and in the crude wax fractions. The amounts were too small to warrant any assumption that

sterols are metabolic products of tubercle bacteria. The slight traces of sterol-like substances found might easily be impurities introduced in the process of handling. Long's synthetic medium, on which the bacteria were cultivated, contained asparagine and glycerol, and it is possible that one or both of these contained minimal traces of sterols. Contamination with sterols might also arise from the cork and rubber stoppers, cotton plugs, filter paper, and the hands during contact with glassware, filter paper, etc. In view of the many possibilities of contamination with natural sterols in working up large quantities of cultures, it is surprising that greater sterol contamination did not occur. Contrary to Hecht (*C. A.* 29, 2902), it is concluded that the tubercle bacillus contains no sterols. A. W. Day

Serological differentiation of hemolytic streptococci of human and animal origin. P. R. Edwards. *Ky. Agr. Expt. Sta. Research Bull.* 356, 3-14 (1935).—These strains can be differentiated by precipitin tests in which acid extracts of the organisms are used as antigens. Buchheim tests check the precipitin results in differentiating human from animal streptococci. C. R. Teller

Bacteriostatic action of dyes with Gram positive cocci. J. E. Fuller and Morrison Rugosa. *Mass. Agr. Expt. Sta. Bull.* 315 (two Repts. 1934), 22 (1935).—Basic fuchsin exerted the most pronounced bacteriostatic effect on staphylococci, and hemolysis and nonhemolytic streptococci, sarcinae and micrococci, followed by crystal violet and gentian violet in the order named. Acid fuchsin had little bacteriostatic activity. Of the organisms studied, streptococci were most resistant to dyes. High acid production by bacteria seems to be correlated with resistance to dyes. C. R. Teller

Variation of *Salmonella pullorum*. Henry Van Roekel. *Mass. Agr. Expt. Sta. Bull.* 310, 40 pp (1935).—The behavior of 163 strains was studied culturally, biochemically and morphologically. Freshly isolated variants in NaCl solns. of different concns and in the presence of pos. and neg. sera differed greatly. Such properties as colony and cellular morphology, Gram-staining, and biochem. reactions revealed little or no variation in most cases. The power of the variants to attack fermentable substances was practically the same as that of the normal type. In general, the agglutinogenic power and the absorptive capacity of the variants tested were not equal to those of the normal type. C. R. Teller

Sensitivity of *Hemophilus pertussis* to vitamin C and hydroquinol. O. Grooten and N. Bersonoff. *Compt. rend. soc. biol.* 120, 121-3 (1935).—*Hemophilus pertussis* is killed by 1000-5000 mc. ascorbic acid per l. and growth is inhibited by 10-100 mc. per l., depending on the age and other conditions of the culture. Reduction is almost without effect. Hydroquinolone is over 10 times as effective as ascorbic acid. Tyroacetal and pyrogallol are somewhat less effective than hydroquinone. L. R. G.

Action of intestinal bacteria on esculin agar medium. A. Rochaix. *Compt. rend. soc. biol.* 120, 217-18 (1935).—R. confirms the work of Jancz, Cressner and Morel (*C. A.* 29, 7373) except that no blackening was obtained with Strong's bacillus. *Proteus X-19* caused blackening of the medium; *Proteus vulgaris* did not. L. R. G.

Investigation of the action of microorganisms on milk by the "milk-on-agar" culture. Constantino Gorini. *Milch. Forsch.* 17, 87-98 (1935).—Milk-on-agar plates were used to detect the production of chymase and protease by 115 cultures of pathogenic streptococci, 33 cultures of mastitis streptococci, 22 cultures of *Esch. coli*, 10 cultures of *Staph. aureus*, 22 cultures of *Staph. epidermidis* and 8 physiological variants of *E. coli*. The method was very satisfactory for detecting the coagulating and peptonizing abilities of these organisms and proved helpful in determining relationships or differences between species and strains. H. Maer

Influence of salt on the growth and cell form in case of lactic acid bacteria. *Es. coli*, *A. aerogenes* and some other common milk bacteria. W. Henneberg and Hanna Kniefl. *Milch. Forsch.* 17, 146-57 (1935).—The effect of the concn of NaCl on the growth and morphology of



68 cultures of common milk bacteria was reported. In most cases, milk, lactose agar or china blue lactose agar was used as the substrate. The individual species and strains were influenced in different ways by the salt concn.

**Purification of suspensions of the virus of vaccinia by means of carbon dioxide.** C. A. Behrens and F. A. Nielsen *Proc. Indiana Acad. Sci.* 44, 100-117 (1934), cf. C. A. 29, 1454.—A method of purifying vaccinia vaccine has been developed, which is based upon the isoelectric ptn of the suspended tissue by means of  $\text{CO}_2$ . These purified preps are  $\text{H}_2\text{O}$  clear and contain a high percentage of virulent virus.

W. J. Peterson

## D—BOTANY

THOMAS G. PHILLIPS

The effects of animal hormones on plants. László Havas and John Caldwell *Ann. Botany* 49, 729-47 (1935).—Various glandular exs. were administered to plants either by the roots or by the petiole stumps. A prep. of estrogenic hormone was found to be toxic to tomato plants. But even concd exs. of ovary, testis, pituitary, suprarenal, thyroid and of thymus appeared to have no marked effect and certainly no toxic action. In a few instances as with exs. of the suprarenal, there was slight evidence of increased flower development in hyacinths and tomato.

R. C. B.

The study of the effect of blue-violet rays on photosynthesis. R. H. Dastur and R. J. Mehta *Ann. Botany* 49, 809-21 (1935).—An increased production of carbohydrates was observed in leaves of plants exposed to a gas-filled elec. lamp of the "daylight" type as compared with leaves exposed to an ordinary gas filled elec. lamp. The former is richer in the blue-violet rays than the latter. With sunlight and with red and blue-violet rays all of equal intensity, the formation of carbohydrates was found to be greatest in the sunlight. Both the red and the blue-violet region seem to be necessary for normal photosynthetic activity.

R. C. Burrell

Effect of certain chemicals on the "combination streak" virus of tomatoes. Michael Shapovalov *Phytopathology* 25, 864-74 (1935).—A great variety of org. and inorg. compds. were tested for lytic effect on the streak virus of tomatoes. Some affected one component of the streak but not the other, others, both components, and still others were without effect on either component.  $\text{H}_2\text{SO}_4$  of about 1.5% concn. was highly toxic to both components of the virus.

R. C. Burrell

Chemical studies on the virus of tobacco mosaic. IV. Some effects of different chemical agents on infectivity. W. M. Stanley *Phytopathology* 25, 899-921 (1935); cf. C. A. 28, 2837.—The effects of 110 chemicals on the infectivity of purified preps of tobacco mosaic were detd. Most of the agents effective in controlling the virus are oxidizing substances, protein precipitants or agents known to cause  $\text{pH}$  changes. V. Determination of the optimum hydrogen ion concentration for purification by precipitation with lead acetate. *Ibid.* 922-30.—The optimum  $\text{pH}$  for the Pb subacetate ptn. was about 9, for the neutral Pb acetate ptn., 5.5, and for the elution of the virus from the neutral Pb acetate ppt., about 7.

R. C. B.

Effect of tannic acid on the infectivity of tobacco mosaic virus. H. H. Thornberry *Phytopathology* 25, 931-7 (1935); cf. C. A. 29, 5878.—Inhibition of tobacco mosaic infection on plants treated with tannic acid before inoculation was proportional to the concn. of the acid. Tannic acid at concns. of 0.01 to 10% had but slight effect upon infection when applied to plants after inoculation. In virus suspensions the inhibiting effect of tannic acid depended upon the concn. of the acid and the time of action.

R. C. B.

Particle diameter of certain plant viruses and *Phytomonas pruni* bacteriophage. H. H. Thornberry *Phytopathology* 25, 938-46 (1935).—Thirteen plant viruses and virus strains in nonpurified samples were estd. by ultrafiltration analysis to have diam. of about 15 m $\mu$ . Purified tobacco virus and particles of *Phytomonas pruni* bacteriophage were estd. to be about 11 m $\mu$  in diam.

R. C. Burrell

Toxicity of low concentrations of ammonia to mycelium and sclerotia of *Sclerotium rolfsii*. L. D. Leach and A. E. Davey. *Phytopathology* 25, 957-9 (1935).—Aq. solns. of  $\text{NH}_3$  in concns. as low as 50 p. p. m. of  $\text{NH}_3$  for exposures of 24 hrs. have been found lethal to the mycelium of *S. rolfsii* in lab. tests. Increasing the concn. of  $\text{NH}_3$  to 150 p. p. m. reduced the necessary exposure to 2 hrs.

R. C. Burrell

Diffusible nature of the inhibitory agent produced by fungi. J. Cedric Carter. *Phytopathology* 25, 1031-4 (1935).—Certain combinations of microorganisms exhibit a sp. antagonistic action toward one another. Stale agar produced by the growth assoc. of *Helminthosporium sativum* and bacterium 9a2 will inhibit the further growth of *H. sativum*. The inhibitory agent is thermostable and diffusible.

R. C. B.

The dependency of the result of a graft on the relationship of stock and scion. Karl Süberschmidt. *Z. Botan.* 29, 65-137 (1935).—The physiol. marks of the growing together of the parts of a graft were the accumulation of nitrogenous compds. in the scion and an increase of proteolytic activity in the press juice from the stock. The proteolytic enzyme of the stock, however, in grafts involving unrelated plants may not act on the protein elaborated by the scion.

R. C. Burrell

Chemical investigations of the tobacco plant. V. Chemical changes that occur during growth. Hubert B. Vickery, Geo. W. Pucher, Charles S. Leavenworth, Alfred J. Waleman and Laurence S. Nolan. *Conn. Agr. Expt. Sta. Bull.* 374, 557-619 (1935), cf. C. A. 28, 571.—A rapid accumulation of a high relative proportion of oxalic acid in the very young tobacco plant was found. This is suggestive of an extremely rapid rate of metabolism in these plants. The 3 chief acids present, malic, citric and oxalic, maintained a nearly const. ratio to one another in the leaves from the 40th to the 110th day, although the total quantity of org. acids present increased about 400% in the interval between 40 and 75 days, and then sharply decreased. The metabolism of these 3 acids is closely related, and oxalic acid shares with the other 2 acids proportionately in the chem. changes. No definite evidence connecting org. acid metabolism with either carbohydrate or protein metabolism was obtained. The predominant malic acid was followed by oxalic and then by citric in order of quantity present. The amt. of unknown acids was intermediate between the oxalic and citric acids. In the stems, the unknown acids predominated, malic and oxalic being present in considerably smaller amts.; citric acid was invariably present but in very small amts. In the pods also, the unknown acids predominated, malic second in quantity, and followed by traces only of citric and oxalic acids. The amide N results suggest that in the leaf of the tobacco plant asparagine and glutamine are the only amides present. On the other hand, the stem tissue contains a considerable proportion of an unstable amide-like substance in addition to asparagine and glutamine. The growth of the plant is divided into 3 stages, e. g., to 3-4 weeks, when the seedling is establishing itself, from the 5th to 10th weeks, a period of rapid growth, and the 3rd or reproductive period which begins after 9-10 weeks and which is characterized by a decrease in wt. of the leaves and an increase in both org. acids and ash in the leaves and stems. In the 1st stage the nitrate N dropped markedly, the  $\text{NH}_4$ , amide and amino N diminished, and the nicotine N increased. The dry matter, org. acids, ash, carbohydrates and N in all forms increased in abs. quantity per plant, but the relative distributions of the org. acids, and of the forms of carbohydrate and of N underwent considerable changes. Malic acid diminished, and oxalic and citric acids increased. The proportion of the total sol. carbohydrate as fermentable carbohydrate diminished. In the second period of rapid growth, the individual org. acids remained remarkably const. in both leaf and stem. In general, the nitrate content of the leaf decreased while that of the stem increased. The

nicotine in the leaves increased but that in the stem remained const. Both amides and  $\text{NH}_3$  in stem and leaf increased. Apparently the total N of the plant decreased in the 3rd stage or mature period. Similarly, nitrate, amide and ammonia N decreased, while nicotine N increased in the leaves but remained const. in the stems. In this final stage of growth there was a striking translocation of org. and inorg. substances from other parts of the plant, particularly the leaves into the developing seed pods. Approx.  $\frac{1}{2}$  of the org. solids of the plant was ultimately located in the fruit, and nearly  $\frac{1}{4}$  of this was  $\text{Et}_2\text{O}$ -sol. material, mostly true fat. There was a marked storage of N in the seed pods amounting to approx.  $\frac{1}{4}$  of the entire N of the plant. Much of it is probably in the form of seed protein. The picture is one of transformation of the plastic materials of the leaves and to a lesser extent of the stems into stable reserves of nutriment for the succeeding generation. The recently reported results of Vladescu are discussed (C. A. 29, 2999). C. R. F.

Isolation of stachyose from peas (*Pisum sativum*). G. Tanret. *Bull. soc. chim. biol.* 17, 1235-6 (1935).—Five kg. of ground dry peas was extd. with boiling alk. and the sugars in the ext. were sepd. by treatment with  $\text{CaO}$ ,  $\text{BaO}$ ,  $\text{SrO}$ ,  $\text{CO}_2$ , etc. About 2 g. of pure cryst. stachyose,  $[\alpha]_D^{20} = +131.5^\circ$ ,  $n_D^{20} = 1.72-3$ , and an addnl. quantity of crude sirupy product was obtained. The peas contained about 3.5% sucrose, 1.7% stachyose and a very small quantity of reducing sugar. L. E. G.

Changes in the chemical composition of developing apples. H. O. Askew. *J. Pomology Hort. Sci.* 13, 232-46 (1935).—Jonathan and Dunn's Favorite varieties were examd. weekly. By the use of I to stain the starch granules in the flesh of the fruit it was shown that, in the 1st sample, traces only of starch were present. Starch formation begins in the layers of tissue near the periphery and progresses toward the core area. With maturing fruit the core areas were the first to be cleared of starch and the last starch to be hydrolyzed was that contained in the cells near the periphery. N and ash (%) decreased steadily throughout the exptl. period according to the equation  $\log C = a + bI$ , where C was the concn. of the constituent concerned, a and b were consts. and I was the time in days from the taking of the first sample. The initial concns. and the rates of fall of these constituents varied with the variety and with the growth conditions of the orchard. The rate of accretion of dry matter, and of intake of N and ash decreased after about 95 days from the date of max. blossom, slightly in the case of dry matter and greatly in the cases of N and ash. The uptake of N and ash ceased after about 120 days. J. O. H.

The physiological action of ammonia gas on plants. Kurt Garber. *Landw. Vers.-Sta.* 123, 277-344 (1935).—The microchem. method for the detection of  $\text{NH}_3$  in leaf tissue (cf. Bredemann and Radeloff, C. A. 27, 3972) does not disturb the natural  $\text{NH}_3$  content of the plant. Expts. were carried out on red clover, tomatoes, cucumbers, beans, lupines and several kinds of young nursery trees. The concn. of  $\text{NH}_3$  gas which the plant can tolerate without injury depends on the  $pH$  value of the cell sap as well as the state of nutrition of the plant. Well-nourished plants with a high carbohydrate content will tolerate the  $\text{NH}_3$  gas better than those which are poorly nourished. Various degrees of sensitiveness to  $\text{NH}_3$  are possible, for the same reasons. As long as the concn. of  $\text{NH}_3$  is not fatal to the plant, that which is taken up by the leaves is deprived of its harmful effects as though it had been absorbed by the roots or formed by chem. changes within the plant. In plants with a cell sap of  $pH$  below 5 the  $\text{NH}_3$  will form  $\text{NH}_4$  salts, while in those with a cell sap of  $pH$  above 5 the  $\text{NH}_3$  is utilized in amide synthesis. In plants with slightly acidic cell sap, the assimilation will be stopped by a comparatively weak concn. of gas (1:7500). If the cell sap is strongly acidic, the assimilation will be increased by this concn. of gas. Respiration is scarcely influenced by the action of  $\text{NH}_3$ . Transpiration is generally decreased by a concn. as low as 1:4000. The harmful action of  $\text{NH}_3$  is first noticeable in the reaction of the cell sap which occurs simultaneously with the pptn. of cell pro-

tein, swelling of the chloroplasts, disturbance of assimilation and decompn. of protein. John O. Hardesty.

[Report on] research botany. H. Evans. Mauritius Dept. Agr., 5th Ann. Rept. *Sugarcane Research Sta.* 1934, 40-8; cf. C. A. 29, 2573.—Control of arrowing in sugar cane. Arrowing did not occur with the DK.74 variety of cane when the plants were heavily fertilized with either nitrate or phosphate; heavy applications of lime reduced arrowing. Arrows in the no-nitrate plots appeared about 3 weeks earlier than those in the heavy lime plots. Soaking of setts before planting. Setts were soaked for 8-16 hrs. in a satd. soln. of lime (I), a satd. soln. of lime contg. 1 lb  $\text{MgSO}_4$  per 50 gal. (II), a complete nutrient soln. (III), and water alone (IV). All treatments improved the rate of germination of the setts. Soaking in either I or II gave decidedly better results than soaking in either III or IV. All treatments caused a significant increase in the cane yields. I gave the best results and there was not much difference in the effect of the other treatments. No marked hydrolysis of sucrose occurred as a result of soaking, at least up to a period of 3 days after the cutting had been removed from the soln. More water was absorbed by cuttings from I than from IV. Cuttings did not absorb a significant amt. of Ca from I in 12 hrs. Only minute traces of minerals entered the cutting when it was soaked in a soln. contg. abundant minerals. The superiority of I seems to be due solely to its effect in increasing the amt. of water absorbed by the cutting. Borer larvae present in the cuttings were completely eradicated by soaking in I. Under field conditions, the rapidity of germination increased with the initial reducing sugar content of the cuttings. For quick and uniform germination it is necessary to obtain cuttings with a high and approx. equal reducing sugar content. K. D. Jacob.

The biochemical bases in plant breeding. N. N. Ivanov. *Theoretical Bases of Plant Breeding, Leningrad Acad. Agr. Sci.* 1, 991-1016 (1935).—I. discusses the subject and presents original data under the following headings: the chem. variability of plants; biochemistry and breeding for quantity and quality of chem. substances; biochemistry and synthetic selection and breeding; biochem. methods for the plant breeder and new problems of biochemistry in its application to plant breeding.

J. S. Joffe. Breeding for chemical composition. N. A. Bazilevskaya. *Theoretical Bases of Plant Breeding, Leningrad Acad. Agr. Sci.* 1, 1017-1043 (1935).—B. presents the subject under the following headings: fundamental trends in chem. variability; chem. variability and the environment; the influence of acclimatization on the chem. compn., genetic data from chem. variability; obtaining new products of biosynthesis in crosses; the role of mutations in obtaining new products of biosynthesis; jarovization; the fundamental paths of plant breeding for chem. compn., the problems and methods of chem. selection; chemistry and morphological attributes; correlation between chem. attributes and microchem. analyses.

J. S. Joffe. Alkaline extract of the anterior hypophysis and germination. E. Pascual. *Soc. biol. Rosario (Argentina) Nov.* 24 (1934); *Rev. sudamericana endocrinol. inmunol. ginecoterap.* 18, 713.—The germination of some seeds is accelerated.

A. E. Meyer. Alkaline extract of the anterior hypophysis and plant growth. E. Pascual. *Soc. biol. Rosario (Argentina) Nov.* 24 (1934); *Rev. sudamericana endocrinol. inmunol. ginecoterap.* 18, 713.—Hypophyseal ext. prep'd. by the method of Evans and Simpson (C. A. 26, 3020) accelerated the growth to *Phaseolus vulgaris*, *Zea mays*, *Vicia faba*, *Lupinus albus* and *Hyacinthus* but was ineffective on *Pisum sativum*, *Lens culiculata*, *Salvinia rotaris* and *Lemna gibba*. The action was marked during the first days of growth. Large doses of the ext. have a toxic action.

A. E. Meyer. Polarity and growth substance. A. Th. Czaja. *Ber. deut. botan. Ges.* 53, 197-220 (1935).—When growth substance is applied to a stem parallel to the main axis, bending takes place and when applied transverse to this

axis a retardation of growth in length and an increase in thickness results. With the *Avena* coleoptile there is no increase in thickness but pos bending takes place on transverse application. These results are explained on the basis of pre-existing streams of growth substance in plant organs in parallel and transverse directions which det. the form of the cells, when added growth substance is applied this balance is altered and various changes in cell shape and therefore the plant organ occur. Lawrence P. Miller

Root growth, growth substance and the theory of growth substance action. A. Th. Czaja. *Ber. deut. botan. Ges.* 53, 221-45 (1935). cf. preceding abstr.—On the basis of the geotropic behavior of roots under the exptl. conditions described C. concludes that there are 2 opposing streams of growth substance in roots in the longitudinal direction, one emanating from the root tip and the other from the growing point of the top. C. discusses the work of Ramshorn (*Planta* 22, 737) on the p.d. between growing points and the rest of the plant and the effect of growth substance on this potential difference, and the action of growth substances in inducing neg. osmosis as a possible explanation of the mode of action of these substances on the cell. Lawrence P. Miller

Investigations on the uptake of fluorescent substances by living plant cells. Helmut Döring. *Ber. deut. botan. Ges.* 53, 415-37 (1935).—With the aid of the luminescence microscope the uptake of eosin, erythrosin, fluorescein, trypanflavine, quinone, hydrastine, acridine, esculin and esculetin by living cells (usually from the outer scale of *Allium cepa*) was studied. The fluorescence of tissues stained by eosin differs in living cells from that of dead cells indicating a difference in the physicochem. union. Fluorescein is taken up from acid soln. by the nucleus, plasma and plastid starch and from alk. soln. by the vacuoles. Lawrence P. Miller

Effect of ethylene on apples at low temperatures: evidence for the production of ethylene by unripe, immature fruit. F. Kidd and C. West. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 119-22 (1935). cf. C. A. 24, 2491.—From the expts. described it is tentatively concluded that  $C_2H_4$  is produced by quite unripe and immature fruit and not only, as has been previously thought, by ripe fruit, and that there must be a threshold value for the stimulating dose, below which no effect is produced. A. Papineau-Couture

Combustible gaseous products of fruits. R. Gane. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 124-8 (1935).—Total combustible gaseous substance produced by a known wt. of oranges at 15° was measured. When ventilation was maintained at a steady rate, the quantity of combustible matter in the airstream remained const. so long as the fruit was sound, but with the onset of fungal attack the rate of output, both of  $CO_2$  and of combustible substance, greatly increased. If the combustible matter is an essential oil with a framework of, say,  $C_{10}$ , the concn. of the substance would be of the order of 1 p. p. m. (by vol.) in the air stream. On removing the outer layers of the peel contg. the oil glands, the amt. of combustible matter in the air stream and the rate of production of  $CO_2$  were both approx. doubled. In similar expts. with bananas, the ratio of  $CO_2$  derived from the combustible gaseous products to that produced directly in respiration was about 1:250 in the unripe green stages and decreased as the peak of respiratory activity was reached. The level of respiratory activity was raised by ventilating the fruit with  $O_2$ , but the amt. of combustible gaseous substances produced was unchanged. A large and rapid increase in combustible gases occurred in the later stages of ripening, at the time when the rate of respiration started to increase once more. Tests so far conducted indicated that injury to green fruit is without effect on the time of ripening. Both the  $O_2$  and the  $IO_2$  methods were found unsuitable for estg. the volatile substances produced by apples, probably because substances with a carboxyl group can react with  $O_2$  or  $IO_2$  to give unstable peroxides which, in presence of  $H_2O$ , yield  $H_2O_2$ , which liberates I from KI. A. Papineau-Couture

Effect of ammonia and of hydrochloric acid on the

respiratory activity and the climacteric in apples. F. Kidd and C. West. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 130-2 (1935).—In contrast with the action of  $CO_2$ ,  $HCl$  (at a concn. of 50 p. p. m.) exercises no appreciable influence on the pitch of respiratory activity of apples and does not stimulate the onset of the climacteric.  $NH_3$  (1000 and 400 p. p. m.), on the other hand, had an immediate effect; respiration was stimulated, and a day or 2 later the climacteric occurred. These results support the suggestion that the onset of the climacteric is connected with changes in acidity in the cell, and that it occurs when, in the presence of  $O_2$ , the acidity has fallen to a crit. value (cf. C. A. 29, 2998). At 27, 5780-1, 29, 4404<sup>1</sup> and of Gane (following abstr.). A. P.-C.

The effects of ozone on bananas. R. Gane. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 128-30 (1935).—The rate of production of  $CO_2$  by green bananas at 15° was measured in atms. contg. different concns. of  $O_3$ . If the fruit is within a few days of its period of rapid ripening, ventilation with air contg. 40 p. p. m.  $O_3$  is not effective in retarding the process, but if exposed at an earlier stage the retardation may be very evident. The rate of production of  $CO_2$  in air contg. 25-30 p. p. m.  $O_3$  is only slightly higher than in ordinary air; ripening in the ozonized air was considerably retarded. With 5-7 p. p. m.  $O_3$  there was no significant change in the rate of respiration or in the rate of ripening. A concn. of 15 p. p. m.  $O_3$  did not appreciably affect the output of  $CO_2$  or delay ripening. A. P.-C.

Acidity and sugar content of bananas during ripening. R. Gane. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 132-3 (1935).—The acidity of the pulp of bananas, as measured by the glass electrode, changes very rapidly during ripening at 15°. The  $pH$  of various samples of green unripe fruit showed little variation, ranging from 5.20 to 5.50. As soon as ripening commenced and respiration started to increase there was an increase in the acidity of the pulp; the fall in  $pH$  continued until well after the peak of respiratory activity, and reached a max. at  $pH$  4.34. Further changes in  $pH$  were small, and the rise which followed occurred at the time when the characteristic aromatic odor was produced. At 0° there was a slight rise in  $pH$  in 16 days, during which time the rate of production of  $CO_2$  was unchanged. A table is given showing the increase in respiration (mg.  $CO_2$  per kg. per hr.), percentage of reducing sugar, sucrose and total sugar over a period of 13 days. A. Papineau-Couture

Effect of temperature on the sucrose/hexose relation in potatoes. J. Barker. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 133-5 (1935).—Curves are given showing the changes in total sugar, sucrose, hexoses and the sucrose/hexose ratio when potatoes were changed from 10° to 1° and vice versa. The results suggest that sucrose and the hexose sugars are readily interconvertible in the potato and that their relative proportions are detd. by a balancing mechanism which is strongly influenced by change in temp. The balance is disturbed in favor of the formation of sucrose by a fall of temp. from 10° to 1°, and vice versa. A. Papineau-Couture

Metabolism of nitrogen by apple fruits during development on the tree and in storage. A. C. Hulme. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 135-43 (1935).—The study was carried out with the previously described method (C. A. 29, 4290<sup>2</sup>). The fresh wt. of pulp per apple continues to increase up to maturity, but the fresh wt. of peel reaches an asymptotic value early in the fruit's development. Presumably when this value is reached the cells of the peel merely stretch longitudinally to allow for the continued swelling of the pulp tissue; this is supported by the cytological observations of Tetley (*Rept. Food Investigation Board* 1932, 75). The change in total N confirms the results of Archbold (C. A. 27, 3234-5) and earlier workers. The rapid decrease in total sol. N (as percentage of total N) which occurs during the early development of the fruit in the case of both peel and pulp is striking. The final decrease in total N, protein and sol. N in the case of the pulp may possibly be

connected with the ripening of the seeds. During the climacteric rise in respiration there do not appear to be any violent changes in the total protein and total sol. N fractions. The increase in protein during storage at the expense of sol. N already reported is also apparent in the present expts. Results for the acid content of the fruit over the period of growth, taken in conjunction with the data for N, show that the acid cannot arise directly from products of the breakdown of protein as has been suggested by Ruhland and Wetzel (C. A. 22, 3182) for members of the Crassulaceae and for *Rheum hybridum*.

A. Papineau-Couture  
Respiration of pea seeds. A. J. M. Smith and R. Gane Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 147-52 (1935).—A study of the respiration of green peas under various humidity conditions and in atmos. of various compns. apparently established the existence of 2 stages in the respiratory phenomena of germination, which it seems reasonable to relate to the stages that have been shown by Kipp (J. Wiss. Bot. 71, 533 (1929)) to occur in the germination of the light-sensitive seeds of *Nicotiana tabacum*, where light is the necessary stimulus to the second phase.

A. Papineau-Couture  
The effect of rubber in the life of the rubber-bearing plant. G. Bosse. *Sovetskii Kauchuk* 1935, No. 4, 32-9 — A discussion

A. Pestoff  
The chemistry of plant respiration. W. O. James School Sci. Rev. 17, 265-81 (1935) — Review with 39 references.

O. Remmuth  
The occurrence of adenosinetriphosphoric acid in living yeast. H. v. Euler, E. Adler and M. Petrusson *Stenssk Kem. Tids.* 47, 249-51 (1935) (in German).—Adenosinetriphosphoric acid is definitely identified in yeast and functions in the same manner as is muscle.

A. R. Rose  
The action of certain metals at a distance, in contact, and in solution on the development of *Thielavia basicola* Zopf. and on that of other fungi. C. Sempio. *Riv. pat. reg.* 24, 413-91 (1934); *Rev. Applied Mycol.* 14, 646-7.—At a distance of 1-2 mm. from the conidia of *Thielavia (Thielaviopsis) basicola* in hanging-drop broth cultures Pb prevented germination altogether or retarded growth considerably; the mycelium developed characteristic swellings and rosette formations, and conidia were scarcely ever produced. If the Pb was removed before or at the commencement of germination the fungus at once began to grow rapidly. Cu and Al very slightly retarded the early stages of development while Pt, Ag and Au had practically no effect on germination and only a slightly depressive effect on growth of the germ tube. When Cu, Al or Ag fungi were placed in contact with the conidia of *T. basicola* in hanging-drop broth cultures, growth was completely inhibited as a result of passage of large amts. of the metals into colloidal soln.; Au and Pt fungi had practically no effect and Pb was only slightly depressive.  $\text{Cu}(\text{NO}_3)_2$  (0.001 M),  $\text{Al}(\text{NO}_3)_3$  or  $\text{Pb}(\text{NO}_3)_2$  (both at 0.0001 M) contg. 2% sucrose and glycerol had practically no effect on the conidia of *T. basicola*;  $\text{AuCl}_3$  had a slight effect, and  $\text{PtCl}_4$  still more;  $\text{AgNO}_3$  (all at 0.0001 M) completely inhibited growth. While there was a certain parallel between the effects produced by the metals in contact with the fungus and those produced by the same metals in soln., no parallel existed between these effects and those produced by the same metals at a distance. When the metals were placed at a distance of 1-2 mm. from the spores of *Erysiphe graminis* and *Puccinia graminis* in water or broth hanging-drop cultures practically no effect was produced. Oden E. Sheppard

Anthocyanins from the flowers of *Pharbitis nil* (Katakoka) 10. Anthocyanin pigment of the flowers of deep-blue hyacinth (Hayashi) 10.

## E—NUTRITION

PHILIP B. HAWK

Ascorbic acid in internal glands. Isolation from hypophysis. Jorge R. Mendive and Venancio Deuloufo. *Z. physiol. Chem.* 236, 208-11 (1935).—Colorimetric detns. on various glands of Argentine steers showed that

hypophysis, corpus luteum and the suprarenal contain considerably more ascorbic acid than the other glands. Thymus and spleen take an intermediate place in the table, then follow testis, pancreas and thyroid with only a small ascorbic acid content. Ascorbic acid was isolated as such from hypophysis, and as vitamase from corpus luteum.

A. W. Dor  
Comparative investigations on the vitamin C (ascorbic acid) content in blood and in cerebrospinal fluid. F. Plaut and M. Bülow *Z. physiol. Chem.* 236, 241-56 (1935), cf. C. A. 29, 5162.—Ascorbic acid occurs in the cerebrospinal fluid of man and rabbit only in its reduced form. In the blood it occurs predominantly in its reversibly oxidized form. Its reduced form is demonstrable after copious feeding of vitamin C. Variations in the ascorbic acid picture of the blood involve only the reduced portion. Blood cannot oxidize ascorbic acid either *in vitro* or *in vivo*. When introduced into the circulation ascorbic acid passes out without being oxidized. Administered orally it reaches the blood in its reduced form and is carried away as such. Spinal fluid cannot reduce reversibly oxidized ascorbic acid *in vitro*. When injected into the spinal canal the oxidized acid does not undergo reduction and is absorbed more rapidly than the reduced acid. Ascorbic acid is able to pass from the blood into the spinal fluid, and when injected into the spinal canal it can pass into the blood; but there appears to be a mechanism which under physiol. conditions controls its release from the spinal fluid. Reduced ascorbic acid entering the blood is very rapidly removed. This explains why with moderate vitamin C intake reduced ascorbic acid is not as a rule demonstrable in the blood, and with copious intake it is found only in small quantities. The oxidized ascorbic acid of the blood is not appreciably affected by the vitamin C metabolism. Ascorbic acid remains a longer time in the spinal fluid, regardless of its mode of access, than in the blood stream. These observations explain why excess and deficiency of vitamin C are more easily recognized by spinal fluid examn. than by blood examn. There is no evidence that the nervous system can reduce ascorbic acid.

A. W. Dor  
Changes in the sensitivity of rachitic rats for vitamin D. J. van Niekerk. *Arch. nêrland. physiol.* 20, 477-80 (1935).—In order to reconcile values obtained in the biol. assay of the substance "D" with the activity in International Units, it is assumed that the sensitivity of the expl. animals has fallen to about 1/4 of the original value over the period 1929/1932.

F. L. D.  
The physiological action of vitamin B<sub>1</sub>. H. G. K. Westenbrink. *Arch. nêrland. physiol.* 20, 481-4 (1935); cf. C. A. 28, 2448.—It is quite probable that polyneuritis resulting from the lack of B<sub>1</sub> is the result of chronic poisoning produced by the degradation products of carbohydrates.

F. L. Dunlap  
Feeding experiments with marrow stem cabbage. H. Binger, A. Werner, J. Schultz, H. Augustin and H. Finzenhagen. *Biedermanns Zentr. B. Tierernähr.* 7, 325-43 (1935); cf. *Ibid.* 5, 450-68 (1933).—In 3 years of feeding tests, 40 kg. of beet roots was compared with 40 kg. of marrow stem cabbage, with dairy cows. In 2 trials, 1-2 kg. of protein-rich concentrates was withdrawn from the ration during the marrow stem cabbage period, while at the same time starch values were added in the form of dried cuttings or of potato flakes. Marrow stem cabbage affected the milk yields favorably, but the percentage of milk fat did not increase. Marrow stem cabbage affected the live wt. of cows less favorably than beets. F. L. D.

Experiments on feeding skim milk powder to laying hens. G. Wiegner and A. Tscherniak. *Biedermanns Zentr. B. Tierernähr.* 7, 344-60 (1935).—Dried skim milk produced a somewhat smaller egg yield than cod meal.

F. L. Dunlap  
The acid-base equilibrium of cattle as influenced by hay and grass silage without mineral acid addition. E. Brouwer and N. D. Dijkstra. *Biedermanns Zentr. B. Tierernähr.* 7, 361-71 (1935).—The pH and total CO<sub>2</sub> content of the urine were somewhat lower in the silage groups than in the hay groups. Nevertheless the urine

in all the silage groups remained strongly or rather strongly alk. From this it may be concluded that the org. acids were for the most part oxidized and that mainly alk. metabolic products were produced from the silage rations. There was no shift in the acid-base equil. as might be caused by an accumulation of the org. acids in the blood before their oxidation, for the  $pH$  and the total  $CO_2$  of the blood plasma were practically the same in both the silage and the hay groups. F. L. Dunlap

Total metabolism experiments on rats with special reference to the influence of vitamin D. W. Schoch. *Biedermanns Zentr. B. Tierernähr.* 7, 382-443 (1935).—A study of energy metabolism under the influence of vitamin D. The expts. were conducted on white rats in a new exptl. cage and in a respiratory app. The results show that vitamin D has no effect on energy metabolism or on the digestibility of protein. The differences between results obtained with rachitic and nonrachitic rats in fasting as well as in feeding expts. were within the exptl. error. In detg. the net energy, measured as protein and fat increase, the thermal energy was calcd. F. L. Dunlap

Treatment of human beriberi with crystalline antineuritic vitamin A. J. Hermano and Froilan Eubanas. *Philippine J. Sci.* 57, 277-87 (1935).—The material employed in this preliminary report was cryst. vitamin B<sub>1</sub> hydrochloride prep'd from rice polishings. One-cc. injections were given by intramuscular injection at each treatment, each cc. contg. 1 mg. of the salt. Twelve cases were treated, 10 adults and 2 infants. The results were such as to give promising indications of curing human beriberi. Nineteen references. F. L. Dunlap

Effect of vitamin B (B<sub>1</sub>) insufficiency upon the nervous tissues of young dogs. C. O. Prickett. *Ala. Agr. Expt. Sta., 45th Ann. Rept.* 19-20 (1934).—The macroscopic and histol. changes due to vitamin B deficiency are outlined. The brain lesions and spasticity were marked. The deficient diet contained 28% fat and 8% autoclaved yeast. C. R. Fellers

Utilization of nitrogen, calcium and phosphorus by the growing chick. C. W. Ackerson, M. J. Blish and F. E. Muschel. *Neb. Agr. Expt. Sta., Research Bull.* 80, 3-16 (1935).—The N, Ca and P contents of 125 newly hatched chicks were det'd. Thirty chicks were fed accurately for 60 days at the end of which time the retention of N was 37.8%, Ca 43.4% and P 27.3% of the resp. elements fed. The av. gain in live wt. per g. of N fed was 12 g.; the rate of gain was 41.5% of the dry matter fed. The coeff. of variability was approx. 5% except for the Ca figures, which had a variability of 8%. Forced feeding of a pelleted ration permits of accurate control of the food intake of baby chicks. No significant sex differences in the utilization of the nutrients were observed. C. R. F.

Vitamin A requirements for growing chicks. A. E. Tepper and F. D. Reed. *N. H. Agr. Expt. Sta., Bull.* 284, 24 (1935).—In a ration deficient in vitamin A little difference in wt. was found in chicks fed 1, 2 or 3% of cod liver oil. One group of chicks fed 0.5% sardine oil plus irradiation showed less wt. gain than the cod liver oil groups. No macroscopic vitamin A injury resulted from any treatment. C. R. Fellers

Sardine oil versus cod liver oil (for chicks). A. E. Tepper. *N. H. Agr. Expt. Sta., Bull.* 284, 24 (1935).—The New England College Conference poultry ration plus 0.5% sardine oil proved equally as efficacious as 1% cod liver oil. There was a slight growth retardation when only 0.25% of the sardine oil was used in the chick ration. C. R. Fellers

Vitamin B in cottonseed meal. F. W. Sherwood and J. O. Halverson. *N. Car. Agr. Expt. Sta., 46th Ann. Rept.* 44-5 (1933).—Five samples of cottonseed meal averaged 3-4 International Units of vitamin B per g. Fleischmann's yeast contained 6 units and Northwestern yeast 12.5 units per g. Peanut meal contained 2-2.5 units per g. Both yeast samples were of equal vitamin G potency. Cottonseed and peanut meals were 12.5% as rich as the yeast in vitamin G. C. R. Fellers

Menhaden fish oil as a source of vitamin D for growing chicks. J. O. Halverson and R. S. Dearystine. *N.*

*Car. Agr. Expt. Sta., 46th Ann. Rept.* 62 (1933).—By supplementing the basal ration of Hart (cf. C. A. 27, 527) by that of Lachat, Halverson and Palmer (C. A. 27, 527) with 1/2 and 1% menhaden fish oil, lots of 20 standardized young chicks showed degrees of bone ash and growth comparable to similar amts. of cod-liver oil.

C. R. Fellers  
Study of the food habits and physical development of preschool children over a two-year period, with special reference to seasonal variations in growth. Hughina McKay and Mary Brown Patton. *Ohio Agr. Expt. Sta., Bull.* 549, 72 pp (1935); cf. C. A. 29, 5487.—Children from 19 to 40 months of age were used as the subjects of careful dietary and phys. studies at 4 seasons during a 2-year period of study. Total protein in relation to height was more significant than in relation to wt. While age had no significant relationship to protein intake, total Ca showed a significant relationship to height but practically none to age and wt. Total P was related to both height and wt. Age, wt. and height were all significant in regard to Fe intake, wt. having a higher value than the other 2. In 9 out of 15 instances, high intakes in regard to cal. per kg., protein per kg., total Ca, total P, P per kg., total Fe and Fe per kg. were found during the periods of greatest gain in wt. C. R. Fellers

Vitamin D requirements of chickens. R. R. Murphy, J. E. Hunter and H. C. Knandel. *Pa. Agr. Expt. Sta., Bull.* 320, 27 (1935).—When denied access to sunlight growing chicks required 34 U. S. P. units of vitamin D per 100 g. of feed; laying hens required double this amt.

C. R. Fellers  
Action of vitamin D on the circulation and blood vessels. H. Handovsky and N. Goormaghtigh. *Compt. rend. soc. biol.* 120, 74-9 (1935).—In dogs large repeated doses of vitamin D first stimulate the cellular activity of the smooth muscles of the blood vessels, then bring about atrophy and necrosis. In the first stage the reaction to small doses of adrenaline is inverted; in the second stage there is sensitization to the hypertensive action of adrenaline and signs of hyperthyroidism. L. E. Gilson

Histological study of the effects produced (in rats) by large doses of carotene, carrot lipides and vitamin A. Tomás Ocaña. *Rev. soc. argentina biol.* 11, 147-54 (1935); *Compt. rend. soc. biol.* 120, 267-9 (1935).

L. E. Gilson  
Biochemistry of vitamin A. I. State of combination of vitamin A in liver oils. Ladislav Reti. *Rev. soc. argentina biol.* 11, 283-90 (1935); *Compt. rend. soc. biol.* 120, 577-80 (1935).—Livers of fish, chickens and various mammals were exam'd. In every case the vitamin A (polyene alc.) was all combined with fatty acids. Methods are described. L. E. Gilson

The anemias of nutritional deficiency. Geo. R. Minot. *J. Am. Med. Assoc.* 105, 1176-9 (1935).—Address

F. P. Griffiths  
Low-calorie, low-fat, ketogenic diet for treatment of infections of the urinary tract. Reed M. Nesbit and C. H. McDonnell. *J. Am. Med. Assoc.* 105, 1183-4 (1935).—A low-fat, low-calorie diet supplying 0.66 g. protein and 0.33 g. carbohydrate (no sugar) per kg. body wt. produced ketosis without gastric disturbances. Ketosis is dependent on the inadequacy of available glucose and not on amt. of fat ingested. F. P. Griffiths

Variations in phosphatase content of bone, kidney and blood in experimental rickets. G. Scow. *Bull. soc. ital. biol. sper.* 10, 823-6 (1935).—During exptl. rickets in rats the phosphatase of the blood increased normally while that of the kidney and bone was below normal. During recovery there was a marked increase in phosphatase in bones and kidney with a diminution from the blood. H. L. G.

Control and reparation of the international standard of vitamin B. Antonio Allegri. *Boll. soc. ital. biol. sper.* 10, 836-9 (1935).—A daily dose of 80 mg. of the international standard was an adequate vitamin B ration for pigeons. In some cases this dose caused severe and even lethal enterorrhagia. A new method of prep'g. the standard is given. Rice husks are ext'd. for 2 days in  $H_2O$  acidified to  $pH$  4 with  $H_2SO_4$ . After filtration fuller's

earth is added to the liquid, shaken for 24 hrs., filtered, dried and washed with  $H_2O$  and alc. Three kg. of the adsorbate represents the vitamin  $B_1$  content of 100 kg. rice husks and 11.25 mg. of the final product corresponds to 10 mg. of the international standard. H. L. Gruehl

Sunflower-seed diet in experimental polyneuritis. Antonio Allegri. *Boll. soc. ital. biol. sper.* 10, 339-43 (1935).—The oil extd. from the seeds had no preventive or curative action on avitaminosis B in pigeons. The vitamin B seemed to be contained entirely in the residue from the extn. After a year of sunflower-seed diet the vitamin B reserve was lower than normal. H. L. G.

Nutritional treatment of acne vulgaris. Charles Lerner. *Arch. Dermatol. Syphilol.* 31, 520-31 (1935).—Twenty acne patients were put on a diet in which the table salt was equilibrated with Ca, K and Mg, for 13 weeks. Good results were obtained with the pustular and indurated types of acne. O. Hartley

Supplementing casein with cystine 12 and 24 hours before and after the casein meal. Lauri Paloheimo. *Suomen Kemistilehti* 8B, 31 (1935) (in German).—A preliminary report. The addn of 3% cystine N to the casein N gave a noticeable displacement in the N balance in the pos. direction in exptl. rats. F. I. Jukkola

Protein supplements in poultry rations. V. Copra meal as a supplement in rations for growing chicks. F. M. Fronda and Manuel P. Mallonga. *Philippine Agr.* 24, 350-56 (1935), cf. C. A. 28, 3436.—Copra meal is not a good source of protein for growing chicks. A. L. Mehning

Chemical constitution and biological properties of the complex lipids of the carrot. D. Romoli-Venturi and A. Pugliese. *Biochim. terrap. sper.* 22, 421-57 (1935).—The following substances were obtained: (1) A phospholipid of vitamin reactions corresponding to A and D and contg. Ca, P and N in org. linkage. R. V. discusses the possible structure of this substance. (2) Pure carotene. (3) A compd. showing the phys.-chem. characteristics of A, free from carotene. (4) A carotene-free compd. with the characteristics of E. Vitamin D was present in all fractions. The biol. properties were substantiated by extensive biol. expts. A. E. Meyer

The maximal concentration of urine, its investigation and diagnostic value in renal insufficiency. Manuel E. Varela. *Serap. id.* (Buenos Aires) 1935, II, 130-5.—Normal persons on a diet rich in protein and low in liquids eliminate a urine of 1 030-40 sp. gr. A decrease of the sp. gr. occurs in beginning renal insufficiency when the nonprotein N in the plasma is not yet increased. There is a parallelism between deficit in concn. and elimination of phenolsulfonphthalein. A. E. Meyer

The dietetic factor determining the glucose tolerance and sensitivity to insulin of healthy men. H. P. Himsworth. *Clin. Sci.* 2, No. 1, 67-94 (1935), cf. C. A. 29, 2582.—The area falling above the resting blood-sugar level or below the insulin depression curve is constant for normal human beings so long as the diet remains unchanged. Improvement in glucose tolerance with the change from a diet low in carbohydrate and high in fat to a diet high in carbohydrate and low in fat is due solely to carbohydrate, and not to caloric intake, fat, protein or the ketogenic ratio. The development of insulin sensitivity is independent of the dose of insulin administered. Changes in glucose tolerance may be accounted for by changes in the pancreatic insulin secretion. The greater the carbohydrate intake in the diet, the greater is the sensitivity to insulin. James C. Munch

The diet of diabetics prior to the onset of the disease. H. P. Himsworth. *Clin. Sci.* 2, No. 1, 95-116 (1935).—The diet of 143 diabetics was compared with that of 258 normal persons. The majority of the diabetics had preferred a diet contg. excess fat, whereas a smaller no. than the normal preferred excess carbohydrate diet. Diabetics had chosen diets with greater caloric value. The habitual ingestion of a low carbohydrate diet may cause progressive impairment of sugar tolerance and insulin sensitivity, resulting in diabetes. James C. Munch

Diet and the incidence of diabetes mellitus. H. P.

Himsworth. *Clin. Sci.* 2, No. 1, 117-48 (1935).—In world-wide studies of distribution of diabetes and its increase during the last 30 years, high incidence was found in countries with a diet low in carbohydrate and high in fat. This explains the higher incidence in the city than in the country. There was no relation to consumption of excess sugar or alc. Caloric value of the diet did not appear to be a factor. Increase in diabetes during the last 30 yrs. was assocd. with increase of fat in the diet. James C. Munch

Preliminary observations on vitamin A deficiency as shown by studies with the visual photometer. Ira O. Park. *J. Oklahoma State Med. Assoc.* 28, No. 10, 357-64 (1935).—The effect of oral administration of carotene in oil to 275 patients was followed by the Jeans and Zentmire technique (*J. Am. Med. Assoc.* 102, 812-5 (1933)). Low A intake was assocd. with the production of rhodopsin. There is a possibility that vitamin A is destroyed by the toxin of measles. Clinical improvement was observed after the administration of carotene in gastrointestinal, sinus, kidney, nervous, mental and circulatory disturbances, diabetes, leucemia, pernicious anemia and night blindness. The addn of vitamin B as brewers' yeast was beneficial in certain cases. James C. Munch

Dilatometric method for studying the digestibility of meals "in vitro". Kamala Bhagvat and Motnahalli Sreenivasaya. *Proc. Indian Acad. Sci.* 2B, 316-21 (1935).—The dilatometer previously described, C. A. 26, 4610, is shown to be suitable for the study of the tryptic digestion of skimmed cow milk and of a casein soln. in phosphate buffer. The 2 solns. are digested at the same rate, if the protein N of cow milk is considered to be 87% of the total N in milk. W. Gordon Rose

Some aspects of dietetics. Anon. *Pharm. J.* 134, 250-60 (1935).—A review of modern principles in dietetics is given, e. g., balanced food, dietary means of preventing constipation, the use of soybean milk in infant feeding (cf. C. A. 27, 2716), ketogenic diet treatment (cf. C. A. 28, 3474, 4456, 6458) etc. S. Waldbott

The effect of diet on the hemoglobin concentration of the blood. L. N. Ellis and O. A. Hessey. *Am. J. Physiol.* 113, 582-5 (1935).—Hemoglobin detrs. on rats were made at 1 month and 1 year of age to study the influence of 5 diets. At 1 month of age the hemoglobin concn. increased along with the Fe content of the ration. A max. of 12.6 g. per 100 cc. was obtained. At 1 year of age the hemoglobin concns. were independent of the Fe content of the diet. E. D. Walter

A comparison of the anemia produced by feeding young rats upon human, cow and goat milk. Howard H. Beard and Thomas S. Bogess. *Am. J. Physiol.* 113, 642-6 (1935), cf. C. A. 27, 5382.—The feeding of human milk did not produce anemia. The drop in erythrocytes and hemoglobin in cow-milk anemia was slower than in goat-milk anemia. Hypertrophy of the heart muscle, atrophy of the spleen and fatty degeneration of the liver were the most consistent gross pathol. findings in the anemic rats. The importance of Fe in preventing these changes was discussed. The pathol. findings in goat-milk anemia are not similar to those in pernicious anemia of man, and any close relationship between both types of anemia must be denied. Fe, with and without Cu, prevented the onset of goat-milk anemia. E. D. Walter

Further observations upon the origin of creatine from proteins and amino acids. Howard H. Beard and Thomas S. Bogess. *Am. J. Physiol.* 113, 647-53 (1935); cf. C. A. 26, 4085.—Rats were fed complete synthetic diets contg. either 4% casein or egg albumin as the sole protein for a period of 5 weeks. Rerfeeding these rats on 25% of casein, or egg albumin, or 21% of glycine or glutamic acid, for a period of 4 more weeks, caused av. increases in the total creatine of the muscles from 20 to 50% above the control animals on the 4% protein diets. Total N, fat and total solid content of the muscles of all rats showed very little change. Creatine is shown to be a product of the exogenous catabolism of proteins and amino acids. E. D. Walter

Synthesis of lactoflavin (Kuhn, *et al*) 10 Titration curves and disson consists of L-ascorbic acid (Kumler, Daniels) 2

## F—PHYSIOLOGY

HOMER W SMITH

The male sex hormone and its artificial preparation in the laboratory. L. Ruzicka *Bull. soc. chim.* [5], 2, 1497-1512 (1935); cf. *C. A.* 29, 3710<sup>1</sup>—Lecture with bibliography of 34 references. C. R. Addinall

Diffusion of lactic acid into and out of the voluntary muscles of the frog. Abdul Ghaffar. *Quart. J. Exptl. Physiol.* 25, 229-39 (1935)—Observations on the diffusion of d-lactate in and out of resting frog muscle during immersion in a Ringer sola. contg. Na d lactate show that only about 1/4 of the muscle H<sub>2</sub>O appears to be involved in the diffusion process, the rest appearing to be shut off by membranes impermeable to lactate. These 2 portions of muscle have been termed "interspaces" and "cells," resp. In the fatigued muscle the interspaces tend to disappear presumably because of swelling of the cells. In heat rigor almost all of the muscle H<sub>2</sub>O becomes available for the diffusion of lactate. An excited state of the isolated muscle does not appear to render the membranes bounding the cells permeable to lactate. The normal lactic acid concn in the cells of isolated frog muscle is found to be lower than that in the interspaces whether the frog is fresh or has been previously cooled or fatigued. The 2 values rise and fall together. This parallelism suggests that the intact animal possesses some mechanism by which a relationship is maintained between the concn. of cells and interspaces. Rachel Brown

Diffusion of iodide into and out of the voluntary muscles of the frog. Abdul Ghaffar. *Quart. J. Exptl. Physiol.* 25, 241-5 (1935), cf. *C. A.* 29, 6944<sup>1</sup>—Observations on the diffusion of iodide into frog muscle when immersed in Ringer sola. contg. NaI show that with fresh muscle only about 1/4 of the muscle H<sub>2</sub>O appears to be involved in the diffusion process. In muscle in heat rigor, iodide diffuses through almost all of the muscle H<sub>2</sub>O. The diffusion const. of iodide through fresh muscle is  $1.2 \times 10^{-4}$  and that through muscle in heat rigor is  $8.5 \times 10^{-4}$ . The diffusion constant of iodide through 2% agar gel is  $9 \times 10^{-4}$ . Rachel Brown

The antagonodotropic effect of epiphysan. Paul Engel *Wien klin. Wochschr.* 48, 1160-1 (1935)—The active agents of the pineal gland are contained in epiphysan (Richter). Biol. tests gave a content of 80 rat units of the antitumorizing substance per ampoule. D. B. Dill

Urne melanogen. Otto Furth and Alfred Friedrich *Wien klin. Wochschr.* 48, 1175-7 (1935); cf. *C. A.* 29, 7447<sup>1</sup>—The mol. relation, with S as unity, is  $C_{60}H_{118}N_4S_{60}$ . D. B. Dill

Experiments on the electrical excitability of the eye. H. D. Bouman. *Arch. neerland physiol.* 20, 430-45 (1935)—Twenty-six references. F. L. Dunlap

Effects of thyroidectomy on the lipide, fat acid, cholesterol and protein contents of the blood serum and the protein-lipide ratio. C. I. Parhon and I. Ornstein *Bull. soc. chim. biol.* 17, 1119-23 (1935)—Male and female rabbits 3-4 months old were thyroidectomized. After 2-3 weeks the total lipide, fat acid and cholesterol contents of the serum had almost doubled while, the protein content was practically unchanged. The protein-lipide ratio had decreased to 19.9 from an original av. value of 31.4. Fifteen references. L. E. Gilson

Fixation of potassium in birds and fish. A. Leulier and F. Paulant *Bull. soc. chim. biol.* 17, 1124-36 (1935), cf. *C. A.* 28, 2762<sup>1</sup>—K detns were made on hen, duck, turkey and pigeon eggs, chick embryos (cf. *C. A.* 29, 3012<sup>1</sup>) and young rainbow trout of various ages. L. E. Gilson

Albumose and peptone contents of muscles of horned cattle. I. A. Smorodintzev and N. N. Krulova *Bull. soc. chim. biol.* 17, 1149-50 (1935)—No albumoses or peptones could be detected in fresh beef muscle or beef stored 1-15 days at 1°. The method used consisted in

detg. the increase, if any, in free amino N after treating the sample with an erepsin prepn capable of digesting albumoses and peptones but not natural muscle proteins. Fifteen references. L. E. Gilson

The ammonia of human and cow milks. Michel Polonovski and Paul Boulanger *Bull. soc. chim. biol.* 17, 1178-83 (1935)—Very fresh cow milk contains 1 mg., or slightly less, per l and fresh human milk slightly over 1 mg. Cow colostrum contains 17-21 mg./l and human colostrum about 5. If the milk is made alk. by adding satd borax soln. (pH 9.3) the NH<sub>3</sub> increases slightly in cow milk but not in human milk. Increase in NH<sub>3</sub> on standing is due to bacterial action. L. E. Gilson

Inactivation of adrenaline by extracts of various organs. J. Toscano Rico and A. Malafaya Baptista *Compt. rend. soc. biol.* 120, 42-5 (1935)—At pH 7.4 and 38°, synthetic L-suprarenine was rapidly destroyed by exts of dog spleen, guinea pig liver and guinea-pig and cat kidneys, much less rapidly by exts of dog kidney, rabbit spleen and dog, calf, hog and rabbit livers; and not at all by exts of cat spleen liver and intestine, hog spleen and various kinds of skeletal muscle. L. E. Gilson

Protein metabolism of *Chaetophractus villosus* B. Brauer. *Compt. rend. soc. biol.* 120, 361-2 (1935)—See *C. A.* 29, 7427<sup>1</sup>. L. E. Gilson

Humoral distribution of sodium chloride in blood and effusions. G. Dell'Acqua *Boll. soc. ital. biol. sper.* 10, 765-8 (1935)—The av. values for NaCl (Rusznayk method (*C. A.* 27, 43, 4533)), from 104 cases, were 613.93 mg./100 cc for effusions (transudates and exudates) and 593.98 mg./100 cc for blood (serum and whole blood). Helen Lee Gruehl

Physicochemical investigations of human sweat. Gustav Illopi *Arch. Dermatol. Syphilis* 171, 301-12 (1935)—The pH of perspiration induced in 20 persons by heat varied with the diet and the alkali reserve of the individual, but followed these less closely than did urine pH. Na and Cl contents of the perspiration increased as the treatment progressed, but K, Ca and Mg concns fell. Twenty-five references. O. Hartley

Chemical study of chicken blood. Arcadio C. Gonzaga *Rept. New York State Vet. Coll.* 1933-34, 53-7.—The concns of sugar and nonprotein N in the blood are high during the early life of chickens, then gradually decrease with age. The increase in age is associated, with a slight increase in the level of the urea N and of Fe with a corresponding increase in hemoglobin and O vol. The uric acid content is higher in 1-day-old chicks and in those 2-4 months of age than in any other age groups. The venous blood has on the av. but slightly more sugar than the arterial blood; it has greater concn of total nonprotein N, urea N and uric acid, but less amts. of Fe, hemoglobin and O. K. D. Jacob

Chemical analysis of blood of five male and five female carabaoes. Lhas G. Posa. *Philippine Agr.* 24, 388-92 (1935)—The av. blood of the 10 carabaoes contained N 30.04, nonprotein N 28.53, urea 13.51, uric acid 1.67, creatinine 1.68, creatine 4.43, Cl as NaCl 477.29, Ca 28.19 and sugar 73.65 mg./100 ml. The serum Ca is more than double that usually given for other farm animals. A. L. Mehring

The mechanism of spontaneous hydrolysis of the organic phosphorus of the blood in vitro. G. De Toni and G. Graf. *Biochim. therap. sper.* 22, 395-410 (1935), cf. *C. A.* 29, 2215<sup>1</sup>—While the inorg. P continuously increases in blood laked with H<sub>2</sub>O, this is not the case when the phosphatase is destroyed with CCl<sub>3</sub>CO<sub>2</sub>H. Spontaneous chem. hydrolysis does not occur. A. E. Meyer

The ratio between dehydroascorbic and ascorbic acids in the liver, the heart and the adrenals in relationship to the administration of sympathetotropic substances. I. Fernando Copello. *Biochim. therap. sper.* 22, 490-506 (1935); cf. *C. A.* 29, 8141<sup>1</sup>—Excitation of the parasympathetic system with eserine or acetylcholine causes a decrease of the ratio dehydroascorbic acid (D): ascorbic acid in liver and heart, because of a diminution of the former. Paralysis of the parasympathetic with atropine causes an increase

of (I) in the liver and heart. The adrenals do not show an appreciable variation.

A. E. Meyer  
Indicanemia during gestation, parturition and puerperium. Roberto A. Ferrari. *Rev. sudamericana endocrinol. inmunol. quimioterap.* 18, 690-701(1935)—The indican content in the blood of normal women varies between 0.03 and 0.12%. An increase during gestation, etc., as asserted by some authors was not observed.

A. E. Meyer  
The metabolism of bone Donald Hunter. *Cambridge Univ. Med. Soc. Mag.* 12, No. 3, 115-32(1935)—11 effects of variation in Ca, P and calciferol intake, thyroxine, parathyroid exts and scurvy upon bone growth were followed by the madder method.

James C. Munch  
The isoelectric point of animal tissues V The isoelectric point of certain cells G Yagurum. *Folia Anatomica Japonica* 8, No. 4, 465-72(1935). cf. *C. A.* 29, 7354<sup>1</sup>—The isoelectric point for old and young rabbit epithelial cells from the esophagus was 4.2 and 3.2, duodenum 4.1 and 3.6, intestine 4.0 and 3.4, kidney glomerulus 7.0 and 4.6. Similar figures were obtained on epithelial cells of mice of various ages. For human erythrocytes of adults the isoelectric point was 0.70, for the fetus, 0.65.

James C. Munch  
The role of heavy metals in animal metabolism J R E. Richardson. *Guy's Hospital Gazette* 49, 239-41(1935)—The spectroscopic identification of Na, K, Ca, Mg, I, e, Zn, Cu, Mn, Al, Rb and Sn is considered.

J. C. M.  
A new study of heat production in man H T W Adams and E. P. Poulton. *Guy's Hospital Reports* 85, No. 1, 50-75(1935)—Carbohydrates and fats are burned in the fixed ratio of 1:1.33. On a fat and protein diet under basal conditions, a rise in R Q is associated with fall in O and const. CO<sub>2</sub>. A carbohydrate diet, under the same condition, produces a rise in R Q with a fall in O, but an increase in CO<sub>2</sub>. CO<sub>2</sub> results only from combustion, whereas O is involved also in the interconversion of fats and carbohydrates. When the R Q falls between 0.775 and 0.875, the correlation coeff.,  $r$ , between CO<sub>2</sub> and heat production was 0.9503  $\pm$  0.00598. If heat is calcd. from CO<sub>2</sub>, the output is too large with a high R. Q value, and too small with a low R. Q. J. C. Munch.

Does normal mental function depend on normal blood-sugar concentrations? Ellis Powell. *Tri-State Med. J.* 7, No. 5, 1421-2, 1431(1935)—Pancreatic involvement leading to hyperinsulinism with a fasting blood sugar of 48 to 60 mg. per 100 cc. produced marked depression of the mental functions.

James C. Munch  
Changes in the muscle pigments. J. Brooks. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 30-4(1935)—In order to det. the influence of pressure of O on the rate of formation of methemoglobin, the rate of oxidation was measured at 30° at const. partial pressure of O ranging from 4.5 to 723 mm., the residue of the gas mixt. being N. The rate of oxidation (given by  $k$ ) increased with decreasing pressure of O, and reached a max. at a pressure of 20-25 mm. The results are decisively in favor of the reaction consisting of reaction of O with reduced hemoglobin. The rate is not a simple function of the partial pressure of O, which may be due to the fact that the reaction between O and reduced hemoglobin is a surface reaction of the Langmuir type. It has been stated that the addn. of 10% CO<sub>2</sub> to air does not increase the rate of formation of methemoglobin in beef muscle and beef fat at 0°, and the above results agree with this conclusion. In the storage of chilled beef in 10% CO<sub>2</sub>, no significant increase in the storage life (from the standpoint of bloom) can be expected by enriching the mixt. with O unless considerable amts. are added, e. g., to give roughly a total concn. of 60% O. For periods up to 60 days the addn. of O to the atm. should be unnecessary if the correct conditions of storage and air circulation and humidity could be defined. That the rate of discoloration of chilled beef stored in high concns. of CO<sub>2</sub> should be markedly greater than the rate in air alone was confirmed by the storage at -0.7° of 2 hind quarters from the same animal, one in 10% CO<sub>2</sub> and the other in 35% CO<sub>2</sub>. A. P.-C.

Bile secretion. Hiroshi Toabe. *Nagoya J. Med. Sci.* 9,

31-56(1935).—A description of the effect of intramuscular, subcutaneous or intravenous injections of several substances on the bile secretion of dogs. Flow of bile to the duodenum was prevented by a ligation of the common bile duct. Secreted bile was collected from a fistula. Secretion of bile is influenced by a neural, humoral or humoral-neural mechanism. The first mechanism is characteristic of substances that are poisons of the autonomic nervous system; these poisons have but slight effect on the secretion of bile. Substances characteristic of the second mechanism are responsible for a large increase in the aq. constituent of bile. Substances responsible for the third mechanism are truly chologogue, since the increased bile secretion includes an increased amt. of bile solids.

W. Gordon Rose  
Lymph sugar J W Heim, R S. Thomson and F. C. Bartter. *Am. J. Physiol.* 113, 549-51(1935). cf. *C. A.* 27, 2491, 28, 5111<sup>1</sup>—A set of conditions which must be observed to establish a true basis for a comparison of the blood- and lymph-sugar levels has been formulated. When these conditions are satisfied, the sugar contents of lymph and plasma were nearly the same. The glucose-tolerance curves for simultaneously collected lymph and plasma of anesthetized and unanesthetized dogs were detd. The curves for lymph very closely resemble those of plasma. When sugar is given either by intravenous injection or by stomach tube, it enters the lymphatic system readily and is removed from the lymph at approx. the same rate as from blood.

F. D. Walter  
Reflex liberation of circulating sympathin. A C Liu and A. Rosenbluth. *Am. J. Physiol.* 113, 555-6(1935). cf. *C. A.* 29, 8118<sup>1</sup>—Stimulation of an afferent nerve in adrenalectomized rats under urethane anesthesia may lead to a delayed contraction of the denervated micturiting membrane. The response depends upon how many sympathetic nerves are intact in the animal and available for reflex activation. The agent responsible for the contractions is sympathin liberated reflexly into the blood. Sympathin may play a role as a hormone in certain physiol. conditions.

F. D. Walter  
Effect of physical training on blood volume, hemoglobin, alkali reserve and osmotic resistance of erythrocytes. John E. Davis and Nathan Brewer. *Am. J. Physiol.* 113, 686-91(1935).

F. D. Walter  
Acidosis as a factor of fatigue in dogs F. W. Schlutz, Minerva Morse and A. B. Hastings. *Am. J. Physiol.* 113, 595-601(1935); cf. *C. A.* 29, 6949<sup>1</sup>—The effect of exptl. alkalosis and acidosis on the capacity of dogs for work was studied. Alkalosis induced by the administration of NaHCO<sub>3</sub> often reduced the capacity of dogs for muscular exercise although the acid-base balance of the blood was normal at exhaustion. Conversely, acidosis following ingestion of NH<sub>4</sub>Cl often exerted a favorable effect and in no case appreciably reduced the capacity of the dogs for exercise. Ingestion of NaHCO<sub>3</sub> promoted the formation of lactic acid in the exercising dog while that of NH<sub>4</sub>Cl depressed its formation. Acidosis accompanying phys. exercise is not to be regarded as a causal factor of fatigue in dogs.

E. D. Walter  
The excretion of phenol red by the dog. James A. Shannon. *Am. J. Physiol.* 113, 602-10(1935); cf. *C. A.* 29, 7441<sup>1</sup>—The excretion of phenol red in the dog was studied with special reference to the effect of plasma level. As the plasma level is raised the phenol red clearance falls, both absolutely and relative to the inulin clearance, until at plasma levels of above 25 mg. % the phenol red/inulin clearance ratio is less than 0.7. The depression of the phenol red clearance effected by raising the plasma level of the dye is reversible. The excretion of phenol red is not specifically influenced by phlorizin. Wide variations in urine flow appear to have no effect upon the clearance of this dye.

E. D. Walter  
The influence of frequency of contraction of the isolated mammalian heart upon the consumption of oxygen. A. E. Cohn and J. M. Steele. *Am. J. Physiol.* 113, 654-8(1935); cf. *C. A.* 21, 1020—Expts. are described which confirm the observation that the rate at which dog hearts beat in heart lung preps. influences the consumption of O<sub>2</sub>.



directly. Both in expts in which the hearts were driven by induction shocks and in those in which they were influenced only by the nature of the prepn. itself, the rate of  $O_2$  consumption progressed about  $1/4$  as fast as change in the rate of contraction. Elec stimulation itself had no effect on the rate of metabolism. E D Walter

Factors concerned in the arrest of contraction in an ischemic myocardial area. Robert Tennant. *Am J Physiol* 113, 677-82(1935).—Simultaneous registration of optical myograms and aortic pressure pulses indicate that abolition of myocardial contraction similar to that following coronary occlusion is produced in the presence of  $O_2$  by KCl and NaCN, but not by  $CH_3CO_2Na$ . Perfusion of a ventricular zone in the normally beating heart with Na lactate in buffered blood-Locke's soln. similarly arrests contraction. This suggests that excess lactate is a factor in preventing contraction under anoxic conditions. This does not exclude change in  $pH$  as another possible mechanism. E D Walter

Sex hormones (Ruzicka, Wettstein) 10 Allo-1-pregnene-3,20-dione—specificity of corpus luteum activity (Butenandt, Mamoh) 10 Sex hormones and related substances (Dirschel) 10

## G—PATHOLOGY

H GIDEON WELLS

Carcinogenic hydrocarbons and their relationship to the sterols. J W Cook. *Chem Weekblad* 32, 563-6 (1935).—Review of historical development of recognition of the cancer-producing constituents of coal tar and earlier investigations, the development of fluorescence spectroscopy and the systematic study of synthetic carcinogenic hydrocarbons including a summary of the results obtained from the examn. of a complete series of tetra- and penta-cyclic hydrocarbons and a moderately wide range of benzanthracene derivs. The speculation is advanced that a meso H atom in a completely aromatic structure plays some role in the carcinogenic process. The isolation of 1,2 benzopyrene from coal tar and its synthesis are re-counted and an account is given of the cyclization of bile acid derivs., and the eventual synthesis of methylcholanthrene, a carcinogenic hydrocarbon related to the sterols. It is inferred that there is some enzymic factor present in the animal body which is capable of effecting dehydrogenation of the sterol ring system and that excessive activity of this factor may conceivably contribute to the production in vivo of carcinogenic compds. C R Addinall

The chemical variability of (tuberculous) primary infection and reinfection and the changes occasioned by carbonic and lactic acids. M. S. Gostev and R. A. Radkevich. *Z. Tuberk.* 72, 97-101(1935).—The chem. compn. of 3 primary infections and 3 reinfection foci are recorded. Qualitatively the foci resembled each other but quantitatively the primary infections showed a greater mineral content. As  $CaO$  and  $P_2O_5$ , the percentages differed in the well known compn. of calcification. As a cause of exacerbation in tuberculosis the accumulation of lactic and carbonic acids in the tissues plays a part. The solvent action of acids on  $Ca$  depositions is significant. H J. Corper

Chloride in blood and urine in rheumatic infection. M I Krynski and I B Schulzko. *Wien klin. Wochschr.* 48, 1065-7(1935).—Cl concn in urine is low in acute rheumatic infections and returns to normal in convalescence. Blood Cl shows variations but these are not closely related to the progress of the disease. D B D

The pathological physiology of the infarct. III. Ammonia and lactic acid in necrosis and autolysis. G. Borger, H. Bayerle, T. Mayr and E. Peters. *Z. physiol. Chem.* 237, 113-20(1935), cf. C. A. 29, 6644'.—Infarcted tissue, resulting from ligation of the blood vessels, undergoes a coagulation necrosis wholly different from the autolysis of excised tissue. The 2 are readily distinguished by the difference in  $NH_3$  content. During the 1st 20 hrs. the infarcted tissue shows a somewhat higher  $NH_3$  content than normal tissue, then the  $NH_3$  returns to the normal value. Probably an incipient autolysis occurs which is

soon checked by the onset of necrosis under control of vital processes. The excised tissue, on the other hand, rapidly undergoes autolysis, so that in a few days its  $NH_3$  content is many times that of infarcted tissue. In infarcted tissue the  $pH$  increases and the lactic acid content decreases, while the reverse is true of autolyzing tissue. A. W. D.

The nature of experimental catalepsy. A. van Harreveld and D. J. Kok. *Arch. nederl. physiol.* 20, 411-29 (1935).—Nineteen references. F. L. Dunlap

Fixation antigens of the tubercle bacillus. I. Fixation antigens contained in the lipid substances extracted from heat killed bacilli. Michel A. Macheboeuf, Georgette Lévy and M. Chambaz. *Bull. soc. chim. biol.* 17, 1194-1200(1935).—See C. A. 29, 5473'. II. Fractionation and purification of the active lipide fraction. Michel A. Macheboeuf and Antoine Bonnesol. *Ibid.* 1201-9. The active material obtained as above was further purified by repeated pptn. from  $CHCl_3$  soln. by  $Me_2CO$  and by cold  $MeOH$ . III. Purification of the alexin fixing hap-

tene. Separation from phosphatides and removal of nitrogen-containing impurities. Some physicochemical properties of the active fraction. Michel A. Macheboeuf, Georgette Lévy and Marguerite Faure. *Ibid.* 1210-34. Other methods of isolating the active substance were tried. The purest product obtained contained 3.4% P and no N, carbohydrate, sterol or unsaponifiable material. It rapidly swelled and dispersed in water and was extd. by water from its  $Et_2O$  soln. L. E. Gilson

Polypeptide contents of blood and spinal fluid in general paralysis. Cyto-polypeptide dissociation. A. Prunelli. *Bull. soc. chim. biol.* 17, 1378-84(1935).—See C. A. 29, 4820'. L. E. Gilson

Relation of cell lysis (hemolysis and leucolysis) to the cholesterol content of pleural fluids. V. de Lavergne and P. Kissel. *Compt. rend. soc. biol.* 120, 151-2(1935).—In pathol. conditions the cholesterol content of the fluids is high, 10-240 mg. %. The cholesterol is liberated by lysis of white and red corpuscles. L. E. Gilson

Production of hyperpolydipidemia in rabbits by intra-peritoneal injections of peptone. O. Lambret and J. Driessens. *Compt. rend. soc. biol.* 120, 184-6(1935). L. E. Gilson

Histological changes in the spleen and in Jensen sarcoma grafts during regression of the latter due to the action of insulin. O. Lambret and J. Driessens. *Compt. rend. soc. biol.* 120, 188-90(1935); cf. C. A. 29, 6644'. L. E. Gilson

Experimental azotemia by injection of diphtheria toxin in the guinea pig. J. Chaher, M. Jeune and R. Fornier. *Compt. rend. soc. biol.* 120, 206-7(1935).—Many times the lethal dose was injected. The blood urea began to increase about 6 hrs. later and continued to increase as long as the animals lived. It averaged 0.16% at the end of the 15th hr. and in one which lived 3 days it reached 0.4% (normal is 0.03-0.04%). The adrenaline content of the adrenals decreased rapidly after the 6th hr. L. E. Gilson

"Index of polydipidemia" in experimental cancer in guinea pigs and in human cancer. René Loucq. *Compt. rend. soc. biol.* 120, 250-2(1935).—In absence of cachexia or infections there was no significant change in blood polydipptides. L. E. Gilson

Changes in the fibrinogen content of the blood in cancer. René Loucq. *Compt. rend. soc. biol.* 120, 253-5(1935).—In human cases and in exptl. cancer in guinea pigs the fibrinogen content was about doubled. L. E. Gilson

Effects of tumor extracts on virus and staphylococcus infections. G. Van Der Schueren. *Compt. rend. soc. biol.* 120, 261-3(1935).—In rabbits, exts. of various animal tumor tissues inhibited vaccine and herpes virus, had no effect on Shope's virus, and stimulated staphylococcus development. L. E. Gilson

The Donagógico reaction in diabetics. Paolo Brocca. *Boll. soc. ital. biol. sper.* 10, 737-8(1935).—Diabetes does not affect the Donagógico reaction which is pos. only when albuminuria accompanies the glucosuria. H. L. G.

Chemical constitution of the lipoma. Emanuele and Guido Stolfi. *Boll. soc. ital. biol. sper.* 10, 742-4(1935).—In a study of 6 cases there were no appreciable differences

in the constitution of the fats of the lipoma and the subcutaneous connective tissue.

Helen Lee Gruelch  
Antigenic power of glycogen and amides. A. Giovannardi. *Boll. soc. ital. biol. sper.* 10, 771-80(1935).—Merkel glycogen and sol. amide produced some precipitins and complement-fixing bodies after repeated injection into rabbits.

Helen Lee Gruelch  
Proteinemia in thyroid dysfunction. R. Olivetti and A. Bobbio. *Minerva med.* 1935, II, 430-46.—Serum proteins (Howe-Cionini method) were detd. on 52 persons. There was a hypoproteinemia with lowering of the albumin globulin ratio accompanying the syndrome of lowered basal metabolism while hyperproteinemia with hyperalbuminemia occurred in myxedema.

Helen Lee Gruelch  
Porphyrin formation by pathogenic fungi of the skin C. Carré and A. St. v. Mallinckrodt-Haupt. *Arch. Dermatol. Syphilis* 170, 521-9(1931).—Forty-two species of skin fungi were investigated for porphyrin formation, as shown by fluorescence and spectroscopy. Species which are elaborated in the deeper skin layers or inside the body, such as sporotrichum and yeasts, produced much more porphyrin than strains which usually remain in surface layers of the skin.

O. Hartley  
The influence of pathological skin conditions on experimental hyperketonemia. Alberto Midana and Luigi Del Grande. *Arch. Dermatol. Syphilis* 171, 203-22(1935).—Twenty-three patients with extensive dermatoses, chiefly psoriasis and eczema, and 10 controls, were given a carbohydrate free diet and tested for induced hyperketonemia. Increase in ketone bodies in normal individuals averaged 8.7% and did not exceed 39%, in skin patients, it averaged 84% and reached 181% in one case. There was high correlation between area of skin involved in the dermatosis and percentage of increase in ketone bodies, irrespective of the nature of the skin disease.  $\beta$ -Hydroxybutyric acid showed the greatest increase. The test was repeated on several patients after recovery and nearly normal values were obtained. Normal liver function was proven in all the patients, so that the increase in ketone bodies is attributed to disturbances in skin metabolism caused by the dermatoses. The ketonemia is much greater than that found in severe liver damage, perhaps because the wt. of the skin is 3 times that of the liver.

O. Hartley  
Investigations of turpentine hypersensitivity with the patch test. Niels Danbolt and W. Burckhardt. *Arch. Dermatol. Syphilis* 171, 252-9(1935).—Fifty-nine persons with a pos. skin test to turpentine were tested with  $\alpha$ -pinene; all reacted. The response produced by  $\alpha$ -pinene was of the eczematous type; that due to  $d$ - $\alpha$ -pinene, the toxic type. When fractionally distd., the turpentine fractions boiling above pinene gave only weak skin reactions. The toxic response is produced chiefly by undiluted turpentine or pinene and never observed with a 10% soln. In 166 persons tested, undiluted turpentine produced a toxic reaction in 58%; with 30% strength, only 15% reacted.

O. Hartley  
The reaction capacity of the skin to chemical eczemas on the basis of investigations on eczematous and healthy individuals with chemical irritants. N. S. Vedrov and A. P. Dolgov. *Arch. Dermatol. Syphilis* 171, 641-6(1935).—Of 324 eczematous patients tested, 28% reacted to white Russian turpentine, 33% to refined petroleum, 11% to 5% formalin and 7% to uncture of arnica. In 100 healthy persons, the percentages were 6, 11, 3 and none, resp. A new test was developed for measuring general skin sensitivity; it made use of (1) phenol in benzene in 3, 5, 10, 15, 20 and 25% strengths, and (2)  $HgCl_2$  in acetone in a duplicate series of concns. Eighty % of the eczema patients reacted to both chemicals in 5-10% solns.; 80% of the healthy persons required 15-20% solns. The greater sensitiveness of allergies is considered due to a lower irritability-threshold, possibly because of increased skin permeability.

O. Hartley  
Specific sensitization of the skin to simple chemicals. N. S. Vedrov and A. P. Dolgov. *Arch. Dermatol. Syphilis* 171, 647-64(1935).—Allergic individuals are much more easily sensitized to specific chemicals than nonallergic, but in some industries a large proportion of

the latter may become allergic. Out of 81 men handling turpentine in their work, 45% tested pos. to it, as compared with 6% of 160 persons not so employed, and 17% of persons with idiopathic eczemas. Of 72 eczema patients tested, 70% were sensitized to dinitrochlorobenzene by a single drop of 10% soln. in acetone. Sixteen references.

O. Hartley  
Sulfur content of hair and of nails in abnormal states. II. Nails. Joseph V. Klauder and Herman Brown. *Arch. Dermatol. Syphilis* 31, 26-34(1935); cf. C. A. 28, 4954.—Of 44 cases of abnormal nails examd., only 10 contained a normal amt of S; the lowest value was 1.1%. Hydrolyzed wool was administered orally for 3 months, with improvement in only 5 cases, although the S content increased in the nails of some of the others. Subnormal values for S were found in 90% of normal nails of patients (33) with various systemic infections. The subnormality was greater in nails than in hair in these cases. Four patients sensitized to sunlight had normal S in their nails.

O. Hartley  
Gastric acidity in acne vulgaris with a consideration of normal gastric acidity. Samuel L. Immerman. *Arch. Dermatol. Syphilis* 31, 343-7(1935).—Fractional gastric analyses of 93 cases of acne vulgaris after bread and water meals failed to show hyp acidity or any correlation between gastric acidity, hemoglobin content and red blood cell count.

O. Hartley  
Minerals in relation to disease of the larger domesticated animals. H. H. Green. *Empire J. Exptl. Agr.* 3, 363-78(1935).—A review is given of the dietary significance of P, Ca, Mg, Fe, Cu, I, F, Cl, Na and K with reference to the occurrence of osteoporosis, osteomalacia, rickets, osteofibrosis, nutritional anemias and hypomineralisms. Forty-three references. K. D. Jacob

Experimental study on the formation of gallstone. I. Influences of fat-soluble vitamins, especially vitamin A (cod-liver-oil and "biostern"), upon the amounts of potassium, sodium, calcium and magnesium in the blood. Tamotsu Maruno. *Japan J. Gastroenterol.* 7, 120-4(1935).—During a period of 2 months different rabbits were fed cod-liver oil (10 cc per diem), olive oil and injected with biosterol. The serum constituents with cod-liver oil changed as follows in the eight-week period: Ca (10.9 to 11.2), Mg (2.6 to 3.6), K (7.3 to 10.5) and Na (455 to 293). All values are in mg. % M. considers these values typical and the changes due to excess of vitamin A since olive oil alone caused no changes.

C. M. McCay  
The function of parathyroid hormone in heat stroke. Variations of calcium and potassium in the serum. E. Sanfilippo and S. Ricca. *Biochim. terap. sper.* 22, 411-20(1935).—The hyperglucemia produced in heat stroke is considerably increased by previous treatment with parathyroid hormone. The hypocalcemia is more accentuated in the initial stage in the treated animals and the high K content of the blood is conspicuous in the terminal stage of hyperthermia.

A. E. Meyer  
The blood sulfur in Addison's disease. Paolo Larizza. *Biochim. terap. sper.* 22, 458-64(1935).—A considerable increase is due primarily to an augmentation of the org. nonprotein S, to a lesser degree to increase of the inorg. S. The protein S is slightly decreased in the serum, but increased in the whole blood and in the corpuscles. The findings were not changed by cortical hormone therapy and intravenous NaCl treatment.

A. E. Meyer  
Blood cholesterol in the preagonic period of tuberculosis. Isidoro R. Stemberg. *Semana med.* (Buenos Aires) 1935, II, 1223-8.—The increase of blood cholesterol in the terminal stage of tuberculosis is an exception.

A. E. M.  
The xanthoproteic reaction in blood as a test of renal function. Håkon Rasmussen. *Acta Med. Scand.* 86, 302-14(1935).—"The xanthoproteic test is of clinical value as a prognostic sign in chronic renal disease since it indicates the onset of true chronic uremia."

S. M.  
Coronary disease and coronary thrombosis in youth. Paul D. White. *J. Med. Soc. New Jersey* 32, No. 10, 690-605(1935).—Specific studies were made on 4 cases of coronary disease under the age of 30, 21 under the age of

40 and 138 under the age of 50 years. Metabolic disturbances caused deposition of fat in the intima of arteries; the presence of these deposits excites fibrosis, calcifications and atheromatous abscesses. Infections did not seem to play a definite part in the production of coronary involvements.

James C. Munch

Contact dermatitis. Robert L. Howard. *J. Oklahoma State Med Assoc* 28, No 7, 259-61 (1935).—H. presents a detailed list of household remedies, animal hairs or emanations, fabrics, cosmetics, dusts, metals, lacquers, varnishes, plants, foods, dyes, chemicals and drugs found to produce contact dermatitis, together with case reports for certain cosmetics, silk and Ni. Among the chemicals reported are  $\text{NiH}_2\text{persulfate}$ ,  $\text{HgS}_2$ , Metol, pyrogallol acid, naphthalene, oil of mirbane, *p*-dichlorobenzene, *p*-toluidine, pyrethrum, pyroxylol and cellulose acetate resin. Among the drugs were benzocaine, butesin, butyn, chloral, cocaine, ephedrine, nupercaine, orthoform and procaine.

James C. Munch

Adsorption of diphtheria toxin and toxoid on colloidal gels. F. A. Miller, Thos DeVries, and Mary Ann Miller. *Proc Indiana Acad Sci* 44, 88-92 (1934).— $\text{Al(OH)}_3$ ,  $\text{Ca}_3(\text{PO}_4)_2$  and silica gels have been studied to det. their adsorptive powers for diphtheria toxin. The gels tested seem to possess properties of adsorbing the toxin in considerable amts and the rate of release when injected into animals was relatively slow, seeming to offer advantages over the unmodified antigen in the production of antitoxin. The method of detg. the degree of immunity developed by toxoid has been modified. Instead of injecting increasing doses of toxin to find the degree of immunity developed in the animals, the guinea pigs are bled 3 and 4 weeks after giving the toxoid and pooled samples of the blood serum are then tested for the actual no. of antitoxic units produced.

W. J. Peterson

Gastric ulcer formation by bile acid salt. Munehide Yoshitomi. *Fukuoka-Ikudaiigaku-Zasshi* 28, 406-14 (1935).—An attempt was made to produce gastric ulcer by bile acid in rabbits. Some ulcers were formed by transfusion with Tyrode soln or with dil rabbit blood, both contg.  $1/1000$  mole of Na taurocholate. The ulcer-forming action of the bile acid is closely related with the acidity of the gastric juice. Lecithin inhibits the ulcer-forming action of the bile acid. The acidity of the gastric juice in the rabbit is slightly decreased by Na taurocholate.

K. Sugura

## H-PHARMACOLOGY

A. N. RICHARDS

The present state of our knowledge of the therapeutic action of organic arsenic compounds. H. Schlossberger. *Ber.* 68A, 149-63 (1935).—An address. G. G.

Fungistatic and fungicidal effects of two wood preserving chemicals on human dermatophytes. Sodium *o*-2-chlorophenylphenolate and sodium tetrachlorophenolate. Lester M. Wieder. *Arch Dermatol. Syphilis* 31, 644-55 (1935). O. Hartley

Treatment of burns with A and D vitamins and camphor oil. Carlos O. Franzetti. *Semana med.* (Buenos Aires) 1935, II, 998.—Combination of local application of 7% camphor oil with medication of A and D vitamins causes disappearance of the general intoxication caused by burns and of edema. A. E. Meyer

## I-ZOOLOGY

R. A. GORTNER

Reducing substances and chlorides of the blood of Orthoptera. Raoul M. May. *Bull. soc. chim. biol* 17, 1045-53 (1935).—Data are given for *Locusta viridissima*, *Diurippus* (*Caraculus*) *marousi* and *Orphania denticauda*. Blood Cl ranged from 0.310 to 0.352% and the glucose equiv. of the reducing substance (Baudouin-Lewis method) ranged from 0.04 to 0.27%, with no great differences between the 3 species. L. E. Gilson

Biochemistry of the aphids (*Pemphigus utricularis*, *P. corniculatus*) infesting *Pastacia terebinthus*. J. Timon.

David and B. Gouzon. *Compt. rend. soc. biol* 120, 164-6 (1935). L. E. Gilson

Microinjection study of the red corpuscles of the teleost, *Cichlasoma fasciatum* Jen. A. Polcard and P. Rojas. *Rev. soc. argentina biol* 11, 164-5 (1935); cf. C. A. 29, 4039\*.—Each cell leaves a ring of brown ash contg.  $\text{Fe}_2\text{O}_3$  enclosing a white dot of Fe-free ash left by the nucleus. L. R. Gilson

A study of the moisture requirements of the eggs of the chicken ascarid *Ascaridia galli*. Anne McKee. *J. Parasitology* 21, 220 (1935).—The chicken ascarid eggs required a relative humidity above 81% at 22° to survive. At 100% humidity and 30° the eggs develop the same as controls in  $\text{H}_2\text{O}$ . F. P. Griffiths

Some marine biotic communities of the Pacific Coast of North America. Early stages of succession from marine conditions to land. Archie MacLean. *Ecological Monographs* 5, 319-24 (1935).—In a lagoon which was undergoing transformation from sea to land, bivalves were favored by or tolerated a certain amt. of org matter and S compds, but  $\text{H}_2\text{O}$  which contained 2038 cc.  $\text{H}_2\text{S/L}$  was unfavorable to their growth. E. D. Jacob

The physiology of the silk-producing gland in the silkworm (*Bombyx mori* L.). I. Rivka Ashbel. *Arch. sci. biol.* (Italy) 21, 192-6 (1935).—The excretory tubes are the most active part from the point of view of respiration and from this it is concluded that it is the predominating part in the function of the gland. The intensity of respiration is a function of the temp. P. F. Mettli

The carbohydrates of the albumin glands of *Rana esculenta*. Fr. N. Schütz and Max Becker. *Biochem. Z.* 280, 217-26 (1935).—Hydrolysis with dil. acids of the dry substance from the albumin glands of *Rana esculenta* sets free 30-35% reducing substance. Of this about 1-1.5% is polysaccharide (glycogen). Galactogen could be detected only in traces. The glycoprotein yielded on hydrolysis 1 mol. galactose and 1 mol. glucosamine. S. Morgulis

Studies on the chemical embryology of amphibia. VIII. Oxidation processes in the giant salamander egg. Fujito Yamasaki. *J. Biochem. (Japan)* 22, 181-4 (1935). cf. C. A. 27, 4851.—An increase in glutathione content during the development of the salamander egg is said to occur. (This statement can only be accepted, if the glutathione content recorded for the early stage is a typographical error.) The indophenoloxidase reaction appears very early not only in the embryo but also in the perivitelline fluid. No glutathione was found either in the perivitelline fluid or in the jelly. IX. Presence of urea and uric acid in the fertilized giant salamander egg. Makoto Takamatsu and Tatsumi Kamachi. *Ibid.* 185-7.—The fertilized salamander egg contains relatively little urea and only traces of uric acid. S. Morgulis

The muscle extractives in hibernating giant salamanders (*Megalobatrachus japonicus*). Takeshi Iwasaki. *J. Biochem. (Japan)* 22, 233-42 (1935).—In muscle ext. from the hibernating giant salamander there was much creatine and an appreciable amt of methylguanidine but no arginine or carnosine. S. Morgulis

The permeability of the skin of frogs to water as determined by  $\text{D}_2\text{O}$  and  $\text{H}_2\text{O}$ . G. v. Hevesy, E. Hofer and A. Krogh. *Skand. Arch. Physiol.* 72, 199-214 (1935).—The permeability is measured by the no. of days which 1 mole requires to pass through 1 sq. cm. at a const. difference of 1 mole concn. In expts with  $\text{D}_2\text{O}$  on isolated skin and frog legs the permeability was found to be the same in both directions and no effect of ions was observed on the permeability. The permeability of the web is about 0.1 as great as that of the skin from the abdomen or thighs. In short expts. the permeability varied inversely with the viscosity of the water. After a longer time (10 hrs.) the permeability at low temp. decreases further. Sectioning of the nerve causes an increase of about 30% in the permeability of the operated leg, which lasts several days. The permeability with  $\text{D}_2\text{O}$  at 21° was 100-170 and with ordinary  $\text{H}_2\text{O}$  22-37. S. Morgulis

The action of thyroxine and similar chemical substances

on the development of sea-urchin larva. Marie-Rose Zellins. *Bull. inst. oceanograph. No. 678*, 10 pp. (1935).—Thyroxine causes characteristic retardations in the growth and differentiation of sea-urchin larva in dilns. of 1/50,000 to 1/600,000 parts. It is without effect before the first mitotic division, but in later stages it modifies general cellular metabolism. Chemically related substances do not have such an effect.

Walter H. Seegers

Larval and imaginal tracheal systems of the odonata and their metamorphosis. Heinrich Wolf. *Z. wiss. Zool.* 146, 591-620 (1935).—Largely morphol. The epidermis of the tracheae, unlike that of the body skin, is strongly pigmented, apparently with hemoglobin, the indications are that this exerts an oxygen-carrying function. Immediately after metamorphosis the imago of eschmids secretes a soln. of uric acid.

K. V. Thumann

Physiology of the movement of Malacodella. Friedrich Eggers. *Z. wiss. Zool.* 147, 101-31 (1935).—Unlike

other Nemertinea, the organism is not free-living, but commensal in *Cyprina islandica*. Probably on this account, it is completely devoid of chemotaxis either to the host or to the other sex. It is also geo- and photo-tactically neg.

K. V. Thumann

Osmoregulation in native crustaceans of water and moist air. Erna Widmann. *Z. wiss. Zool.* 147, 132-69 (1935).—By f.-p. detns the blood of a no. of crustaceans was found to undergo an annual cycle, the salt content reaching a max. from November to March and a min. from May to September for all animals. The effect is due to temp. The decrease in concn. of blood salts for 10° rise in temp. varies for different animals and sometimes for different sexes of the same animal, it causes an increase in f.-p. depression of 0.1-0.3° on the total  $\Delta$  of 0.6-2.2°. In hunger the salt concn. of the blood is lowered by a  $\Delta$  of 0.2°, thus opposing the winter effect of low temp. Salt feeding raises the blood salt concn.

K. V. Thumann

## 12—FOODS

F. C. BLANCE AND H. A. LITPER

Analyses of common foods. E. M. Bailey. Conn. Agr. Expt. Sta., *Bull.* 373 (39th Rept. on Food Products) 529-51 (1935).—These collected proximate analyses of the several classes of foods are brought up to date in convenient form for use.

C. R. Fellers

Metallic contaminations of foods. II. Effect of cooking and storage on foodstuffs in aluminum vessels. N. C. Datta. *Proc. Indian Acad. Sci.* 2B, 322-32 (1935). cf. C. A. 29, 1889°.—The Al in foods cooked and stored in Al vessels was detd. gravimetrically as the phosphate. Milk and milk products dissolve only traces of Al from vessels. The small amt. of Al dissolved by fruit and vegetable juices during storage in Al vessels depends on the nature of the org. acid present and on the buffering capacity of the food. The presence of NaCl increases the corrosive action of acid foods on Al. The amt. of Al dissolved by tamarind soln. contg. NaCl almost equals the sum of the amt. dissolved by each separately. A max. of 50 mg. of Al can be added daily to the diet by the inclusion of acidic foods contg. NaCl and cooked and stored in Al vessels. Feeding expts. with rats show that foods prepd. in Al vessels have no harmful effect on the growth, reproduction and general well-being of the animals. Rachel Brown.

Rachel Brown

Aluminum possesses wide adaptability to many conditions and requirements of the food industries. J. R. Akers. *Food Ind.* 7, 587-8 (1935).—General.

C. R. Fellers

Glass-lined equipment. P. S. Barnes. *Food Ind.* 7, 590-2 (1935).—The value of vitreous and glass equipment in the food industries is pointed out.

C. R. Fellers

Rubber products find increasing use in food manufacture. A. E. Juve. *Food Ind.* 7, 594 (1935).

C. R. Fellers

Plastics find increasing utility in food equipment and food plant accessories construction. James A. Lee. *Food Ind.* 7, 587-6 (1935).—General.

C. R. Fellers

Paints and painting to protect the equipment and plant interior from corrosion; paints must be chosen and applied with care. Victor Buhr. *Food Ind.* 7, 597 (1935).

C. R. Fellers

Paints for plant and equipment. R. C. Sheeler. *Food Ind.* 7, 598 (1935).

C. R. Fellers

Steel alloys (in food manufacturing). Walter M. Mitchell. *Food Ind.* 7, 577-9 (1935).—Stainless steels of the 18-8 type give satisfactory results in food processing where the metal comes in contact with foods.

C. R. Fellers

Nickel and its alloys. F. L. LaQue. *Food Ind.* 7, 550-2 (1935).—Ni and its alloys satisfy the exacting requirements of food-processing equipment. Their non-corrosiveness is especially stressed.

C. R. Fellers

Copper, brass and bronze in the food industries. Carter S. Cole. *Food Ind.* 7, 583-4 (1935).—The uses are outlined.

C. R. Fellers

Copper alloys in food manufacture. James T. Kemp. *Food Ind.* 7, 584 (1935).—Greater strength is imparted by alloying Cu, and with some alloys, greater resistance to corrosion.

C. R. Fellers

Tin as an anticorrosion coating. Bruce W. Gosner. *Food Ind.* 7, 585 (1935).—General.

C. R. Fellers

Lead (in the food industries). F. E. Wormser. *Food Ind.* 7, 586 (1935).—Pb is never used in contact with foods because of its toxic properties. The use of Pb and Pb compds. in building construction and maintenance is outlined.

C. R. Fellers

The fermentable glucides of flour and dough. R. Guillemet. *Compt. rend. acad. agr. France* 21, 899-92 (1935); cf. C. A. 29, 6654°.—These glucides, more or less hydrolyzable, with high enzymolytic index, slightly sol in alc., and in part clarified by Pb, are fructohololoses. It is not a question of Tanret levosins, stachyose, raffinose nor gentianose. In dough in fermentation, the sucrose from the living yeast with fructose forms 1/3 of the fermentable stock of white flours and regulates in a large measure the course of the bread making fermentation. The previously formed hexoses and the sucrose and fructose detached from the fructohololoses ferment first; the maltose is attacked more and more rapidly.

J. R. Adams

The application of fats and oils to the baking industry. Geo. F. Garnatz. *Oil and Soap* 12, 290-3 (1935).—The application of fats and oils in the production of bread, cakes, biscuits and crackers is discussed.

E. S.

What is a quality dairy product? M. E. Parker. *Natl. Butter and Cheese J.* 26, No. 20, 26-7 (1935).

A. H. Johnson

Further investigations on the determination of the hydrogen-ion concentration of milk by the colorimetric method. G. Schwarz and Ottmar Fischer. *Milchsch. Forsch.* 17, 158-69 (1935). cf. C. A. 23, 3991°.—As standards for comparison, instead of inorg. buffer solns., milk serums were prepd. by using normal sweet milk, adding to portions increasing amts. of sour milk, treating with methanol (20 ml. milk, 30 ml. methanol), placing the mixt. in a refrigerator for 30 days and then filtering. The pH of serums was detd. electrometrically, indicator added and the serum placed in sealed glass tubes to fit in a comparator device. These standards were good for 6 months. The indicator soln. consisted of 20 mg. bromocresol blue, 50 mg. methyl red and 100 mg. bromocresol purple dissolved in 1000 ml. neutral methanol. One ml. indicator was added to 10 ml. serum. Samples of serums to be tested were prepd. by addn. of methanol in the ratio given above. Measurements were made with an accuracy of about 0.1 pH within limits of pH 5.6-6.7.

H. Macy

A gravimetric micromethod for determination of milk fat and dry matter. G. Gorbach and R. Kadner. *Milchsch. Forsch.* 17, 100-2 (1935).—A new app. is described for the

microdetn of milk fat and dry matter by ether ext. The method is much faster than macromethods with a Soxhlet extractor. The max. error was 0.07% for fat content, but somewhat greater for dry matter. H. Macy

Composition of media for the bacteriological analysis of milk. C. S. Bowers and G. J. Hucker. N. Y. Agr. Expt. Sta., *Tech. Bull.* 228, 3-42 (1935).—A total of 1142 samples of milk of types normally encountered in control work was studied, with agar of varied compns. Comparative bacterial counts were made to det. their relative efficiencies. The addn of yeast ext. to standard agar or its substitution for beef ext. did not increase the efficiency of the medium. However, yeast ext. is desirable in media for the cultivation of *Leuconostoc* and *Lactobacillus*. The addn of a fermentable carbohydrate and 0.5% skim milk to standard agar increased its efficiency. The colonies were more numerous, larger, and more readily counted. The best all round medium for milk control consisted of 0.5% tryptone (Digestive Ferments Co.), 0.1% glucose, 0.5% fresh skim milk and 1.5% agar. C. R. P.

"Milking through" cows, the qualities and composition of their milk and the suitability of an Austrian Alpine breed for the "finish milking" industry. Wolfgang Schempfing. *Milchforsch* 17, 118-45 (1935).—Expts are reported concerning companions in compn and properties of the milk from cows beyond their normal lactation period and those under normal conditions of lactation. Percentage of fat, size of fat globules, creaming ability, sp. gr., total dry matter, fat-free dry matter, viscosity, lactose, protein, cond., Ca, Cl, K, Na, H<sub>2</sub>PO<sub>4</sub>, acidity, 1-p depression, rennet coagulability, bacterial flora, reductase, Schardinger enzyme and diastase were all studied and data reported for each group of cows.

H. Macy  
The relation of mastitis to rennet coagulability and curd strength of milk. H. H. Sommer and Helene Matsen. *J. Dairy Sci.* 18, 741-9 (1935).—Subclinical mastitis causes milk to have a lower curd strength and to coagulate more slowly with rennet. Philip D. Adams

What causes most common off-flavors of market milk? Wm. H. Chilson. *Milk Plant Monthly* 24, No. 11, 24-8 (1935).—About 25 to 30% of the cows in the herd studied gave milk which developed off-flavor during the months from February to June. When milk was pasteurized by heating at 143°F. for 30 minutes, the off-flavor developed to a more pronounced degree than in raw milk. If the milk which usually gave the off-flavor is heated at 170°F. for 10 min., the off-flavor does not develop. This was taken to indicate that there is an oxidizing enzyme in the milk which catalyzes oxidation and this enzyme is destroyed by heating for 10 min. at 170°F. When skim milk which had been held at 170°F. for 10 min. was mixed with raw cream to form a milk contg. 4% of fat, this milk did not develop the oxidized flavor. When raw cream was added to the heated skim milk to form 15 or 20% cream, the oxidized flavor sometimes developed. The development of the occasional oxidized flavor in the latter case and none in the former was due to the greater amt. of raw skim milk (contg. the active oxidizing enzyme) contained in the cream. Oxidized flavor was never found in skim milk unless the fat content was too high. Oxidized flavor was more likely to develop in cream of low fat content than in cream of high fat content because of the higher proportion of oxidase-contg. skim milk in the former. There was also a difference in susceptibility of fat to oxidation by the enzyme. The addn of CuSO<sub>4</sub> to cream which did not develop the oxidized flavor caused the off-flavor to develop. A. H. Johnson

Tallowy flavor in milk. C. D. Dahle. Pa. Agr. Expt. Sta., *Bull.* 320, 22 (1935).—An oxidized or tallowy flavor which developed in 48-60 hrs. at 4.5° was intensified by pasteurization, lnt heating to 80° eliminated it. The active principle, probably an oxidizing enzyme, was present in the milk plasma. The L no. of the fat was decreased in proportion to the degree of flavor present. The flavor is absent in summer when cows are on pasture.

C. R. Fellers

Investigation of resazurin as an indicator of the sanitary

condition of milk. Guy A. Ramsdell, Wm. T. Johnson and F. R. Evans. *J. Dairy Sci.* 18, 705-17 (1935).—Only 1 hr. is required to complete the resazurin test. By means of it milk can be classified into 4 groups as regards sanitary condition. Milk from diseased udders and from physiologically abnormal cows has significant effects on the reduction of resazurin. The rate of color change of resazurin-milk mixts. on incubation yields considerable information concerning the bacterial flora. Philip D. Adams

Standardization of the Borden Body Flow Meter for determining the apparent viscosity of cream. J. C. Henning. *J. Dairy Sci.* 18, 751-6 (1935).—Standardization was accomplished with sugar solns. and the seconds of flow showed a straight-line relationship to viscosity in centipoises. Philip D. Adams

Experimental cream canning. Alfred H. Loveless. *Food Sci.* 52-8, 87 (1935).—The rate of heating and the viscosity of cream were studied. The viscosity changes considerably during the 1st day after sterilizing, and continues to change at a decreasing rate over a period of many days. Variations in agitation seem to be the cause of variation in viscosity. The use of a preheating temp. of 140°F. (instead of 110°F. as generally used in com. practice) produces an increase of about 50% in viscosity after sterilization and reduces the tendency of the cream to form grains by coagulation of the protein, this gives a smoother cream. A. Papineau-Couture

What happens to cream in paper milk-containers? Thomas Duffee, W. S. Arnott and P. R. Nelson. *Milk Dealer* 25, No. 2, 40-2 (1935).—A cream layer forms less readily on milk in paper containers than on milk in glass bottles. The difference in cream-rising phenomena in the 2 types of containers was more pronounced for pasteurized milk than for raw milk. Thus the fat content of the top 50 cc. of cream from a glass bottle was 22% while the fat content of a similar sample from a paper container was 6.75%. The use of various types of paper containers, coating glass bottles with paraffin, vaseline, halowax, etc., impressing pos. or neg. charges on the milk affected the fat content of the top 50 cc. of cream. Attempts were made to explain the exptl. findings on the basis of electrostatic phenomena. Any static charge of electricity induced in the milk to create a potential sets up an electrostatic force in the fat globules. Pasteurization changes the structure of these globules sufficiently to permit the induction of a higher potential than is developed with raw milk. When milk is bottled, the container acting as a Leyden jar prevents the discharge of this potential. The container, whether glass, paraffined glass or paper, has an opposite charge to that on the fat globules in the milk. Therefore, a certain elec. attraction is created depending on the capacity of the particular container material. Since the electrostatic capacity of glass is greater than that of paraffin which in turn is greater than that of paraffined paper, the ability of glass to counteract the residual potential in the fat globules is greatest, that of paraffin being less and paraffined paper least. The above ideas were used to explain the observed data. A. H. Johnson

The keeping quality of butter depending on different packing materials. Autodetactive spoilage of butter. F. Kieferle and A. Seusz. *Milchforsch* 17, 181-9 (1935).—See C. A. 29, 6659. H. Macy

Butter cultures. W. Ritter and M. Christen. *Landw. Jahrb. Schweiz* 49, 749-60 (1935).—Volatile acids, biacetyl and acetylmethylcarbinol, formed as by-products of lactic fermentation, vary in quantity with the length of time of incubation of the milk culture. These substances are formed in much greater quantities if citric acid or citrates are added to the milk. After successive transfers, the aroma-producing streptococci may disappear from the culture and a harsh acid flavor or a malt flavor may appear. Sometimes the proportion of volatile acids will increase and that of acetylmethylcarbinol decrease without apparent effect on the flavor of the culture. F. O. W.

The diketone produced when butter cultures are steam distilled with ferric chloride added. B. W. Hammer. *J. Dairy Sci.* 18, 769-71 (1935).—It appears that the

diketone is biacetyl rather than one of the homologs. If homologs are present they are in relatively insignificant amounts.

Philip D. Adams  
International agreement for the unification of methods for the sampling and analysis of cheese in International trade. *Anon. Ann. fals.* 28, 450-6 (1935).—Text of the agreement with appendices describing the procedure to be used for sampling and the methods to be used for the determination of  $H_2O$  and fat.

A. Papineau-Couture  
The detection of preservatives in process cheese. I. G. Schwarz, Ottmar Fischer and O. Kahlert. *Milchforsch.* 17, 170-80 (1935).—Three distinct and independent processes are described for the detection of benzoic acid in process cheese (1) microscopic observations of crystal building, (2) microdeton of m. p. and (3) color reaction. Benzoic acid was found in 23 of the 135 samples.

H. Macy  
Use of hydrogen-ion determinations on young cheese in predicting acid development in Cheddar cheese during storage. Delmar W. Spicer and L. H. Burgwald. *Natl. Butter & Cheese J.* 26, No. 21, 14-15, 33-4 (1935).—The determination of the  $H$ -ion concentration during the cheese-making process did not indicate significant changes in acidity any more accurately than did the usual titration method. The amount of acid that developed in cheese while in storage was closely related to the titratable acidity at the time of milking. The amount of acid that American cheese will develop within 6 weeks time could be predicted quite accurately by determining the  $H$ -ion concentration of the cheese when from 3 to 10 days old. In a majority of cases cheese having a  $pH$  value of 5.07 or above did not develop acid in storage, while those having a  $pH$  of less than 5.07 did develop acid in storage.

A. H. Johnson  
The bacteriology of Swiss cheese. IV. Effect of temperature upon bacterial activity and drainage in the press. L. A. Burke, G. P. Sanders and K. J. Matheson. *J. Dairy Sci.* 18, 719-31 (1935). *cf. C. A.* 29, 7514.—The area just beneath the rind cools more rapidly than the interior. Bacterial growth and acid production correspond in general with the decrease in temperature of each part of the cheese.

Philip D. Adams  
Vitamin A in eggs and food. Walter C. Russell and Milton W. Taylor. *New Jersey Agr.* 17, No. 4, 3 (1935). *cf. C. A.* 29, 1465.—Eggs from hens that received no pigmented foods in the diet contained 300 units of vitamin A per yolk, as compared with 425 units in those from hens receiving a ration containing 35% of yellow corn and 3% of dried alfalfa. Both groups received cod liver oil as 1% of the total ration. Egg yolk color was not a reliable indicator of vitamin A content. The vitamin A content of the eggs amounted to 18% of the vitamin A intake in the 1st group and to 31% in the 2nd group. The livers of birds affected with leucemia, bronchitis or cholera were definitely low in vitamin A. The eggs contained 2-10 Steenbock units of vitamin D per yolk. In 1 experiment the vitamin D content of the eggs amounted to 10% of the intake in the feed.

K. D. Jacob  
The sardine canneries of California. The fish and its by-products. V. Cahalan. *Food* 5, 59-63 (1935).—A brief description of present-day sardine-canning practice in California.

A. Papineau-Couture  
The vitamin A and D contents of canned salmon. Grace M. Devaney and Lydia K. Putney. *J. Home Econ.* 27, 658-62 (1935).—The vitamin A content of salmon varies between 0.3 and 0.25 international units per g. for chum to 8 units per g. for Chinook. The vitamin D content varies from 1.9 and 2.6 units for chum and Chinook to 8 units for a red salmon.

A. L. V.  
Composition of miscellaneous tropical and subtropical Florida fruits. A. L. Stahl. *Fla. Agr. Expt. Sta., Bull.* 283, 20 pp. (1935).—The chemical composition of 5 varieties of Florida mangoes, *Mangifera indica* 7 times, *Citrus aurantiifolia* 6 lemons, *Citrus limonia* 2 limequats, 1 calamondin, *Citrus reticulata* 1 Japanese persimmon, *Diospyros kaki*, sourpump, *Annona muricata* papaya, *Carica papaya* white sapote, *Cassipouira edulis*, fig, *Ficus carica*, Surinam cherry, *Eugenia uniflora*; guava, *Psidium cattleianum*, pomegranate, *Punica granatum*, tamarind, *Tamarindus*

*indica*; and jujube, *Ziziphus jujuba*. The moisture, oil, acid, protein, ash, reducing sugars, hydrolyzable sugars and total sugars are given. Twenty-one references.

C. R. Fellers  
Changes in apples during storage. E. J. Rasmussen. *N. H. Agr. Expt. Sta., Bull.* 284, 18-20 (1935).—The effect of fertilization, especially  $NaNO_3$ , on storage life was uncertain. The fruit grown on nitrate-fertilized trees was a little softer than that from unfertilized trees and had the greenest ground color and highest acidity.

C. R. Fellers  
The vitamin B and G contents of Arizona-grown grapefruit and broccoli. Gladys Harilevich. *J. Home Econ.* 27, 663-4 (1935).—Grapefruit pulp is rich in vitamin G (0.4 unit per g.) and poor in vitamin B (0.16 unit per g.). The peel is rich in vitamin G. Broccoli has 3-4 vitamin G units per g. or 8 times more than milk. Its vitamin B content is the same as that of other leafy plants.

Amy Le Vesconte  
Presence of yeasts in fruit juices that are sold for beverages. J. M. Brannon and R. J. Pollitt. *Milk Dealer* 25, No. 2, 35 (1935).—Yeasts were found in samples of orange and grapefruit juice. If the juices were kept cold, they remained in good condition for several weeks which is much longer than juices are held before being consumed. The presence of yeasts in fruit juices should cause no particular concern as yeasts are consumed with several fresh fruits.

A. H. Johnson  
Vitamin B content of raw Pinto beans. Mary L. Greenwood. *N. Mex. Agr. Expt. Sta., Bull.* 232, 3-10 (1935).—The vitamin B ( $B_1$ ) value as determined by the Chase technique was 208 Sherman units per oz. The relation of vitamin B to health is discussed.

C. R. Fellers  
Examination of sweet pickles. E. M. Bailey. *Conn. Agr. Expt. Sta., Bull.* 373 (34th Rept. on Food Products) 509-13 (1935).—Of the 114 samples examined, 3 contained saccharin, 19 contained benzoates and 7 contained both saccharin and benzoates.

C. R. Fellers  
Stability of provitamin A in alfalfa and silage. M. W. Taylor. *New Jersey Agr.* 16, No. 6, 3 (1934); *cf. C. A.* 28, 6788.—Machine-dried alfalfa lost 50-67% of its carotene content during storage in the barn for 3-7 months, the greatest losses occurring during the 1st 3 months. Immediately after the hay was cut and dried it contained 70,000-90,000 international units of carotene per lb. Samples of silage contained 7000 units of carotene per lb. of wet material. There were indications that the carotene in silage is quite stable under silo conditions over long periods of time.

K. D. Jacob  
Molasses hay silage. C. B. Bender. *New Jersey Agr.* 17, No. 5, 3-4 (1935).—Green alfalfa and mixed grasses, averaging 68%  $H_2O$ , were chopped and packed into silos within 1-6 hrs. after cutting in the field. The green material was mixed with a 40% solution of molasses in water at the rate of 100 lb./ton. The temperature in the silos seldom exceeded 100°F. When the silos were opened in the following spring the material was in excellent condition, of good odor, quite green and succulent, and the average chemical analysis compared favorably with that of the fresh green material. When the green material was allowed to lie in the field for 12-24 hrs. before ensiling the  $H_2O$  content averaged 44%, the temperature in the silos reached a maximum of 160°F and the material was quite decomposed and moldy when the silos were opened.

K. D. Jacob  
The comparative digestibilities of artificially dried pasture herbage by sheep and rabbits. C. J. Watson. *Empire J. Exptl. Agr.* 3, 346-50 (1935).—Mixed pasture grasses, cut in August when 5 weeks old and artificially dried, contained  $H_2O$  9.07-10.0%, ash 9.27-9.37, protein 20.76-20.93, Et-O. ext. 5.11-5.15, crude fiber 21.33-21.56 and N-free ext. 43.16-43.36%. The material was a highly digestible feeding stuff for sheep but was not so satisfactory for rabbits because of the inability of the latter to make efficient use of the crude-fiber fraction.

K. D. Jacob  
Utilization of some by-products of Puerto Rican industries as feeds for livestock. F. Pico and J. H. Ramirez. *Puerto Rico Agr. Expt. Sta., Ann. Rept. Director* 1933-4, 163-4 (1935).—Grapefruit bran made from the dried

ground skin of the grapefruit has the following percentage compn.: moisture 7.53, carbohydrates 78.65, fat 1.09, protein 4.94, fiber 4.82 and ash 4.97. The product was very palatable to dairy cows. C. R. Fellers

Acidification experiments with the addition of ammonia and ammonium salts C. Windheuser, O. Hoffmann and E. Ohlmer. *Biedermanns Zentr. B. Tierernähr.* 7, 372-81 (1935).—This paper deals with the influence of admixtures of  $\text{aq. NH}_3$ ,  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{NH}_4\text{HCO}_3$  on the fermentation of feed plants. Meadow grass, red clover, soybeans, beet roots, marrow stem cabbage and maize were tested with  $\text{aq. NH}_3$ . Maize was tested in a silo; the others were tested in glass vessels. The  $\text{aq. NH}_3$  contained 2.5-7%  $\text{NH}_3$  and the added amts of  $\text{NH}_3$  were 0.12 to 0.3%. The silages from the marrow stem cabbage and maize were of unobjectionable quality, while the others—though free from butyric acid—were less satisfactory foods because of their high content of  $\text{AcOH}$  and their low content in free lactic acid. The expts. with  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{NH}_4\text{HCO}_3$  were carried out in glass vessels. Grass silage with 1% of  $(\text{NH}_4)_2\text{CO}_3$  was less good as a food—though free from butyric acid—because of its low lactic acid content and its high content in  $\text{AcOH}$ . The maize silage with 0.4%  $(\text{NH}_4)_2\text{CO}_3$  and that with 0.65-2%  $\text{NH}_4\text{HCO}_3$  were of unobjectionable quality. The digestion coeffs. of pure protein were higher and the losses of pure protein and of digestible protein were lower, but that of dry matter was somewhat higher than in the corresponding silage without admixts. F. L. Dunlap

Cosmetic colors [foodstuff colors] (Redgrove) 17 Supplying pure water for beverage manuf. (Stalder) 14 Iodine in Westphalia [detn. in spinach and milk] (Balks) 7. Some African oil seeds (Aron) 27. Influence of the reaction and fertilizing on the compn. and digestibility of meadow grass (Nehring) 15. Combination of catalysts to reduce digestion time in the detn. of N II Dairy products (Poe, Schafer) 7. Corrosion of Al [by fruit exts.] (Bryan) 9. Chocolate and cacao products—their relation to pharmacy (Churchman) 17. The action of microorganisms on milk (Gorini) 11C Sterilizing liquids [milk] (Fr. pat. 781,959) 13 Phosphatide compds. [products used in the foodstuff industry] (Ger. pat. 619,235) 25

Food flavoring materials containing lecithin. Stroud Jordan (to American Lecithin Co.). U. S. 2,019,494, Nov. 5. Lecithin is added to a normally volatile essential oil such as lemon oil or to an oil-sol. raspberry flavoring or the like, for flavoring candy, cakes, etc.

Composition for coloring foods. Hugh E. Allen and Albert G. McCaleb. U. S. 2,021,621, Nov. 19. A product suitable for coloring meat juices, meats, etc., is prep'd by reaction of animal blood hemoglobin with mono Na glutamate (suitably by heating together at about 70-75°) and may be associated with curing salts such as  $\text{NaNO}_2$ ,  $\text{NaNO}_3$  or  $\text{KNO}_3$ .

Apparatus for deaerating liquid foods (suitably as a step preparatory to irradiation with ultraviolet rays). Henry C. Stephens and Stedman B. Hoar (to Natural Food Products Co.). U. S. 2,020,250, Nov. 5. Various structural and operative details of a cascade deaerator. Amino acid salt sirup (concentrated food product). Chyosaku Wada. Brit. 432,895, Aug. 6, 1935. See Fr. 771,095 (C. A. 29, 890°).

Treating rice, rice offals or paddy, etc. Sam H. Gibbon (one-half to Steel Bros. & Co. Ltd.). U. S. 2,021,721, Nov. 19. For improving the food value of the material its water content is brought up to about 15-30% (as by soaking in warm water) and it is then maintained for a short time (suitably 5 to 10 min.) at a temp. below the freezing point of water (suitably about -40°), and is then heated in water and may be dried. App. is described.

Improving grain meal. Rudolf Rüter. Ger. 608,947, Aug. 10, 1935 (Cl. 53c 602). The baking qualities of flours are improved by treating the flour with org. amines or amides, halogenated at the N atom, e. g., toluenesulfochloroamide, amyldibromoamine

1 Determining rope infection in bread. Herbert H. Bunnell. U. S. 2,019,950, Nov. 5. Freshly baked bread is incubated and its catalase activity is then measured. App. is described.

Testing milk. Max Gebetsrother. Austrian 142,913, Oct. 10, 1935 (Cl. 53a). In detg. milk fat by the Gerber method with the aid of  $\text{H}_2\text{SO}_4$  and amyl alc., use is made of red-colored amyl alc. of sp. gr. 0.815-0.816 to which  $\text{CH}_2\text{O}$  has been added, suitably in a proportion of 5 cc. of 30-50%  $\text{ClH}_2\text{O}$  soln to 1 l. of alc. A clear dividing line between acid and fat in the butyrometer is thus obtained.

Preserving milk, cream, cheese, etc. Ralph L. Eagles. Brit. 432,894, Aug. 6, 1935. Dairy products are preserved by enclosure in air-tight substantially full receptacles, each provided with a vent designed to vent at about 4 lb. per sq. in. above atm. pressure; the product is kept therein out of contact with air and in contact with the gas, principally  $\text{CO}_2$ , generated therefrom.

Electrolytic treatment of milk, etc. "Elaet" Gesellschaft für elektrische Apparate G. m. b. H. Brit. 433,570, Aug. 18, 1935. Acid is eliminated from milk, cream, butter, whey, etc., by upward passage through the compartments formed by the end electrodes of a cell and a series of vertical plates that serve as intermediate bi-polar electrodes.

Precipitating proteinaceous matter from whey, etc., by use of chlorine. Stefan Ansbacher, Geo. E. Flanagan and Geo. C. Supplee (to Borden Co.). U. S. 2,021,712, Nov. 19. For pptn. of nitrogenous matter (as from whey in the manuf. of milk sugar),  $\text{Cl}_2$  gas is added (suitably to the point of satn. and while the material has a  $\text{pH}$  below 7.0).

Cleansing milk containers such as those lined with glass. Geo. B. Ashton (to Victor Chemical Works). U. S. 2,020,228, Nov. 5. Various details are described of a process involving rinsing the containers with water, treating them with a detergent such as one contg.  $\text{Na}_2\text{PO}_4$  and  $\text{NaOCl}$  which is allowed to remain in contact with the container for at least an hr., and then removing the detergent with water.

Preserving meat, fish, etc. Karl A. Johansson and Johan A. Wallström. Brit. 431,994, July 18, 1935. In treating meat and like food materials prior to storage with vacuum for extg. blood and other liquids and with  $\text{NaCl}$  which is forced into the materials by pressure, the materials are first placed in cases, etc., e. g., of tar-free wood or rustproof sheet metal, with salt and then subjected to the vacuum and pressure treatment. App. is described.

Preserving fish by cold storage. Hugues M. J. de Mené de Bellefont and Joseph E. A. Folliot (to Société René Mauballarg & Cie.). U. S. 2,020,109, Nov. 5. Fish are placed in containers which are hermetically sealed and subjected to a refrigerating treatment which brings the contents to a temp. below 0° but does not freeze them. App. is described.

Salting fish. Institut für Seefischerei, Otto Bähr and Otto Wille. Ger. 589,388, Sept. 30, 1935 (Cl. 53c 603). Fish-salting processes are improved by addn. of a phenol. Thus, 0.1-0.2% of hydroquinone may be added to the salt used.

Extracting fatty oils, deodorizing liquids. Wilhelm Kelle and Franz Carlsson. Brit. 432,771, Aug. 1, 1935. Oil or fat is extd. from meat, fish, fruits, etc., and waste waters or other liquids contaminated by fish, meat, etc., are deodorized by exposing at water-bath temp., e. g., 77-85°F., to the action of cryptococcus fungi or other saccharomyces types and of such bacteria as are contained in wheat and rye flours and then centrifuging or otherwise sepg.

Pectin from plant and fruit materials. Wm. C. Platt (to Calif. Fruit Growers Exchange). U. S. 2,020,872, Nov. 12. The  $\text{pH}$  of a pectin ext. contg. solid substances is adjusted (suitably to about 1.2-1.6 for filtering under pressure with use of a freely flowing siliceous filter-aid) to a  $\text{pH}$  at which clear filtrates may be obtained with a filter-aid capable of high rate of flow but normally incapable of producing clear filtrates from the exts. without such adjustment of the  $\text{pH}$ .

**Pectin preparations.** Hans Schauweker. Fr. 785,151, Aug. 3, 1935. Dry pectin is made readily sol. in water by mixing finely powd. sugars, e. g., dextroses, with the finely powd. pectin. One or more acids such as citric or tartaric may be added according to the use to which the pectin is to be put.

**Pectous material.** Mutual Citrus Products Co., Inc. Brit. 432,244, July 23, 1935. Material contg. pectose, e. g., fruit pulp, is washed to remove  $H_2O$ -sol. ingredients and the pulp is then mixed with a filter-aid, e. g., Lueselguhr, pressed and dried. The washed material may be finely ground before addn. of the filter-aid and  $H_2SO_4$  may be added at this stage. Jellies may be obtained by hydrolyzing the pectose to pectin.

**Wax emulsions suitable for coating citrus fruits.** John R. MacRill (to Calif. Fruit Growers Exchange). U. S. 2,019,758, Nov. 5. A compn which forms an emulsion when mixed with a solvent such as  $Na_2CO_3$  soln. and with water is formed of ingredients such as paraffin 553, carnauba wax 68, a vegetable oil such as cottonseed oil 98, oleic acid 183 and triethanolamine 98 parts.

**Solidified honey.** Victor J. Hampton. U. S. 2,021,450, Nov. 19. Natural honey is transformed into a hard, glassy solid product, by subjecting it to a partial vacuum, simultaneously heating it to about  $70^\circ$  and allowing the resultant viscous mass to solidify in an atm. the relative humidity of which is 45% or less.

**Increasing the viscosity of gelatin.** Jay Bowman and Vernon L. Harnack (to United Chemical & Organic Products Co.). U. S. 2,020,234, Nov. 5. Gelatin for

use as a food or for other purposes is treated to increase the ratio of its viscosity to its jelly strength by heating to about  $95-125^\circ$  for several hrs.

**Condiments for seasoning foods.** Erich Brust. U. S. 2,021,403, Nov. 10. A mixt. of citric acid with a larger proportion of salt is slowly heated, with const. stirring, to the m. p. of the citric acid and the stirring is continued to coat the particles of salt with a film of citric acid, followed by cooling, with continued stirring, and addn. of substances such as paprika, pepper and sugar.

**Treating nuts such as pistachio nuts in the shell.** Joseph A. Zaloom. U. S. 2,020,533, Nov. 12. Salted nuts in the shell are provided with an outer coating such as edible shellac to seal the shell and assist in preserving the kernel in good condition.

**Coffee.** Ricofin (S. A. r. l.). Fr. 785,418, Aug. 9, 1935. A better extn. of the alkaloids and aromatic substances is made by adding to the boiling water used or to the roasted ground coffee  $NaH_2PO_4$  in the presence of compds of  $NH_4$ , alkali carbonates or bicarbonates and compds of Fe or Al.

**Preserving green fodder.** Friedrich A. Henglein (to I. G. Farbenind. A.-G.). U. S. 2,021,996, Nov. 26. Green fodder, e. g., crimson clover, is treated with a soln. of "wood-sugar" which serves as a preservative, while the material is kept in a silo.

**Preserving green fodder in silos, etc.** Georg Meder and Erich Leger (to I. G. Farbenind. A.-G.). U. S. 2,022,139, Nov. 26. Formic acid and a formate such as that of Na, K or  $NH_4$  are used together as preservatives.

## 13—CHEMICAL INDUSTRY AND MISCELLANEOUS INDUSTRIAL PRODUCTS

(PLASTICS, RESINOIDS, INSULATORS, ADHESIVES, ETC.)

HARLAN S. MINER

**Present-day material problems in the chemical industry.** E. Rabald. Chem. Fabrik 1935, 441-6.—A review of imports and exports of Cu, Pb, Zn, Sn and Al; of German production from domestic and imported ores; of methods of economizing in construction and of combating corrosion by coating and alloying; of the recovery of scrap metal, and the substitution of other materials for metals. Thirty-five references. J. H. Moore

**Progress in engineering knowledge during 1935.** P. L. Alger. Gen. Elec. Rev. 38, 546-57 (1935).—A review of the contributions by General Electric Co.'s engineers. C. G. F.

**Size distribution of industrial dusts.** J. J. Bloomfield. U. S. Pub. Health Repts., Suppl. No. 115, 9 pp (1935).—17 references. J. A. Kennedy

**Increasing the production of plastic materials.** M. K. Shcherbakov. Uspekhi Khim. 4, 773-99 (1935).—A review of various production processes, sources of raw materials and the chem., phys. and elec. properties of the products obtained. F. H. Rathmann

**Progress in the chemistry of synthetic resins.** B. V. Maksorov. Uspekhi Khim. 4, 539-72 (1935).—A review of the manuf. and properties of the various types of synthetic resins. F. H. Rathmann

**Preparation of artificial resins by benzoylation of aluminoids.** B. V. Maksorov and K. A. Andrianov. Rev. gen. mat. plastiques 11, 82-3, 115, 211-13 (1935).—Casein,  $CH_3O$ -treated galalith, horn, yeast, fish scales, castor-seed cake, gelatin and glue can be benzoylated by treating with NaOH and refluxing with  $BzCl$  at  $80-110^\circ$ ; the paste reaction product can be steam distd. to remove volatile products, washed with warm dil. AcOH and then with  $H_2O$  till free from Cl, and finally dried in vacuum at  $80-90^\circ$  yielding a product that is sol. in mixts. of alc. with  $Me_2CO$  or aromatic hydrocarbons, and in chlorinated hydrocarbons, less sol. in EtOH and BuOH, very difficultly sol. in  $Et_2O$ , spirit of turpentine and oils, and completely insol. in benzene and water; in dil. solns. of bases the product swells and finally dissolves. Fractional pptn. of  $C_2H_5$ -alc. soln. by means of xylene showed that products

of considerably higher m. p. and viscosity can be sepd. from the total reaction products. Lacquers produced by prep. 4 15-25% soln. of the resin in alc.- $C_2H_5$  mixt., with or without addn. of a plastifier (6% tritoly phosphate on the wt. of resin), were applied to thin paper and to glass, dried at atm. temp. for 50-60 min. and then 30 min. at  $100^\circ$ . The resulting films were not scratched by a sharp point under a load of 300 g., and could be wound around a rod 5 mm. in diam without showing any signs of cracking. The dielec. properties of freshly prep. films were comparable to those obtained under the same conditions by the usual insulating lacquers (phenol-formaldehyde resins, copals, glyptals, etc.); but the resistivity falls off rapidly when treated with  $H_2O$  or acid or alk. solns., especially HCl. A. Papineau-Couture

**Thermal insulators and their application.** A. Clauset. Verre silicates ind. 6, 425-7 (1935).—An elementary theory of thermal insulation. Herbert S. Willson

**Asbestos in electrical insulation.** H. Warren. India Rubber J. 90, No. 17a (Intern. No.), 19-22 (Oct. 31, 1935).—A review and discussion. C. C. Davis

**Recent progress in dielectric strength.** John B. Whitehead. Elec. Eng. 54, 1288-9 (1935).—A summary of some 1934-1935 results indicating the scope and significance of research involved in the formulation and application of elec. insulating materials. Fifty-five references. W. H. Boynton

**The adhesive power of adhesives.** Max Delpy. Kunststoffe 25, 165-8 (1935).—The use of various adhesives for sticking paper to paper, patching tire inner tubes, making safety glass and soldering metals is discussed. To obtain good adhesion, the adhesive must penetrate and fill surface pores. J. W. Perry

**The distribution of the plasticizer in the artificial leather film.** Walter M. Münzinger. Kunststoffe 25, 249-51 (1935).—Artificial leather is apt to become sticky, when solvent residues or relatively nonvolatile solvent impurities or excessive amts. of plasticizers diffuse to the surface. J. W. Perry



Coated fabrics in construction industry. Dorman McBurney *Ind. Eng. Chem.* 27, 1400-3 (1935). G. G. Turkey-red oil from grapeseed oil. Welwart. *Seifen- und Ztg.* 62, 933 (1935) — Practical experience has convinced W. that sulfonated grapeseed oil is a poor substitute for sulfonated castor oil. J. W. Perry. Sulfonated oils. A. E. Sunderland. *Soap* 11, No. 10, 61-4, 71, No. 11, 61-4, No. 12, 67-9 (1935) — Manuf. properties and applications of sulfonated oils are discussed. E. Scherubel.

Dressings, polishes and abrasive preparations for automobile finishes. Carl Becker. *Jr. Seifen- und Ztg.* 62, 709-11, 732-3, 752-3, 772-4 (1935) — Raw materials, recipes, production methods and the proper use of various preps are discussed. J. W. Perry.

Shoe dressings—yesterday and today. Karl Dopf. *Kunststoffe* 25, 168-9 (1935).

Five recipes. Floor wax, rust removing paste, liquid metal cleaner, liquid furniture polish and liquid black stone polish. Leo Ivanovsky. *Seifen- und Ztg.* 62, 708-9, 732 (1935). J. W. Perry.

Iron in common sponges. P. Robin. *J. pharm. chim.* 21, 600-4 (1935) — The quantities of Fe occurring in sponges of different origin are recorded. They vary with the geographical source, being larger in Mediterranean, smaller in West Indian sponges. S. Waldbott.

Paint removers. R. Forster. *Paint Manuf.* 5, 314-16 (1935) — There are 4 types of removers for paint films. (1) mech., (2) caustic alkali, (3) elec. method and (4) org. solvent. The 4th type is subdivided into 3 groups: (1)  $(CH_3)_2CO$ , (2) high flash or nonflammable and (3) Me-Cl base. Waxes and solvents are discussed. A paint remover may contain wax, a protective colloid, a wax precipitant and a wax solvent. Special measures are necessary for removal of coatings other than oil paint. A formula is given for each of the 3 types of org. solvent paint removers and several patent nos. are appended. W. H. Boynton.

Synthetic detergents and their practical application. Oskar Uhl. *Seifen- und Ztg.* 62, 687-8, 719-20 (1935). — A review emphasizing practical problems. J. W. P.

Plastics used in food equipment (Lee) 12. Fire risks and their prevention (Wood) 30. Gas filters (Fr. pat. 751,773) 1. Lacquers and plastic masses (Brit. pat. 432,762) 26. Urethans [derivs as dispersing, softening or sizing agents] (Ger. pat. 619,500) 10. Alkali metal tetraphosphates [products used as detergents] (U. S. pat. 2,019,665) 18. Quaternary ammonium salts [emulsions for treatment of fibrous materials] (Brit. pat. 433,356) 10. Cellulose foils [gas mask windows] (Brit. pat. 432,772) 23. Ester-like compds. [as soaps and active O-yielding agents] (Ger. pat. 616,847) 10.

Effecting reactions with circulating gases. Kenneth Gordon and Imperial Chemical Industries Ltd. Brit. 432,574, July 30, 1935. In carrying out reactions with circulating gases, the gases are purified by being passed in rapid co-current flow with a liquid absorbent, which removes in each operation only a small proportion of the undesired constituents. Specific applications are dehydrogenation,  $NH_3$  synthesis and  $MeOH$  synthesis.

Filters for gas masks and like protective apparatus. Decca A.-G. (Auerger). Ger. 619,278, Sept. 26, 1935 (Cl. 61a. 29.30). A paste contg. gas-absorbing reagents is kneaded into a porous support, e. g., rubber sponge. The paste may contain also a binding or stiffening agent, e. g., waterglass, cement or gelatin, and (or) a powder adsorbent, e. g., active C.

Plastic materials. Deutsche Hydrierwerke A.-G. Fr. 755,009, July 31, 1935. Artificial albuminoid materials in gelatin, strong glue, casein, albumin, etc., are softened and made elastic by adding aliphatic, aromatic or cycloaliphatic alcs. contg. 6 or more C atoms or ethers or esters of these compds., or cyclic ethers contg. an OH group or an ether or ester thereof. Thus, a soln. of oleic alc. in

1 MeOH is incorporated in fused gelatin, and acetone-glycerol benzoate in BuOH is added to an artificial casein material.

Plastic materials. I. G. Farbenind. A.-G. Fr. 755,522, Aug. 12, 1935. In making films, threads and molded articles from plastic materials sol. in org. solvents, particularly cellulose derivs., haloalcohol esters of polyhydric alcs. in which all the free OH groups are esterified with aliphatic carboxylic acids of straight chain contg. at least 8 C atoms, are used as emollients. Examples are ethylenechlorohydrin naphthenate and -phenylsearate, monochlorohydrin-ditrate and -erythritolpalmitate, and dibromohydrin ester cocinic acid ( $CH_2Br-CHBr-CH_2-O-COR$ , R is cocinic acid).

Plastic substances. I. G. Farbenind. A.-G. Fr. 755,705, Aug. 17, 1935. Plastic substances of high mol. wt. are made by causing alkali, alk. earth or  $NH_4$  sulfides, particularly polysulfides, to act on polyhalo compds. having the halogen atoms attached to C atoms of lower aliphatic chains and contg. the group  $CO$ , e. g., glycerol-trismonochloroacetate, the di-ester of glycol and  $CHCl_2COOH$ , sorbitol tetrakismonochloroacetate, dichloroacetone,  $\beta$ -chloroethyl ester and  $\beta$ -chloroethylamide of  $CH_2ClCOOH$  and dichloroacetyl ethylenediamine.

Plastic substances. I. G. Farbenind. A.-G. Fr. 755,861, Aug. 21, 1935. Rubberless products resembling rubber are made by causing alkali, alk. earth or  $NH_4$  sulfides, particularly polysulfides, to act on aromatic compds. contg. at least 2 halogen atoms in aliphatic side chains, e. g., benzylidene chloride, benzotrichloride or *p*-xylene dichloride. A diluent such as EtOH or water is used, and if water is used dispersing or emulsifying agents are added.

Testing thermosetting plastic materials. Balclutha Corp. Brit. 432,646, July 31, 1935. The materials are tested by subjecting a predet. quantity thereof to heat and pressure in a mold provided with a channel along which the material may flow, maintaining the heat and pressure until the material sets and measuring the extent and (or) rate of flow along the channel. App. is described.

Apparatus for extruding plastic material under pressure. Imperial Chemical Industries Ltd. Fr. 755,788, Aug. 19, 1935. Means for removing gas by reduction of pressure before extruding.

Plastic compositions containing resins and cellulose derivatives. Harry B. Dykstra and Walter E. Lawson (to E. I. du Pont de Nemours & Co.). U. S. 2,021,121, Nov. 12. Compds. such as those contg. cellulose esters and ethers, etc., are plasticized with esters of the general formula  $R'OOCOR''COOR''COOR''$  in which  $R'$  and  $R''$  are univalent radicals derived from monohydric alcs.,  $R'$  is a bivalent hydrocarbon radical and  $R''$  is a bivalent aliphatic hydrocarbon radical, such as butyl phthalyl vinyl glycolate or the like.

Artificial resins. Wilhelm Kraus. Brit. 432,637, July 25, 1935. Condensation products are prep. by condensing a phenol, e. g., PhOH or cresol, 1, urea 1-2 and an aq. acid  $CH_2O$  soln. of  $fu$  below 3, 2-4 mol. proportions in the presence of such quantity of  $(CH_3)_3N$ , that the reaction mixt. is acid. The  $(CH_3)_3N$  may be added as a whole or in parts or it may be prep. from  $NH_3$  and  $CH_2O$  added separately. The urea may be replaced by mints thereof with other compds. that react with  $CH_2O$ , e. g., thiourea, urethan, formamide, acetamide and oxamide. The products may be worked up with fibrous or filling materials, e. g., cellulose.

Synthetic resins. Walter Wollaston (to Brunswick-Balke-Collender Co.). U. S. 2,019,591, Nov. 5. A compn. of stiff consistency is formed contg. a urea formaldehyde resin mixed with a phthalic anhydride glycerol resin, a phenol-formaldehyde resin and a filler such as lithopone, ZnO or barytes and articles such as billiard balls, toys, etc., are molded from the compn. under heat and pressure, and are then finally shaped and finished.

Synthetic resins. F. I. du Pont de Nemours & Co. Brit. 433,432, Aug. 12, 1935. Resinous substances are prep. by causing to react at elevated temp. and in the presence of alkali 1 or more dihalo derivs. of formula halo-

X-halo, wherein X is  $\text{CH}_3$  or an aliphatic hydrocarbon chain, which is preferably broken by an ether linkage, with 1 or more dimuclear phenols of formula  $\text{R}(\text{HOAr})\text{C}(\text{ArOH})\text{R}'$ , where Ar and Ar' are phenylenes, which may carry alkyl, alkoxy or halo substituents or, preferably, a secondary or tertiary alkyl group in  $\beta$ -position to the phenolic OH, and R and R' are H, alkyl or aryl groups. A portion of the phenol may be replaced by an equiv. amt. of a dihydroxydithiosulfone or by 1 or more monohydric phenols and a proportion of the dihalo compd. may be replaced by a monohalo compd. Among examples, resins are prep. by condensing, in presence of  $\text{H}_2\text{O}$  and  $\text{NaOH}$ , dichlorodimethyl ether and (1) di-(4-hydroxyphenyl)-dimethylmethane (I), with or without the addn of di-(4-hydroxyphenyl)-sulfone, (2) di-(4-hydroxy-3-methylphenyl)-dimethylmethane, (3) di-(4-hydroxy-3,5-dichlorophenyl)-dimethylmethane, and (4) *o*-cresol and I, or by condensing I, in presence of  $\text{H}_2\text{O}$  and  $\text{NaOH}$ , with ethylene or amylene dichloride. The resins may be used in various compns in conjunction with solvents, pigments, fillers, plasticizers, etc., as adhesives, impregnating, coating, sizing, glazing or water-proofing agents, binders for lunoleum plastics, glass substitutes, cements and sealing waxes, varnishes, etc.

Synthetic resins. Wilhelm Kraus Austrian 142,893, Sept. 25, 1935 (Cl. 39a) Urea and  $\text{N}_2\text{H}_4$  are condensed with  $\text{CH}_3\text{O}$  in the presence of an acid condensing agent. The preferred proportions are 1 mol. of  $\text{CH}_3\text{O}$  or slightly more for each mol. of urea, 2 mols. of  $\text{CH}_3\text{O}$  for each mol. of  $\text{N}_2\text{H}_4$ , and less than 0.05 mol. of  $\text{N}_2\text{H}_4$  for each mol. of urea.  $\text{N}_2\text{H}_4$  may be taken as its hydrate or as a salt. Other reagents which condense with  $\text{CH}_3\text{O}$ , e. g., thiourea or phenol, may be added to the mixt. The reaction is effected at a raised temp. in the presence or absence of water or other solvent. Sp. processes are described. The products are more easily molded than the known resins of the urea- $\text{CH}_3\text{O}$  type.

Synthetic resins. Soc. pour l'ind. chim. à Bâle Ger 612,626, Sept. 16, 1935 (Cl. 12a 17 05). Resins insol in water are prep. by heating 1 mol. of urea with 2 mols. of  $\text{CH}_3\text{O}$ . The aq. soln. of the condensation product is further heated in the presence of free OH ions with substances (except dicyanodiamine) which, with  $\text{CH}_3\text{O}$  or its condensation products and free OH ions, form insol. resins, till the insol. resins sep. Examples of the substances used in the second stage are phenylurea, thiourea,  $\text{PbNH}_2$ , urea, biuret, and  $\beta$ -naphthylamine, all with  $\text{NaOH}$ .

Synthetic resins. Allgemeine Elektricitäts-Ges. Ger 619,439, Oct. 1, 1935 (Cl. 26 02). See Brit 393,034 (C. A. 28, 3529).

Synthetic resins. Elly Pollak (nee Hissberger). Fr 785,763, Aug. 19, 1935. A soft resin is prep. by heating together urea and  $(\text{CH}_3)_2\text{N}$ , in the presence or not of a solvent and, after removal of the liquid, is transformed into a hard sol. resin by sepn. of water.

Synthetic resins from urea (or thiourea) and formaldehyde. I. G. Farbenind. A.-G. (Karl Eisenmann and Hans Scheuermann, inventors). Ger. 618,743, Sept. 10, 1935 (Cl. 12a 17 05). Addn. to 588,426 (C. A. 28, 1560). The ester added to the reaction mixt. in the process of Ger. 588,426 is replaced by a higher alc. contg. more than 8 C atoms in the mol., e. g., cetyl alc. or mixts. of alcs. obtained by reducing animal or vegetable oils, fats or waxes or by oxidizing paraffin wax. The proportion of higher alc. must be at least 20%, calcd. on the urea- $\text{CH}_3\text{O}$  product. The soly. properties of the products vary with the proportion of higher alc. The products are useful as components of lacquers or, after addn. of an acid hardening agent, for making molded articles. Examples and numerous details are given.

Resin compositions. Carbide Chemicals & Carbon Corp. Fr. 784,681, July 22, 1935. Vinyl resins resulting from the joint polymerization of a vinyl halide with a vinyl ester of an aliphatic acid have added thereto heat stabilizers composed of metals or metal compds. which form insol. Cl. derivs. and (or) substances having a moderate reducing action. Examples are oxides of Sb, Bi, Cu and Hg, sulfite

of Ag, Cd, Ca, Ce, Na and Sr, silicates of Cu and Mg, metallic Cu, Cd, Al and Mg, mixts. of basic Pb sulfate and Pb sulfite, and "sublimed blue lead." The compns. may be heated to a much higher temp. than the resin alone without decompos.

Vinyl resins. Soc. Nobel française. Fr. 785,569, Aug. 13, 1935. Condensation products of polyvinyl alcs. and aldehydes having a chem. structure of acetals are stabilized by antioxidants such as hydroquinone, pyrogallol and "antioxygen RR5."

Resinous composition suitable for coatings, etc. Caryl Sly (to I. I. du Pont de Nemours & Co.). U. S. 2,022,011, Nov. 26. A resinous reaction product is formed from a noncellulosic polyhydric alc. such as glycerol and the product obtained by heating together an org. polybasic acid such as phthalic anhydride and a cellulose deriv. such as cellulose acetate or ethyl or benzyl cellulose.

Cyanamide resin. Palmer W. Griffith (to American Cyanamid Co.) U. S. 2,019,490, Nov. 5.  $\text{CH}_3\text{O}$  is added to an unaltered soln. of cyanamide of a  $\text{pH}$  between 4 and 8 and the ppt. formed is sepd. and chemically combined with water to form a product which is suitable for molding.

Polyhydric alcohol-polybasic acid resins. Ben E. Sorenson (to E. I. du Pont de Nemours & Co.). U. S. 2,019,510, Nov. 5. See Can 351,817 (C. A. 29, 6975).

Electric insulating compositions. Siemens & Halske A.-G. Ger. 619,253, Sept. 30, 1935 (Cl. 21c 2 11). Rubber is mixed with a water-insol. cellulose deriv. e. g., cellulose laurate, without the aid of a solvent or dispersing agent. A softening agent, e. g., a mineral wax, is included in the compn., and is preferably mixed separately with the rubber or the cellulose deriv. The proportion of cellulose deriv. may be about 40%. The compns. are particularly useful for insulating submarine cables.

Heat-insulating material. Insulations (London) Ltd. Fr. 784,802, July 25, 1935. Diatomaceous earth is ground, mixed with ground furnace slag and a binder, such as  $\text{CaCO}_3$  and a small amt. of  $\text{NaCl}$ . The mass is moistened, compressed to the desired shape and heated under steam pressure until a chem. reaction takes place between the  $\text{CaCO}_3$ ,  $\text{NaCl}$  and slag. Glucose may be used as a temporary binder.

Cables, insulation. Allgemeine Elektricitäts-Gesellschaft (to The British Thomson-Houston Co. Ltd.). Brit. 432,284, July 24, 1935. Cables, condensers and other elec. app. are impregnated with a compd. contg. a considerable amt. of  $\text{CO}_2$  that has been dissolved therein under pressure and which escapes to fill any voids formed in the impregnated dielec. during operation of the app.

Nonconducting coverings for heat. Franz Wm. Seiving, Abel Bergqvist and Karl E. Olsson. Brit. 432,019, July 15, 1935. In heat insulation comprising spaced sheets of a transparent material having the mech. properties of paper, e. g., cellulose acetate, the sheets are impregnated with particles of material having a metallic luster, e. g., metals, alloys, metallic compds. Air bubbles may be blown in the mass during the prepn. of the sheets.

Dielectric materials. The British Thomson-Houston Co. Ltd. Brit. 433,070, Aug. 8, 1935. Elec. app. is provided with a dielec., insulating and (or) cooling medium comprising a non-cryst. halogenated diphenyl ketone in which the mol. proportion of the halogen is at least equal to that of the H. Diphenyl ketone, prep. by causing  $\text{C}_6\text{H}_5$  to react with  $\text{CCl}_4$  in presence of  $\text{AlCl}_3$ , hydrolyzing the product and fractionating, is halogenated in presence of Fe or Fe chloride as catalyst to give mixts. of isomeric halogenated diphenyl ketones. In Brit. 433,071, Aug. 8, 1935, the medium comprises a non-cryst. solid or liquid halogenated dibenzyl. Such products may be prep. by treating chlorobenzenes with ethylene dichloride in presence of  $\text{AlCl}_3$ .

Dielectric material suitable for condensers. Raymond H. Hobrock (to Western Elec. Co.). U. S. 2,020,468, Nov. 12. Material such as paper is impregnated with an insulation compn. comprising a halogenated naphthalene or halogenated stearic acid and a substantially amorphous paraffin hydrocarbon material in minor proportion (the

components of the mixt. being sol in the liquid phase but insol in the solid phase).

**Adhesives** I G Farbenindustrie A-G Brit 432,977, Aug 7, 1935. Addn to 401,200 (C A 28, 24811). Low polymerized halogenated polyvinyl chlorides that are obtained according to 401,200 by further chlorination of polyvinyl chlorides in the presence of solvents or agents promoting the formations of suspensions are used as a basis for adhesives, particularly for leather cements. In an example, a leather cement is obtained by dissolving low polymerized polyvinyl chloride 20 in methylene chloride 80 parts Cf C A 29, 22629.

**Dry powdered adhesives** Henkel & Cie G m b H Brit 432,480, July 15, 1935. This corresponds to Fr. 766,504 (C A 28, 74457).

**Testing the adhesive properties of coatings** such as asphalt, paint or lacquer Geo R Hoover and George E. Shafer (to American Rolling Mill Co.) U. S. 2,020,891, Nov. 12. A jet of fluid such as water or other inert liquid is projected from a nozzle so as to impinge at the edge of an isolated area of the coating material (the coating support and nozzle being at an angle of about 45°). App is described.

**Cements** Alfred S. Colling Brit 432,493, July 25, 1935. A cement that will stick rubber or fabric to metal consists of Paris white 40, rosin 3, dammar or copal gum 15,  $\text{CH}_2$  15, naphtha 23 and rubber 15 parts. Whiting may be added to make the cement suitable for sticking vitreous tiles to plaster cement surfaces.

**Wetting and other agents** Soc. pour l'ind. chim. à Bâle Fr 785,475, Aug. 10, 1935. Products having excellent capillary properties are made by acylating the  $\text{NH}_2$  group or groups of aromatic amines which contain at least one hydroxyalkyl or hydroxyalkylaryl sulfonamide group, esterified if desired with  $\text{H}_2\text{SO}_4$ , with aliphatic carboxylic acids contg more than 3 C atoms. The  $\text{OH}$  group is then esterified with  $\text{H}_2\text{SO}_4$  if not previously esterified. Thus, the hydroxymethylamide of 1-nitro-3-benzenesulfonic acid is esterified with  $\text{H}_2\text{SO}_4$ , reduced to the 1-amino compd., and acylated with oleic acid chloride or lauric acid chloride. Other examples are given.

**Wetting and other agents** Compagnie nationale de matières colorantes & manufactures de produits chimiques du nord réunies établissements Kuhlmann Fr 785,561, Aug. 13, 1935. These are prep from ketones of the formula  $\text{RCOR}$  and  $\text{RCOR}'$  (R being an aliphatic chain of more than 8, preferably 12-18, C atoms,  $\text{R}'$  being a chain of less than 5 C atoms), by halogenation. One or more of the halogens may be replaced by a solubilizing group or a short chain contg such a group. Thus, a ketone derived from stearic acid is dissolved in  $\text{CCl}_4$  and  $\text{Cl}_2$  is passed through the soln. Other examples are given.

**Wetting and washing agents** Herbert Fleisch, Carl Fleisch and Leonore F. Abelmann (trading as Farb- und Gerbstoffwerke Carl Fleisch, Jr.) Brit 433,206, Aug 7, 1935. See Fr 767,788 (C A 29, 5322).

**Wetting, washing and dispersing agents, soaps** I G Farbenindustrie A-G Brit 433,305, Aug 8, 1935. Oxidation products of nonaromatic hydrocarbons of high mol wt are dissolved in a  $\text{H}_2\text{O}$ -insol solvent or mixt. of solvents, the soln is treated with an alk. substance dissolved in  $\text{H}_2\text{O}$  in such amt that the acid portions of the oxidation products are neutralized, the aq soap soln is sep'd and the unaponifiable constituents may then be ext'd from it by means of the same or another  $\text{H}_2\text{O}$ -insol solvent. The soap soln may be evap'd. to dryness and formed into shaped soaps, soap powder, etc. Among examples, hard paraffin is oxidized with air by heating in the presence of Na and Mn palmitates, the product is dissolved in a mixt of benzene and alc and stirred with warm  $\text{Na}_2\text{CO}_3$  soln, the soap soln is removed after settling, ext'd with benzene and evap'd in a roll drier to form a soap powder.

**Compositions for use as wetting agents** Wm Todd (to Imperial Chemical Industries Ltd.) U. S. 2,020,385, Nov. 12. A dry mixt readily sol in water is formed of a wetting agent such as one of the sulfonated alkylated naphthalene series and an alkali metal sulfonate of ben-

zene, aniline or an N-substituted aniline such as Na benzyl-aniline sulfonate or the like.

**"Assistants"** suitable for use as wetting agents in the textile and related industries Hans Deller and Hermann Schuette (to I G. Farbenind. A-G.). U. S. 2,020,453, Nov. 12. A product from the incomplete liquid phase oxidation of nonaromatic hydrocarbons of high-mol wt. such as paraffin or gas oil from which non-oxidized hydrocarbon material has been sep'd and which contains at least 15% of alcs and olefins is caused to react with a sulfonating agent. Various examples are given.

**Phosphoric esters** Bohme Fettechemie-Ges m. b. H. Ger. 619,019, Sept. 20, 1935 (Cl. 12a 5 04). Aliphatic alcs. contg more than 8 C atoms are treated at atm or raised temp. with acetylphosphoric acid or a reagent which yields it, e. g., a mixt of  $\text{AcCl}$  and  $\text{H}_3\text{PO}_4$ . Phosphoric esters of the alcs., useful as wetting, emulsifying and cleansing agents, are obtained. Examples are given of the manuf. of esters from stearyl, lauryl and oleyl alcs.

**Moldable composition** Harold S. Holt (to E. I. du Pont de Nemours & Co.) U. S. 2,022,001, Nov. 26. A thermoplastic, substantially water insensitive  $\text{CH}_2\text{O}$ -treated casein compn is used together with a polycarboxylic acid-polyhydric alc. condensation product.

**Molding powder** suitable for making brushes of electric machines Carleton N. Smith and Newcomb C. Chaney (to National Carbon Co.) U. S. 2,020,053, Nov. 5. A finely divided conductive material, principally C, is used with a minor proportion of a vinyl resin such as that formed by the conjoint polymerization of vinyl chloride and vinyl acetate, the vinyl resin being distributed on the surface of the conductive material in extremely fine subdivision.

**Urea-formaldehyde molding composition** Kenneth N. Francisco (to American Cyanamid Co.) U. S. 2,020,024, Nov. 5. A cellulose filler such as paper pulp is impregnated with a urea- $\text{CH}_2\text{O}$  condensation product and the material is then dried and ground to a powder. Agglomerated into lumps with an aq. soln of a urea- $\text{CH}_2\text{O}$  condensation product, the lumps are disintegrated into relatively dense granules, and the granules are preformed under pressure to give a product which can be molded under heat and pressure to form strong and uniform products.

**Molded book covers of rubber and other materials** Arthur G. Rendall (to Morland & Impey Ltd.) U. S. 2,020,214, Nov. 5. A layer of rubber is used adjacent at least one layer of another thermoplastic material such as a synthetic resin compn, which is united with and harder than the rubber. Various mfg details are described.

**Artificial leather** Horace Johnson Martin Fr. 785,102, Aug. 2, 1935. Waste and crude leather are reduced to a uniformly colloidal state by mech. disintegration in the presence of water, e. g., in a colloid mill, and the resulting product is tanned giving a leather having the properties of the natural product but which can be made in any desired shape or length.

**Fabric-coating apparatus for making artificial leather** Atlas Agrochem Fab A-G (Otto Spreckelsen, inventor) Ger. 619,124, Sept. 23, 1935 (Cl. 84 7).

**Condensation products** Henkel & Cie G m b H Fr 785,490, Aug. 10, 1935. Products which are emollients, artificial resins, etc., and may be used in lacquers, varnishes, etc., are made by causing oxidation products of natural resins or resinic acids contg at least 1  $\text{COOH}$  group to react with org. compds contg at least 1  $\text{OH}$  group, or their functional derivs capable of reacting, e. g., glycerol, glycol,  $\text{BuOH}$ , hexanol, benzyl alc.,  $\text{PhOH}$ , creosol, epichlorohydrin, aminopropandiol, triethanolamine and their halides and alkylene oxides.

**Phenol-amine-aldehyde condensation products** Aug Nowack A-G, Richard Hessen and Karl A. Schuch Brit. 433,766, Aug. 19, 1935. Condensation products are prep from  $\text{CH}_2\text{O}$ , a phenol or homolog thereof and an aromatic amine by condensing the  $\text{CH}_2\text{O}$  with 1 of the aromatic components to yield a sol. resinous product and further condensing this with the other aromatic compd and  $\text{CH}_2\text{O}$  or with a preformed sol. resinous condensation

product derived from the other aromatic compd. and  $\text{CH}_2\text{O}$ , the reaction taking place in a liquid or viscous medium and the condensation being continued until formation of layers takes place. Among examples, (1) a viscous  $\text{PhOH}\cdot\text{CH}_2\text{O}$  resol is dissolved in  $\text{PhNH}_2$  and the product condensed with  $\text{CH}_2\text{O}$  in the presence of  $\text{Na}_2\text{CO}_3$ , and (2) an acid-condensed  $\text{PhNH}_2\cdot\text{CH}_2\text{O}$  resin is dissolved in  $\text{PhOH}$  or cresol and the product condensed with  $\text{CH}_2\text{O}$  in presence of  $\text{Na}_2\text{CO}_3$  or  $\text{NH}_3$ .

**Phenol-formaldehyde condensation products** Alan A. Drummond, Howard H. Morgan and Imperial Chemical Industries Ltd. Brit. 433,539, Aug. 12, 1935. Resins are made by causing  $\text{CH}_2\text{O}$  to react with a cresol or a xyleneol, or a mixt. thereof, in the presence of more than 0.5% of  $\text{NH}_3$  (calcd. on the amt. of phenol), the reaction being continued sufficiently long and the proportion of phenol to  $\text{CH}_2\text{O}$  being sufficient for the reaction product to be permanently fusible and sol. in drying oils.

**Urea-formaldehyde condensation products** I. G. Farbenindustrie A.-G. Brit. 433,536, Aug. 12, 1935. These are made by subjecting the products obtainable by condensation, preferably in the presence of acid condensing agents, of urea and (or) thiourea and  $\text{CH}_2\text{O}$  or its polymers, or of methylol derivs. of urea and (or) thiourea or alkyl ethers thereof, or of amorphous products of high mol. wt. obtainable from the said methylol compds. by splitting off  $\text{H}_2\text{O}$ , or of mixts. of said substances, in a monohydric alc. solvent, to condensation in the presence of an aliphatic alc. contg. more than 8 C atoms, neutralizing the reaction mixt., expelling the excess of alc. solvent and heating the remaining resinous product until it has become sol. in aromatic hydrocarbons. The products may be worked up alone or together with cellulose esters or ethers and the usual softening agents or with drying oils to form lacquers and adhesives. The resins, after adding a suitable acid or acid-forming hardening agent, and, if desired, with the addn. of softening agents, may be used for precast articles that harden on heating and are suitable as substitutes for glass. Among 8 examples, (1) dimethylol-urea is heated with  $\text{BuOH}$ ,  $\text{EtOH}$ , urea nitrate and the mixt. of alcs. obtained from the acids of palm kernel oil, the reaction mixt. is neutralized with  $\text{Na}_2\text{PO}_4$ , the excess  $\text{BuOH}$  is removed in a vacuum kneading-machine and the resin is further kneaded at 85–95°, and (2) a mixt. of dimethylolurea and dimethylolthiourea is heated with  $\text{BuOH}$ ,  $\text{EtOH}$  and octadecyl alc., as in (1) and the product is kneaded with phthalic anhydride and hardened in molds.

**Resinous polymerization products** Harold J. Tattersall and Imperial Chemical Industries Ltd. Brit. 433,540, Aug. 12, 1935. Polymerized acrylic esters or their homologs are made in sheet form by polymerizing the ester or esters or mixts. thereof by heat, light or other known means, preferably in the presence of an O-catalyst, if desired with the addn. of plasticizers, dyes or other effect materials, interrupting the polymerization when the mixt. has the consistency of a thick sirup just capable of pouring, placing the sirup between 2 horizontal sheets of glass or other material, preferably polished, of which the upper 1 is counterpoised, allowing polymerization to proceed slowly at not above 50° or, if necessary, with cooling down to about 5° until solidification is complete, and removing the sheet of polymerized material, advantageously after 1st heating the plates with the solid polymer between them. In examples, (1) *Me methacrylate* is heated to not above 80° in the presence of  $\text{Bz}$  peroxide until the mol. wt. is 3000 by Staudinger's method, cooled to 30°, polymerization completed at 20–25° between glass plates, the sandwich heated to 80–90° and the sheet of polymer detached, and (2) the materials and app. of (1) being used, a 1st layer is polymerized without the top sheet of glass, which is applied only after a 2nd layer of partly polymerized material has been poured on top of the 1st.

**Sheets made from polymerized substances** Imperial Chemical Industries Ltd. Fr. 785,506, Aug. 12, 1935. Polymerized acrylic esters or their homologs are obtained in the form of sheets by polymerizing the ester or esters by heat, light or other means, preferably in the presence

of an O-catalyst such as benzoyl peroxide, with the addn., if desired, of dyes, plasticizers, etc., interrupting the polymerization when the mixt. has reached sirupy consistency, just capable of flowing, placing the sirup between 2 sheets of glass or other polished material, and allowing the polymerization to proceed slowly at not above 50° until solidification is complete.

**Chlorinated polymers of acrylic acid and derivatives** for making molded articles, etc. Walter Bauer (to Rohm & Haas Co.) U. S. 2,021,763, Nov. 19. Various examples are given of the production of chlorinated derivs. from compds. such as polymerized acrylic acid methyl ester, polyacrylic acid, polymerized methacrylic acid ethyl ester, etc., which may be used for the production of artificial materials such as lacquers, films, intermediate layers for safety glass, articles to be used in electro-technology, threads, artificial leathers, oil-cloth, rayon, fabrics of various kinds.

**Compositions containing collagen products** Wilfred Graham Dewsbury and Arnold Davies Fr. 785,114, Aug. 2, 1935. See Brit. 431,360 (C. A. 30, 1934).

**Sheets and threads from polyvinyl chloride** Max Hagedorn (to Agfa-Ansco Corp.) U. S. 2,020,642, Nov. 12. A soln. of an after-chlorinated polymerized vinyl chloride contg. a firmly bound quantity of Cl above that corresponding to the formula  $\text{CH}_2\text{Cl}\cdot\text{CHCl}$  and needing for solution of 1 gm. less than 15 cc. of a mixt. of  $\text{PhCl}$  and epichlorohydrin mixed in the ratio of 3:1 at 100°, said mixt. keeping the polyvinylchloride in soln. for at least 5 min. when cooled to 20°, is used for casting to form films or sheets or for forming threads.

**Emulsions.** H. Th. Böhme A.-G. Fr. 785,006, July 31, 1935. Stable emulsions of fatty materials are made without the addn. of special protective colloids by mixing electroequiv. amts. of 2 different colloidal electrolytes, one having a colloidal cation and the other a colloidal anion giving rise to an electrostatically neutral compd. insol. in water and adding to this compd. an excess of one of the constitutive elements or any other colloidal electrolyte until the material gives a stable emulsion with water. The fatty material is preferably added to one of the constituents before mixing with the other constituent. As electrolytes with colloidal cation may be used quaternary  $\text{NH}_4$  compds. of the aliphatic, aromatic, aliphatic aromatic, hydroaromatic and heterocyclic series, or salts of alkyliso- and alkylisothio-urea. As electrolytes with colloidal anion may be used alkyl esters of strong mineral polybasic acids, e. g., dodecyl-, hexadecyl-, octadecenyl- and octadecyl-sulfates or their alkali salts, alkali salts of fat acids, free fat acids and products having a basis of Turkey-red oil. Thus, paraffin oil or soft paraffin is emulsified with an alkali salt of dodecyl sulfate, lauryl-pyridinium bisulfate is added in amt. such that the paste obtained has max. viscosity. The salted water is sepd. by heating, and a high percentage emulsion of pos. charge is obtained by a fresh addn. of lauryl-pyridinium bisulfate. By adding dodecyl sulfate an emulsion of neg. charge is obtained. Other examples are given.

**Emulsifying agents, etc.** Henryk Cohn and Conrad Siebert. Fr. 785,078, Aug. 16, 1935. Waxes, such as wool fat, are saponated at ordinary temp. by means of an alc. soln. of KOH contg. petroleum ether. The product insol. in alc. is sepd. and from the dissolved product is sepd. by means of water an insol. substance, which, on swelling, absorbs a large amt. of water which may be removed again by heating. The product has various technical uses but is particularly suitable for the prepn. of emulsions, and for cosmetics and pharmaceutical products.

**Dispersing agents, etc.** Imperial Chemical Industries Ltd. Fr. 785,500, Aug. 19, 1935. Products which are used for emulsifying and dispersing or as auxiliary agents in the textile industry are obtained by causing degreas to react with ethylene oxide. The reaction is carried out in the presence of a substance having surface activity or a caustic alkali.

**Cleansing compositions.** Johann Bertram and Erich

Schieweck. Fr. 785,941, Aug. 22, 1935 See Ger. 616,443 (C. A. 29, 7531<sup>1</sup>).

Cleaner Rudolf Wille. Ger. 616,885, Aug. 5, 1935 (Cl. 30k 13) A compn for removing black grease or oil, etc., from the hands, etc., consists of leather felt pads soaked in a mixt. of soap soln and acetone or other solvent miscible with water.

Bleaching agents Henkel & Cie G. m. b. H. Fr. 785,329, Aug. 7, 1935 Bleaching agents contg percompds and salts of pyro- and meta phosphates are stabilized in alk medium by adding Al hydroxide, or salts of Al by which the hydroxide is formed. One example contains soap 57, neutral Na pyrophosphate 12, Na perborate 12, Na<sub>2</sub>CO<sub>3</sub> 14 and Al hydroxide 5 parts.

Penetrating liquid suitable for removing dirt, grease and rust from metal and lacquered surfaces, etc. Dudley K. French. U. S. 2,021,448, Nov. 19 A mixt. of kerosene 60, sulfonated castor oil 20 and sulfonated coconut oil neutralized by rosin soap 20% is stabilized by less than 1% of alc. and forms an emulsion when mixed with water.

Polish Henri J. S. Cuvilliez. Fr. 785,300, Aug. 6, 1935 The polish contains mineral oil 2.5, turpentine oil 2.5, alc 1, glycerol 0.11, CaCO<sub>3</sub> 0.6, shellac 0.3 and animal wax 1 kg.

Polishing composition for use on furniture or automobiles John Rong. U. S. 2,020,125, Nov. 5. A polishing compn is formed of motor lubricating oil 1 oz, kerosene 0.5 oz, gasoline 0.5 oz, "ammonia oil" 1 oz, japan varnish dryer 1 oz, furniture glue 0.5 oz, "liquid bronze" 1.5 oz, Javel water 1.5 oz, "turpentine 0.25 oz, "plain white ammonia" 0.25 oz, a "commercial disinfectant" 4 drops and camphor oil 7 drops.

Polishing devices such as wheels or belts comprising resilient rubber Frank H. Manchester (to Wingfoot Corp.). U. S. 2,021,371, Nov. 19. A body of resilient rubber carries a thin outer layer of treated rubber, difficultly separable and substantially inelastic, having very small fissures and carrying an outer coating of adhesive and abrasive material.

Aqueous oil emulsions such as automobile polishes. Foster D. Snell and Cyril S. Kimball. U. S. 2,021,027, Nov. 12. An emulsifier compn suitable for making aqueous emulsions of oils with 1 part of emulsifier to 50 parts of oil such as a lubricating oil to 200 parts of the emulsion as a whole comprises at least 3 colloidal substances including (a) starch, dextrin, glycogen or flour, (b) gum arabic or gum senegal, gum tragacanth or gum karaya and (c) algin or pectin (the carbohydrate material predominating). A metal soap such as Ca or Al oleate or stearate and an unorg abrasive material such as diatomaceous earth may be added to form a polishing compn.

Cores for electric apparatus Haristoff-Metall-A.-G. (Hametar). Ger. 619,064, Sept. 23, 1935 (Cl. 21g 31.03) Powd. ferromagnetic material of a granule diam below 0.2 mm. is mixed with a binder, e. g., a soln of borax or resin, and with finely ground asbestos of a filament thickness below 0.001 mm. The mixt. is then compressed in the usual way.

Cores for electric apparatus Siemens & Halske A.-G. Ger. 619,095, Sept. 23, 1935 (Cl. 21g 31.03) Powd. ferromagnetic material is compressed into a rigid form with the aid of an insulating binder in a proportion below that necessary for the required degree of insulation. The compressed material is then further insulated by impregnation with an oil or fat in a nonviscous liquid form.

Magnets Mitsubishi Denki K. K. Yogo Kato and Takehiko Takei. Brit. 432,152, July 22, 1935 In a magnet consisting of a magnetized mass of particles of oxides of Fe and other metals, disintegration is prevented by an exterior protective covering or by a reinforcement embedded in the material or by both these means. The material may be oxides of Fe and Ni or Co and has an external cover of gauze, sheet metal, enamel or lacquer or, alternatively or in addn, reinforcing bars of Ni, porcelain or earthenware, embedded therein.

Spark plug with an ionizing catalytic element Charles Michel. U. S. 2,022,140, Nov. 26 The bottom end of a central electrode is surrounded by a grounded body and

1 is covered with a layer of radioactive substance of ionizing catalytic character incorporated in a porous enamel of high m. p.

Envelopes or containers for chemical heating compositions Karel Gutwirth. Brit. 431,255, July 3, 1935

Optical polarizers Edwin H. Land. Brit. 433,455, Aug. 15, 1935 A polarizing body comprises a set suspending medium having a multiplicity of dispersed particles of an inorg. polarizing periodide embedded therein with their polarizing axes oriented into substantial parallelism. The prepn. of the suspending medium, which is preferably a cellulose compd., the incorporation of the polarizing particles therein, and the orientation of the particles are effected as described in Brit. 412,179 (C. A. 29, 1907<sup>1</sup>). Purpureocobalt chloride sulfate periodide is preferably used.

Laminated material suitable for container cap facings George Goebel (to Crown Cork & Seal Co.). U. S. 2,020,776, Nov. 12 A cap shell contains a cushion liner such as cork and a facing comprising a backing such as adhesive united to the liner and carrying a coating of sprayed metal such as Al, Zn, Ni or Cu.

Laminated composition gaskets suitable for engine manifolds, etc. John H. Victor, Wm. A. Heunze and Joseph B. Victor (to Victor Mfg. & Gasket Co.). U. S. 2,021,571, Nov. 19. Layers of preliminary oil-treated and backed packing material and other layers of untreated material, of various thicknesses, are built up, with an adhesive such as a phenol resin, to a desired thickness, with use of heat and pressure, the exterior surfaces of the material having indentations in which a graphite compn is placed.

Composite gasket sheets Claude B. Bailey (to McCord Radiator & Mfg. Co.). U. S. 2,019,474, Nov. 5 Various details of app. and operation are described for the manuf. of sheets with outer layers of discrete particles such as those of asbestos and an insert layer of sheet metal such as sheet steel.

Laminated sheet material suitable for decorative purposes Benjamin Ames (to Dennison Mfg. Co.). U. S. 2,020,928, Nov. 12 Sheets of material such as paper, cloth or regenerated cellulose are adhesively united, one sheet being more wavy than the other so that when the material is stretched crosswise of the waves the other sheet will tear along lines extending transversely to the direction of stretching.

Decorative material suitable for use on table tops, etc. Amerigo F. Caprio (to Celluloid Corp.). U. S. 2,019,951, Nov. 5. A relatively thin metal foil such as Al foil has a design embossed on it and is united, by an adhesive, to a transparent facing sheet of a thermoplastic material contg. a cellulose deriv. such as cellulose acetate or nitrate and to a backing sheet of a thermoplastic deriv. of cellulose such as celluloid.

Plywood, etc. Karl Vierling, Matthias Schmiling and Hugo Klingenberg (to I. G. Farbenind. A.-G.). U. S. 2,019,834, Nov. 5. There is applied to the surfaces to be joined a soln. of a water-sol. condensation product of a urea and CH<sub>2</sub>O to which an acid-reacting agent or acid-forming agent inert to CH<sub>2</sub>O has been added shortly before use of the soln., and the surfaces are united by heat and pressure.

Moistureproofing thin, flexible transparent cellulose acetate sheeting adapted for wrapping purposes Norman F. Beach and James G. McNally (to Eastman Kodak Co.). U. S. 2,019,645, Nov. 5. The material is first treated with an adhesive layer comprising a gum such as ester gum and on the gum layer there is superimposed a layer mainly formed of wax such as paraffin or candelilla wax. An arrangement of app. is described.

Mineral fiber from clay and limestone. Isaiah B. Eagle. U. S. 2,020,403, Nov. 12 A mixt. of clay and waste limestone is formed into briquets or the like which are subsequently fused to produce a molten stream of material, and a stream such as one of steam is directed against this molten stream to produce a fibrous product. App. is described.

Apparatus for making artificial pumice from slag. Wilhelm Benzinger. Fr. 777,782, Aug. 19, 1935.

Decolorizing mineral substances such as fine sand, clay or kaolite. Frederick W. Birn (to Virginia Smelting Co.) U. S. 2,020,192, Nov. 5. The mineral matter is treated in water containing an acid to reduce the pH of the resulting mix. to 2.5 or less and this mix. is then mixed with a coarse mix. of Zn hypochlorite or the like such as one which has been prepared by treating Zn dust with  $SO_2$  in the presence of water.

Disintegrating diatomaceous earth. Guy C. Pierce (to Fila L. Pierce) U. S. 2,021,576, Nov. 19. A mass of particles of the material of given size is compressed by a pressure less than required for disintegration of the particles, and a shearing force is then applied to the compressed mass to shear the particles into particles of smaller size. App. is described.

Dental impression material. Laurence F. Harrison (to Ormold Products Corp.) U. S. 2,021,036, Nov. 12. An embossed impression material is formed from ingredients such as resins, oil, Pentaclak gum, triethanolamine, agar-agar, coral gum, water and wax or the like. U. S. 2,021,036 relates to an impression material containing dusted cement vulcanized rubber filaments together with other ingredients such as water, agar-agar, glycerol, a rubber lower mix,  $K_2SO_4$ , menthol and oil of wintergreen.

Thermoplastic compositions. Wingfoot Corp. Fr. 745,841, Aug. 14, 1935. A boiling mix. of rubber in an organic solvent is treated with an amphoteric metal halide, e. g.,  $SnCl_4$ ,  $FeCl_3$ ,  $CrCl_3$  or  $AlCl_3$ , and the product is poured into a large amt. of water containing a reducing agent, e. g., hydroquinone,  $Na^+$  or  $Na_2SO_3$ . The solvent is removed by steam distn. and a thermoplastic condensation product of rubber is obtained.

Thermoplastic compositions suitable for making dental impressions, etc. Laurence F. Harrison (to Ormold Products Corp.) U. S. 2,020,211, Nov. 12. A resin is fused with glycerol at a temp. of 200-220°, the temp. of the mix. is lowered to about 170° and there is then incorporated with it a substantially water-insol. soap such as Al or Zn stearate and a triethanolamine soap. Various modifications are described.

Coating-compositions. Oskar Schnor (to Metallgesellschaft A.-G.) Brit. 423,101, Aug. 8, 1935. Adds to 400,224 (C. 28, 63289). A form of the liquid coating-compn. claimed in 409,234 contains 25-60% of an Al-Si pigment contg. 7-95% Si, in such fine state of division that it passes through a sieve of 10,000 meshes per sq. cm., together with 25-70% of 1 or more binding media or vehicles. Up to 10% of diluents and up to 35% of inert mineral fillers may be added. Driers may be added. The addn. of alk., BuOH, hexamine or PhNH<sub>2</sub> prevents thickening.

Coating-compositions. Hubert-Lambert and Edmond Hubert. Fr. 764,732, July 23, 1935. Coating- and binding-compos. are made by mixing a natural or artificial emulsion of rubber or like vegetable milks with powder material to vary the viscosity of the mix., dyes, an emulsifying agent and a vulcanizer. The start of the vulcanization may be obtained by the addn. of a certain amt. of  $SiO_2$ , e. g., in the form of fine sand.

Coated fabrics. Wm. H. Hale. Brit. 423,205, Aug. 8, 1935. Fabric is reinforced by coating with a plastic compound composed of silicates of Na,  $Fe_2O_3$ , soap, soda, carnauba wax and gum arabic. The coated fabric may be used as a lining between the inside and outside of boots and shoes, etc.

Coated projection screen. David E. Knapp. U. S. 2,020,787, Nov. 12. To produce an uneven light-reflecting and distributing surface, a light-colored priming coating is applied to a sheet of fabric, the sheet is embossed, the entire surface is coated with a lustrous coating comprising powd. metal such as Al and flaky light-reflecting particles such as those of mica and the raised portions of the embossed sheet are wiped off while still wet so as to leave the depressed portions coated with the lustrous coating

and the raised portions of contrasting appearance beneath a transparent glass or the wiped surface.

Nonflammable coatings for sliding-clasp fastener parts. Gen. H. C. Corner and Lightning Fasteners Ltd. Brit. 422,847, July 31, 1935. The parts, made of non-metallic material, e. g., celluloid, are rendered nonflammable by applying a thin film of a nonflammable material that evolves carbonation-suppressing gases when heated, e. g., a chlorinated rubber compn. is sprayed thereon, the stinger tapes being protected by masks. If necessary, a bonding coating, e. g., a gelatin or synthetic resin soln., is appld. previously.

Protecting coatings such as automobile finishes during shipping, handling or storage. Lloyd G. Copeman (to Copeman Laboratories Co.) U. S. 2,020,220, Nov. 5. Various details are described relating to the use of a protective coating formed of paper and rubber deposited from an aq. dispersion, which serves to protect the permanent coating and is readily removable when desired. U. S. 2,020,227. See Brit. 420,250 (C. 29, 34284).

Metal-coated articles. R. I. du Pont de Nemours & Co. Brit. 422,218, Aug. 12, 1935. Base materials of a cellulosic nature are treated with a permanently-setting adhesive compn., to which, when partially dried but still in a tacky state, a metal powder, the individual particles of which have light-reflecting faces, is applied, said faces being then oriented by burning with brush rolls while the adhesive is still incompletely dried. The adhesive is finally hardened by baking.

Ornamentation comprising pyroxylin and metal ornaments. Morris H. Wagman. U. S. 2,020,607, Nov. 5. An article such as a lady's hand bag is provided with a pyroxylin coating on which metal initials or ornaments are secured by a cellulose backing and use of a solvent such as  $EtOAc$  and BuOH.

Ornamenting plastered or whitewashed surfaces. Xavier Meier. Ger. 619,578, Sept. 28, 1935 (Cl. 756 6). Use is made of a paint prepd. in a medium comprising a decoction of Iceland moss, animal glue and a small proportion of glycerol. A design is painted on paper with this paint, and the moist paper is then applied to the surface with the aid of a rubber roll.

Protective foil. Wolf & Co. Kommandit-Gesellschaft auf Aktien, Hans I. Schulz and Julius Appel (to Transparent Paper Ltd.). Brit. 423,011, Aug. 7, 1935. A protective foil for application to cards, pictures, maps, etc., comprises a foil of cellulose hydrate having a coating that becomes plastic and adhesive when warm and which is composed of a cellulose deriv. of low viscosity and a resin that retains the same in soln. in the warm, with or without plasticizers. The coating layer should contain cellulose deriv. 30-60, natural or synthetic resin 10-60% and 15-60% of plasticizer referred to the dry products. If the resin is a solvent for the cellulose deriv. in the liquid condition, the plasticizer may be omitted. The foil may be waterproofed with a lacquer coating prior to the application of the adhesive layer.

Paper impervious to moisture and grease. Edmond H. Bucy (to Atlas Powder Co.). U. S. 2,021,172, Nov. 19. Flexible paper sheets are treated with an oil-sol. condensation product of the  $PhOH-CH_3O$  type in soln. in a drying oil such as linseed oil and castor.

Ultramarine blue. Henry Dourif (to Standard Ultramarine Co.). U. S. 2,020,539, Nov. 12. An ultramarine gel for laundry purposes capable of reverting to liquid form upon agitation comprises a mix. of finely divided ultramarine blue particles with monoethanolamine, diethanolamine or triethanolamine and water, forming a nonsetting compn. readily dispersed in water. App. is described.

Gramophone records. Abraham van Paap, Henrij van Paap, Rozalie van Paap-Maykels (representatives of Salomon van Paap, deceased). Pnt. 422,763, Aug. 1, 1935. Defective or obsolete sound-record disks are reconditioned by coating them with a thermoplastic sound-record compn. having a basis of a polyvinyl compd., e. g., a polyvinyl ester such as polyvinyl acetate, and simultaneously or subsequently subjecting them to pressure between matrices. Intermediate heat-insulating layers of

paper, fabric, wood, etc., may be interposed between the disk and the coating

Sealing compositions for containers Crosse & Blackwell Ltd., Wm Clayton and Robert I. Johnson. Brit. 432,571, July 30, 1935 The compns comprise an alkali silicate, alk earth silicate or org silicate in soln or dispersion in an aq system, 1 or more turgescant materials of colloidal character, e.g., starches, bours, proteins, hydrophilic colloids,  $H_2O$ -sol cellulose derivs, or aq suspensions of  $H_2O$ -insol cellulose derivs, and an org hygroscopic substance, e.g., glycol, glycerol or diethylene-glycol Natural or synthetic resins and fillers, e.g., kaolin or diatomaceous earth, may be added

Stencils Sydney G. Pipe Brit. 432,358, July 25, 1935 A sheet for use in the manuf of stencils for screen stencil printing comprises a sheet of transparent paper coated with cellulose acetate (I). A sheet of oiled or waxed paper is coated with I and, optionally, with an outer coating of adhesive, e.g., shellac, the stencil is cut in the coating and the necessary parts are removed, the sheet is applied to a silk screen with the aid of heat or a solvent and the paper backing is removed to leave parts of the screen blocked out by I.

Isinglass Charles F. Carpenter and Morrison, Carpenter & Co Ltd Brit 432,159, July 22, 1935. In the production of isinglass finings by dissolving isinglass in  $H_2O$ , the material is subjected to a steady agitation as the soln proceeds The isinglass may be so treated for about 12 hrs after standing in acidulated  $H_2O$  about 3 hrs The soln is then filtered App is described

Photomechanical printing processes Bekk & Kaulen chem Fab G. m. b. H. Ger. 617,749, Aug 24, 1935 (Cl 57d 2 03). Addn to 605,296 (C. A. 29, 1183). The alk. shellac soln. used in the process of Ger. 605,296 is prep'd. with the aid of an aq soln of an alk comp'd. of Li, e.g.,  $LiOH$  or  $Li_2CO_3$ . Cf. C. A. 30, 198.

Flexible planographic printing element ("offset blankets or plates") Benjamin L. Sites (to the Miehle Printing Press and Mfg Co.). U. S. 2,020,479, Nov. 12 A backing of paper is united with a sheet of fabric on which is cemented a top sheet of rubber

Etching solid areas on intaglio printing cylinders. Irving Gurwack (to Shellmar Products Co.). U. S. 2,021,687, Nov. 19 An etching fluid is applied through a preliminarily screened carbon tissue or resist, and a desired deep etch is obtained without breaking down or undercutting the screen by placing solid  $CO_2$  in an etching bath to reduce its temp. to below  $5^\circ$ , first applying to the resist an etching fluid of normal temp adapted to penetrate the thin areas of the resist and then continuing the etching with the chilled etching fluid

Transferring designs, etc Bakelite Corp Brit. 432,508, July 29, 1935 Compd paper sheet material, etc., for use in the manuf of panels, table tops, etc., is coated with a compn requiring baking by applying the compn in liquid form to a metallic surface, baking, transferring the baked film to the material and fixing it thereto by an interposed bonding-sheet impregnated with adhesive. In an example, a synthetic resin lacquer (prep'd by causing glycerol to react with 2 parts of phthalic anhydride with the application of heat till  $200-10^\circ$  is reached, when the reaction is arrested by adding a solvent, e.g., diethyl oxalate, and dilg with  $Me_2CO$ ) is applied to a Cr-plated metal sheet or to Al foil, dried and baked Designs may be printed on the film before baking and may be covered by an enamel coating, or the metal sheet may be etched so that a relief or intaglio design is formed on the film; an ornamental film may be formed also by applying differently-colored compns, superposed or side-by-side. The coated metal sheet is placed on top of material consisting of a no. of paper sheets impregnated with synthetic resin, a paper sheet impregnated with rubber or an alkyl resin being interposed, and heat and pressure are applied, the metal sheet being finally removed. Dyes and pigments may be added to the lacquer.

Composition for blocks or surfaces of brakes James N. Longley (to Ferodo Ltd.). U. S. 2,020,701, Nov. 12 A synthetic resin binder in finely divided form is mixed with 5-7 times its volume of a nonmetallic inert and inert finely divided water-insol filler such as "rottenstone" having an av. particle size not less than that of diatomaceous earth, and the mixt. is consolidated by heating to above  $132^\circ$  and subjecting to a pressure of 4 to 5 tons per sq in. Asbestos fiber, etc., also may be used

Apparatus for testing the hardness, etc., of brake lining Wm. S. James (to Bendix Aviation Corp.). U. S. 2,022,040, Nov. 26 Various structural, mech and operative details

Brake operating liquid. Franklin P. Frey (to Johnson Oil Refining Co.) U. S. 2,020,543, Nov. 12. Castor oil 40 and EtOAc 60% are used together.

Uniting pieces of leather stock as in shoe manufacture Max W. Tetlow (to United Shoe Machinery Corp.). U. S. 2,019,585, Nov. 5 Pieces of stock are stitched together with thread composed at least in part of sol fibers (such as those of cellulose acetate) the material of which when dissolved is adhesive in character, and the thread is treated with a solvent which cements the stitches in place.

Stiffened shoe upper material Charles E. Reynolds (to Cambridge Rubber Co.). U. S. 2,021,870, Nov. 19. Material such as canvas, twill or duck is impregnated with a starchy sizing agent which adheres firmly to the fibers of the material and is rendered stiff and stable but somewhat flexible, and, after squeezing out the surplus, the material is further treated with an aq dispersion of a waterproofing agent such as "Vulter" and is dried, using sufficient waterproofing constituents to protect both the fiber of the goods and the stiffening agent.

Preserving animal and vegetable substances. John Bleck Fr. 785,524, Aug. 12, 1935. An aq soln. of  $Na_2SO_4$  and  $NaF$  (neutral or acid) contg. a suspension of kaolin is used.  $Na_2SiF_6$  may also be present. An example contains in water  $Na_2SO_4$  80-85,  $NaF$  2-6,  $Na_2SiF_6$  0.25-2 and kaolin 12-17%.

Dispersions such as those of insecticides or pigments Wm. S. Calcott and Ira Williams (to E. I. du Pont de Nemours & Co.). U. S. 2,021,143, Nov. 19. Material to be dispersed, such as S (or various other named materials) and a dispersing medium, in liquid condition, such as water and glycerol or  $NH_4$  caseinate, are passed through a tube at a velocity greater than the crit velocity and in unobstructed flow Various operative details are given.

Fly killer I. G. Farbenund A.-G. (Kaspar Pfaff and Michael Erleben, inventors) Ger. 616,770, Aug. 5, 1935 (Cl 45f, 3 01). Flies are destroyed by spraying closed rooms with a soln. or emulsion of aromatic hydroxy-carboxylic acids esterified or etherified in both the  $COOH$  and  $OH$  groups Other insecticides may be present In an example, a spray of the methyl ester of 1-phenoxy-benzene-carboxylic acid, soft soap and the oleic acid ester of Na hydroxyethanesulfonic acid emulsified in water is used. Other examples are given

Sterilizing liquids Jesús Cambón Fr. 784,959, July 30, 1935. Metals such as Ag or Au are deposited in colloidal form on pulverulent inert material such as clay, infusorial earth,  $Al_2O_3$  or  $MgO$  and this is used for sterilizing wines, beer, milk, etc

Fireproofing agents Günther Schwedler and Hans Schwinzer. Brit. 432,819, Aug. 2, 1935 See Fr. 774,664 (C. A. 29, 2268).

Fire extinguishers Établissements Bouillon Frères Fr. 785,180, Aug. 3, 1935 Powders which are bad conductors of electricity, such as  $NaHCO_3$ , talc,  $CaCO_3$ ,  $MgCO_3$ , etc., are mixed with substances which under the action of heat form a resistant insulating layer on the article on which the powders are sprinkled, e.g.,  $Al_2(SO_4)_3$ ,  $Na_2SO_4$ ,  $NaHSO_4$  and Na tetraborate

## 14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

Water investigations. Uniform methods and standardization Haase. *Chem. Fabrik* 1935, 421. J. H. M. The Stuttgart water supply. E. Link. *Deut. Wasserv.* No. 7, 141-2(1935); *Wasser u. Abwasser* 33, 229-30.—Details F. P. Griffiths

Russian water-supply systems. V. E. Kojnov. *Water Works Eng.* 88, 1234-7(1935). W. A. Moore

Mineral water of the springs in Bad Deutsch-Altenburg J. Gangl and R. Posega. *Wien. klin. Wochschr.* 48, 1157-60(1935) —The content of electrolytes and gases is given D. B. Dill

Thermal springs at Aix-les-Bains Georges Schneider *Ann. mines* 8, 5-64(1935) —The sulfur spring, in 1927, flowed about 12 l.  $H_2O$  per sec. at 40° of the following compn., Ca 111, Mg 22, K 2, Na 28,  $SO_4$  167, 7 and  $Cl_{25}$  8mg per l. The alum spring flowed about 29 l. per sec. at 43°. The solids contained Ca 07, Mg 14, K 0, Na 28,  $SO_4$  131, 9 and  $Cl$  16, 6 mg. per l. H. E. M.

Supplying pure water for beverage manufacture. E. S. Stetler. *Food Ind.* 7, 635-6(1935). —The ground water is softened by zeolite treatment, mineralized, adjusted for pH value, filtered, treated with  $O_3$  to destroy bacteria, refrigerated to 33°F. and treated with ultraviolet irradiation for a final sterilization C. R. Fellers

The occurrence of iron in the water of the northeast Pacific Ocean Thomas G. Thompson and Raymond W. Bremner. *J. conseil intern. exploration mer.* 10, 39-47(1935); cf. *C. A.* 30, 4074. —The inland waters of the San Juan Archipelago averaged 0.0053 mg. atoms total iron per kg. of unfiltered sea water during the spring and summer and 0.00147 mg. atoms per kg. during the fall and winter Sol iron (filtered water) averaged 0.00026 mg. atoms per kg. for the spring and summer and 0.00075 mg. atoms for the fall and winter. Most of the iron contributed by the rivers to the sea is in the suspended form. The sol and total iron content of the water over the continental shelf in the Gulf of Alaska is similar to that of the previous water. The sol iron content of the ocean varied but little vertically and was about half that in the coastal waters during the spring and summer. The total iron content of the open ocean was about 0.00035 mg. atoms per kg. in the water layer near the surface. Max. values of about 0.0007 mg. atoms iron were found at 800 to 1000 m. which is the zone of very low O values. Below 1000 m. the total iron content decreased but slightly. Rex J. Robinson

Step-photometric determination of silicic acid and the application of the method to the examination of water and mineral water. R. Strohecker, R. Vaubel and K. Breitwieser. *Z. anal. Chem.* 103, 1-12(1935). —Most natural waters contain 5-50 mg. of dissolved  $SiO_2$  per l. probably present in a state of mol. dispersion and, according to Fresenius (*C. A.* 26, 541; 28, 5557) the dissolved  $SiO_2$  has a therapeutic action. A silicomolybdic acid suitable for analysis can be obtained by adding 0.2 cc. of 50%  $H_2SO_4$  by vol and 2 cc. of 10%  $NH_4$  molybdate soln. to 50 cc. of the water. The most suitable filter for the step-photometric measurement is the violet filter S 43. The Lambert-Beer law holds rigidly for the yellow color measurement only up to about 10 mg.  $SiO_2$  per l. From then on there is a slow but perceptible decrease in the extinction power. The  $K_2CrO_4$  and picric acids soln. used for measuring the color of silicomolybdic acid show the same phenomena at almost the same color value and consequently their suitability for colorimetric comparisons can be based upon the step photometer. With respect to the blue reduction product of the silicomolybdic acid complex, the color is more intense than should be the case for dil. solns. Data are given to show the accuracy of the measurements as compared with the much more tedious gravimetric detn. of  $SiO_2$  W. T. H.

The determination of the  $CO_2$  pressure of natural water. Aino Pekkarinen *Suomen Kemistilehti* 8B, 34(1935)(in German) —A preliminary report. The method of Kauko

(*C. A.* 28, 3337) can be used to det. the  $pH$  after passing  $CO_2$ -satd. air through the water. E. E. Jukkola

The corrosive effect of ground water on concrete. Toivo Karttunen. *Teknillinen Aikakauslehti* 25, 301-2(1935). —Analysis of the water from a concrete-lined well showed decided changes in compn. depending on the length of time the water stood in the well. E. E. Jukkola

Ozone solves color, odor and taste problem in Hobart (Ind.) plant. Theodore O. Gerkinhoff. *Am. City* 50, No. 11, 47-8(1935) —Ozonization of  $H_2O$  after filtration removes vegetable taste and odor and reduces color index from 25 to approx. 2. Germicidal action is appreciable, a reduction in *Es. coli* per ml. on 37° agar from 1000 on 3 raw  $H_2O$  (8 after filtration) to zero after ozonization being obtained. Ozone is prep'd by applying a potential of 18,720-20,800 v. to a stream of dry air, approx. 1000 gals.  $H_2O$  is ozonized per kw-hr. Expts with other methods for taste, odor and color removal showed ozonization to be the most effective G. H. Young

Permanent chlorine standards Francis E. Daniels. *Public Works* 66, No. 11, 26(1935), cf. *C. A.* 29, 1546. —Directions are given for prep.  $K_2Cr_2O_7$ - $CuSO_4$  standards for use in field testing sets (1/2-2-in. depths in comparator tubes). In checking the standards the o-tolidine was added to the diln.  $H_2O$  before adding the measured amts. of carefully titrated  $Cl_2$  soln., only in this way could consistently accurate checks be obtained. Necessary tables for diln. are included G. H. Young

Improved standards for the residual chlorine tests. R. D. Scott. *Water Works and Sewerage* 82, 399-400(1935); cf. *C. A.* 29, 2634. —Use of buffered chromate-dichromate standards rather than the conventional dichromate- $CuSO_4$  mixts. is recommended on the basis of greater flexibility; color matching is exact regardless of whether viewed through the length or breadth of tubes over the broad range of 0.01-3.0 p. p. m.  $Cl_2$  Buffering is necessary to prevent changes in color of standard solns., a pH of 5.2-5.7 being required for max. stability. Directions for prep. of the chromate-dichromate soln., and the  $KH_2PO_4$ - $NaOH$  buffer (pH 6.5) are detailed together with the necessary table of dilns. for a  $Cl_2$  range from 0.01 to 3.0 p. p. m. Standards thus prep'd will stand as much as 6 weeks' direct exposure to sunlight without fading. G. H. Young

The development of the ammonia chlorine treatment for water disinfection. Paul D. Haney. *Kansas Water Sewage Works Assoc.* 4, 76-82(1934-5). —A review, with 9 references. M. G. Moore

Plant tests for residual chemicals resulting from water treatment. Selma Gottlieb Kallis. *Kansas Water Sewage Works Assoc.* 4, 80-2(1934-5). —The various methods for the detn. of residual  $Cl$ , pH hardness, Cu, Fe, alkyl, and Al are discussed. W. A. Moore

Standardization work in the field of water softening Splittergerber. *Chem. Fabrik* 1935, 421-7. —Proposed analytical methods and reagents. J. H. Moore

\$1,000,000 water-softening plant. Robert W. Wolfenden. *Water Works Eng.* 88, 1238-9(1935) —The raw water at the Hamilton, Ohio plant is first aerated, then softened by the lime, alum, and soda ash treatment. Provision is also made for the use of chloramine or  $Cl_2$  if necessary. W. A. Moore

Madisonville (Ky.) new filter plant. Hall Arnold. *Public Works* 66, No. 11, 11-12(1935). G. H. Y.

A well filter of ball-shaped stoneware. F. Hosemann. *Deut. Wasserv.* No. 3, 58-9(1935), *Wasser u. Abwasser* 33, 240-1. —The wall of the well is built of a double layer of balls of masonry cemented at points of contact. The advantage of greater porosity is claimed. F. P. G.

The use of activated carbon at Garden City. A. H. Rogers. *Sewage Works J.* 7, 601-5(1935). E. H.

Pure water and sewage. Experiences with sprinkling systems. Gusovius. *Berliner Börsen Ztg.* No. 317, 15(1935); *Wasser u. Abwasser* 33, 270-1. —Results of the



use of purified effluent for crop fertilization are discussed. Care in application is essential and moderate increases in crop yields result. F. P. Griffiths

Primary elements for sewage and water-works meters L. D. Carlyon *Water Works and Sewerage* 82, 375-8 (1935).—Discussion of the fundamental principles, applications and relative merits of the (1) Venturi tube, (2) flow nozzle and (3) orifice, as applied to the measurement of sewage, sludge, water and air. G. H. Young

Water supply and sewage disposal at Singapore, Straits Settlements Isadore W. Mendelssohn *Water Works and Sewerage* 82, 371-4 (1935). G. H. Young

A day at a sewage treatment plant Frank W. McCann *Sewage Works J.* 7, 694-5 (1935). E. H.

Experiences at Newark sewage-treatment plant Thomas J. Smith *Sewage Works J.* 7, 684-91 (1935), cf. C. A. 29, 7539<sup>1</sup>. E. Hurwitz

Economic operation and power development at the Springfield, Ill. sewage-treatment plant W. B. Walraven *Kansas Water Sewage Works Assoc. J.* 134-41 (1934-5). W. A. Moore

Disposal of sewage from the South Essex Sewerage District, Salem, Massachusetts. Edward Wright *Sewage Works J.* 7, 663-72 (1935). E. Hurwitz

Construction of a sewage treatment plant under relief program Robert C. Wheeler. *Sewage Works J.* 7, 706-12 (1935). E. Hurwitz

Sewage and waste water disposal in the Niers (Germany) area Schmitz-Lenders *Zentr. W. Bauverw. No.* 22, 422-31 (1935). *Wasser u. Abwasser* 33, 264-5. F. P. Griffiths

The Stuttgart sewage-disposal system Maier and Sohler *Deut. Wasserw. No.* 7, 143-7 (1935). *Wasser u. Abwasser* 33, 275-6.—Details are given. F. P. G.

The present knowledge of sewage disposal P. Langbein *Der Bauingenieur* No. 13-14, 147-53 (1935). *Wasser u. Abwasser* 33, 260-2, cf. C. A. 29, 5209<sup>1</sup>—A review. F. P. Griffiths

Successful sewage-plant operation. Ralph E. Fuhrman *Kansas Water Sewage Works Assoc. J.* 119-21 (1934-5), cf. C. A. 28, 6903<sup>1</sup>.—The value of the lab in the operation of a sewage plant is stressed. W. A. M.

The patent situation in sewage treatment Paul Hansen *Public Works* 66, No. 11, 13-14, No. 12, 49-50. *Water Works and Sewerage* 82, 425-6 (1935). G. H. Y.

The use of chemicals in the treatment of sewage F. M. Veatch *Kansas Water Sewage Works Assoc. J.* 122 7 (1934-5).—Twelve recently advanced methods for the chem. coagulation of sewage are discussed. The Scott-Darcy process for the prepn. of  $FeCl_3$  is also described. V. states that this process is the most economical source of  $FeCl_3$  for use in medium and large size treatment plants. W. A. Moore

Chemical mechanical treatment of sewage Louis P. Booz *Am. City* 50, No. 11, 65, 67 (1935).—The new Perth Amboy (N. J.) sewage plant is described. G. H. Young

Chemical mechanical treatment of sewage V. Sludge treatment Factors governing choice of method—results obtainable Philip B. Streadwell and Michael J. Blew *Public Works* 66, No. 11, 22-4 (1935), cf. C. A. 30, 199<sup>1</sup>.—Under sludge treatment are discussed (1) dewatering on drying beds, (2) vacuum-filter dewatering, (3) centrifuge dewatering, (4) chem. sludge digestion, (5) ultimate disposal (1) Choice of methods, (2) patent situation, (3) design procedure and (4) degree of treatment possible and results are discussed. G. H. Y.

Chemical treatment of sewage evaluated in A. P. H. A. committee report Langdon Pearse *Water Works and Sewerage* 82, 392-3 (1935).—(1) Chem. treatment usually accomplishes a degree of treatment intermediate between plain sedimentation and biol. processes (2) By addn. of a zeolite filter, following chem. pptn. the Guggenheim process substantially equals an activated-sludge process. (3) Seasonal or occasional use of chemicals attracts favorable notice where sedimentation alone may suffice for the greater part of the year or for many years out of a cycle

(4) Otherwise overloaded plants find the use of chemicals in addn. to sedimentation, ahead of biol. filters, worth while. (5) Fe salts, particularly  $FeCl_3$  and  $Fe_2(SO_4)_3$ , seem most satisfactory for chem. treatment; such salts may be economically manifold at the treatment plant from Fe scrap and  $Cl_2$ . (6) Use of  $FeCl_3$  for sludge conditioning prior to vacuum filtration has been generally adopted where practicable (7) Chem. treatment in handling industrial wastes is practicable where cost and performance justify the procedure as compared with other methods. G. H. Young

Plant experiments on the filtration of settled sewage W. Rudolfs, J. H. Brendlen and Wm. T. Carpenter. *Sewage Works J.* 7, 628-39 (1935).—Filtration through a mechanically cleaned rapid sand filter was effective in reducing the required settling time. The filter rates were varied from 0.58 to 3.33 gal. per min. without effecting materially the biochem. O demand, suspended solids or turbidity of the effluent. Companson is made with results obtained in a plain settling tank not equipped with filter. E. H.

The possibilities of the digestion of garbage in a sewage-treatment plant Harold E. Babbitt. *Sewage Works J.* 7, 658-62 (1935); cf. C. A. 29, 3757<sup>1</sup>. E. Hurwitz

Effects of sewage gases on concrete A. F. Pistor *Sewage Works J.* 7, 697-705 (1935).— $H_2S$  and  $CO_2$  react with concrete to cause its deterioration. E. Hurwitz

Putting sewage effluent to work Charles C. Agar. *Water Works and Sewerage* 82, 398 (1935).—Portable pump installations enable plant effluents to be used for (1) flushing tank walls, (2) breaking up solids in gas vents and (3) washing screen chambers, etc. G. H. Young

Determination of suspended solids in sewage by light absorption Glenn W. Holmes *Sewage Works J.* 7, 642-57 (1935).—The electronic cell was used with excellent results in the detn. of suspended solids. Communion of the larger suspended particles greatly improves the relationship between the suspended solids and light absorption. E. Hurwitz

Activated-sludge plant in Helsingfors R. Graugvist. *Tek. Foren. Finland Forh.* 55, 179-87, 215-17 (1935).—The plant consists of 4 units. Unit I is built according to Activated Sludge Ltd. specifications, which have been modified in Units II and III. Unit IV is an Imhoff system.

Operating data on the Units I, II, III and IV are, resp.: aeration time, 12.8, 9.2, 6.7, 10.8 hrs.; air consumption, 18.2, 15.8, 13.8, 9.1 cu. m./cu. m. water, biochem. Oxygen consumption in 5 days, 405 on all units, purification effect 94, 94.5, 94.4, 92.8%; capacity 3.55, 5.25, 6.15, 3.84 cu. m. water/sq. m. area/24 hrs.; sludge concn. 51.2, 54.0, 76.9, 71.2%; power consumption 306, 276, 234, 294 kw. hr./1000 cu. m. water. The gases with a heat effect of 6900 kg. cal. per cu. m. are collected and used for fuel on gas engines used for power. H. C. Duus

Sewage and sludge pumping Henry Ryon *Sewage Works J.* 7, 673-83 (1935); cf. C. A. 29, 8191<sup>1</sup>. E. H.

Thermal considerations in the design of heated sludge-digestion tanks Edward W. Moore. *Sewage Works J.* 7, 618-27 (1935).—M. has collected and summarized the data available on heated digestion tanks in light of the fundamental laws governing heat of flow and has developed a method of calcg. the approx. thermal balances in digestion tanks. E. Hurwitz

Adaptability of sewage sludge as a fertilizer E. E. DeTurk. *Sewage Works J.* 7, 597-609 (1935).—Activated sludge cannot be considered more than a low-grade fertilizer. Digested sludge has very little fertilizer value. Both types of sludge would be most valuable as fillers for synthetic fertilizers. Activated sludge contains 2 to 3% available N, 2 to 2.5% available phosphoric acid and approx. 0.2% potash. Digested sludge contains approx. 2% N of very low availability, 1.5%  $P_2O_5$  and 0.17%  $K_2O$ . E. Hurwitz

Public-health significance of sewage sludge when used as a fertilizer. Fred W. Tanner *Sewage Works J.* 7, 611-17 (1935).—Longevity studies indicate the presence of viable *Eberthella typhosa* in sludge sufficient to require a caution as to its use on truck-garden crops. The danger

appears to be minimized in well-digested sludge.

**Pollution of the Volga River by the Pravdinskii paper combine.** R. M. Pavilnova. *Bumazhnyia Prom.* 14, No. 8, 48-59 (1935).—The pollution of the Volga River by the waste waters of the paper mill and ways of economical elimination of the nuisance are discussed. C. Blanc

**Recent trade waste-treatment methods.** Willem Rindolls. *Sewage Works J.* 7, 713-26 (1935).—The literature published during the years 1932-34 on treatment of trade wastes.

**The disposal of industrial waste water in the central (city) sewer system.** G. Jordan. *Z. Gesundheitswesen u. Gesundheitsfürsorge* 5, 553-60 (1935). *Wasser u. Abwasser* 33, 256-7.—A discussion

**Grease removal at Hamilton, New York.** Leon Waldron. *Sewage Works J.* 7, 606 (1935).

**The disinfection of swimming pools with chlorine.** P. Alborno. *Rev. méd. del Rosario* 24, 1174-80 (1931). *Wasser u. Abwasser* 33, 241-5.—The use of a Cl-contg prep'n (Chlorox) in the disinfection of pools.

**Use of hypochlorous acid in the Stadion swimming pools, Breslau.** P. Wolf. *Das Bad* 30, 13-4 (1935). *Wasser u. Abwasser* 33, 247-8.—Application of NaOCl soln. twice daily to the sides and inlets of these open-air pools prevented growth of algae

**Rept. on cane [wo] constituents of water (Hance) 15. Iodine in Westphalia [detn in water] (Balks) 7. Se in the Colorado River (Williams, Byers) 8. Alkal metal tetraphosphates [products used for softening water] (U. S. pat. 2,019,667) 18**

**Purifying water.** Albert Schneider and Friedrich Gaisner. *Brit.* 432,123, July 22, 1935. In purifying  $H_2O$ , e. g., boiler feed water, by freeing it from hardness formers, the raw water is heated to about  $100^\circ$  and caused to trickle down in fine distribution in a cylindrical vessel into which a gas consisting chiefly of carburetted H and (or) N is introduced in counter current from below,  $NH_3$  being previously added to the  $H_2O$  or the gas. App. is described.

**Purifying water.** Rudolf Adler. *Ger.* 610,180, Sept. 25, 1935 (Cl. 85b, 1.01). A water-clarifying reagent is prep'd by digesting a soln. of  $Al_2(SO_4)_3$  with more than an equimol. amt. of freshly ppt'd.  $Al(OH)_3$  or  $Fe(OH)_3$ , and evap. the product, preferably *in vacuo*. Examples are given

**Purifying water.** Léon Drin. *Fr.* 784,780, July 21, 1935. Water is purified for drinking by an excess of hypochlorites and the excess is removed by a filter of active charcoal which also has a clarifying effect.

**Purifying water.** Robert Cambier and Marcel Cambier. *Fr.* 785,131, Aug. 2, 1935. Water contg. fermentable org. material is purified by suspending active charcoal therein and beating energetically with a current of air. App. is described.

**Apparatus for biological purification of waters.** Soc. d'entreprises et d'applications sanitaires. *Fr.* 781,705-6, July 23, 1935.

**Apparatus for proportional mixing of treating agents such as water-purifying reagents with water or other materials.** Harvey C. Waugh (to American Water Softener

Co.). U. S. 2,021,247-8, Nov. 19. Various structural, elec. and operative details.

**Water-softening apparatus.** Gustav F. Gerdt. *Brit.* 432,947, Aug. 6, 1935. App. comprising a vessel through which feedwater flows to the boiler and to which  $H_2O$  from the bottom of the boiler is returned for renewed chem. action has the return pipe provided with 2 throttles and with means between the throttles to indicate the flow of the return  $H_2O$ .

**Apparatus for softening water as by treatment with lime or soda and for separating resultant sludge.** Charles H. Spaulding. U. S. 2,021,072, Nov. 10. The raw water is mixed with a pptg. agent such as lime or  $Na_2CO_3$  in an agitating zone and the resulting mixt. is flowed upwardly through a quiescent pptg. zone open at the bottom and free from undissolved pptg. agent and through which the water rises at a constantly decreasing velocity, the velocity at the bottom being sufficient to support all but large sludge particles and that at the top being insufficient to support any substantial sludge particles, so that a considerable volume of sludge remains suspended in the lower part of the pptg. zone and acts as seed and the larger particles as formed descend out of the pptg. zone

**Apparatus for making a continuous record of the oxygen content of flowing water such as that fed to boilers.** Clark Pettinill and Elmer J. Harlow. U. S. 2,019,871, Nov. 5. The water is passed through a transparent vessel and suitable reagents such as pyrogallol acid and  $Na_2CO_3$  are introduced into the water whereby the light-absorptive quality of the water is changed by reaction between the O and the reagents, the water is passed through a second transparent vessel, and light from a common source is passed through the vessels and onto light-sensitive electric generative cells, currents from which are used for making the desired continuous record.

**Chlorinating apparatus formed of transparent material such as glass and suitable for chlorinating water, etc.** Dudley J. Lambert (one-half to Guy Ilearn). U. S. 2,021,116, Nov. 12. Various structural and operative details.

**Apparatus for treating waste waters for clarifying.** Lucien Linden. *Fr.* 785,936, Aug. 22, 1935.

**Removing salt from sea water.** Mutosel. *Fr.* 784,767, July 21, 1935. The halides are transformed by base-exchange agents into halides of heavy metals, e. g., of Cu, which are afterward transformed to insol. compds. and sep'd. from the water. The salts of Cu obtained are re-transformed to neutral Cu salts capable of being used to regenerate the base-exchange agents

**Sewage treatment.** John G. Reyan (to Guggenheim Bros.). U. S. 2,021,679, Nov. 19. Sewage is aerated in the presence of added sewage sludge produced in the process and a water-sol. comp'd. of a trivalent metal such as Fe and Al sulfates to coagulate putrescible matter in the sewage in the form of a settleable sludge, the sludge is sep'd. from associated liquor, and is used as an addn. to sewage to be subjected to the aeration. App. is described.

**Filtering sewage or other materials applied to vertical fibers of a brush mat filter.** Gilbert J. Fowler. U. S. 2,021,122, Nov. 12. Various details of app. and operation are described.

**Apparatus (with rotary drums) for incinerating sewage, etc.** Roger Granger. U. S. 2,020,652, Nov. 12. Structural, mech. and operative details.

## 15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

M. S. ANDERSON AND K. D. JACOB

**Ontario soils and farm chemical problems.** G. N. Ruhnke. *Can. Chem. Met.* 19, 287-90, 292 (1935).—A study of the different kinds of soils, the detn. of their dominant morphological and chem. characteristics, also their classification into soil types. Pasture problems, tobacco fertilization and animal diet are other problems for the agricultural chemist.

**Variations in soil reaction.** K. Nehring. *Z. Pflanzen-*

*ernähr. Düngung Bodenk.* 40, 137-41 (1935); cf. C. A. 29, 1558<sup>h</sup>.—Polemical against Fehr (C. A. 29, 6312<sup>h</sup>). Some of the seasonal changes in  $pH$  reported by F. are improbably large, e. g.,  $pH$  7.6 in summer and 4.6 in winter, in the case of a cultivated soil with 1.7%  $CaCO_3$ . Compared with this, the changes observed by N. were slight; records over a period of 3 years indicate that forest soils tend to become more acid in summer. C. J. S.

Plant associations as indicators of the value of sandy soils. D. Fehér. *Z. Pflanzenernähr., Düngung Bodenkd.* 40, 129-37 (1935).—Plant assocns on 28 sandy soils of the Hungarian plain and contents of citric acid sol.  $P_2O_5$  and  $K_2O$ , nitrate N and humus were not in general very closely correlated. The lab. examn of soil samples is still an indispensable supplement to ecological indications of a soil's adaptability to forestry. C. J. Schollenberger

The acidity index G. Deines and P. Kürbis. *Z. Pflanzenernähr., Düngung Bodenkd.* 40, 141-8 (1935).—Fehér's claim (C. A. 28, 5570) that the  $pH$  at any soil- $H_2O$  ratio can be calcd from a single detn at any other ratio is not justified, since it requires the assumption that soil acids dissociate in a simple manner, like common soil acids. It is possible to calc. the change in  $pH$  with variation in soil  $H_2O$  ratio (C. A. 27, 4332), best by a graphic method but approx. by formula, but the general course of the curve for the soil under investigation must first be ascertained. C. J. Schollenberger

Study of base-exchange power and base content of typical Connecticut Valley soils as influenced by heavy liming and fertilization. J. L. Haddock and H. H. Coyle, Jr. *Mass Agr. Expt. Sta., Bull.* 315, (Ann. Rept., 1934) 14 (1935).—In spite of heavy fertilizer and lime treatments the exchange bases in soils remain remarkably const., even on light, sandy types. The greatest influence on the availability of these bases seems to be the total exchange power of the soil, which is influenced by texture and soil humus. C. R. Fellers

Analysis of soils for copper. T. F. Manns and Raymond Russell. *Del. Agr. Expt. Sta., Bull.* 192 (Ann. Rept., 1934) 50-1 (1935).—Analysis of 25 soils for Cu by the Biazotti method (C. A. 29, 4092) showed only 4 exceeded 20 p.p.m. An application of  $CuSO_4$  of 50 lb. per acre on Del. soils increased the yields of corn and potatoes. C. R. Fellers

The manganese content of soils and its influence on the determination of  $pH$  by the quinhydrone electrode. L. Günter. *Landw. Jahrb. Schweiz* 49, 735-48 (1935).—The oxidizing action of Mn compds. is the cause of drift of potential in  $pH$  detns on soil suspensions. Some of the hydroquinone of the quinhydrone is oxidized to quinone, and the 1 to 1 ratio thereby altered. Consequently, potential readings should be made within a minute after the addn. of the quinhydrone, on soils strongly alk. and those low in humus, within a half minute. The addn. of  $Ca(OH)_2$  in quantity almost equiv. to the exchangeable Mn in a soil suspension causes changes in apparent  $pH$  analogous to those caused by the addn. of quinhydrone. E. O. W.

Nitrification studies with soil types of Northern Puerto Rico. Juan A. Bonnet. *J. Agr. Univ. Puerto Rico* 19, 73-103 (1935).— $(NH_4)_2SO_4$  should not be used as a source of N for plants in Lares clay loam, Sabana Seca clay, and Catalina clay, and in some cases Múrcia clay loam, unless special attention is given to lime broadcasting. For the most part, some nitrate is beneficial in these soils as well as in other types.  $Ca(NO_3)_2$  is the preferred form of nitrate.  $(NH_4)_2SO_4$  is well nitrified in Toa silt loam, Coto clay light texture phase, and in Espinosa clay, even in the absence of lime. Nitrification tests were conducted on 21 different soil samples representing 9 soil types. Partial chem. analyses of the soils are given. C. R. Fellers

Nitrogen fixation in soils. N. R. Dhar and S. K. Mukherji. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 4, 330-41 (1935).—When cane sugar was added to an unsterilized soil and exposed to light and air, the ammonia N increased from 0.00192 to 0.00437% in 13 days. Appreciable increases were also obtained on sterilized samples. The max. increase, 0.01444%, was reached in 28 days followed by a decrease to 0.00642% in 63 days at which time the trax amt. of nitrate N was found. The amt. of ammonia N found in samples exposed to light was always greater than in those kept in the dark. Proper aeration of soils increased the ammonia N content. Insufficiently aerated soils treated with molasses showed increases in acidity. In completely sterilized soils, the increase of ammonia N is due to photooxidation of the energy-rich compds. K. C. Beeson

Nitrogen fixation in soils on the application of molasses. N. R. Dhar, S. K. Mukherji, and P. K. Kar. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 4, 175-8 (1934).—A quantity of sieved soil was sterilized at  $150^\circ$  for 3 hrs. Two hundred and fifty-g. portions each of sterilized and unsterilized soil were mixed with pure cane sugar and 50 ml.  $H_2O$  and placed in shallow enameled dishes 10 in. in diam. Exposure to sunlight for 6 to 7 hrs. daily was allowed, and the ammonia N and nitrate N were detd. periodically. In the unsterilized soil an increase of ammonia N from 0.00092 g. to 0.0091 g. was found in 10 days when 20 g. of sugar was added. Ten g. of  $Na_2HPO_4$  in addn. to the sugar increased the ammonia N to 0.0140. No increase in nitrate N was found. At the end of 55 days only 0.00151 g. of ammonia N was found, and when  $Na_2HPO_4$  was added only 0.001008 g. of ammonia N was present. At this point the nitrate N had increased from 0.000728 to 0.00242 and 0.00176 g., resp. Much smaller increases in both forms of N were found in the sterilized soils. The ammonia N increases up to a limiting value until the nitrate N begins to increase, then the ammonia N as well as the sum of the 2 tends to decrease. The addn. of 35 kg. of molasses to 500 sq. ft. of soil caused a ten-fold increase in the ammonia N when the soil was properly aerated. K. C. Beeson

The effects of soil liming on the composition of sugar cane. G. Rodriguez. *Proc. Sugar cane Investigation Comm. (Trinidad)* 4, 389-94 (1934).—A study was made of the effects of lime (10 tons per acre) on the compn. of sugar cane grown on a heavy acidic clay. Liming caused a marked increase in mineral uptake, especially of phosphates, by the cane plant and significantly increased the total ash and P contents of the juice. Liming tended to decrease the sucrose and increase the glucose content of the juice. The relative amts. of phosphate fixed on the cellulose framework were significantly higher in the unlimed cane; Ca and Mg contents, considered together, showed pos. correlation with phosphate content. K was higher, but not significantly so, in limed cane. K. D. Jacob

Evaluation of limestone used as a [soil] supplement. L. Tschumi and J. Stalé. *Landw. Jahrb. Schweiz* 49, 726-34 (1935).—A method of evaluation of agricultural limestone consists of stirring mechanically for a half hour in 500 cc. 0.1 N disodium citrate soln. a quantity of the substance equiv. to 1.59 g.  $CaCO_3$ , and detg. the quantity of Ca dissolved. The soly. increases in inverse proportion to the diam. of the grains, showing a sharp increase for particles of a diam. less than 0.15 mm. It is therefore advantageous in practice to apply finely ground limestone. The soly. depends also on the geologic origin of the sample. Results obtained on dolomitic limestone must be corrected by multiplying by a factor, percentage of  $CaCO_3$  plus percentage of  $MgCO_3$ /percentage of  $CaCO_3$ . E. O. W.

Soil conditions determining response [of sugar cane] to fertilization with potash. P. E. Turner. *Proc. Sugar cane Investigation Comm. (Trinidad)* 4, 394-6 (1934); cf. C. A. 29, 1923\*.—Soils contg. less than 0.005-0.008% exchangeable  $K_2O$  gave significant responses to K fertilizers in the presence of 2-3 cwt./acre of  $(NH_4)_2SO_4$ . In lab. expts. with K-deficient soils which did not respond to K fertilizers, it was found that over 50% of the K applied as  $K_2SO_4$  was fixed in a comparatively unavailable form when the soil temp. was allowed to rise to  $40^\circ$  but practically no fixation occurred at  $30^\circ$ . It is not unusual in Trinidad for the temp. of the top in. of an exposed soil to rise to  $40^\circ$ . It therefore appears advisable to work K fertilizers below the surface of the soil. K. D. Jacob

Further research on permanent pastures as influenced by flood water containing potash waste liquor. Stahlin, Zapfe and E. Klapp. *Landw. Vers.-Sta.* 123, 237-75 (1935); cf. C. A. 24, 1456.—Analyses of flood waters, soils and crops gave results which agreed with previous evidence that soils and plants were not damaged by the constituents of flood waters of streams richest in waste liquor from the potash industry. John O. Hardesty

A growth and respiration factor for certain Rhizobia. Sam R. Hoover and Franklin E. Allison. *Trans. 3rd*

*Intern. Congr. Soil Sci., Oxford, 1935 1, 158-60(1935).*—The results of further studies on the "coenzyme R" (C. A. 27, 5345) found essential to the growth of *Rhizobia* of various strains are reported. It occurs not only in legumes, but in practically all vital materials examd.; yeast and liver are good sources. Addns. of adequate amts. to synthetic media increase the growth rate of *Rhizobia* 20-30 times; a great excess is not toxic. These effects are evident upon other soil bacteria also. Expts have not indicated that this growth factor has any function in N fixation. The effects upon bacterial respiration are remarkably rapid, a 2-5-fold increase in rate within an hr. The substance is produced in quantity by *Asiobacteria*, and can be extd. from the culture with acetone and recovered in aq. soln. Attempts at crystn. were unsuccessful. The impure prepn. at 0.4 p. m. produced half max. growth increase. The substance is dialyzable and resistant to hydrolysis in 25%  $H_2SO_4$  for 24 hrs. and in 0.1 N NaOH to 15 mins. autoclaving at 15 lb. It is insol. in ether,  $CHCl_3$ , and  $CaH_2$ . The isoelec. point is near  $pH_4$ , but the substance is not identical with the pantothenic acid of Williams and Saunders (C. A. 29, 2199) nor is the relationship to other growth factors settled. C. J. S.

*The microbiology of Australian soils. III. The Rosi-Cholodny method as a quantitative index of the growth of fungi in the soil, with some preliminary observations on the influence of organic matter on the soil microflora.* H. L. Jensen. *Proc. Linnean Soc. N. S. Wales* 60, 145-54 (1935).—The influence of different kinds of org. material, mostly hay meal, on the microorganisms of various soils was studied at temps. from about  $10^\circ$  to  $40^\circ$  and at varying degrees of moisture. Plate counts showed that the multiplication of bacteria was generally greatest at the lower temps., but the reverse was true of actinomycetes. The ratio of actinomycetes was narrowest at high temp. and low moisture. Fungi generally attained their most abundant vegetative development at  $16-21^\circ$ . Plate-counting, as applied to fungi, seems satisfactory only for the detn. of the no. of fungal spores in the soil. The figures for the density of mycelium, obtained by the microscopic method, as well as the plate-counted nos. of bacteria and actinomycetes, showed a correlation with the rate of  $CO_2$  production. J. R. Adams.

*Microbiological study of podzol soil profiles II. Laurentian soils.* P. H. H. Gray and C. B. Taylor. *Can. J. Research* 13C, 251-5(1935). cf. C. A. 30, 2085.—Microbiol. studies of samples from the sep. horizons of 2 soils from the Laurentian uplands showed that, as with soils previously studied from the Appalachian uplands, biol. activity was dependent upon the org.-matter relations of the horizons.  $A_1$  (org.-matter) horizons were the most active, as shown by the evolution of  $CO_2$ , nitrification of the soil's own N, by nos. of microorganisms (bacteria, actinomycetes and fungi) and by the production of  $NH_4$  from urea. Bacterial nos. in the illuvial horizons were, however, higher than those in the eluvial (leached) horizons above, a condition that did not obtain with the Appalachian soils. The org. matter of a soil bearing a coniferous (spruce) flora was deficient in nitrifying power. The biol. activity at different levels of a virgin clay soil was found to be in sharp contrast with that of the podzol soils. III. Bacteria found in separate horizons. P. H. H. Gray. *Ibid.* 250-62.—An investigation has been made to det. the presence of bacteria concerned in certain important biochem. reactions in the sep. horizons of 5 heavily leached (podzol) soils of Quebec. Cellulose-decomp. bacteria were represented by a species of *Micrococcus*, and by *Cytophaga hutchinsoni*. Organisms of the group *Mycobacteriaceae* were found in media devised to isolate bacteria able to decompose phenol and naphthalene. Of the N-fixing bacteria the aerobic organism *Asiobacteria* was absent, but the anaerobic organism, *Clostridium butyricum*, was present in all of the horizons. The decompn. of urea was due to Gram-negative spore forming rods whose systematic position has not been detd. J. W. Shipley.

[Report on] botany, forestry and pathology. [Determination of available potash in soils by the Aspergillus niger method]. H. L. Lyon. *Hawaiian Sugar Planters'*

*Assoc., Proc. 54th Ann. Meeting (Rept. Committee in Charge Expt. Sta.) 1934, 27-37.*—In samples of 14 soils the amts. of available  $K_2O$  as detd. by the 1% citric acid method and by the *Aspergillus niger* method were 0.019-0.047 and 0.020-0.044%, resp. The methods gave closely agreeing results on all samples. K. D. Jacob.

The relation between plant growth, soil and nutrient ratio in fertilization. I. K. Rackmann. *Z. Pflanzen-ernähr., Düngung Bodenkd.* 40, 148-78(1935).—Detailed data on yields and assimilation of nutrients by oats grown in pots of sand with systematic variations in fertilization with  $NH_4NO_3$ ,  $CaH_2(PO_4)_2$ ,  $KCl$  +  $K_2SO_4$  and a uniform addn. of salts with a soil suspension are reported. The effect of variation in  $CaCO_3$  supplied was also investigated. Conclusions: Uptake and action of one nutrient are largely dependent upon the abs. and relative amts. of others available. The nutrient requirements of plants change with growth. At first, N is in greatest demand, then  $K_2O$ , and  $P_2O_5$  at ripening, but all nutrients accelerate the rate of formation of org. matter.  $CaO$  is in greatest demand at a late stage, although its rate of uptake parallels the formation of org. matter. With a const. ratio, the proportional amts. of nutrients assimilated vary with the level of supply. At high level, relatively more of the limiting nutrient is absorbed. In the field, this is usually N. Lack of a single element diminishes uptake of others, so that demand for all is greater at a late stage of growth. At maturity, oats have the highest requirement for  $P_2O_5$  with 1.5-2 parts  $K_2O$  to 1 part N, and the highest requirement for  $K_2O$  when 0.75-1 part  $P_2O_5$  to 1 part N is supplied. At a normal level of fertilization, young plants produced most dry matter when  $N:P_2O_5:K_2O = 1:0.25:1$ ; at maturity, the highest yields of grain and of straw were produced when the ratios were 1:1:1.5 and 1:0.5:2, resp. The effects of lack of N and  $K_2O$  are evident in the first weeks of growth, lack of  $P_2O_5$  may be evident only from decreased yield of grain. Delay in supplying nutrients until the approach of maturity does not prevent their utilization, but the effect is to decrease the yield, especially of straw. Under these circumstances, N accumulates in the grain and  $K_2O$  in the straw. The rate and total uptake of nutrients is greater from an acid than from a neutral or alk. soil, with young plants, this is true even of  $CaO$ . The nutrient content of plants is not an indication of the supply in the soil. Soil  $pH$  increases with growth of the crop, but falls again as the crop matures, because of greater uptake of N at earlier stages and of  $CaO$  later. With a high level of  $CaCO_3$  supply, this effect may be obscured. In these pots of sand, the effects of fertilizer chemicals upon  $pH$  were marked. C. J. Schollenberger.

Origin of combined nitrogen in the atmosphere. The analysis of tropical rain and its importance in agriculture. A. Ram. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 4, 147-58(1934).—Rain water falling at Allahabad contains 0.10-1.450 p. p. m. ammonia N and 0.28-2.56 p. p. m. of nitrate N with an av. content of 0.469 and 0.881 p. p. m., resp. The ratio of nitrate N to ammonia N is 1.9. The chief source of ammonia N present in rain water seems to be the soil and the decompn. of org. matter on the surface of the soil, and very little of it seems to come from the sea. The high ratio of nitrate N to ammonia N appears to be due to the increased photo-oxidation of the  $NH_3$  present in the atm. and the action of ultraviolet rays coming from the sun. The max. amt. of nitrate N present in the atm. is found in the summer. The amt. of nitrate N has no connection with the incidence of thunderstorms. K. C. Beeson.

The effect of temperature on the bacterial ammonification of urea. S. P. Tandon. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 4, 169-72(1934).—Estimation of urea.—Urea was detd. in the presence of  $NH_3$  as follows: To a 5-ml. aliquot add a known vol. (excess) of a standard soln. of  $NaOBr$  (standardized against standard  $H_2AsO_4$  and I soln.). When the reaction has ceased add an excess of the standard  $H_2AsO_4$  soln. to neutralize the unused hypobromite. The excess of  $H_2AsO_4$  is titrated with the I soln. The  $NaOBr$  is a measure of the total urea and  $NH_3$ . Det. the  $NH_3$  present colorimetrically with

thunderstorms. K. C. Beeson.

Nessler's soln. Twenty g. of air-dried fresh soil was sieved and put into a 200-ml. Erlenmeyer flask with 80 ml. of a 2% urea soln. The flasks were plugged with cotton wool and maintained at a const. temp. Samples were withdrawn at intervals for the urea detn. The results indicate that the optimum temp. for bacterial ammonification of urea is about 40°. K. C. Beeson

Influence of lime on the availability of potash. H. C. Harris. Del. Agr. Expt. Sta., *Bull.* 192 (*Ann. Rept.* 1934) 18-21 (1935).—The Na cobaltumitrite method (C. A. 27, 2904) for the detn. of K was thoroughly satisfactory for soil K analysis. The Neubauer and PtCl<sub>4</sub> methods were also used as controls. Conclusion: If the Neubauer method and a detn. of the exchangeable K give an indication as to the availability of K in the soil, liming has had little influence in that respect. The continued use of K fertilizers and farm manure has not materially increased the replaceable K of this soil. C. R. Fellers

Effect of fertilizers and cropping upon the nature and amount of electrolysable bases in the soil with particular reference to potash. G. M. Gilligan. Del. Agr. Expt. Sta., *Bull.* 192 (*Ann. Rept.* 1934) 25 (1935); cf. C. A. 28, 6225<sup>1</sup>.—Preliminary findings show that liming increases the availability of K and P when electroanalysis is used as an index to availability. When base-exchange (leaching with neutral NH<sub>4</sub>OAc) is employed as an index to availability of K, the results are similar but of a lower order. This variation may be due to equal changes since the reaction with NH<sub>4</sub>OAc takes place at approx. *pH* 7.0 whereas the *pH* decreases as electroanalysis progresses. It is questionable whether or not electroanalysis is capable of giving a true index to the availability of K since it is well known that liming decreases the amt. of K in the soil soln. When subjected to electroanalysis the soils from the limed portions of the plots yield more K than the soil from unlimed portions. Without exception the soils from the limed plots yield the higher content of K. Limed soils yield a greater amt. of Mg, Mn and P than the unlimed soils of the same plots when subjected to electroanalysis. Of the elements added, P is the most readily reflected in the dialyzates. C. R. Fellers

Effect of fertilizer treatment on germination of sugar cane under field conditions. P. E. Turner. *Proc. Sugarcane Investigation Comm. (Trinidad)* 4, 404-16 (1934).—A dressing of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> applied at planting time had an adverse effect on the germination of sugar cane in certain expts. but not in others. The adverse effect tended to become more serious as the size of the dressing was increased. The magnitude of the effect was reduced by previous treatment of the soil with ground limestone. The beneficial effect of ground limestone on germination was independent of the size of the initial dressing of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> had no deleterious effect when it was applied 3 weeks after the cane was planted. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> nitrifies very rapidly in Trinidad soils and its deleterious effect on germination of cane setts may be due to the formation of a high concn. of H ions in the surrounding soil soln. Germination was also adversely affected by the application of either superphosphate or K<sub>2</sub>SO<sub>4</sub> at the time of planting. Previous treatment of the soil with ground limestone had a favorable effect on germination in most of the presence and absence of superphosphate and K<sub>2</sub>SO<sub>4</sub>. K. D. Jacob

Small scale fertilizing experiments by the Azotobacter method. A. Stockl. *Landw. Jahrb. Schweiz* 49, 697-725 (1935).—The Azotobacter method (C. A. 26, 6048), as a rapid method for detg. fertilizer requirements of soils, shows the greatest possible agreement of its results with those of field expts., is usable for most types of soils, and is easily carried out in a short time and at slight expense. While the Durks and Sheffer method (C. A. 28, 3164<sup>1</sup>) depends on the assumption that, with increasing percentages of available P<sub>2</sub>O<sub>5</sub> in the soil, the fertilizer requirement decreases, the Azotobacter method takes into account the soil reaction and the quantity of CaCO<sub>3</sub> present. The chief disadvantage of the Azotobacter method is that it indicates as slightly deficient some soils adequately supplied with P<sub>2</sub>O<sub>5</sub>. To carry it out carefully requires double

the time that the D and S. method requires, but much less time and expense than the Neubauer method requires. The Azotobacter method is not recommended for peat soils, nor for soils fertilized for many years with liquid manure. It does not indicate K<sub>2</sub>O requirements. E. O. W.

Studies on humus production from stall manure. Kurt Simon. *Z. Pflanzenernähr., Düngung Bodenk.* 40, 178-89 (1935); cf. C. A. 29, 888<sup>1</sup>.—A review and general discussion of the nature and production of humus and its functions in the soil, with a brief account of expts. with manure proving the formation of H<sub>2</sub>O-sol. humus substances by fermentation. Twenty-two references. C. J. S.

The influence of some potash fertilizers and associated salts upon the lime status of the soil. O. Lemmermann and L. Fresenius. *Z. Pflanzenernähr., Düngung Bodenk.* 40, 189-99 (1935).—Porcelain percolators were filled with 2.3 kg. sandy loam treated with various salts at the rate of 350 lb. per acre and leached with 100 ml. H<sub>2</sub>O daily until 12 l. had been collected. Ca was detd. in the percolate. The data indicated that Mg salts cause the greatest loss of Ca; K salts came next and Na salts caused the least loss. The sulfates are slightly more active than chlorides. Kamite, 40% K<sub>2</sub>O salt and sulfate of potash-magnesia to furnish equiv. K<sub>2</sub>O caused losses of Ca in proportion to Mg supplied; Na assoc. with Mg in these salts had little protective effect. C. J. Schollenberger

The influence of the [soil] reaction and fertilizing on the composition and digestibility of meadow grass. I. The influence of the reaction. K. Nehring. *Biedermanns Zentr. B. Tierernähr.* 7, 444-62 (1935).—In pot tests with timothy and red clover, besides an influence of the reaction on the yields, there was a change in compn., especially in relation to the content of crude protein and of the different mineral constituents (CaO and P<sub>2</sub>O<sub>5</sub>) which increased distinctly under the influence of increased lime adds. Corresponding field tests with timothy and red clover showed similar results. In 2 test series, lasting several years, the influence of the reaction on compn. and digestibility of natural meadow crops was studied. Addn. of lime effected a considerable yield increase and a distinct change in compn. As to crude nutrients, the addn. of lime increased the ash, crude fat and crude fiber, while there was a corresponding decrease in the amt. of N-free ext. The effect on the protein was not const.; in one series it was not essentially affected, while in the other it was favorably influenced. The content of CaO increased considerably; an increase in P<sub>2</sub>O<sub>5</sub> was not noticed in all cases. In the first series (on an acid soil of an elevated fen) digestibility was only slightly influenced. In the other series (on an exchange acid sand soil) digestibility was considerably increased by the addn. of lime, especially with physiologically acid fertilizing. Physiologically alk. fertilizing was able to reduce partly the unfavorable effect of the acid reaction, but not as to the yields. The addn. of lime effected the following av. increases in the yields per year per hectare: first expt., starch values 700, digestible crude protein 120 kg.; second expt., starch values 500, digestible crude protein 130 kg. F. L. Dunlap

[Report on] chemistry. P. E. Hance. Hawaiian Sugar Planters' Assoc., *Proc. 54th Ann. Meeting (Rept. Comm. in Charge Expt. Sta.)* 1934, 69-90; cf. C. A. 28, 6503<sup>1</sup>.—Growth-failure of cane.—A spectrographic study of a soil sample from a virgin portion of a growth failure area showed the absence of B and P. Cu was present in fair concn., together with Fe, Al, Ti, Ca, Na, Mg, Mn, Si and K. The soly. of native Fe and Mn in an alk. growth-failure soil was markedly increased by the application of a mixt. of equal parts of S and filter press cake at the rate of 2 tons/acre. The increases amounted to as much as 500 lb./acre-ft. of Fe as Fe<sub>2</sub>O<sub>3</sub> and 250 lb. Mn as MnO<sub>2</sub>. Inorg. constituents of water.—Distd. water from different stills varied widely in Cu content. Sea water was shown spectrographically to be less concd. than well water in Ti, Mn, Cu, Al and P. *Detn. of available Fe and Mn in Hawaiian soils*.—Procedures involving the use of NH<sub>4</sub>OAc and NH<sub>4</sub>Cl solns. were unsatisfactory. A procedure was developed involving the use of neutral N NH<sub>4</sub> citrate (prepd. by mixing equal vols. of 2

NH<sub>4</sub>OH and citric acid solns. and adjusting to neutrality with bromothymol blue) as an extg. medium. It possesses the advantage that the amts. of Fe and Mn extd. by neutral NH<sub>4</sub> citrate are of such magnitude as to make possible the detection of small fluctuations resulting from responses to soil treatments. *Arsenic in refined sugar*—Refined Hawaiian sugars did not contain As in amts. detectable by either the spectrographic or Gutzeit methods. The small quantity of As (2 p. p. m.) absorbed by the cane plant remained in the mill by-products as a result of refining operations. K. D. Jacob.

*Asparagus investigations* Robert E. Young. *Mays Agr. Expt. Sta., Bull. 315 (Ann. Rept. 1934) 62-4 (1935)*.—On *Merrimac* coarse sandy loam at N. Estham a 3-year field expt. showed that fertilizer contg. 8% N applied at the rate of 1 ton per acre gave max. results in the N series. NaNO<sub>3</sub> gave the best results of any N source. A fertilizer mat contg. 12% P<sub>2</sub>O<sub>5</sub>, where the P was derived from superphosphate, gave best results in the P series. Fertilizers contg. 6% K<sub>2</sub>O produced crop increases of 32% over the no-potash control plots. KCl was preferable to other K salts. The substitution of NaCl for KCl reduced yields. Even when NaCl was added to complete fertilizers, no yield increases were noted. N fertilizers should not be added all at once, but 1/2 should be applied at the end of the cutting season and 1/2 about a month later. Asparagus was greatly benefited by the addn. of lime to an acid soil. The resulting yield increase was 22%. Cyanamide, in both powd. and granular forms, was an excellent weed killer in asparagus fields. C. R. Fellers.

*Cane ecology* F. Hardy. *Proc. Sugarcane Investigation Comm. (Trinidad) 4, 352-3 (1934)*.—Cane plants were grown in quartz sand and watered with pure nutrient solns. Cultures lacking in either N or P produced crops weighing only about 5% of those receiving a complete nutrient soln. In the absence of K the crop was reduced approx. 50%. Growth was not affected by the absence of either Ca or Mg. The highest sugar content (percentage wt. of cane material) occurred in the culture lacking N, closely followed by that lacking P. The glucose ratio (glucose accompanying 100 parts of sucrose) was very much lower in the cultures lacking either N or P than in any of the others, but was relatively high in the culture lacking K. K. D. Jacob.

*The mineral nutrition of grape vines* E. Vinet. *Compt. rend. acad. agr. France 21, 911-10 (1935)*, cf. C. A. 29, 873<sup>1</sup>.—Fertilizers have a marked effect on the development of the wood, its mineral compn. and its productivity. Fertilizers increase the density of the wood and the best developed and densest woods are obtained in plots which receive the greatest amt. of K<sub>2</sub>O. In this regard K<sub>2</sub>SO<sub>4</sub> is not superior to KCl but it is superior in enriching the wood in K<sub>2</sub>O and increasing the sugar production. K<sub>2</sub>O fertilizers also improve the fruit-bearing aptitude of the vines. The absence of K<sub>2</sub>O in the fertilizers causes a physiol. unbalance which leads to poor development of the wood and smaller production. J. R. Adams.

Responses of the tomato in solution cultures with deficiencies and excesses of certain essential elements. Paul L. Fisher. *Md. Agr. Expt. Sta., Bull. 375, 283-98 (1935)*.—Tomato plants of the Marglobe variety were grown in solns. deficient in, and in solns. with excesses of, the following essential elements. B, Ca, Mg, K, N, P and S. Generally there was decreased growth of plants grown in solns. deficient in each of the elements. This same decrease was not produced in every case by an excess of these elements in solns. Plants can make relatively good growth in concns. double that of control cultures, with the exception of Ca, N and S. The distinctive symptoms of tomato plants grown in solns. deficient in each of the essential elements studied are very similar to those reported by McMurtrey (C. A. 27, 2712) for tobacco. In general, the effects were observed much earlier in tomatoes. Because of the ability of the tomato to stand high salt concns., no distinctive differences or responses were obtained as a result of growing tomato plants in excessive salt concns. of the elements studied. C. R. Fellers.

Rate of fertilizing cotton with and without poisoning

(from arsenic). R. Y. Bailey and J. M. Roberson. *Ala. Agr. Expt. Sta., 45th Ann. Rept. 13-14 (1934)*.—Four different rates of a 4:8:0 6:4:8 fertilizer made from NaNO<sub>3</sub>, superphosphate, and KCl were compared for cotton on Norfolk sandy loam over a 10-yr. period. The rates of application of the fertilizer varied from 500 to 2000 lb. per acre per year. Some of the plots were dusted annually with Ca arsenate to control boll weevil while other plots were left as controls. The As dust gave an av. increase in cotton yield per acre of 40 lb. per year on the unfertilized plots and of 203 lb. on the fertilized plots. There was no evidence of poisoning or incompatibility by the fertilizer ingredients or Ca arsenate. C. R. Fellers.

*Fertilizer requirements for Irish potatoes, wheat and soybeans when grown in rotation* C. B. Williams, W. H. Rankin and S. C. Clapp. *N. C. Agr. Expt. Sta., 46th Ann. Rept. 18-19 (1933)*.—Larger yields of soybean hay were produced from 400 lb. of 12-24 fertilizer contg. K from low-grade potash salts like kainite than from a complete fertilizer of the same analysis contg. equiv. amts. of K from either KCl or K<sub>2</sub>SO<sub>4</sub>. P influenced the crop most, and N least. For potatoes, K<sub>2</sub>SO<sub>4</sub> proved a better source of K than kainite or KCl. Scabby tubers resulted from potatoes fertilized with kainite. C. R. Fellers.

*Fertilizer and lime requirements for tobacco grown in rotation with oats, soybeans and rye* E. G. Moses. *N. C. Agr. Expt. Sta., 46th Ann. Rept. 21 (1933)*.—Long-time expts. on plots showed that cottonseed meal and NaNO<sub>3</sub> ranked 1st and 2nd as sources of N for tobacco. KCl was preferred to K<sub>2</sub>SO<sub>4</sub>. Bone meal and basic slag as sources of P injured the quality of the tobacco and delayed maturity. C. R. Fellers.

*The question of the fertilizing action of brown coal* H. Kappen, R. W. Beling and G. v. Strinck. *Z. Pflanzen-ernähr., Düngung Bodenk. 40, 215-28 (1935)*.—Pot expts. with mustard on sandy loam, comparing urea, Leunassal-peter (NH<sub>4</sub> sulfate-nitrate) and 3 ammoniated brown coal preps. contg. 1.0, 4.7 and 3.5% N from NH<sub>3</sub> as sources of N in a complete fertilizer, are described. The results indicated that the value of these preps. was solely in the NH<sub>4</sub> salt content; neither the N nor the org. matter of the coal had any effect. Treatment of coal with gaseous or liquid NH<sub>3</sub> causes some combination of N in unavailable forms, hence it is not economical. The products made from coal and dil. NH<sub>4</sub>OH or NH<sub>3</sub> salt solns. contain the added N in available form, but carry so little that they are at a disadvantage in comparison with other N fertilizers. C. J. Schollenberger.

*Nonacid-forming mixed fertilizers* II. The value of dolomitic limestone supplements of different degrees of fineness as measured by the increase in water-soluble magnesium in the soil. J. R. Taylor, Jr., and W. H. Pierre. *J. Am. Soc. Agron. 27, 764-73 (1935)*; cf. C. A. 29, 6904<sup>1</sup>.—After a period of 24 days, tests on a DeKalb silt loam soil of pH 5.4 showed that the finer than 100-mesh dolomitic limestone in a mixed fertilizer liberated in the soil-fertilizer zone practically as much Mg as did pure MgSO<sub>4</sub> contg. an equiv. amt. of Mg. As measured by the concn. of sol. Mg, the finer the division of dolomitic limestone used in the production of nonacid-forming fertilizers, the greater the rate of decompn. in the soil-fertilizer zone. To obtain any benefit during the year in which it is applied, the dolomitic limestone should all pass the 20-mesh sieve and a large part should pass the 60-mesh sieve. The rate of decompn. of W. Va., Ohio, Tenn., and Ala. dolomitic limestones as indicated by the concn. of Mg, nitrate N and H ions in the soil-fertilizer zone was practically the same. It appears that in many cases dolomitic limestone used in this manner should supply a large part, if not all, of the Mg needs of plants, even when grown on Mg-deficient soils. J. R. Adams.

*The determination of phosphoric acid in Nitrophoska containing lime* W. Lepper. *Landw. Vers.-Sta. 123, 345-8 (1935)*.—The shortened method of Petermann, as described previously (C. A. 29, 2111<sup>1</sup>), compares favorably with other methods for the detn. of citrate-sol. P<sub>2</sub>O<sub>5</sub> in Nitrophoska. John O. Hardesty.

Rock phosphate, superphosphate, soft phosphate and

basic slag compared as sources of phosphoric acid for corn, wheat and soybeans grown in rotation. C. B. Williams, W. H. Rankin and S. C. Clapp. *N. Car. Agr. Expt. Sta., 46th Ann. Rept.* 19-20(1933).—Superphosphate produced larger yields than other forms of P in a 5-yr. expt.

Improving superphosphate. P. A. Baranov. *J. Chem. Ind. (Moscow)* 12, 832-5(1933).—The addition of 5-10% of bone meal to superphosphate greatly improves the physical properties and fertilizing power of the product. Addition of phosphorite meal is also helpful, but less so than the addition of bone meal.

Absorption by food plants of chemical elements of importance in human nutrition. A. B. Beaumont and E. B. Holland. *Mass Agr. Expt. Sta., Bull.* 315, (Ann. Rept. 1934), 14-15(1935).—By use of a fine sandy loam at pH 4.4, toxicity symptoms appeared when  $\text{FeSO}_4$  was added at the rate of 1280 lb per acre for spinach, and at 640 lb per acre for turnips. For  $\text{Fe}_2(\text{SO}_4)_3$ , the toxic contents were, resp., 640 and 320 lb., and for  $\text{CuSO}_4$ , 320 and 160 lb. At a soil pH of 5.4,  $\text{FeSO}_4$  became toxic for spinach at contents of 5120 lb per acre, and for turnips at no content. Using  $\text{Fe}_2(\text{SO}_4)_3$  became toxic for spinach at 2560 lb.;  $\text{CuSO}_4$  was toxic for spinach and turnips at a content of 1280 lb. In soil-tank expts., the use of 16-24 kg per hectare of KI increased the yield of turnips but not of spinach.

Control of the bacterial wilt disease of tobacco, pepper and Irish potato. R. F. Poole. *N. Car. Agr. Expt. Sta., 46th Ann. Rept.* 24-5(1933); cf. C. A. 28, 6918<sup>18</sup>.—The addition of 200-600 lb. of S per acre for the control of bacteria wilt reduced the soil pH from 5.6 to 3.2-4.5 and greatly injured the plants. Even after  $\text{Ca}(\text{OH})_2$  or dolomitic lime was added to the soils before replanting, the injury still persisted. Conclusion: The use of S as a control has little practical value.

[Report on] entomology. C. E. Pemberton. Hawaiian Sugar Planters' Assoc., *Proc. 54th Ann. Meeting (Rept. Committee in Charge Expt. Sta.)* 1934, 19-26, cf. C. A. 28, 6514<sup>18</sup>.—Good control of the cane root grub (*Anomala orientalis*) was obtained by applying to the soil a mixt. of mud press cake and  $\text{As}_2\text{O}_3$ . Neg. results were obtained with  $\text{As}_2\text{O}_3$  incorporated in Oliver filter mud and spread over the fields in irrigation water; the As was fixed at the surface of the soil and was almost completely retained there for at least 1.5 yrs. Cane plants did not absorb As from the soil except in exceedingly minute amounts, which were taken up even where no As was applied. Canes from untreated fields, as well as those from fields treated with  $\text{As}_2\text{O}_3$  at the rate of 1890 lb/acre (worked into the soil), contained 2 p.p.m. of As on the dry basis. K. D. J.

The coding moth problem. S. L. Allman. *Agr. Gas. N. S. Wales* 46, 459-63(1935).—Addn. of white oil (1 gal/80 gal of spray) to the last 3 cover sprays of Pb arsenate gave effective control of the insect. Decidedly poorer results were obtained when either kerosene, casein or fish oil was substituted for white oil. Of the nonarsenicals tested, nicotine sulfate plus white oil was the most effective but was decidedly inferior to Pb arsenate. Poorer results were obtained with erythrite sprays. The highest spray residue (0.0114 grain  $\text{As}_2\text{O}_3$  per lb of fruit) was obtained with white oil in the last 3 cover sprays. The lowest residue (0.0076 grain/lb.) occurred when nicotine sulfate was substituted for white oil. The Pb arsenate-fish oil spray caused a very heavy fruit drop. K. D. Jacob.

Studies on brown root rot of cereals. III. Phosphorus nitrogen relations of infested fields. IV. Effects of fertilizer amendments. V. Preliminary plant analyses. T. C. Vanterpool. *Can. J. Research* 13C, 220-60(1935).—These papers deal with the elucidation of the factors which predispose wheat seedlings following summer fallow to *Pythium* root rot in certain parts of Sask. The chem. soil analyses on 63 sample pairs reveal that available P in the soil is lower and nitrate N higher in brownish patches than in normal patches. Greenhouse expts. and field observations show that phosphate amendments at the time of seeding, and farmyard manuring, permit the development of normal plants in brownish soil; cereal

straw applications inhibit the disease to some extent, while inorg. nitrogenous fertilizers usually have no effect or occasionally may prove detrimental. Preliminary plant analyses indicate that the total P in diseased plants is lower than in healthy ones, and that there is relatively more water-sol. N and relatively less protein N in diseased than in healthy plants. The soil and crop-culture factors, especially the practice of bare summer fallowing, which may influence the available P-nitrate N ratio in the soil, are discussed in relation to disease incidence. It appears that any crop practice which tends to increase the available P-nitrate ratio in the soil inhibits the disease, and conversely. This is used as a basis for control recommendations. It is considered that an improper balance of available P and nitrate N in the soil leads to an unbalanced metabolism of the wheat seedlings at a crit. stage in their development, thereby predisposing them to fungal attack. The effect of the unbalanced nutrients on the parasitic vigor of the pathogen still requires elucidation.

J. W. Shipley. Influence of certain dusts and sprays upon the growth, yield, quality and general characteristics of peanuts. H. B. Mann. *N. Car. Agr. Expt. Sta., 46th Ann. Rept.* 41-3(1933).—Pot expts. showed that the addition of S to the soil at the rate of 94 lb per acre made the foliage a bright green color and hastened maturity in peanuts. The S increased the soil acidity an av. of 1.4 pH.  $\text{CaSO}_4$  increased the soil acidity only 0.5 pH.  $\text{CaCO}_3$  decreased it by 1.1 pH. The rate of application of the  $\text{CaSO}_4$  and  $\text{CaCO}_3$  was 400 lb. per acre. S was decidedly injurious to the foliage and to yield.  $\text{CaSO}_4$  and Bordeaux mixt. were ineffective.  $\text{CaCO}_3$  gave greatly increased nodulation.  $\text{CaSO}_4$  decreased nodulation.

C. R. Fellers. Investigations on derris preparations. II. Evaluation of derris root and stability of rotenone in commercial preparations. 1. Evaluation. P. W. Danckwort and G. Baumgarten. *Arch. Pharm.* 273, 385-9(1935), cf. C. A. 28, 4836<sup>18</sup>.—This is a criticism of methods at present in vogue, notably those based on EtO extn. 2. Stability of rotenone preparations. P. W. Danckwort and H. Patzsch. *Ibid.* 387-8. Results obtained in a series of stability tests indicate that the rotenone content over a period of 4 months is slightly lowered. With old preps even in nonalk. solns some loss of rotenone may be expected.

W. O. E. Changes in apples during storage (Rasmussen) 12. Sewage sludge as a fertilizer (De Turk) 14. Public health significance of sewage sludge when used as a fertilizer (Tanner) 14. Iodine in Westphalia (detn. in soils) (Balks) 7. Some African oil seeds [used as fertilizer] (Anon.) 27. Pure water and sewage [fertilization] (Gusovius) 14. Testing rind hardness in sugar cane [and cohesion and penetrability of soils] 28. Dispersions such as those of insecticides (U. S. pat. 2,021,143) 13.

Fertilizer. Charles K. Lawrence (to Atmospheric Nitrogen Corp.) U. S. 2,010,713, Nov. 5,  $\text{NH}_4\text{NO}_3$  45-60 is used with ammoniated triple superphosphate 55-40 parts. Cf. C. A. 29, 4125<sup>18</sup>.

Phosphate fertilizers. Hoersch-Köln-Neussens A.-G. für Bergbau und Hüttenbetrieb (Friedrich Heinrich, inventor). Ger. 616,940, Aug. 15, 1935 (Cl. 16 2). A continuous wet process for producing  $\text{CaH}_2(\text{PO}_4)_2$  consists in: (a) treating crude natural or synthetic phosphate with HCl to obtain  $\text{CaH}_2(\text{PO}_4)_2$  or free  $\text{H}_3\text{PO}_4$ , and passing in  $\text{NH}_3$  to ppt.  $\text{CaH}_2(\text{PO}_4)_2$ ; (b) heating the  $\text{CaH}_2\text{-NH}_4\text{Cl}$  soln. of (a) with  $\text{CaO}$  to liberate  $\text{NH}_3$ , which is dried ready for re-use in stage (a); the  $\text{CaH}_2$  lye of (b) is freed from excess of  $\text{Ca}(\text{OH})_2$ , evapd. and heated to 750° with steam to generate HCl lye, used in stage (a) and the residual  $\text{CaO}$  is re-used in stage (b). Examples are given.

Fertilizers. Lonza Elektrizitätswerke und chem. Fab. A.-G. (Fmil. Lüscher and Ernst Sturmann, inventors). Ger. 618,791, Sept. 16, 1935 (Cl. 16 5). Addn. to 598,446 (C. A. 28, 5590<sup>18</sup>). Known fertilizers comprising  $\text{Ca}(\text{NO}_3)_2$  and soil phosphates are warned and mixed with a basic substance which has been produced in a finely sub-

divided state by chem. reaction, e. g., pptd.  $\text{CaCO}_3$  or slaked lime. Strewable compns. stable to storage are obtained.

**Fertilizers.** *Gewerkschaft Victor and Wintershall A.-G.* (H. Schmalfeldt, inventor). *Ger.* 619,397, Oct. 2, 1935 (Cl. 16.4). Products contg.  $\text{KH}_2\text{PO}_4$  and  $\text{MgH}_2(\text{PO}_4)_2$  are prep'd. by heating mixts. of  $\text{KCl}$  and  $\text{MgCl}_2$ , e. g., natural or synthetic carnallite, to about  $180^\circ$  with the appropriate amt. of  $\text{H}_3\text{PO}_4$  soln. of 30–40% concn. Traces of  $\text{HCl}$  in the products may be neutralized by treatment with  $\text{NH}_3$  or addn. of a solid alkali.

**Mixed fertilizer.** *Hubert Kappen.* *Ger.* 616,947, Aug. 8, 1935 (Cl. 16.6). Solid  $\text{Mg}$  salts contg.  $\text{K}$  salts are dampened and treated with excess of  $\text{NH}_3$  and  $\text{CO}_2$  or gas mixts. contg. these. The product is powd., exposed to air, warmed and retreated with  $\text{NH}_3$  and  $\text{CO}_2$  to give a product rich in ammonia  $\text{N}$ . *Cl. C. A.* 29, 3102<sup>1</sup>.

**Peat-containing fertilizer.** *Werner Liehr and Eduard Dyckerhoff* (to *Lduard Dyckerhoff*). *U. S.* 2,019,824, Nov. 5. A mixt. including dry peat and plant fertilizing material such as an aq. fertilizer soln. is subjected to a pressure of not less than 130 kg. per sq. cm. and sufficiently high to cause a colloidal change in the peat without the application of external heat. The product may be formed into pellets.

**Nitrogen fertilization of soil.** *Cornelis B. de Bruyn* (to *Shell Development Co.*) *U. S.* 2,020,824, Nov. 12.  $\text{NH}_3$  gas is passed into irrigation water to produce a concn. of  $\text{NH}_3$  in the water which will avoid burning of vegetation, and the treated water is supplied to the soil.

**Ammonium sulfate.** *Andreas v. Kreisler.* *U. S.* 2,021,093, Nov. 12. For the production of coarse cryst.  $(\text{NH}_4)_2\text{SO}_4$  from synthetic  $\text{NH}_3$ , the sulfate is crystd. from its aq. soln. with addn. of 2–6 parts carbamide per 100 parts of  $\text{NH}_4$  sulfate. The cryst. product may contain about 0.1–0.5% of carbamide and is suitable for use as a fertilizer.

**Superphosphates.** *Lewis B. Skinner.* *U. S.* 2,021,–

671, Nov. 19. Coarsely comminuted calcareous material such as crushed limestone is subjected to attrition (suitably in a rotary pebble mill or the like, of a described app.) in the presence of  $\text{H}_3\text{PO}_4$  employed in an amt. required to convert the  $\text{Ca}$  content of the material substantially all into mono- $\text{Ca}$  phosphate as a substantially dry product at the end of the reaction.

**Insecticides.** *I. G. Farbenindustrie A.-G.* *Brit.* 432,–188, July 23, 1935. See *Fr.* 754,735 (*C. A.* 28, 1459<sup>4</sup>).

**Insecticides.** *Chemische Fabrik Marienfelde G. m. b. H.* *Brit.* 432,626, July 30, 1935. Concd. derris root and other exts. emulsifiable with  $\text{H}_2\text{O}$  are obtained by adding weakly acid alkali-resin soaps to derris root and other exts. obtained by means of and contg. di- or tri-chloro-ethylene.

**Insecticides, etc.** *Soc. anon. des établissements Nifo.* *Fr.* 785,049, July 31, 1935.  $\text{AcH}$  in various forms is used as insecticide, bactericide, fungicide, etc.

**Germicide, insecticide and fungicide.** *Julius Hyman* (to *Velsicol Corp.*). *U. S.* 2,020,648, Nov. 12. A fraction of a polymer derived from vapor-phase cracked gasoline and boiling in the kerosene to gas-oil range is subjected to mol.  $\text{O}$  oxidation in the presence of a seccative metallic soap such as  $\text{Mn}$  linoleate or soap-forming compd. of a seccative metal such as  $\text{Co}$ ,  $\text{Pb}$  or  $\text{Mn}$  until pptn. substantially ceases, the ppt is sepd. and the filtrate is recovered.

**Destroying parasites.** *Gesellschaft für Neuzeitliche Bodenbehandlung m. b. H.* *Fr.* 785,481, Aug. 10, 1935. Vegetables, leaves, etc., are sprinkled with finely powd. siliceous mineral substances having absorbent properties, e. g., concreted quartz, flint, quartz sand or firestone having a high content of amorphous silicic acid.

**Weed killers.** *Fahlberg-List A.-G.* *Chem. Fab.* *Ger.* 619,237, Sept. 25, 1935 (*Cl.* 45.5). Partly dehydrated  $\text{CuSO}_4$  is mixed with the double salt  $\text{ZnCl}_2 \cdot 2\text{NH}_4\text{Cl}$  or with a mixt. of the double salt and free  $\text{ZnCl}_2$ . An acid salt, e. g.,  $\text{NaHSO}_4$ , may also be added. The products are stable to storage.

## 16—THE FERMENTATION INDUSTRIES

C. N. FREY

**Double-effect distillation of alcohol under vacuum.** *E. Barbet.* *Intern. Sugar J.* 37, 434–5 (1935).—The merits of the vacuum system of distn. are discussed. It not only economizes in steam but gives a definitely purer product contg. much smaller quantities of volatile acids, aldehydes, ethers and higher alcs.

**Alcoholic fermentation of saccharine materials.** *D. Sidersky.* *Bull. assoc. chim.* 52, 753–9 (1935).—The max. theoretical yield of alc., 64.33 l. from 1 kg. of sucrose, is never reached in practice, but 60.1 l. has been obtained by S. in the fermentation of beet diffusion juice. The alc. usually lost by entrainment in the  $\text{CO}_2$  gas may be recovered by passing the gas through activated  $\text{C}$  and releasing the absorbed alc. by steaming the  $\text{C}$ . The yield of alc. has been increased 0.7% by this measure.

**Scientific aspects of cacao fermentation.** *A. W. Knapp.* *Bull. Imp. Inst.* 33, 31–49, 147–61, 306–19 (1935).—A review with 49 references. *A. P.-C.*

**The rum industry of Martinique.** *J. Guillaume.* *Ann. zymologie* [2], 2, 125–32 (1935). *A. P.-C.*

**Barleys of 1935.** *J. Raux.* *Brasserie & malterie* 25, 257–66 (1935); cf. *C. A.* 29, 8224<sup>1</sup>.—Av. compns. of barleys from different regions of France are tabulated and discussed. *A. Papineau-Couture.*

**Spirituous liquors from grains: gin.** *E. Dorchie.* *Ann. fals.* 28, 452–6 (1935).—A description of the manuf. and compn. of gin, with analyses of a no. of com. samples of known genuineness. Considerable variations occur in the products of different distilleries, but the variations are much narrower with different samples from the same distillery. Constituents other than  $\text{EtOH}$  never fall below 322 mg. per 100 cc. of abs. alc.; higher alcs., the most important of these constituents, do not fall below 274

mg. per 100 cc. of abs. alc. furfural was present in undeterminable traces in all samples except those from one distillery which yielded a product contg. 1.0–9.6 mg. per 100 cc. abs. alc. Artificial gins are low in non- $\text{EtOH}$  constituents and contain not more than traces of higher alcs.

*A. Papineau-Couture.*

**Wines of the 1934 vintage in the Champagne area.** *Lebrun and Radet.* *Ann. fals.* 28, 465–77 (1935).—Analyses of 65 wines are tabulated and discussed. *A. Papineau-Couture.*

**The preparation and the determination of trehalose in yeast.** *A. Steiner and C. F. Cori.* *Science* 82, 422–3 (1935).—Treatment of starch-free bakers' yeast with  $(\text{NH}_4)_2\text{SO}_4$  followed by pptn. with  $\text{H}_2\text{SO}_4$  +  $\text{Fe}_2(\text{SO}_4)_3$  in 7.5%  $\text{H}_2\text{SO}_4$  exts. the trehalose (I) completely. Neutralization with  $\text{BaCO}_3$  ppts. the small amt. of polysaccharide present while I remains in soln. The filtrate is freed of  $\text{Ba}$  and heavy metals and concd. *in vacuo*. Addn. of 20 vols. of alc. ppts. some salts, which are filtered off. The soln. is placed in the refrigerator. After standing overnight, or in a few days, the typical rhombic crystals of I are formed, which grow considerably in size in the next 10 days. During glucose fermentation the I content of yeast increases markedly. In one prepn. in which 300 g. of yeast was allowed to ferment 150 g. of glucose, 1.5 g. of I was obtained in the 1st crystn. and 0.6 g. by working up the mother liquors. Directions are given for the detn. of I. Fresh bakers' yeast contains 0.1–1.5 g. I per 100 g. moist wt., the amt. depending upon the medium upon which the yeast was grown. *Felix Saunders.*

**Amber sorghum from Minnesota as a source of alc.** (Grossi) 28. Sterilizing liquids [wines and beer] (*Fr. pat.* 784,959) 13.



Alcohol. *Aktiebolslaget Separator* and Nils E. Svensson. Brit. 432,387, July 23, 1935. In processes for producing alc. from sugar-contg liquids produced by extrn. from sugar beets, etc., the yeast, after fermentation in vats, is extrd. by means of centrifuges having a conveying device or pile of disks arranged within the bowl and a sludge space outside the conveying device or pile of disks, the septed yeast being discharged to a point immediately outside said device, etc., or, when the outer periphery of said device, etc., is at a smaller distance from the center than 7/10ths of the inner radius of the bowl, to a point at least 7/10ths of this radius from the center. The yeast is then subjected to a further fermentation in a vat contg nonfermented sugar-contg liquid. App is described.

Alcohols from sugars. I. G. Farbermand A-G (Johannes Muller and Ulrich Hoffmann, inventors). Ger. 534,074, May 18, 1935 (Cl. 12a 5 03). Addn to 544,606 (C. A. 26, 3514). The method of 544,606 for obtaining alcs by reducing sugars with H in the presence of an activated Ni or Cu catalyst is extended to include the use of nonactivated catalysts, OH ions being present in the latter case. In example, unactivated Ni is used with  $\text{Ca}(\text{OH})_2$ ,  $\text{Ba}(\text{OH})_2$ , and  $\text{MgSiO}_3$ .

Propionic acid production by fermentation. Hugh R. Stiles (to Commercial Solvents Corp.). U. S. 2,020,251,

Nov. 5. Fermentation, such as that of molasses or corn mash, is conducted in the presence of a bentonite clay or hydrous Al oxide or activated alumina which serve as catalysts.

Use of sucrose octa-acetate as a denaturant for alcohols. Herbert G. Stone (to Eastman Kodak Co.). U. S. 2,019,744, Nov. 5.

Fermentation of sugar-cane juice. Albert Lepelletier-Baufond. Fr. 745,221, Aug. 5, 1935. The juice is purified by bringing its  $\text{pH}$  value successively to the isoelec. points of the org. and mineral impurities and is then seeded with a suitable yeast, with the addn. of yeast nutrients if necessary. A wine like product is obtained.

Treatment of wine, must, etc. Edvard Muller. Ger. 619,191, Sept. 23, 1935 (Cl. 6c. 1). The acidity of wine, must and other fruit juice preps. is reduced by electrolyzing the materials in a partitioned cell. Material of reduced acidity is withdrawn from the cathode compartment. Exptl. results are given.

Temperature-controlling means for malt kilns and like drying apparatus. Heinrich Muger. Ger. 619,384, Oct. 2, 1935 (Cl. 82a 26).

Apparatus for hopping beer wort. Weigelwerk A-G. Ger. 617,842, Aug. 23, 1935 (Cl. 6b. 14). Addn. to 602,371 (C. A. 29, 544).

## 17—PHARMACEUTICALS, COSMETICS AND PERFUMES

W O EMERY

Determination in the presence of tertiary alcohols of the free primary and secondary alcohol contents of essential oils by acetylation in pyridine. Raymond Delaby and Sebastien Sabets. *Bull. soc. chim.* [5], 2, 1710-24 (1935).—Acetylation of primary and secondary alcs. present in essential oils contg. tertiary alcs. by means of  $\text{Ac}_2\text{O}$  and pyridine gives results comparable to those furnished by phthalization. The procedure is rapid and economical. Results, satisfactory from a practical standpoint, are obtained in 1-2 hrs. Phenols and primary alcs. and amines are quantitatively acetylated in 0.5-1 hr.; secondary alcs. are almost totally acetylated in 1 hr. while tertiary alcs. and aldehydes are scarcely affected. Pyridine acts as a neg. catalyst which paralyzes the acetylation of tertiary alcs. by  $\text{Ac}_2\text{O}$  whereas  $\text{P}_2\text{O}_5$  acts as a pos. catalytic agent. Tech. details as to app., materials, technique and calcn. are given. The acetylating mixt. consists of 1 part of  $\text{Ac}_2\text{O}$  with 2 parts of anhyd. pyridine dried over and distd. from  $\text{Ba}(\text{OH})_2$ . It is found essential to use an excess, 2-3 times the theoretical amt. of the acetylating mixt. This method gives 99, 98.7 and 99.3% results with  $\text{PhCH}_2\text{OH}$ , geraniol and l-citronellol. The accuracy of this method shown for acyclic terpenes has also been demonstrated for santalols (C. A. 26, 4727). The method gives 97, 96 and 93% results with the secondary alcs. menthol, borneol and cyclohexanol but only 0, 1, 2.3 and 2.2% with the tertiary alcs. MePr(PhCH<sub>2</sub>)COH, linalol, terpineol and MeEt(PhCH<sub>2</sub>)COH and 4.7, 5.7, 7.5 and 0% with the aldehydes citronellal, citral, hydroxycitronellal and Balil. The primary amine Me anthranilate was acetylated 99.6%. Tests made on various essential oils were sensibly in agreement with results found by the Radcliffe-Chadderton phthalization technique. The method outlined is more selective than the pyridine- $\text{AcCl}$  method published by Smith and Bryant (C. A. 29, 1749).

Azochloramide. Anon. *J. Am. Med. Assoc.* 105, 1191(1935).—The following dosage form has been accepted for admission to New and Non-Official Remedies by the Council on Pharmacy and Chemistry of the Am. Med. Assoc. *Azochloramide in Triacetin* 1.500—A soln. contg. 1 g. of azochloramide in triacetin, a bitter colorless oily liquid, b. 238-9°, has  $d_4^{20}$  1.154-1.158°,  $n_D^{20}$  1.4205-1.4310, sapon value 70-72 and contains approx. 95%  $(\text{ClH}_2\text{OAc})_2\text{CHClOAc}$ . Tests for  $\text{pH}$  and  $\text{H}_2\text{O}$  content are also given. C. R. Addinall.

Cetyl alcohol—the universal cream base. Joseph

Kahsh. *Drug Cosmetic Ind.* 37, 595-6, 598(1935).—The properties and uses with formulas are given. H. M. B.

Use of the methyloones in perfumery. Paul Jellinek. *Rieschhoff Ind. u. Kosmetik* 10, 177(1935).—A discussion with a table showing the amts. of the 4 methyloones necessary to produce the various odor types. H. M. B.

New procedures in the chemistry of aromatics. A. Lewinson. *Rieschhoff Ind. u. Kosmetik* 10, 187-9(1935); cf. C. A. 30, 2357.—A review dealing with the ketones of value in perfumery. H. M. Burlage.

Alkaloids of *Cytisus caucasicus*. A. P. Orshov, S. S. Norkina and T. Makomova. *Arch. Pharm.* 273, 319-72 (1935).—Leaves collected in the summer of 1934 yielded from  $\text{Et}_2\text{O}$  extrn. an alkaloidal mixt. (0.4%), from which a liquid base was isolated as the perchlorate m. 210-11° and having  $[\alpha]_D^{20}$  50°, probably identical with lupanine whose perchlorate m. 210° and has  $[\alpha]_D^{20}$  61°. Comparison of other salts as the hydriodide, methiodide and chloroplatinate gave addnl. confirmation. From the mother liquors of the lupanine perchlorate a beautifully crystalline salt was isolated having the formula  $\text{C}_{24}\text{H}_{39}\text{N}_2 \cdot \text{C}_2\text{H}_5\text{H}_2\text{O}_7$  (OH)(NO<sub>3</sub>)<sub>2</sub> and m. 204-5°, which identifies the salt with pachycarpine picrate. From the  $\text{CHCl}_3$  extrn. a min. amt. of long cryst. needles, m. 120-2°, was obtained, too small, however, for further investigation. W. O. E.

Estimation of essential oils in drugs, and the oil content of peppermint, sage, fennel and caraway. L. Koffer and G. v. Herrenschwand. *Arch. Pharm.* 273, 348-402 (1935).—A simple procedure is described, according to which the essential oil is driven over and detd. volumetrically. For reception of the distillate a 60-cc. flat-bottom flask contg. 20 g. NaCl is employed, on the neck of which a hood-like attachment is placed at the end of distn., this attachment develops into a narrow tube graduated into 0.01 cc. The lower side of the flask carries a wide tube closed by a rubber stopper, which on pushing in drives the NaCl soln. into the graduated column for subsequent reading. The entire operation requires only 1 hr. This receiving and measuring app. presupposes a distn. app. of the type suggested by previous investigators. It requires not more than 60 cc. distillate for exhaustion of the drug. With certain drugs like peppermint and sage the yield of oil is less from the powder than the whole drug. Most of the com. samples of the 4 previously noted drugs in powder form proved on examn. to be inferior or worthless. W. O. E.

Behavior of drugs of high oxidation potential. P. W.

Danckwort, W. Dietrich and G. Wilkens. *Arch. Pharm.* 273, 403-8(1935).—Some 49 drugs have been examined, the results obtained are comparatively reported. W. O. E.

Adsorption of nicotine from tobacco smoke by different adsorbents. J. Bodnár, Vitéz Ladi-laus Nagy and Tibor Vecsey. *Pharm. Zentralhalle* 76, 657-61(1935).—The elimination of nicotine from cigarette smoke by tips containing silica gel; by various chemicals as tartaric, tannic and phosphotungstic acids, iron alum and  $\text{FeCl}_3$  contained in pledgets of cotton in the cigarette mouth piece has been studied. It is shown that such pledgets impregnated with tartaric acid, in an amt. of about 25 mg, do not influence the smell and taste of cigarette smoke as much as other materials, but a 45% adsorption of the nicotine resulted. It is further shown that the cotton pledgets of certain com. cigarette tips are practically worthless as nicotine adsorbents, the paper insets of certain cigarette tips adsorb more nicotine (45% at the most), but much less than that guaranteed. W. O. E.

Incompatible drug mixtures K. Brunner. *Pharm. Zentralhalle* 76, 885-92(1935).—Numerous examples are cited of incompatible mixtures by double decomposition through acid or basic components, of acid and basic material, formation of difficultly sol. salts, oxidation and reduction phenomena, incompatible powder mixts. W. O. E.

Discoloration and filtration under pressure C. Rohmann and J. H. Ehlers. *Pharm. Zig* 80, 1190-7(1935).—An illustration and description are given of a specially constructed app. designed to impart pressure to a discolored extract of the Bredin type. Precise directions for its manipulation are given. W. O. E.

Certain simple drug tests L. Rosenthaler. *Scientia Pharm.* 6, 109-10(1935).—Swiss pharmacopeial and more simple tests are comparatively recorded, for citric and salicylic acids, acetanilide, antipyrine, salicylate, pptd.  $\text{CaCO}_3$ , caffeine and theophylline, glucose, Hg salicylate, tamarind pulp, tartar emetic, theobromine and theophylline. W. O. E.

Homeopathic preparations VIII. Occurrence of enzymes (desmolases). A. Kuhn and G. Schifer. *Pharm. Zig* 80, 1029-31(1935), cf. C. A. 29, 237.—The results obtained in the examn. of some 50 odd plant preps. with respect to their content of oxidases, peroxidases and catalases are reported. W. O. E.

Emulsions, emulsifiers and new saline bases. G. Wallrabe. *Pharm. Zig* 80, 1147-9(1935).—An address. W. O. E.

Morphine. R. Fischer. *Pharm. Zig* 80, 1055-7(1935).—In sterilization as commonly effected, a possible decompn. of the alkaloid is not to be ignored, since long storage in unsuitable glass containers may represent an undesirable factor on account of alkali from the glasses. This problem may best be solved through recourse to suggestions made by Th. Paul. Thirty-six references. W. O. E.

Fluidextracts, now and in the past. E. Kessler. *Pharm. Zig* 80, 1080-2(1935); cf. C. A. 29, 6222<sup>1</sup>. W. O. E.

Chemical and biological experiments and reflections on homeopathy. Th. Sabatichka. *Pharm. Zig* 80, 1002-5, 1108-11(1935).—An address. Thirty references. W. O. E.

Diagnosis and prognosis during the period of uroscopic medicine. M. Weiss. *Säident. Apoth.-Zig* 75, 870-9(1935). W. O. E.

Estimation of ipecacuanha alkaloids. Lajos David. *Pharm. Zig* 80, 1121-5(1935).—The various procedures official in pharmacopeias and certain text books on alkaloids are reviewed in connection with a suggested method involving extrn. of the alkaloids with  $\text{CHCl}_3$  and their titration with standardized HCl. W. O. E.

Percolation and percolators C. Koch. *Pharm. Zig* 80, 1170-2(1935); cf. C. A. 29, 5221<sup>1</sup>. W. O. E.

Certain chapters of the homeopathic pharmacopeia. Wolfgang Brandrup. *Pharm. Zig* 80, 1160-1(1935).—Data are presented on the primary tincture of *Drosera*. W. O. E.

Estimation of soap in pharmaceutical preparations.

W. Stüwe. *Apoth. Zig* 50, 1545-8(1933).—Certain procedures are outlined and discussed for the examn. of various soaps as potash, glycerinated liquid, potash spirit, liq. cresoli sapon., spirit sapon., camphorated spirit, medicated, jalap, camphorated liniment, ammon. liniment, liq. formaldehyde and  $\text{H}_2\text{O}$  in medicinal. W. O. E.

Operations with a discolator. Karl Höll. *Pharm. Zig* 80, 1155-7(1935).—The operations involve the prepn. and evaluation of cinchona, ipecacuanha, strophanthus and strychnos tinctures, as also the application of the discolator in forensic analysis, as the detection of strychnine in various org. tissues. W. O. E.

New drugs and pharmaceutical specialties during the 3rd quarter of 1935. F. Zernik. *Säident. Apoth.-Zig* 75, 957-9(1935), cf. C. A. 29, 7017<sup>1</sup>. W. O. E.

The estimation of essential oils in drugs and plant material R. Wasieky, Irida Graf and Stella Bayer. *Scientia Pharm.* 6, 101-6(1935).—For the estn. of oils in drugs and plant material a simple and rapid method is described. It utilizes the principle recommended by A. Kuhn (C. A. 28, 2125<sup>1</sup>) involving the estn. of moisture with xylene. The defects inherent in app. heretofore employed are discussed and the elimination of such defects through appropriate changes is indicated. The type of app. suggested consists of a l. flask and rung tube in which a reflux condenser empties and which possesses a U-shaped branch; the one shank is quite narrow and carries a scale divided into 0.01 cc., the U-tube carries in the convex bend a short tube with glass cock. In carrying out the operation 2-20 g. of the sample (corresponding to the oil content) and 150-500 cc. satd. NaCl soln. are introduced into the flask, then allowed to boil vigorously for 1 hr. Toward the end of the distn. the boiling is interrupted a few min., the flask rotated and the distn. continued 10 min. After appropriate cooling, the vol. of the distd. oil is noted, the result indicating the cc. of oil per g. of drug. In the case of drugs with oil of a d. greater than or about 1, also with certain other drugs as *Flos Chamomillae vulgaris*, a small quantity of pinene is pipetted into the distn. flask; allowance for such addn. is made when the vol. of oil is noted. The influence of the fineness of the sample, as also its compn. on the analytical result is discussed. The results obtained in the examn. of certain drugs are reported. The app. is suited to the prepn. of aqueous aromatics. W. O. E.

Albumin tannate. F. Reimers. *Scientia Pharm.* 6, 100-9(1935); cf. C. A. 29, 6362<sup>1</sup>.—The results obtained in the examn. of a series of com. samples obtained in Denmark indicate unsatisfactory qualities in this commodity. W. O. E.

Absorption of the most important cinchona alkaloids in the ultraviolet. Leopold Fuchs and Anni Kampitsch. *Scientia Pharm.* 6, 113-22(1935).—The ultraviolet absorption spectra of pure quinine and cinchonine, and their stereoisomers quinidine and cinchonidine have been detd. under different conditions and thereby 2 absorption types noted. The aq. and alc. solns. of the neutral salts correspond likewise to the type of the absorption spectra of their bases. The not very far reaching changes in the spectra observable within this type of absorption, for example through addn. of  $\text{H}_2\text{O}$  to the alc. soln., are traceable to the influence of the solvent or to the formation of heterogeneous assoc. combinations (solvat formations via Hantsch), resp., while the influence of the  $\text{pH}$  only appears in the solns. of acid salts or in excess acid. The latter form the 2nd type of absorption spectra; in between are found the aq. solns. of acid salts, which in alc. soln., corresponding to the lower  $\text{pH}$  concn., show a very similar spectrum to the base. Acid concns. stronger than 0.1 N only change the spectra of both alkaloids very slightly. In all the solvents examined a distinct difference between the quinine and cinchonine spectra was noted, conditioned on the chromophore MeO group of quinine as opposed to the cinchonine. Solns. of the chlorides and sulfates, the latter calcd. to half the mol. wt., yield the same spectrum, also solns. in 0.1 N  $\text{H}_2\text{SO}_4$  and HCl. Kundt's rule—red shifting of the spectra with increasing  $n$  of the solvent—could only be partially

corroborated, and indeed with solns. in 0.1 N HCl acid alc. as compared with the soln. in aq. 0.1 N acid. Of the absorption curves of alkaloidal solns. in 90% and in dil. (15 to 20%, resp.) alc. the latter in contradiction to Kundt's rule are moved toward the long wave length. Under like conditions quinidine and cinchonidine possess almost the same absorption spectra as quinine and cinchonine, higher quant. divergencies in the magnitude of extinction coeffs., as deduced from the curves of J. Manta, were not observed. In connection with the authors' expts. the findings of H. Fischer are discussed. W. O. E.

Precipitation of alkaloids with sodium glycerophosphate. L. Rosenthaler (Bern) *Scientia Pharm.* 6, 122-3 (1935).—Reference is made to a micromethod by D. v. Klobuzitsky (*C. A.* 28, 48377), in connection with the pptn. of 14 different alkaloids, a description of which is given. W. O. E.

Oleates. Luis de Prado *Rev. centro estud. farm. bogum* 25, 403-18, 453-72 (1935).—The chemistry of oleates, fixed oils and oil solns. used in pharmacy is discussed. L. E. Gilson.

Alkaloidal content of infusions and decoctions of the seeds of *Lupinus albus*. D. Torrisi *Boll. soc. ital. biol. sper.* 10, 807-9 (1935).—The amt. of alkaloid extractable by simple infusion or decoction of the seeds of *Lupinus albus* is greater from the roasted seeds than from the raw because of the greater absorptive power of the latter. The alkaloidal content of 100 g. raw or slightly roasted seeds is about 1 g. while after intense roasting it is only about 0.90 g. The prolonged intense heat destroys a small portion of the alkaloid. Helen Lee Gruhl.

Importance of applying a correction in the determination of morphine in opium in the method of analysis prescribed by the British Pharmacopoeia of 1932. Jitendra Nath Rakshit *Ann. chim. anal. chim. appl.* 17, 315-16 (1935).—The pptn. of morphine by  $\text{NH}_4\text{Cl}$  in the presence of alc. and ether in solns. contg. morphine that has been treated with milk of lime is incomplete. From results obtained in the recovery of morphine from the mother liquor after the above pptn., the conclusion is drawn that, for every 100 g. of opium taken, 0.66 g. should be added to the wt. of morphine obtained in the analysis by the British method. W. T. H.

Combating epidermophytes of feet and hands. H. O. Loos *Arch. Dermatol. Syphilis* 170, 602-14 (1934).—Eleven dyes and 26 other chemicals were tested for fungistatic and fungicidal value against *Epidermophyton interdigitalis*. Brilliant green was by far the most effective. O. Hartley.

The "membrane method" for determining the fungicidal action of chemicals. Its clinical implications. Herman Sharlit *Arch. Dermatol. Syphilis* 31, 217-23 (1935).—Samples of collodion were impregnated with each of the following tetraiodohexamethylenetetramine (I), thymol (II), I plus II, iodine (III), salicylic acid (IV), benzoic acid (V), boric acid (VI) and Arning's tincture (VII). When the tubes were completely lined with the medicated collodion, I through V were fungistatic against all fungicides tested except *Aspergillus niger*, on which II only was effective. When only the upper half of the tube was covered with collodion, with no contact between medium and membrane, II alone was fungistatic. Tests showed that II volatilized from the membrane and was taken up by the medium, and that the chemicals in the contact expts. diffused into the medium appreciably. Analogies are drawn to diffusion through human skin membranes. Salicylic acid is considered the ideal fungicide and boric acid the ideal fungistat because they diffuse rapidly through the skin and appear unchanged in the urine. O. Hartley.

Further studies in fungicides. Comparative evaluation of phenol derivatives by modified laboratory procedure. Lyle B. Kingery, Roger Williams and Glen Woodward *Arch. Dermatol. Syphilis* 31, 452-9 (1935).—The fungicidal powers of 38 phenol and thymol derivs. were detd. on *Monilia tropicalis*. None was as effective as I in vitro, but hexylresorcinol ranked highest, with 3,5-dibutylphenol second. In the presence of hide dust, 3 times as much I

and hexylresorcinol were required to kill the fungi as before and with blood serum, 10 fold concns. were needed. NaOCl and I were the only popular ringworm remedies which retained fungicidal power in the presence of these proteins. O. Hartley.

Mycostatic studies on certain monilias and related fungi. Paulina Gomez-Vega *Arch. Dermatol. Syphilis* 32, 49-58 (1935).—Ten dyes and 8 disinfectants were tested in vitro for fungistatic properties. Crystal, gentian, methyl violet and cresol were most effective. Clinically, crystal violet proved therapeutic in several dermatomycoses, and mercurochrome cured a case of tinea and one of onychia when the treated part was exposed to sunlight. Mercurochrome was fungistatic in 1:10,000 diln. when accompanied by sunlight, but ineffective in 1:500 strength without it. O. Hartley.

Preliminary observations on the chemistry and pharmacology of the alkaloids of *Duboisia hopwoodii*. C. S. Hicks and H. Le Messurier *Australian J. Exptl. Biol. Med. Sci.* 13, 175-88 (1935).—The chief alkaloid was isolated and fairly well identified as *d-nornicotine*. C. G. King.

Yield and characteristics of digitoxin obtained from *Digitalis purpurea*. Nahuel-Huapi. G. Spagnol and N. Manzano *Rev. sud americana endocrinol. inmunol. quimioterap.* 18, 683-9 (1935).—The dried leaves from the drug cultivated in Argentine yielded 0.317% digitoxin. The physiol. activity was one cat unit to 0.96 mg. A. E. Meyer.

Ethereal oil of *Torilis anthriscus*. Gmel. Tatsuo Kariyone and Atsuko Majima *J. Pharm. Soc. Japan* 55, 887-93 (1935).—(In German 168-70).—K and M. obtained from the fruits of *Torilis anthriscus*, Gmel. an ethereal oil which had the following phys. properties:  $d_4^{20}$  0.8774,  $n_D^{20}$  1.4504,  $n_D^{25}$  1.4343, acid no. 0, ester no. 8.23, I no. 70.7 and methoxy no. 0. The fractional distn. of oil (100 g.) gave the following constituents: cadinene dihydrochloride,  $\text{C}_{15}\text{H}_{24}\text{Cl}_2$ , m. 116°,  $[\alpha]_D^{25}$  -37.67° (in  $\text{CHCl}_3$ ), hydrobromide,  $\text{C}_{15}\text{H}_{24}\text{Br}_2$ , m. 122-3°. *Torilene*, as sulfate,  $\text{C}_{15}\text{H}_{24}\text{SO}_4$ , m. 145°. *Torilene* sulfate on boiling with 10% alc. KOH for 20 min. gave *torilene* hydrate,  $\text{C}_{15}\text{H}_{24}\text{O}$ , m. 51-5°. The fruit of *Torilis anthriscus* contains about 10% oil. The oil was hydrolyzed with 10% alc. KOH and treated with MeOH. The Me ester was then fractionated and each fraction was hydrolyzed and sepd. according to the method of Twitchell. The fatty acid thus obtained was petroselinic acid,  $\text{C}_{15}\text{H}_{30}\text{O}_2$ , m. 33°; amide,  $\text{C}_{15}\text{H}_{29}\text{NO}$ , m. 70.5°. Oxidation with  $\text{KMnO}_4$  of the crude acid obtained from the alc.-sol. lead salt gave dihydroxy-stearic and sativnic acid through which the presence of oleic and linoleic acid was proved. F. I. Nakamura.

Antiseptic action of physical and physicochemical factors. Sterckx *Ann. hygiénique* [2], 2, 132-40 (1935).—A brief review of work published in 1934 and 1935. A. Papineau-Couture.

The accurate determination of the rotatory power of essential oils. Y. R. Naves and M. G. I. *Parfums de France* 13, 253-8 (1935). (In French and English).—A discussion of the chief sources of error in the accurate detn. of the  $\alpha$  of essential oils, and the precautions required to eliminate or minimize them. A. Papineau-Couture.

Natural and artificial musks. Ignaz Herold *Seifen-Industrie-Zig* 62, 695-7, 767-9, 849-51 (1935).—A historical review. J. W. Perry.

Modern perfume compositions. Fritz Schulz. *Seifen-Industrie-Zig* 62, 693-9 (1935).—Recipes for 5 joqual compns. are given. J. W. Perry.

Almecerin as the basis of skin creams or as an agent for improving them. Josef Augustin. *Seifen-Industrie-Zig* 62, 702, 726 (1935). J. W. Perry.

Modern fat free skin creams made with triethanolamine. I. Bell and Julius Hubbscher. *Seifen-Industrie-Zig* 62, 702-4, 728-9 (1935).—The use of triethanolamine in skin creams is discussed and 7 recipes are given. J. W. Perry.

Hair oils. Hans Schwarz *Seifen-Industrie-Zig* 62, 610-12 (1935).—The selection of ingredients and their proper proportioning are discussed. J. W. Perry.

**Cosmetic colors.** I. H. Stanley Redgrove. *Pharm. J.* 134, 288-9(1935).—Pigments used in cosmetics are discussed under the heads of natural earths, synthetic inorg. white, yellow, blue, green and black pigments, and carmine, the Al lake of the cochineal color. Emphasis is laid on the question of toxicity in contact with the skin. II. *Ibid.* 606-8.—Colors of natural org. origin (chlorophyll, cochineal, alkanin) are considered, then synthetic dyestuffs, with Fuchner's table of classification into harmful and harmless dyes (F in Handbuch der experimentellen Pharmakologie, Vol. I, cf. *C. A.* 14, 2031), foodstuff colors, with table of 61 dyes permitted in different countries are also discussed (cf. Hesse, *C. A.* 6, 3293). III. *Ibid.* 135, 533.—The toxicities of certain lake colors, especially those contg Ba even if "insol.," are discussed (cf. E. Klarmann, *Aromatics*, April, 1931). Suitable dyes or pigments for cream rouges, creams, lipsticks and nail enamels are recommended. Synthetic insol pigment dyes present a subject for further study in cosmetics.

S. Waldbott

**Cholesterol in cosmetics.** E. A. Lum. *Pharm. J.* 134, 291(1935); cf. *C. A.* 27, 5892, 28, 17731.—A tentative antiskunk formula contains almond oil 50%, anhyd lanolin 6.5, white beeswax 13.0,  $H_2O$  26.0, borax 1.0, cholesterol 1.0, lecithin 1.5, Na benzoate 1 or nipagin 0.2%. The heated aq. soln. of borax and preservative is slowly added to the mixed and melted fats while stirring until cool.

S. Waldbott

**Constitution and action of drugs.** F. L. Pyman. *Pharm. J.* 134, 619(1935).—The relation between chem. constitution and physiol. action was pointed out in an address on arsenicals, Bi compds and the chemotherapy of certain antiseptics, e. g., harmine derivs (cf. *C. A.* 27, 5419; 28, 4481), plasmochin and atetrin (cf. *C. A.* 28, 8261<sup>1</sup>, 8271<sup>1</sup>, 37941<sup>1</sup>, 48371<sup>1</sup>), etc.

S. Waldbott

**The British Pharmaceutical Codex, 1934; comments.** Horace Finnemore. *Pharm. J.* 135, 131-2(1935).—For the prepn. of Easton's syrup, an improved procedure is suggested based on the use of formulas for soln. of ferrous phosphate and syrup of quinine and strychnine adopted in the Australian and New Zealand official formulary. For internal remedies, the unit method of prescribing, i. e., the quantities necessary for one dose, should be used. Other criticisms refer to elixirs, incompatibilities and certain special preps.

S. Waldbott

**Troublesome emulsions.** James Hall. *Pharm. J.* 135, 133(1935).—Improved processes are suggested for limentum terebinthinace, Brit. Pharm., haustus filices, emulso petrolei cum laolini, emulso petr. c. agar c. phenolphthalein of the National Formulary, and limentum alba, lotio calaminea oleosa of Nat. Form. 2nd ed.

S. Waldbott

**Chocolate and cacao products, their relation to pharmacy.** A. Churchman. *Pharm. J.* 135, 134-5(1935).—The characters of the cacao bean and mfg. methods are described, with photographs. The uses of the shell, its theobromine content (cf. *C. A.* 12, 2633; 13, 3244; 14, 3114; 15, 1170, 16, 1995) and its high vitamin D content (*C. A.* 28, 5897<sup>1</sup>) are referred to.

S. Waldbott

**A method of ampoule filling.** F. A. Hudson. *Pharm. J.* 135, 157(1935).—To avoid contamination of the filtered soln. by dust in filling ampoules contg 50 cc. or more, the flask contg. the filtered soln. is used in inverted position as the filling reservoir. Upon opening a pinchcock, the liquid runs into a corked cylinder below, graduated into 100 cc. From this, the liquid runs through a 1-ft. rubber tube and a surgical hollow needle into the ampoule. Before removing the needle, the ampoule with the needle above the liquid is raised to the level of the liquid in the cylinder; this causes the liquid in the needle to recede, and no liquid will adhere to the neck of the ampoule disturbing the sealing of it.

S. Waldbott

**Structural standards for crude drugs.** T. E. Wallis. *Pharm. J.* 135, 207-8(1935).—Structural standards for pharmacopoeial drugs, unground or powd., are classified and discussed. The lycopodium method (cf. *C. A.* 14, 93) permits approx. quant. examn. of powd. drugs.

S. Waldbott

**Some new preparations for the hair.** Frank Atkins. *Pharm. J.* 135, 531-2(1935).—The preps. and modes of uses are given for permanent waving solns., "friction lotions," setting lotions of the non-gummy and gummy types, hair fixatives for men, brillantines, fixative creams and perfume compns.

S. Waldbott

**Propylene glycol extracts; stability of vegetable coloring matters.** John Rae. *Pharm. J.* 135, 539(1935); cf. *C. A.* 29, 4901<sup>1</sup>.—Exts. of 14 vegetable colors prepd. with propylene glycol were examd in 1% aq. solns. for their resistance to light during 6 months' exposure; stabilities of color in descending order were for henna 95%, curcuma 90, carmine and red pine 85, madder and logwood 70, cudbear 45, defatted cochineal 20 and saffron 10%. When 1 cc. of about  $NH_4OH$ ,  $NaOH$  or  $H_2SO_4$  was added to the 1% soln.,  $NH_4OH$  gave for carmine, red sanders and red pine 90%, Brazil wood 85, henna, rose leaves 80, litmus 65, cudbear 50, cochineal 10 and saffron 0%. With dil.  $H_2SO_4$ , only litmus (90%) and madder (70%) were quite permanent.

S. Waldbott

**Ba salt [used in prepn. of cosmetics]** (Fr. pat. 785,713) 19 Emulsifying agents, etc. [used in prepn. of cosmetics and pharmaceutical products] (Fr. pat. 785,678) 13

**Castor-oil preparation for medicinal use.** John C. Bird (to John Wyeth & Bro.) U. S. 2,021,044, Nov. 12. A medicine for internal use comprises castor oil emulsified with  $Al(OH)_3$  gel.

**Ricinoleate medicament for intestinal detoxification.** Theodore H. Rider (to Wm. S. Merrell Co.) U. S. 2,019,933, Nov. 5. Mg and Ca ricinoleates are used for oral administration (suitably emulsified with mineral oil).

**Antanemia product (incipiently self-digested and dehydrated and defatted whole-stomach material such as that from hog stomachs).** Geo. B. Walden (to Eli Lilly and Co.) U. S. 2,020,267, Nov. 5. Various details are given for prepn. a product for treating pernicious anemia.

**Quinine compounds.** Hans P. Kaufmann. Brit. 433,261, Aug. 12, 1935. See Ger. 614,882 (*C. A.* 29, 8239<sup>1</sup>).

**Aliphatic diamines.** Boot's Pure Drug Co. Ltd., 6 Frank L. Pyman and Hyman H. L. Levene. Brit. 433,086, Aug. 8, 1935. Diamines of formula  $(CH_2)_n(NRR')$ , where  $n$  is 7-13 and  $R$  and  $R'$  are alkyl groups which must not both be Et, are prepd. from compds. of formula  $(CH_2)_nX$ , where  $X$  is halogen, by heating with secondary amines of formula  $NHRR'$ . Among examples, 1,10-bis-(diamylamino)decane is prepd. from 1,10-dibromo- or -dichloro-decane and diamylamine. The products are amebicides.

**Derivative of 2-phenylquinoline-4-carboxylic acid.** Hirsch Gregor Jacobson. Brit. 433,040, Aug. 2, 1935. See U. S. 2,006,020 (*C. A.* 29, 5602<sup>1</sup>).

**Cyclopentylbarbituric acids and their salts.** Horace A. Shonle (to Eli Lilly & Co.) U. S. 2,019,936, Nov. 5. Various details and examples are given of the production of intermediates and of final products such as cyclopentyl-ethylbarbituric acid, m. 181-3° its Na, NH<sub>4</sub>, methylammonium and diethylammonium salts, etc., suitable for therapeutic purposes.

**Bz-Alkoxy-2-aminobenzothiazoles.** Max Engelmann (to E. I. du Pont de Nemours & Co.) U. S. 2,019,529, Nov. 5. Cryst. compds. having more than 2 C atoms in the alkoxy group are obtained by a process (of which various examples are given, with details of procedure) involving reacting with a halogenating agent (such as Br to form the hydrobromide or sulfonyl chloride to form the hydrochloride of the product) upon the corresponding Bz-alkoxyphenylthiourea. 6-Isobutoxy-2-aminobenzo-thiazole m. about 140° and its hydrochloride m. about 171° and may be used as a local anesthetic, having less toxicity than cocaine. 6-Isomethoxy-2-aminobenzo-thiazole m. about 122° and its hydrochloride m. about 135°. 6-Isopropoxy-2-aminobenzo-thiazole m. about 135°. 4-Butoxy-2-aminobenzo-thiazole m. about 142-3° and its hydrobromide m. about 166-7°.

Substituted halo phenols. Emil Karmann and Louis W. Gates (to Lehn & Fink, Inc.). Brit. 432,955, July 29, 1935. See U. S. 1,935,911-12 (C. A. 28, 1472).

Cyclically disubstituted tetrazoles. Zoltan Földi (to the firm Chinoin Gyógyszer és Vegyszereti Termékek Gyára R. T. (Kereszty & Wolff)). U. S. 2,020,937, Nov. 12, 1936. See Ger. 611,692 (C. A. 29, 5995).

Complex salts of pyridine derivatives. Albert Rothmann. Ger. 665,526 and 566,694, July 9, 1935 (Cl. 12 p. 1,01). Water-soluble complex metal and metalloids salts of 2-amino-6-mercaptopyridine-4-carboxylic acid and 2,6-dimercaptopyridine-4-carboxylic acid are prep'd by the action of the oxide or hydroxide of the metal or metalloid on solns of the acids or their salts. The new complex salts are obtained from the resulting solns by evap'n or pptn. by an org solvent. Examples describe the prep'n of  $(\text{C}_4\text{H}_4\text{O}_2\text{N}_2\text{S}_2\text{Na})_2\text{Ca}$ ,  $\text{C}_4\text{H}_4\text{O}_2\text{N}_2\text{S}_2\text{KCl}$ ,  $\text{C}_4\text{H}_4\text{O}_2\text{N}_2\text{S}_2\text{KAs}$ , and other similar compds., the structural formulas being given in some cases, (565,526). Other mercapto- or polymercaptopyridine carboxylic acids may be used (566,694). The compds. are used in therapy.

Purified neoparsphenamine. George W. Raunz and Abraham I. Kremens (to Abbott Laboratories). U. S. 2,020,655, Nov. 12. An "improved neoparsphenamine" produced from Na formaldehydesulfoxylate and arsenamine, having a good color, low toxicity, high therapeutic efficiency and stability and which is substantially free from uncombined  $\text{CH}_3\text{O}$ , is obtained by including in the reaction mixt. a relatively small proportion of a reducing substance such as Na hyposulfite or  $\text{NaHSO}_3$  which will convert any free  $\text{CH}_3\text{O}$  present into formaldehydesulfoxylate.

Ethereal sulfur-containing oils. Friedrich Boedecker. U. S. 2,021,530, Nov. 19. Oils such as mustard-seed oil, asafetida oil or garbe oil together with desoxycholic or apocholic acid form cryst. addn. products of odorless character and suitable for therapeutic purposes. Several examples are given.

Detoxified pollen extract. Edgar B. Carter (to Abbott Laboratories). U. S. 2,019,088, Nov. 5. A detoxified pollen ext. suitable for treatment of hay-fever is prep'd. by incubating a pollen ext. (suitably at a temp. of about 37.5° and for a time of about 2 weeks) with the addn. of about 0.1% of a 40%  $\text{CH}_3\text{O}$  soln or an equiv. amt. of methanamine or the like.

Antituberculosis extract. I. G. Farbernd. A.-G. Ger. 616,884, Aug. 7, 1935 (Cl. 30k 6). Heat-killed tuberculous bacilli are boiled for 1 hr with 96% alc. The cooled ext. is filtered and the filtrate ext'd with a readily volatile solvent such as  $\text{CHCl}_3$ , pyridine, petr. ether, etc., and the ext. evap'd to dryness. The residue is heated for 1 hr with acetone and the insol. part, after removal of the acetone, is taken up in  $\text{C}_4\text{H}_6$  or petr. ether and made up into a 1% soln for use as an antituberculosis ext.

Serum. F. Vagoras J. Georgiou. Ger. 616,716, Aug. 3, 1935 (Cl. 30k 6). A prep'n for treating malignant swellings such as cancer is obtained by pptg. serum from the blood of a highly pregnant animal by alc., sepg. and sterilizing the ppt., taking it up in a physiol. NaCl soln, and removing the last traces of alc. by evap'n. *in vacuo* at 60°. Examples are given, dog and rat serum being used.

Hormones. Soc. pour l'ind. chim. à Bâle. Ger. 616,612, Aug. 1, 1935 (Cl. 12p 17 10). The corpus luteum hormone is obtained in pure form by prep'g. a crude ext. of the organ with an org. solvent nonmiscible with water, and extg. this with strong mineral acid. The hormone is ppt'd. from the acid by adding water and re-ext'd. by an org. solvent nonmiscible with water. The solvent is then driven off. An example is given.

Follicle hormones. Schering-Kahlbaum A.-G. Brit. 432,851, July 26, 1935. Divided on 432,435 (C. A. 30, 2509) and addn. to 432,435 and 432,474 (C. A. 30, 2509). The hormones are isolated from urine or preps. made therefrom, or from other starting materials, e. g.,

placenta ext. or exts. from vegetable matter, by 1st removing volatile phenols, e. g., by steam distn., dissolving the residue in a  $\text{H}_2\text{O}$ -immiscible solvent, e. g.,  $\text{Et}_2\text{O}$ , extg. the soln. with alkali hydroxide soln., e. g., KOH, which takes up the hormones but not the neutral constituents, causing the hormones to react with an acylating agent, e. g.,  $\beta$ -toluenesulfonyl chloride, isolating the acylation product, e. g., with  $\text{Li}_2\text{O}$ , and saponif. it to yield the hormones.

Purifying enzymes. Schering-Kahlbaum A.-G. Fr. 785,490, Aug. 10, 1935. Intermediate enzymes are ppt'd. from Lebedev juice or ext. or like preps. obtained from yeasts or other kinds of cells by regulating the  $pH$  value of the liquid, addn. if necessary, to an optimum value (4). The soln. is afterward purified by cataphoresis.

Bacterial solutions, endotoxins. Roderick F. Hunwick. U. S. 2,020,647, Nov. 12. See Brit. 425,216 (C. A. 29, 5601).

Cyclic esters. Wallace H. Carothers and Julian W. Hill (to E. I. du Pont de Nemours & Co.). U. S. 2,020,298, Nov. 12, Brit. 433,632, Aug. 19, 1935. Cyclic esters having rings of more than 7 atoms, suitable for use in perfumes, toilet waters, etc., are produced by depolymerizing the corresponding linear polyester; e. g., decamethylene carbonate is produced by depolymerizing polymeric decamethylene carbonate (suitably by heating in the presence of Na or Sn, etc.). Numerous examples are given.

Germicidal detergent and cosmetic preparations. Lever Brothers Co. Brit. 432,689, July 31, 1935. Addn. to 427,324 (C. A. 29, 6366). Germicidal soaps, cosmetics, etc., are made by mixing soaps or similar saponaceous substances with a Hig comp'd. of type  $(\text{RHg})_2\text{R}'$ , in which R is an aromatic nucleus, with or without side chains, in which the nuclear and side chain C atoms are not directly connected to any element other than C, H or Hg, and R' is a radical contg. 1 or more imido groups the N atom of which is directly connected to the RHg group. Hig comp'ds. of hydrazobenzene, guanidine, phthalimide,  $\alpha$ -benzoylsulfonide, succinimide, pyrrole, naphthalimide, indole, isatin, carbazole, piperidine, 3-nitrophthalimide, diketopiperazine, acridone, pyrrolidine, emetine, 1,3-dimethylxanthine, barbituric acid, parabanic acid, urea acid, theobromine, alloxan, quinonimide, allantoin and such dyes as auramine, indigo and indanthrene red violet are specified. Dyes and perfumes may be added. After mixing, the soap may be plodded, milled or stamped.

Skin protective coating composition. Omer McDaniel (to Mountain Varnish & Color Works). U. S. 2,021,131, Nov. 19. Lemenone 1, Na silicate 900, glycerol 1155, Na stearate 288 and water 1600 parts are used together.

Cosmetic composition. Albert Ebel, Sauerkohlfabrik and Max Platsch. Ger. 605,941, June 5, 1935 (Cl. 30k 13). A stable product for cleaning and feeding the skin is obtained by pulverizing fermented sauerkraut, removing the sap by pressing, drying, grinding and mixing with perfume, etc. The sap may be deodorized and preserved.

Shaving compositions. Fritz Feigl and Hugo Gleich. Austrian 142,762, Sept. 10, 1935 (Cl. 23b). Solid, paste or liquid shaving compns. are improved by addn. of a secondary or tertiary phosphate of an org. base, e. g.,  $(\text{HOC}_2\text{H}_4)_3\text{N}$ , in a proportion of 15-30% calcd. on the soap and other solids in the compns.

Hair waving. Elie Soussa. Fr. 785,878, Aug. 21, 1935. A soln. for application to the hair before permanent waving contains  $\text{Na}_2\text{SO}_3$  35,  $\text{NaHCO}_3$  20, Na borate 7, powd. soap 4 g. per l. of soln.

Nicotine. Soc. de participation à l'industrie cuprique. Fr. 784,837, July 25, 1935. Crude material contg. nicotine is treated with water contg. an oxide or carbonate of an alkali or alk. earth metal and then submitted to distn. with steam or a gas. The nicotine is absorbed by a dil. acid.

Nicotine removal from tobacco. Hall Tobacco Chemical Co. Fr. 784,795, July 24, 1935. Nicotine is removed or reduced by submitting tobacco to the action of ethylene oxide in the presence of moisture, or by treating the tobacco with an aq. soln. of ethylene oxide.

## 18—ACIDS, ALKALIES, SALTS AND OTHER HEAVY CHEMICALS

E. M. SYMINGTON

Manufacture of sulfuric acid from hydrogen sulfide. Wolfhart Seckel. *Chem. Fabrik* 1935, 416-18.—Gases contg.  $H_2S$  from coke ovens, hydrogenation processes, etc., are burned at 750-800° with excess air and moisture and without further treatment are passed at 350-450° over a V catalyst to convert  $SO_2$  into  $SO_3$ , and the  $H_2SO_4$  is fractionally condensed. The yield is about 98% of theory, the acid contains 80-90%  $H_2SO_4$ , and is pure enough for accumulator acid. No acid pumps are required, a fan draws the gases through the system, and little floor space and attention are required. About 30 kw-hrs and 13 cu m of cooling  $H_2O$  are required. About 35% of the  $S$  in crude  $NH_3$  liquor can be recovered, the remainder at an addnl cost. *Cl C A* 29, 1592, 3783\*, 4906; J. H. Moore

Leaching alunite with sulfuric acid. L. C. Pan. *Chem. Ind. (China)* 10, No. 1, 35-42 (1935).—The best strength of  $H_2SO_4$  for leaching alunite calcined at 500° is about 15% by wt. Little advantage is gained by heating the leaching soln to 100°. The yield of alum by crystn is highest when the acidity is at a min. The size of the alum crystals formed is smallest from a soln contg 47%  $H_2SO_4$ . Solns of 0.133 N and 3 N  $H_2SO_4$  give about the same size of crystals, but the size increases as the acidity is further increased. *C. L. Tseng*

Conversion of chlorides to nitrates by means of nitric acid. R. Frankowski. *Przemysł Chem* 19, 154-6 (1935).—To dry  $NaCl$ ,  $KCl$  and  $BaCl_2$  from 1 N to 8 N  $HNO_3$ , in proportions of 1 0.5, 1 1, 1 1.5 and 1 2, was added, the mixt. evapd and yields were calc'd in terms of the starting salts and of  $HNO_3$ . For  $NaCl$ , the best yields on salt were obtained with 1 2 acid proportion and for the most dil. acid. The most dil. acid gave also the best yields with  $KCl$  and  $BaCl_2$ , the effect of concn. being the least for  $NaCl$  and the greatest for  $BaCl_2$ . The sepn. of the products depends on temp. coeffs. of soly. Excess acid can be used with a second or even a third batch of the starting salt, to give better acid yield. Sepn. of  $KNO_3$  from  $KCl$  is easy. Results point to a successful com. production of  $KNO_3$  from  $KCl$  and synthetic  $HNO_3$ . *A. C. Zachlin*

Production of hydrogen from natural gas. V. A. Karzhavin, N. P. Elektronov and B. M. Ovchinnikov. *Khimistrol* 7, 459-64 (1935); *cf C A* 26, 5399, 28, 3189\*—A mixt. of 1 cu m of natural gas (contg 87.4%  $CH_4$ ) with 1.9 cu m. of water vapor reacted at about 1350° over porous prog. treated with  $N_2$  catalyst produced 3.3 cu. m. of gas composed of  $CO_2$  9,  $CO$  22,  $H_2$  64,  $CH_4$  0.8 and  $N_2$  4.2%. The semicon. procedure of conversion and app. are illustrated and described. *Chas. Blanc*

The rare gases. Mathias. *Refrigerating Eng* 30, 196-8 (1935).—A review of the industry and new applications are discussed in the cases of He, Ne, Ar, Kr and Xe. *A. H. Johnson*

Some properties of activated carbon. E. R. Sutcliffe. *India Rubber J.* 90, No. 17a (Intern. No.) 37-8 (Oct. 31, 1935).—A review and discussion, with special reference to the plant at Leigh, England, and the properties of the C-manufd there. *C. C. Davis*

Action of decolorizing earths. Edward Erdheim. *Przemysł Chem.* 19, 150-7 (1935).—Preliminary results show that fuller's earth NY and Montana Z, on extn. after decolorization, act in a very different manner from that exhibited by activated charcoals. The decolorizing action of the earths is quite distinct from that of the charcoals. *A. C. Zachlin*

App. for cooling gases as in oxidation of  $NH_3$  (U. S. pat. 2,019,533) 1. Effecting reactions [ $NH_3$  synthesis] (Brit. pat. 432,574) 13.

Hydrocyanic acid. Russell W. Mullar and Herbert P. A. Groll (to N. V. de Bataafsche Petroleum Maatschap-

py). Brit. 433,398, Aug. 14, 1935. See Fr. 774,011 (*C. A.* 29, 2316\*).

Nitric acid. E. I. du Pont de Nemours & Co. Brit. 432,898, Aug. 6, 1935. Gases obtained in the demitration of residual acid from the manuf. of liquid nitric esters such as nitroglycerin are converted into  $HNO_3$  by scrubbing in countercurrent with dil.  $HNO_3$ , in an app. resistant to the corrosive action of the gases, whereby the corrosive impurities are removed, and absorbing the treated gases in  $H_2O$  or dil.  $HNO_3$  in an absorption tower made of Cr-Fe alloy. App. is described.

Phosphoric acid. Marx in J. Udy (to Swann Research, Inc.) U. S. 2,020,976, Nov. 12. A phosphatic material such as phosphate rock, carbonaceous material such as coke and a flux such as silica are melted in a furnace under conditions to form a gas comprising V vapor together with impurities, and there is formed within the gas a dispersed phosphoric oxide-water reaction product by reaction of part of the V vapor, the reaction product is removed, and elemental P is recovered from the vapor. App. is described, including an elec. furnace and elec. pptn. app., etc.

Phosphoric acid from ammonium phosphate. Charles L. Levermore (to General Chemical Co.) U. S. 2,022,050, Nov. 26.  $NH_3$  is driven off from an  $NH_4$  phosphate such as  $NH_4H_2PO_4$  at a temp. above about 300° while passing steam in contact with the material.

Sulfuric acid. Cyril B. Clark (to American Cyanamid Co.) U. S. 2,019,893, Nov. 5. An acid sludge, such as that produced by the  $H_2SO_4$  treatment of petroleum oils, is subjected to thermal decompn. at a temp. at which  $SO_2$  comds. of the sludge are reduced to  $SO_3$ , the  $SO_2$  is removed from the zone of decompn. together with water vapor and gaseous substances contg. H, the gas stream is subjected to dehydration, substances contg. H, capable of reacting with O to form water, are removed to an extent such that the remaining H content, after adjustment of the gas stream to the O content necessary for catalytically oxidizing the  $SO_2$ , is not greater than 3 mg. of H per cu. ft. of gas, and the gas stream is heated to a temp. at which it can be catalytically oxidized and is then catalytically converted to  $SO_3$ . An arrangement of app. is described.

Dehydrating caustic alkalis. Arnold Hanchett (to Solvay Process Co.) U. S. 2,022,037, Nov. 26. An aq. soln. of NaOH or the like in regulated quantity is brought into contact with a quantity of substantially anhyd. caustic of sufficient mass and temp. to vaporize the water from the liquid. App. is described.

Purifying caustic soda solutions. Pennsylvania Salt Manufacturing Co. Fr. 785,399, Aug. 8, 1935. NaCl is removed from concd. solns. of NaOH by heating the latter to 70-80°, adding Na $_2$ SO $_4$  in amt. depending on the desired degree of elimination of NaCl, mixing until the formation of an insol. salt contg. NaCl and Na $_2$ SO $_4$  is complete, cooling to about 30° and agitating slowly until the state of equil. of the supersatd. soln. is completely destroyed. The Na $_2$ SO $_4$  is added in an anhyd. finely divided state and in excess. The Na $_2$ SO $_4$  is recovered by adding sufficient water to dissolve the NaCl and NaOH.

Alkali metal phosphates. Louis Preisman (to General Chemical Co.) U. S. 2,021,699, Nov. 19. A mixt. of phosphatic material and an alkali metal sulfate is furnaceed in the presence of a reducing agent and from the furnaceed product is obtained an alkali metal phosphate and a mother liquor contg. alkali metal comds. including alkali metal sulfate, and the mother liquor is treated with a phosphoric acid soln. contg. sol. Ca phosphate for reaction with the sulfate.

Alkali metal tetraphosphates. Augustus H. Fiske and Charles S. Bryan (to Rumford Chemical Works). U. S. 2,019,665, Nov. 5. A tetraphosphate is formed by heating an alkali metal metaphosphate such as Na $PO_3$  with an alkali such as NaOH (at a temp. of 150° or higher, preferably to a temp. of redness) and the products may be

used as *detergents* in the laundry, dyeing, or leather industries, for *softening water*, etc. U. S. 2,019,666 relates to the production of tetraphosphates by reaction of an alkali metal acid pyrophosphate such as  $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$  with an alkali such as NaOH.

Pure aluminum-alkali double fluorides. Friedrich W. Hülcher and Veit Schwemmer (to Saline Ludwigshalle). U. S. 2,021,601, Nov. 19. Raw aluminous material such as bauxite contaminated with Fe and Si is treated with HF to dissolve Al, Fe and Si, and an alkali salt such as NaCl is added to the soln in only sufficient amount to ppt fluosilicic acid as alkali metal fluosilicate without use of a reducing agent, the pptd fluosilicate is sepd and an alkali metal salt such as NaCl is added to form a sol Fe salt and an insol Al double fluoride which is pptd and sepd.

Beryllium compounds. Carlo Adamoli. Ger. 616,671, Aug. 3, 1935 (Cl. 12m. 4). Addn to 541,544 (C. A. 27, 570). The method of 541,544, for obtaining Be compds. by treating finely ground Be minerals with  $\text{CO}_2$  and water under high temp and normal or elevated pressure in the presence or absence of acid or basic catalysts, is modified by pre-treating the ground mineral with a small amt of alkali carbonate and alk earth oxide at 700-750°, to disintegrate the mineral. Thus, Be ore contg 0.6% BeO is ground and heated with  $\text{Na}_2\text{CO}_3$  and CaO. The product is then heated to 850-900° with water and  $\text{CO}_2$  to give basic BeCO<sub>3</sub>.

Stabilizing hypochlorites. Electrochlore (Soc. anon.) Fr. 785,562, Aug. 13, 1935. Solns of alkali or alk earth hypochlorites are kept out of contact with the air and any O in soln is removed.

Salts from polyhalite. Arthur Lambert (to Rita Vinay). U. S. 2,020,322, Nov. 12. Polyhalite is treated with an acid, such as hot 10% Be  $\text{H}_2\text{SO}_4$ , the filtered soln. is neutralized with an excess of  $\text{Na}_2\text{CO}_3$ , the resulting mixt. is filtered and the filtrate is treated with  $\text{NH}_3$  and  $\text{CO}_2$  to ppt  $\text{NaHCO}_3$ , the soln is filtered and is further treated with an acid phosphate, if desired, and salts formed are crystd. Various modifications of procedure are described.

Magnesium products from brine. Wm. H. Farnsworth and Montel Montgomery (to Marine Chemicals Co., Ltd.). U. S. 2,021,501, Nov. 19. A brine contg convertible Mg salts such as sea water is treated with Ca(OH)<sub>2</sub>, particles carrying a film of Mg(OH)<sub>2</sub>, formed separately from the main body of brine to be treated and which serve to effect pptn of Mg(OH)<sub>2</sub>.

Sulfates. Metallgesellschaft A-G (Conway Freiherr von Gurswald and Erich Stahl, inventors). Ger. 610,925, Sept. 18, 1935 (Cl. 12m. 7). Addns to 571,494 (C. A. 27, 3041). See U. S. 1,992,681 (C. A. 29, 2671). According to Ger. 612,113, Sept. 18, 1935, the process of Ger. 610,925 is improved when the gases passed into the mixt. in a finely divided state are passed at such a rate and in such a state of subdivision that the mixt. is caused to foam. Methods of procedure are indicated, and examples are given. According to Ger. 618,890, Sept. 18, 1935, the process of Ger. 610,925 is applied to the manuf. of  $(\text{NH}_4)_2\text{SO}_4$  from the products, mainly  $\text{NH}_4\text{HSO}_4$ , obtained by treating  $\text{SO}_2$  or gases contg  $\text{SO}_2$  in the presence of water and preferably of air, with  $\text{NH}_3$  (or gases contg  $\text{NH}_3$ ) in the proportion of 1 mol of  $\text{NH}_3$  to 0.5-1 mol of  $\text{SO}_2$ . The addnl  $\text{NH}_3$  required for the manuf. of  $(\text{NH}_4)_2\text{SO}_4$  may be added before or during the oxidation Sp. processes are described.

Packaging acid sulfates such as bisulfates. Leo Löwenstein. U. S. 2,020,072, Nov. 5. An outer container is used of material such as metal or impregnated paper contg alkali metal carbonate compd. such as a bicarbonate capable of reacting with the sulfate, together with an inner container which may be formed of paper and is completely surrounded by the bicarbonate or the like.

Reaction of chlorine with metal bearing solids such as in making aluminum chloride. Sidney G. Osborne and Jasper M. Rowland (to Hooker Electrochemical Co.). U. S. 2,020,431, Nov. 12. Relatively coarsely comminuted ungraded solid material such as aluminous material for making  $\text{AlCl}_3$  is introduced into a stream of gas

contg. Cl flowing upwardly in a reaction chamber (of a described app.) of gradually increasing and abruptly decreasing cross-sectional area, and the rate of supply of solids and gases is adjusted to cause a progressive reduction in velocity and to maintain at substantially the point of greatest cross-section of the reaction chamber a zone in which the velocity is sufficient to support the solids only after they have been reduced in size to relatively fine particles consisting principally of materials unreactable with the Cl.

Alumina. Charles B. Willmore and Conrad C. Callis (to Aluminum Co. of America). U. S. 2,019,553, Nov. 5. Aluminous material such as calcined bauxite is treated with  $\text{HNO}_3$  in the presence of a F compd. such as  $\text{CaF}_2$ , and the resulting Al nitrate is decomposed by heat to form alumina.

Alumina. Ralph B. Derr (to Aluminum Co. of America). U. S. 2,019,554, Nov. 5. Aluminous material such as bauxite tailings contg. Fe is digested with a soln. contg.  $\text{HNO}_3$  and the resulting Al nitrate liquor is concd. and increased in basicity (suitably by evapn. of  $\text{HNO}_3$ ) and is cooled to crystallize Al nitrate which may be heated to form alumina. Mother liquor from the Al nitrate crystn. is added to liquor from the digestion stage.

Alumina. Wilhelm Fulda, Erich Wiedbrauck and Rudolf R. B. Wittig (to Vereinigte Aluminium-Werke A-G and Th. Goldschmidt A-G). U. S. 2,021,548, Nov. 19. Clay or the like is disintegrated by calcination and treatment with an aq. soln. of  $\text{SO}_2$ , thus forming an aq. soln. contg. Al sulfate; alumina is pptd. from the soln. by heating, the pptd. alumina is calcined, the calcined material is dissolved in alkali lye such as with NaOH and a pure alumina is pptd. from the resulting soln. by heating.

Alumina. The Electric Smelting & Aluminum Co. Ger. 619,129, Sept. 25, 1935 (Cl. 12m. 5). See Can. 324,768 (C. A. 26, 5390).

Alumina. I. G. Farben und A-G. Fr. 785,459, Aug. 10, 1935.  $\text{Al}_2\text{O}_3$  which is easily filtered is made by causing  $\text{NH}_4\text{HCO}_3$  to act on solid Al salts, sol. in water, e. g.,  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  or  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$  in the presence of a min. amt. of water.

Alums. Svend S. Svendsen (to Clay Reduction Co.). U. S. 2,022,012, Nov. 26. A material such as kaolin or bauxite contg. an alum-forming trivalent metal, silica, and a basic material capable of yielding a univalent sulfate is heated with  $\text{NH}_4\text{F}$  in the absence of water in the liquid state to form volatile  $\text{NH}_3$ -Si-F compds. and with reactive sulfates such as  $(\text{NH}_4)_2\text{SO}_4$  to produce sulfates of the trivalent and univalent bases; the volatile  $\text{NH}_3$ -Si-F compds. are volatilized, and the alum produced is extd. from the residue. An arrangement of app. is described, and several examples are given.

Removing iron from aluminum salt solutions. J. R. Geigy A-G. Ger. 619,018, Sept. 20, 1935 (Cl. 12m. 6). Addn to 570,376 (C. A. 27, 2540). Acidified solns. of  $\text{Al}_2(\text{SO}_4)_3$  contg. Fe are treated at an elevated temp. with the Ca or Ba salt of  $\beta$  naphthalenesulfonic acid during or after the reduction of the Fe to the ferrous form. The soln. is then cooled and filtered.

Ammonium chloride. Société d'études pour la fabrication et l'emploi des engrais chimiques. Brit. 432,770, Aug. 1, 1935. This corresponds to Fr. 779,317 (C. A. 29, 5608).

Calcium carbide containing calcium nitride. Soc. anon. pour l'exploitation des Brevets Jullien. Ger. 619,074, Sept. 21, 1935 (Cl. 12m. 30). See Fr. 750,039 (C. A. 28, 5877).

Calcium cyanamide. Bayerische Stickstoff-Werke A-G. Brit. 433,254, Aug. 12, 1935. This corresponds to Fr. 782,739 (C. A. 29, 70274).

Calcium cyanamide and phosphorus. Nikodem Caro and Albert R. Frank (Karl Briegleb, Ludwig Gscheidmeyer, Franz J. Kaess and Karl Zieke, inventors). Ger. 616,958, Aug. 30, 1935 (Cl. 12m. 9). Addn to 609,730 (C. A. 29, 5230).  $\text{CaCN}_2$  and P are produced simultaneously by reducing  $\text{Ca}_3(\text{PO}_4)_2$  with SiO<sub>2</sub>-free carboniferous material such as peat or anthracite.

**Lime from limestone.** Wm. H. Knox, Jr. (to Victor Chemical Works). U. S. 2,021,663, Nov. 19. Limestone contg. about 20-300 parts of F per million parts of CaO is heated to drive off substantially all CO<sub>2</sub>, and the heating is then continued at 1000-1500° for 1-12 hrs (the lower the temp. the longer the time) so that a lime is produced having a F content below 5 parts per million.

**Hydrating lime.** Carlos M. Bird. U. S. 2,020,098, Nov. 5. An app. is described in which milk of lime under pressure is added to unslaked lime at a plurality of hydration stages, in each of which the pressure is controlled, milk of lime from the same source being supplied for condensation of vapors resulting from the hydration, and milk of lime in excess of that required for hydration and condensation being returned to the source of supply.

**Dicalcium phosphate and fertilizers containing it.** Robert Suchy and Emil Reubke (to I. G. Farbenindustrie A.-G.). U. S. 2,021,527, Nov. 19. Raw tri-Ca phosphate is dissolved by treatment with a soln. contg. mono-Ca phosphate and free H<sub>2</sub>PO<sub>4</sub> in such proportions that the resulting soln. still contains a substantial excess of H<sub>2</sub>PO<sub>4</sub> over the ratio prevailing in mono-Ca phosphate, the resulting soln. is heated to the b. p. to ppt. di-Ca phosphate, the ppt. is sep'd from the soln. without substantial cooling, and there is added to the soln. the amount of H<sub>2</sub>PO<sub>4</sub> required for converting a fresh batch of raw phosphate into di-Ca phosphate.

**Hydrated ferric chloride.** Edward T. Ladd (to Isco Chemical Co.). U. S. 2,021,791, Nov. 19. Water and Cl are introduced into a container contg. Fe, in such proportion as to produce a soln. contg. about 4.9 mols. of water per mol. of FeCl<sub>3</sub>, and the product is withdrawn from the container. App. is described.

**Ferroso-ferric oxide.** Joseph W. Ayers. Brit. 433,333, Aug. 13, 1935. Black oxide of Fe is obtained from pptd. ferrous compds. by oxidation with air or other O-contg. gas at elevated temps., e. g., 220-300° T., and at pressures above about 40 lb. and up to, say, 100 lb. per sq. in. The ppt. may be obtained by treating FeSO<sub>4</sub> or FeCl<sub>3</sub> with an alkali carbonate or hydroxide, preferably in excess and pptn. may be at 125-200° T. An autoclave for the pptn. and oxidation is described.

**Lead oxide.** Paul Kemp and Emanuel Feuer. U. S. 2,019,530, Nov. 5. See Brit. 421,297 (C. A. 29, 3292<sup>24</sup>).

**Magnesium hydroxide.** I. G. Farbenindustrie A.-G. Brit. 433,233, Aug. 12, 1935. See Fr. 780,141 (C. A. 29, 6002<sup>29</sup>).

**Removing soluble salts from magnesium hydroxide.** Wm. H. Farnsworth (to Marne Chemicals Co., Ltd.). U. S. 2,019,488, Nov. 5. Filamentary floccs are formed from a hydrous slurry of Mg(OH)<sub>2</sub> (suitably by extrusion through small apertures) and the floccs are caused to be immersed in a body of wash water by which sol. salts are extd. without substantial change in the phys. form of the floccs and without substantial diffusion of the Mg(OH)<sub>2</sub> in the wash water; the floccs are then caused to merge into a substantially undivided mass. An arrangement of app. is described.

**Magnesium sulfate solution.** Kali-Forschungs-Anstalt G. m. b. H. (Benno Wandrowsky, inventor). Ger. 616,543, July 31, 1935 (Cl. 12m. 3). Highly conc'd MgSO<sub>4</sub> solns. are obtained by treating the 33% solns. made by shaking kieserite with water, with a hydrated sulfate, e. g., kieserite contg. epsom salt, at 90-110°. An example is given.

**Platinum oxides.** Paul Lafitte and Pierre Grandadam. Fr. 785,082, Aug. 1, 1935. Pt is oxidized to PtO and PtO<sub>2</sub> by heating it in O to 100-560°, under pressure of 8-200 atm.

**Cyclic manufacture of potassium carbonate from potassium chloride.** Friedrich Rühsberg (to Kali-Chemie, A.-G.). U. S. 2,020,801, Nov. 12. In the cyclic manuf. of K<sub>2</sub>CO<sub>3</sub> from KCl by the Engel-Frech method, the Mg carbonate trihydrate spent in the manuf. of Engel salt in the cycle is compensated by introduction of corresponding amounts of Engel salt into the cyclic process, which is decompd. together with the Engel salt produced in the

cycle, the regenerated MgCO<sub>3</sub> being introduced into the cycle.

**Sodium azide.** Westfälisch-Anhaltische Sprengstoff-A.-G. chem. Fab. Ger. 619,017, Sept. 20, 1935 (Cl. 12a. 30). A fine suspension of NaNH<sub>2</sub> in an inert liquid, e. g., a hydrocarbon oil, is treated with N<sub>2</sub>O at about 150-180°.

**Acid sodium pyrophosphate.** Campbell R. McCullough (to Swann Research, Inc.). U. S. 2,021,012, Nov. 12. Mono-Na orthophosphate is heated to a temp. above 284° and at which substantially complete conversion to pyrophosphate will take place and in an atm. comprising water vapor of such concn. that the partial pressure of such water vapor is greater than atm. pressure and greater than that expressed by the relation  $\log p = \frac{-5263}{T} +$

12.32, where  $p$  is the partial pressure of the water vapor in mm. of Hg and  $T$  is the temp. of heating expressed in abs. degrees C. App. is described.

**Zinc and sodium sulfides.** Thomas A. Mitchell and Royal L. Sessions (to Hughes-Mitchell Processes Inc.). U. S. 2,020,323, Nov. 12. A black ash formed by heating BaSO<sub>4</sub> with C and contg. BaS is leached with a soln. of Na<sub>2</sub>SO<sub>4</sub>; pptd. BaSO<sub>4</sub> is filtered out, and the filtered soln. is caused to react with ZnSO<sub>4</sub> to form pptd. ZnS and leave Na<sub>2</sub>SO<sub>4</sub> in soln. The leached black ash is reheated with more C to produce more Ba sulfide for the process, and the black ash is leached with the Na<sub>2</sub>SO<sub>4</sub> obtained from the pptn. of the ZnS.

**Zinc oxide.** Carl H. Bunce, Clarence J. Lentz and Geo. T. Mahler (to New Jersey Zinc Co.). U. S. 2,021,284, Nov. 19. Various details of app. and operation are described for making ZnO by introducing a blast of air into a stream of Zn vapor and gaseous products of combustion issuing into an oxidizing chamber from the upper end portion of an elongated porous column consisting of an agglomerated charge of mixed zinciferous material and carbonaceous reducing agent, the base of the column being in communication with the atm. and the sides of the column being enclosed. Cf. C. A. 29, 3790<sup>9</sup>.

**Zinc sulfide.** Arne J. Myhren and Byron Marquis (to New Jersey Zinc Co.). U. S. 2,020,325, Nov. 12. See Can. 353,104 (C. A. 29, 8367<sup>9</sup>).

**Zinc sulfide.** American Zinc, Lead & Smelting Co. Fr. 785,594, Aug. 13, 1935. Basic ZnS is made by treating a soln. of ZnSO<sub>4</sub> with an alkali thiosulfate contg. Na<sub>2</sub>S.

**Purifying hydrogen peroxide.** E. I. du Pont de Nemours & Co. Brit. 432,915, Aug. 6, 1935. Aq. solns. of H<sub>2</sub>O<sub>2</sub> are purified by pptg. Sn(OH)<sub>2</sub>, therein after adjusting the  $pH$  of the soln. so that a  $pH$  of over 1.4 is obtained during pptn., and removing the ppt. The pptn. may be effected by adding a sol. Sn compd., e. g., SnCl<sub>4</sub> or Na stannate. If the pptn. of Sn(OH)<sub>2</sub> tends to be incomplete, a small amt. of a sol. substance that furnishes multivalent pos. ions may be added to promote pptn. of Al compds., e. g., AlCl<sub>3</sub>, potash alum, or Ba compd. may be thus used. Cf. C. A. 29, 8241<sup>9</sup>.

**Stabilizing hydrogen peroxide solutions.** E. I. du Pont de Nemours & Co., Harvey N. Gilbert and Joseph S. Reichert. Brit. 433,470, Aug. 15, 1935. H<sub>2</sub>O<sub>2</sub> solns. having a  $pH$  not greater than 0.5 and preferably below 0.5 are stabilized by the addn. of a small amt. of a preformed compd. or compds. obtained by combining Sn with H<sub>2</sub>PO<sub>4</sub>. A suitable compd. may be prep'd by heating hydrated SnCl<sub>4</sub> with 85% H<sub>2</sub>PO<sub>4</sub> for about 1 hr. at 300°. Cf. C. A. 29, 5230<sup>9</sup>.

**Apparatus for distilling hydrogen peroxide from solutions of persulfate.** Gesellschaft zur Verwertung Chemisch-Technischer Verfahren. Fr. 785,876, Aug. 21, 1935.

**Removing acid from hydrogen peroxide solutions.** Joseph S. Reichert (to E. I. du Pont de Nemours & Co.). U. S. 2,021,354, Nov. 19. A H<sub>2</sub>O<sub>2</sub> of  $pH$  less than 1.8 and contg. H<sub>2</sub>SO<sub>4</sub> is treated with Ba(OH)<sub>2</sub> to reduce the acidity, but not to a  $pH$  materially greater than 1.8, pptd. BaSO<sub>4</sub> is sep'd., alkali such as NaOH is added to bring the  $pH$  to 2.2-3.5, and Sn(OH)<sub>2</sub> is pptd. in the soln. and removed with assoc. catalytic impurities.

**Sulfur dioxide.** Metallgesellschaft A.-G. Brit. 432,-



714, July 25, 1935  $\text{SO}_2$  is obtained from refuse  $\text{H}_2\text{SO}_4$ , such as obtained from the refining of mineral oil by introducing it together with carbonaceous fuels and air into a rotary furnace where the fuel and the C content of the refuse acid are eliminated by combustion App is described

Sulfur dioxide Christian J Hansen Ger 616,824, Aug 6, 1935 (Cl 12/ 21)  $\text{SO}_2$  is obtained from gases such as foundry waste gases by washing the gases with a thiocyanate soln. The  $\text{SO}_2$  is then driven from the thiocyanate soln by heating under ordinary or reduced pressure. Preferably  $\text{NH}_4\text{CNS}$  soln is used

Sulfur dioxide recovery from mixed gases Frederick E Lee, Robert Lepsoe and Francis H. Chapman (to Consolidated Mining and Smelting Co. of Canada, Ltd.) U S 2,021,555, Nov 19 For the recovery of  $\text{SO}_2$  and production of cryst. Al sulfate, the  $\text{SO}_2$  in a gas is combined with  $\text{NH}_3$  monosulfite, in one or more absorption stages (the monosulfite soln being obtained from the reaction of  $\text{NH}_3$  with  $\text{NH}_4$  bisulfite soln) and solns resulting are decompd. with  $\text{H}_2\text{SO}_4$  to liberate a pure  $\text{SO}_2$ . Various details of a cyclic process are described.

Sulfur trioxide from acid sludge. Wm C Mast (to Chemical Construction Corp.) U S 2,021,372, Nov 19 Sludge such as that from the use of  $\text{H}_2\text{SO}_4$  for purifying petroleum fractions is subjected to thermal decompn with the production of a gas contg  $\text{SO}_2$ , water and hydrocarbons, water and condensable hydrocarbons are condensed out, sufficient air is introduced for combustion of the hydrocarbons and they are burned in a furnace with sufficient addnl. fuel such as oil to maintain ignition temp. The gases entering the furnace are caused to pass in heat-exchange relation to, but not in direct contact with, the combustion gases, the combustion gases are treated to condense water vapor and the purifier gases are converted in a  $\text{H}_2\text{SO}_4$  converter App is described

Hydrogen N V de Bataafsche Petroleum Maatschappij Ger 616,976, Aug 9, 1935 (Cl 12/ 1 01)  $\text{H}_2$  or gases contg  $\text{H}_2$  are obtained by heating  $\text{CH}_4$  or gases contg  $\text{CH}_4$  to temps above  $800^\circ$  in the presence of catalysts comprising compds of oxides of metals of the Ie group with acid forming metal oxides of the 4th, 5th and 6th groups, of m p over  $800^\circ$ . Examples of the catalysts are titanates, vanadates and tungstates of Ni, Fe and Co

Gases containing hydrogen International Hydrogenation Patents Co Ltd Fr 785,617, Aug 14, 1935 Gases which have been used for the destructive hydrogenation of carbonaceous materials are preliminarily purified and washed at a low temp by means of liquefied hydrocarbons which are gaseous at ordinary temp. The hydrocarbons introduced into the gases by the operation of washing are removed by washing the gases with oils in which the hydrocarbons are readily sol

Purifying hydrogen Lee S Twomey U S 2,022,165, Nov 26  $\text{H}_2$  is liquefied and assoc. impurities such as those from water gas are frozen, the liquefied  $\text{H}_2$  is collected in a pool from which solid particles settle as sediment, and purified  $\text{H}_2$  is evapd. from the upper part of the pool App is described

Krypton and xenon L'air liquide (Soc. anon pour l'etude et l'exploitation des procedes Georges Claude) Brit 432,644, July 31, 1935 Kr and Xe are obtained from atm. air by rectifying air cooled to near its liquefaction point by washing liquid in amt. sufficient to condense practically all the Kr and Xe, but less than sufficient to condense the max. amt. of  $\text{O}_2$ , the washing liquid may be about 10% of the air treated App is described Cf C A. 29, 3792

Lithium recovery from its ores Walter Rosett and Francis R. Buchowsky U S 2,020,954, Nov 12 A comminuted Li ore such as lepidolite is intimately mixed with a Ca-contg. material such as  $\text{CaCO}_3$  which yields  $\text{CaO}$ , at least in part, on roasting, the mixt. is roasted

at  $800$ - $1000^\circ$  until clinkered, the coarse clinker is reduced to a suitable size for leaching, leached with hot water until free of alkali salts, Ca is pptd. as a carbonate, the liquor is filtered from the ppt. and is treated with an acid such as  $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$  or  $\text{HNO}_3$  to form the desired salt of Li

Sulfur Metallgesellschaft A-G Tr. 784,942, July 29, 1935  $\text{SO}_2$  is withdrawn from gases contg. a small amt. thereof by a mixt. of org. bases, such as xylidine, with water, in the presence of substances capable of decomposing the difficultly sol. sulfates of the org. bases formed as side reactions. The  $\text{SO}_2$  is driven off again by heating, caused to react at  $800$ - $1200^\circ$  with a carbonaceous reducing agent, submitted to a complementary treatment at  $400$ - $700^\circ$  by catalysts to destroy attached S compds, and the S is pptd. by a high-tension electrostatic field.

Removing oxygen from gas mixtures Arthur B Ray (to Carbide and Carbon Chemicals Corp.) U S 2,019,632, Nov. 5 For removing O from large volumes of gas mixts. contg. not more than about 4% of O, the gas mixt. is brought into contact with highly reactive carbon heated to about  $600^\circ$  for a time only sufficient to combine substantially all O into compds. other than CO

Rotary furnace for producing carbon by catalytic decomposition of carbon monoxide. Georg Gros and Theodor W. Pfirrmann Ger 619,137, Sept. 23, 1935 (Cl 23f. 14) Addn. to 565,053 (C. A. 27, 1110)

Carbon black Edmund Billings and Harold H. Offutt (to Godfrey L. Cabot, Inc.) U S reissue 19,750, Nov 12 A reissue of original pat. 1,957,314 (C. A. 28, 4190\*)

Active carbon Franz Krczil Austrian 142,780, Sept. 10, 1935 (Cl 85c). In the manuf. of active C by carbonizing crude org. materials contg. water, e. g., peat or nutshells, and activating the carbon by means of gases, use is made of a system comprising a no. of activating vessels, the gases contg. water vapor evolved from the first vessel of the series being passed through the other vessel or vessels without intermediate condensation

Active charcoal Franz Krczil Fr. 785,427, Aug 9, 1935 Wood, sawdust, lignin or peat is impregnated with an activating agent such as a soln. of  $\text{ZnCl}_2$  or  $\text{H}_3\text{PO}_4$ , and is submitted in the presence of a carbonizing substance such as  $\text{H}_2\text{SO}_4$  to a temp. above  $100^\circ$  until a moldable mass is obtained. The mass is molded under pressure, and heated if necessary, dried and baked at activation temp., if desired in the presence of activating gases

Adsorbents Moric Pickl Austrian 142,753, Sept. 10, 1935 (Cl 12d). Raschig rings made from compressed hygroscopic salts are used as adsorbents for drying gases. Raschig rings of compressed silica gel are used for drying gases or for recovering volatile solvents

Porous sound absorbing material Dorne N. Halstead U S 2,021,350, Nov 19 Porous articles such as sound-absorbing pads contain a binder such as starch, bitumen or cement and aggregations of felted fibers which are waterproofed and have voids between them. Various details of manuf. are described

Catalytic material Paul S. Greer (to Carbide and Carbon Chemicals Corp.) U S 2,020,411, Nov. 12 For producing a catalyst comprising a metal such as Cu or Ni supported on inert carrier material such as siliceous material, the carrier is immersed in a hot soln. of a sol. metal salt such as  $\text{Cu}(\text{NO}_3)_2$ , coned to a point of incipient crysln, then drained and dried, roasted with a blast of preheated air to convert the salt material to oxide and the material may then be subjected to reduction to complete the formation of the desired catalyst

Catalytic gas reactions Studien- und Verwertungs-Ges. m. b. H. Fr. 784,885, July 27, 1935 The catalytic mass is divided into thin layers of uniform thickness and the reaction takes place in spaces formed by the exterior walls of chambers contg. a circulating liquid used to control the temp.

## 19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

C. K. BARTON AND C. H. ASBEE

Fundamental research in glass. Bernard P. Dudding. *Ceram. Ind.* 24, 342 (1935).

The inaugural lecture of the course on the physical chemistry of glass at the University of Liege. P. Gilard. *Verre silicaté* vol. 6, 414-19 (1935).—A discussion of the value of phys. chemistry in glass making. H. S. W.

The kinetics of the photochemical change of glasses through ultraviolet radiation and their regeneration by heat. A. Klemm and E. Berger. *Glastech. Ber.* 13, 349-68 (1935).—The solarization of glasses and regeneration by heat appear to be reversible processes which follow the same law. The change in transmission on solarization with the logarithm of time is approx. proportional to the 4th power of the abs. temp. It is also proportional to the difference of the initial and final transmissions and depends also on the wave length. The logarithm of the time necessary to reach the final value likewise depends on the wave length and is proportional to the 4th power of the abs. temp., decreasing rapidly with increasing temp. Thermoluminescence is not observed in Ce-contg. glasses and is therefore not essential to the regeneration processes. Thirty-one references. J. I. Hyde.

Chemical properties of glass. Geo. W. Moore. *Ceram. Ind.* 19, 203 (1935).

The use of cadmium sulfide and selenium in making ruby glass. Wm. J. Mathews. *Ceram. Ind.* 24, 334, 336, 342 (1935).

Combination furnace for flat glass. Thomas B. Hart. *Ceram. Ind.* 24, 340 (1935).

Colored fire-polished opaque glass. Rudolf Hehlbaum. *Spektrum* 67, 731-3, 747-9 (1934).—The polished surface of the glass is a natural polish which is assumed as the glass solidifies. This surface is harder and more resistant to mech. and chem. action than glass polished mechanically. The density of the surface is such that moisture will not penetrate. Fire-polished opaque glass was found quite resistant to the action of acids, alkalis, etc. Other properties of this glass are high resistance to pressure, good adherence to mortars and ease of production. Many uses and applications are suggested. The av. compn of this glass is given as  $SiO_2$  72,  $CaO$  3,  $Al_2O_3$  5,  $NaO$  15 and  $CaF$  and the remainder 2%. Manuf. and production of this glass are described. C. B. Jenni.

A study of sagger clays and sagger bodies. Raymond A. Hendl. *J. Research Natl. Bur. Standards* 15, 235-70 (1935) (Research Paper No. 827). cf. C. A. 28, 2781.—*Filter-press clays and 22 conc. bodies have been tested for thermal and mech. properties. Any sagger body having a total linear expansion from room temp. to 250° of 0.15% or greater cannot be expected to have a high resistance to thermal shock. The life of the sagger is more sensitive to changes in thermal expansion than to changes in extensibility or strength. Great increases in modulus of elasticity may accompany changes in gross sizes from coarse to fine, or a higher temp. of heating, with but little change in porosity. Porous grog, or loosely bonded grog, imparts greater resistance to thermal shock than dense grog. Plastic deformation at 1000° is less in fine-grogged bodies and decreases with increase of heating temp. Adding  $MeO$  either as talc or as magnesite leads to increased sagger life, but the amt. must be limited.* P. S. Roller.

Carbon brick. M. A. Uralov and A. S. Berezhnii. *Ukrain. Nauch.-Issled. Inst. Ozerapovne Khimichesk. Uprav. No. 31*, 76 pp (1934); *Referativnyi Sbornik* No. 1372 (1935).—Details of production are given. The typical compn. is: coke 80, tar 15.7, anthracene oil 3.6 and bitumen 1.7%. The brick are fired in special furnaces or in saggers in ordinary furnaces with overhead flame to 1100° and in some cases to 1430°. Specifications of carbon brick are given. M. V. Kondodov.

Production of bottom brick for Ajax electric furnaces. *Referativnyi Sbornik* No. 1098 (1934).—Ural font contg.  $SiO_2$

97.45,  $Al_2O_3$  0.90,  $FeO$  1.27,  $MgO$ ,  $CaO$ ,  $TiO_2$  traces and loss on ignition 0.04% was used for refractories for Ajax furnaces. They withstood 2000 brass and red brass melts. Details of production are given. M. V. K.

Production of dynamite brick from bauxite. N. P. Kuchel. *Tekhn. Zh.* 7099, Ser. 70, 4 pp., *Referativnyi Sbornik* No. 1224 (1935).—Tikhvin bauxites contg. hydromillite and diaspore were used in the production of dynamite brick. The brick were made from raw and calcined bauxite, and fired to 1335° for 3 hrs. They did not shrink on firing, had a 10% water absorption, and a resistance to pressure of 630 kg./sq. cm. Their resistance to sudden temp. changes was much greater than that of grog and silica brick. These brick were tested as refractories for Krupp furnaces and contained 30% corundum after use, the rest being grog (kaolin) and bonding clay. The abrasion loss was small. The softening under load began at 1530°; at 1690° the brick contracted 4%; the refractoriness was 1820°. M. V. Kondodov.

Destruction of grog brick by carbon monoxide. P. P. Dudnikov. *Novyi Izhivis* No. 26, 11 (1934); *Referativnyi Sbornik* No. 612 (1934).—It was found that (1)  $Fe_2O_3$  in the clay changes into oxide when fired. (2) Iron oxide acts as a catalyst during the reaction  $2CO \rightleftharpoons CO_2 + C$  and promotes the course of reaction to the right side.  $Fe_2O_3$  is reduced to  $FeO$ . The pptd. C deposits in the pores of the brick and destroys them. The iron spots show the beginning of the destruction. (3) The oxidation of  $CO$  to  $CO_2$  with the pptn. of C occurs at 450° to 500°. At higher temp. the equil. moves to the left side of the equation and C does not ppt. (4) With a definite iron content and degree of multilayering, the vol. porosity becomes an important factor in the stability of the products. (5) An addn. of small amt. of  $CuSO_4$  to the mix increases the resistance of grog brick to the effects of  $CO$ . It is recommended therefore: (a) to reduce the iron content of the brick to 1 or 1.5%, (b) that the remaining iron be uniformly distributed in the brick; (c) porosity should be not over 18%; (d) the brick should be fired between 1300° and 1350° in a reducing atm.; the  $FeO$  formed increases the d. of the body and promotes the formation of mullite. A "protective liver" is thus formed which increases the chem. resistance of the brick. M. V. Kondodov.

Optical instruments in the study and control of ceramics. Everett W. Melson. *Ceram. Ind.* 24, 263-2, 264 (1935).

Special ceramic bodies. Marcel Leungue. *Verre silicaté* vol. 6, 337-9, 372-4, 383-6 (1935).—Properties and uses of such refractories as  $Al_2O_3$ , corundum, spinel,  $Be$ ,  $ZrO_2$ ,  $MeO$ ,  $ThO_2$  and  $ZrSiO_4$  are given. H. S. W.

Use of syenite in ceramic bodies. C. J. Koenig. *Ceram. Ind.* 24, 338-9 (1935).—Bibliography.

Causes of reddish and other discolorations in ceramic bodies, which can be removed by a second firing. Günter Hammer and Lotte Schulz. *Fer. dest. keram. Ges.* 16, 70-3 (1935).—Addnl. evidence is presented in support of the chlorination theory. Cl impregnates the body and does not combine with the body material. H. G. S.

Drying problems of the ceramic industry. John L. Carruthers. *Trans. Am. Soc. Mech. Engrs.* 57, 489-92 (1935).

Red and yellow colors in ceramics. I. Red colors. H. Kohl. *Fer. dest. keram. Ges.* 16, 103-9 (1935).—The Ostwald standards are used in a study of ceramic colors. These standards are compared, the Pt-Bk photometer is used. All ceramic reds were remarkable because of their high content of black and their low content of full color, which seldom exceeded 50% even with selenium red. Copper reds, pinks, chrome-alumina pinks, reds contg. neodmium phosphate, high-fire porcelain pinks, uranium reds and enamel colors, iron reds, purple, coral reds and cadmium-selenium reds were analyzed. Selen-

ium reds were found to be more resistant to acids than coral reds. Iron reds showed a moderate resistance. Ordinary purples were found to be good. Special acidproof purples showed complete resistance. H. G. Schurecht

Ceramics in the telephone. A. G. Johnson and L. I. Shaw. *Ind Eng Chem* 27, 1326-32 (1935) G. G.

Teeth from ceramic material. R. Riecke and W. Bilke. *Ber. deut. keram. Ges.* 16, 91-104 (1935) —Bodies used for false teeth are incorrectly called porcelain, they are composed of mixts of feldspar and quartz. The relations between compns and phys properties were studied. In general, in pure feldspar-quartz frits the thermal expansion increases with an increase in feldspar contents with a min at 40% quartz. Substitution of quartz sand for gang quartz resulted in a slight increase in thermal expansion, probably because of the lower soly. of the sand. Addns. of 5% uncalcined kaolin increased the expansion slightly. Kaolin fritted with the body reduced the expansion in proportion to the amt added. Adding unfritted feldspar to the fritted body produced no effect on expansion. Rapidly cooled bodies showed marked stresses as compared with slowly cooled bodies, the crit. temp. being about 700°. Increasing the quartz content and substituting sand for gang quartz reduced the transparency in the pure feldspar-quartz frits. The addn. of kaolin and of unfritted components produced a similar effect. Mech strength was max with about 40% quartz in feldspar-quartz mixts. The strength was reduced by adding sand instead of quartz, unfritted kaolin or feldspar. Increased strength was usually accompanied by a decrease in translucency. H. G. Schurecht

Alsmag 196—a new ceramic insulating material for high frequency purposes. Hans Thurnauer. *Radio Eng* 15, 15-16, 20 (1935) —A group of steatite materials possessing accuracy of dimensions and low cost has been developed. Steatite is a ceramic material based on the minerals lava, talc or soapstone, which differ principally in cryst. structure. The finely pulverized raw materials are mixed with fluxes and dried or fired as required. The steatite bodies possess high elec resistance and very low dielec loss. The phys and elec properties of various materials used as insulators for high frequency purposes are tabulated. In Alsmag 196 porosity has been cut down to nil even in pressed pieces, the power factor is nearly as low as that of transparent fused quartz. Uses for this new steatite product include receivers and transmitters where high-frequency app., low dielec losses and high dielec strength are required. W. H. Boynton

Effect of grain size and of firing schedule on properties of feldspar as a vitrifying agent. Edmund A. Durbin and Arthur S. Watts. *Ceram. Age* 26, 135-6 (1935) —Decreasing the grain size of the feldspar produces increased vitrification in a whiteware body. The more gradual the temp. increase, the more nearly the glassy phase represents a satn. of clay and flint in the feldspar solvent. Herbert S. Willson

Air-compressor faults. E. I. Lloyd. *Glass* 12, 461-2 (1935). Herbert S. Willson

Refractory clays of Ohio. E. J. Bognar. *Ceram. Age* 26, 183-6, 194-5 (1935). Herbert S. Willson

German raw materials for the preparation of magnesia refractories. A. Berge. *Keram. Rundschau* 43, 471-3 (1935) —The utilization of carnallite is described. P. S. Roller

Silicon carbide refractories and their application in boiler furnaces. J. Walker. *Steam Eng* 3, 341-2 (1934), *Referatkarles Silikaliteratur* No. 713 (1934) —SiC refractories are excellent for boiler furnaces. They do not deform at high temp. under load, they resist the attack of slag with a low Fe or Ca content, however, they are attacked by liquid slag high in basic oxides. Because of their hardness they resist erosion by flue gases. M. V. Kondoidy

Chromite lining for industrial furnaces. Grigor'ev. *Zhurn.-So Ser* 79, No. 1828, *Referatkarles Silikaliteratur* No. 624 (1934) —For the production of chromite refractory mortar, chromite (80 to 95%) is mixed with aluminous cement (5 to 2%) and a little water. This mixt shows a

high refractoriness, good resistance to pressure, considerable chem. resistance and does not shrink. M. V. K.

Caustic magnesia lining for arc furnaces. V. Bogolubov. *Novosti Tekhniki* No. 37, 1-2 (1934); *Referatkarles Silikaliteratur* No. 606 (1934) —In contrast with sintered magnesite (firing temp. 1615° to 1750°), caustic magnesia (fired at 800° to 1000°) shows hydraulic properties. Chemically and physically the 2 kinds are similar with the exception that caustic magnesia has a finer crystal lattice.

Its advantage lies in the fact that it crystallizes more easily than sintered magnesite. The rammed linings, used in electrometallurgy, contg. tar, molasses or water glass are unsatisfactory. The Moscow Steel Inst. developed a rammed mix for arc furnaces contg. 80% sintered and 20% caustic, which recrystallizes with every melting and changes finally into a monolithic structure resisting highly oxidizing melts. Such linings were used successfully in Herault arcs for melting steel and ferrochrome. M. V. K.

Test for refractory for glass melting pots. E. Bayer. *Keram. Rundschau* 43, 544-5 (1935) —A 5-cm. cube of refractory is placed under a small load in a crucible contg. glass at 1000-1300°. Characteristic differences are found in the deformation, speed and degree of soln. P. S. Roller

Gas permeability of refractories. N. Lomskii and S. German. *Ukrain. Nauch.-Issledovatel. Inst. Ognenoporo. Kisloloporo. 30*, 24 pp. (1934), *Referatkarles Silikaliteratur* No. 1000 (1934), cf. C. A. 29, 1221\* —A simple app. for measuring gas permeability of refractories is described. The coeff. of gas permeabilities at room temp., of grog and silica brick, fluctuated between 1.40 and 3.919 and 0.146 and 0.807, resp. The difference in pressure was not more than 6 or 7%. Attempts were also made to det. gas permeability between 20° and 8000°. It was found that an absolutely uniform heating of the sample is required when measuring permeability at high temps., that the temp. of the gas must be detd. before its conduction through the product, and that loss of gas must be prevented. M. V. Kondoidy

Sintering and melting processes in porcelain glazes. R. Riecke and C. Tanne. *Ber. deut. keram. Ges.* 16, 147-58 (1935) —The porosities and linear shrinkages of glazes were detd. for different firing temps. The whole melting process, the sintering temp. and the duration of the sintering temp. were found to be as important as the melting temp. The melting behavior of a glaze of a given chem. compn. may differ considerably according to the raw material used. This point was studied on 5 glazes with different chem. compns and compounded with different amts. of feldspar, marble, kaolin and sand. H. G. S.

Insulator porcelain testing. Horst v. Treufels. *Ber. deut. keram. Ges.* 16, 74-80 (1935) —Porcelain insulators were shown to develop fatigue. H. G. Schurecht

Pin holes in glazed ware. Maurice Barrett. *Brit. Clayworker* 44, 226-8 (1935) —Defects may be caused by the following: gas evolution from a sulfate radical, high Zn content, plaster from the molds, org. matter, high Al<sub>2</sub>O<sub>3</sub> content, Fe, pyrites or SnO<sub>2</sub>. The kiln should be free from CO<sub>2</sub> or steam. Finer grinding of the clay and grog will reduce the tendency to pin hole. Dust and dirt should be avoided. BaCO<sub>3</sub> can be used to an advantage. Washing with H<sub>2</sub>O and the application of compressed air are effective as remedies. E. C. Petrie

Opacifiers used in the vitreous enamel industry. W. A. Liles. *Foundry Trade J.* 53, 351-4 (1935) D. S.

Pickling in enameling. M. Pecraux. *Verre industriel* 5nd 6, 428 (1935) Herbert S. Willson

Adherence of ground enamels containing cobalt and nickel oxides. Friedrich Heimes. *Ceram. Age* 26, 480-2 (1935), cf. C. A. 29, 3478\*. Herbert S. Willson

Specifications for enameled cooking vessels. Richard Aldinger. *Keram. Rundschau* 43, 431-3, 498 (1935) —Resistance to heat, boiling solns. and impact is discussed. Cf. C. A. 28, 6961\*. P. S. Roller

Glass-lined equipment for the food industries (Barnes) 12 Kaolin and clay (Vasel) 8 Kinetics of solid phase reactions of certain carbonates with mullite, silica and

alumina (Pole, Taylor) 2. App. for sepg. dust and other light material from heavier matter, e. g., from abrasive after its use in sand-blast app. (Brit. pat. 432,222) 1.

Glass. Erich Heinz. U. S. 2,020,467, Nov. 12 In compounding glasses of the usual soda-lime type, there is added to the usual glass-batch ingredients a prefused Ba borosilicate flux having a compn. which comprises about 8-20% of  $B_2O_3$  and about 15-30% BaO together with silica in proportions at least chemically equiv. to the  $B_2O_3$  and BaO.

Glass Corning Glass Works. Fr. 784,667, July 22, 1935. Glass useful for rods and mountings of elec. lamps contains 2-3 times as much  $K_2O$  as  $Na_2O$ , the proportion of  $B_2O_3$  is 1-1.1 and BaO 6.5-7.3%. An example contains  $SiO_2$  55-60,  $Na_2O$  3-4,  $K_2O$  19-21.5, BaO 6.5-7.3 and  $B_2O_3$  1-1.1%.

Cellular glass Soc anon des manufactures des glaces et produits chimiques de St Gobain, Chauny & C<sup>ie</sup> Brit. 433,423, Aug. 14, 1935. Articles of cellular glass are made by placing molten glass that is seedy or bubbly in a mold and creating a partial vacuum in the mold whereby the glass is caused to swell and take the shape of the mold. App. is described.

Apparatus for circulating the glass in gathering basins of glass furnaces Alexander F McNish U. S. 2,021,013, Nov. 12 Structural, mech and operative details.

Automatic glass-gathering and -shaping machine for manufacture of bottles or the like Etienne Fechoz U. S. 2,020,462, Nov. 12 Mech and operative details.

Apparatus for feeding mold charges of molten glass Edwin E. Slack, Jr. U. S. 2,021,623, Nov. 19 Various mech and operative details.

Apparatus for feeding molten glass to molds Geo R. Haub U. S. 2,020,143, Nov. 5 Structural, mech and operative details.

Apparatus for gathering glass blanks for the manufacture of bottles or jars August Kadow and Willard L. Van Ness (to Libbey Glass Mfg. Co.) U. S. 2,020,032, Nov. 5. Mech and operative details.

Forming hollow glass blanks such as bottles or jars August Kadow (to Libbey Glass Mfg. Co.) U. S. 2,020,031, Nov. 5. Various details of app. and operation.

Apparatus for manufacture of hollow glass products such as bottles or flacons. Emile Rourant (to Soc. anon. d'études et de constructions d'appareils mécaniques pour la verrerie) U. S. 2,021,670, Nov. 19. Various mech and operative details.

Evacuated blown glass articles such as double-walled jars Roy E. Swan. U. S. 2,020,590, Nov. 12. Various details of app. and operation are described.

Means for feeding predetermined weights of molten glass. John Thomas Wood. Ger. 619,306, Sept. 28, 1935 (Cl. 32a. 5). This corresponds to Brit. 389,923 (C. A. 27, 5918).

Fitter or shaper for glass articles suitable for trueing glass tumblers. John C. E. Schwab (to Hazel-Atlas Glass Co.) U. S. 2,019,736, Nov. 5. Mech features.

Marvering glass to fit a mold prior to entering the mold Thomas Stenhouse (to Hazel-Atlas Glass Co.) U. S. 2,020,623, Nov. 12. A charge of glass substantially circular in cross section is dropped vertically and passes by its own momentum through a shaping die which shapes it to fit a mold, as in the formation of articles such as bottles. App. is described.

Glass-working apparatus suitable for fusing tubes into lamp bulbs, etc. Alvin R. Knoepfel (to General Elec. Vapor Co.) U. S. 2,020,729, Nov. 12. Mech. and operative details.

Apparatus for supporting glass plates while they are case hardened Lloyd V. Black (to Pittsburgh Plate Glass Co.) U. S. 2,019,595, Nov. 5. Structural and mech features.

Apparatus for edging glass sheets and plates by grinding. Wm. Owen (to Pittsburgh Plate Glass Co.) U. S. 2,021,193, Nov. 19. Structural, mech. and operative details.

Glass furnaces Fours et appareils Stein (Soc. anon.) and Charles Royer. Fr. 785,143, Aug. 2, 1935.

Electric furnace suitable for melting glass. John Ferguson. U. S. 2,022,112, Nov. 26. Structural features. Induction heater for fusing and refining glass Forges et ateliers de constructions électriques de Jeumont. Fr. 785,564, Aug. 13, 1935.

Canal cooling furnace for glassware. Otto Maetz and Hermann Heinrichs. Ger. 616,606, Aug. 1, 1935 (Cl. 32a. 29).

Drawing kiln and associated apparatus for sheet-glass manufacture Karl G. Kutchka (to Pittsburgh Plate Glass Co.) U. S. 2,021,180, Nov. 19. Various structural and operative details.

Leer, with heating, tempering and cooling zones for treating glassware. Geo. W. Batchell. U. S. 2,019,839, Nov. 5. Structural and mech features.

Apparatus for tempering sheets of glass. Compagnie réunies des glaces et verres spéciaux du nord de la France. Fr. 784,733, July 23, 1935.

Mirrors Duro-Ray Ltd. Fr. 785,270, Aug. 6, 1935. Colloidal Ag is deposited on the surface of glass, then a film of Cu and finally a protective coating of Pb.

Silvering Duro-Ray Ltd. and Albert M. Barnes Brit. 432,687, July 30, 1935. In silvering glass or similar transparent or translucent materials, films of colloidal Ag only are formed to a predet. linear thickness on their surfaces to facilitate the electrodeposition of metals for protective purposes. Sufficient  $NH_4OH$  is added to an aq.  $AgNO_3$  soln. to redissolve the pptd.  $Ag_2O$ , an aq.  $NaOH$  soln. is added and sufficient  $NH_4OH$  to redissolve any pptd.  $Ag_2O$ , the mixt. is added to a concn. corresponding to 5 g.  $AgNO_3$  and 4.68 g.  $NaOH$  per l. and equal vols. of the resultant soln. and an aq. soln. of cane sugar and tartaric acid are fed through nozzles so as to mix to form a single jet which falls on the glass, etc. The Ag deposit is backed by an electrolytic deposit of Pb. The electrolyte may be an aq. soln. of  $Pb(ClO_4)_2$ , contg. 37.5 g. Pb and 0.05 g.  $HClO_4$  per l. Clove oil or peptone may be added to reduce the grain size of the deposited Pb to a min.

Colored optical glass Murray R. Scott (to Bausch & Lomb Optical Co.) U. S. 2,021,244, Nov. 19. A colored optical glass which is suitable for spectacle lenses has a  $n$  of 1.50-1.55 and a dispersion, as expressed by  $\nu$ , between 61.0 and 58.5 and contains compds. of Pb, Mn and Ti.

Glass permeable to ultraviolet rays. Chemische Fabrik Joh. A. Benckiser G. m. b. H. and Fritz Draibach. Fr. 785,966, Aug. 23, 1935. Orthophosphates of Be, Ca, Mg, Na or mixts. of these phosphates are used exclusively in making glass of good permeability to ultraviolet rays.

Laminated ("safety") glass Jean Haas (to Société générale d'optique (Soc. anon. des anciens établissements Huot & Cie. et jumelles Flammarion)). U. S. 2,020,178, Nov. 5. A viscous material is forced upwardly under pressure between glass sheets to be united. App. is described.

Safety glass Deutsche Celluloid-Fabrik. Fr. 785,422, Aug. 9, 1935. Plates of glass are coated by projection with an aq. paste or emulsion of a polymerization product, the water is removed and the plates of glass are brought together and joined by heat and pressure.

Purifying clay. English Clays, Lovering, Pochin & Co. Ltd. and Reginald J. Davies Brit. 433,416, Aug. 14, 1935. A dil. soln. of sulfite lye with or without an addn. of alk. electrolyte is used as a deflocculating agent in the purification of clay.

Molding clay pipes, etc. Wm. M. W. Sutton. Brit. 432,579, July 30, 1935. Clay and similar single-piece pipes, conduits, etc., are formed with holes or slots in the thickness of the walls so that when the pipe is subsequently fired, the slots provide air ducts for circulation of hot air and so reduce the time required for firing.

Fire-clay sewer pipe, etc. Willard K. Carter and Geo. H. Duncombe, Jr. (to National Aluminate Corp.). U. S. 2,019,619, Nov. 5. Fire clay is tempered with water contg. an amt. of Na aluminate not more than about 1% the wt. of the clay and the resulting plastic mixt. is molded, dried, and fired at a temp. equiv. to about cone 8.

Firebrick John M. McKinley and Willard K. Carter

(to National Aluminate Corp.). U. S. 2,019,618, Nov. 5. Highly heat-resistant, strong firebrick of decreased porosity and firing shrinkage and of good resistance to spalling are produced by mixing fire clay with aggregates such as ground brickbats and an amt. of Na aluminate substantially equal to 1-5% the amt. of the clay, and with water to form a plastic mass, shaping, drying and firing.

Coloring bricks Wm S Damhorst U. S. 2,020,137, Nov. 5. The cavity walls of a brick-forming mold (as used in connection with the sand-mold process of making soft-mold brick) are covered with a relatively thin coat of a mix formed of fine sand, fire-clay dust and coloring materials such as "manganese," "metallic red" or powder Zn (this mix being applied while dry to the wet mold walls) and plastic clay is forced into the mold cavity under heavy pressure (various other details of treatment and final burning being also described).

Tamping-mechanism for brick making machines. Wm G Chapman and Dunns' Engineering Works Ltd. Brit. 433,187, Aug. 9, 1935.

Rotary mold-table machines and method for molding bricks, tiles, etc. Geo Head Brit. 432,799, July 29, 1935.

Apparatus for applying color to tiles, etc. Harry F Wood and Wm Coulter. Brit. 432,193, July 18, 1935. The tiles are carried along on a horizontal band or conveyor beneath 1 or more color containers having a plurality of color outlet nozzles from which small quantities of color are deposited on the tile surfaces, the color, before it sets, being subjected to an air or like blast from a plurality of nozzles so as to spread the color or to mix different colors on the surface of the tiles.

Ceramic articles. Walter J. Scott (to Western Elec. Co.) U. S. 2,020,476, Nov. 12. An article such as a telephone dial number plate is formed with a base having a groove in such proximity to an edge as to form a sharp ridge at the edge and with a ceramic coating extending to the ridge. The base may be formed of iron or steel plated with Cu and Ni.

Ceramic articles such as telephone dial number plates. Walter J. Scott and Sumner R. Mason (to Western Elec. Co.) U. S. 2,020,477, Nov. 12. A ferrous metal base is plated with Cu, then plated with Ni, and an enamel coating is applied over the Ni.

Red glaze for ceramic products. Andrew Malnovsky and Albert L. Bennett (to Mahinite Products, Inc.) U. S. 2,020,559, Nov. 12. A brilliant glaze is provided by applying a glazing material containing a selenium stain and a frit containing  $B_2O_3$  about 7.5-15 and  $PbO$  about 10-22%, not more than about 5%  $Al_2O_3$ , and practically free from Sn, the balance being mainly  $SiO_2$ , and then heating to cause the glazing material to reach a maturing temp in a time between 10 min and 4 hrs, then cooling.

Bismuth salt. Le progrès scientifique. Fr. 785,713, Aug. 7, 1935. A bismuth salt which is soluble in mineral and fat oils in all proportions is made by causing a Bi salt or oxide to react with diallylactic acid at about 40°. The product may be used in cosmetics for making colors called "Luster" and in the prepn of cosmetics.

Fusing metals into pottery. Karl Schindhelm. Ger. 613,327, May 17, 1935 (Cl. 80b 10 02). Adds to 610,422 (C. A. 29, 6099\*).

Tunnel kiln. Warmestelle Steine und Erden G. m. b. H. (Karl Beckenbach, inventor). Ger. 612,524, July 20, 1935 (Cl. 80c 5). A kiln or furnace for firing earthenware, roasting or sintering ores, etc., is described.

Continuous tunnel kiln suitable for treating ceramic ware. Philip D. H. Dressler (to Swindell Dressler Corp.) U. S. 2,020,140, Nov. 5. Structural and operative details.

Kiln for drying and burning bricks, tiles, etc. Alois Habla U. S. 2,020,641, Nov. 12. Structural and operative details.

Preventing formation of carbon deposits on bricks of regenerators, etc. Hans Wolf and Hermann Leuchs (to I. G. Farbenindustrie A.-G.). U. S. 2,020,713, Nov. 12. Ceramic masses such as those used for regenerators contain Fe or Fe oxide and also contain a Cu compd cor-

responding to a Cu content in the material of not over 0.2% (suitably about 0.12%).

Electric furnace suitable for producing fused refractory and abrasive materials. Raymond C. Benner and Geo. J. Easter (to Carborundum Co.). U. S. 2,021,221, Nov. 19. A carbon resistor is embedded in a charge of material which is to be fused by heat from the resistor, the resistor being surrounded by a high-frequency elec. heating coil for heating its central portion and being connected with leads for passing an elec. current of lower frequency through the carbon to heat its terminal portions preferentially when it is desired to release it from the charge of material. Various operative details are described.

Refractory compositions. Camillo Konopicky. Brit. 432,303, July 24, 1935. Sintered ferruginous  $MgO$ , in which the total quantity of fluxing agents is greater than 2 but not greater than about 12%, is produced by adjusting the relative proportions of  $CaO$ ,  $SiO_2$ , and  $Al_2O_3$  in the ferruginous  $MgO$ , e. g., by adding these oxides or  $MgO$ -material contg. them, so that the said proportions fall within an area indicated on a ternary diagram and then firing the mixt. The  $Fe_2O_3$ , preferably, amounts to at least 1.5 times the  $CaO$ . Among examples, 1.5 parts of a sintering magnesite contg.  $CaO$  3.4,  $SiO_2$  0.7,  $Al_2O_3$  0.7 and  $Fe_2O_3$  9.1% is added to 1 part of a magnesite contg.  $CaO$  0.4,  $SiO_2$  3.2,  $Al_2O_3$  0.6 and  $Fe_2O_3$  0.2%.

Refractory materials. Harbison-Walker Refractories Co. Fr. 784,832, July 25, 1935. Between the grains of magnesite refractory material is formed a binder containing a network of forsterite in amt. such that the material is made mechanically resistant to high temps as compared with material not containing the network. The forsterite is formed *in situ* on heating.

Siliceous refractories. John M. McKinley and Willard K. Carter (Carter to National Aluminate Corp., and McKinley to North American Refractories Co.) U. S. 2,019,542, Nov. 5. Gansiter particles are mixed with an aq. dispersion of Ba acetate, hydroxide, nitrate, peroxide or carbonate and the mixt. is molded, dried and fired to produce a product which is suitable for furnace linings, etc.

Refractory material suitable for metallurgical furnaces. Geo. D. Cam (to Republic Steel Corp.) U. S. 2,021,222, Nov. 19. About 5-15 parts of olivine contg.  $MgO$  about 48,  $SiO_2$  42 and  $FeO$  2-20% is used with about 5-15 parts of chromite contg.  $FeO$ ,  $Cr_2O_3$ , and about 5-15 parts of calcined dolomite contg.  $CaO$  and  $MgO$ .

Refractory cylindrical filter suitable for filtering liquid in refrigerating systems. Walter A. Kuenzli and Clyde E. Ploeger (to Servel, Inc.) U. S. 2,021,452, Nov. 19. Various structural details.

Porous articles such as plates for surface combustion. Reinhold Reichmann (to Siemens & Halske A.-G.) U. S. 2,021,520, Nov. 19. Prefired refractory material is sintered with grains of the material, the sintering mixt. is mixed with a slip consisting of finely divided calcined metallic oxide material activated with dil. acid to form a moldable crumbly mass, this material is molded under pressure and sintered at a temp. above 1600°.

Refractory and abrasive materials. The Carborundum Co. Fr. 784,820, July 25, 1935. A product is obtained by fusing  $Cr_2O_3$  and  $Al_2O_3$  to form a solid soln. The content of  $MgO$  present should not be above 1.5%. Cf. C. A. 29, 7037\*.

Coated abrasive articles. Richard Paul Carlton (to Minnesota Mining and Manufacturing Co.) Brit. 433,128, Aug. 6, 1935. In mfg. sand paper, emery cloth, etc., particles of the abrasive are propelled into position by subjecting them to the combined action of mech. and elec. or magnetic forces which move them against the force of gravity so that they are deposited with their longer dimensions perpendicular to the surface of the article. App. is described.

Abrasive articles such as leather-buffing wheels surfaced with embossed abrasive fabric. Oliver Lanier (to Carborundum Co.) U. S. 2,021,160, Nov. 19. Structural details.

**Enamel.** Vereinigte chemische Fabriken Kneidl, Heller & Co. Ger. 610,534, July 31, 1935 (Cl. 48: 2 02). A turbidity-producing agent for enamel is obtained by fusing  $ZrSiO_4$  with an equimol. amt. of  $CaO$ ,  $SrO$ ,  $BaO$ ,  $MgO$  or  $ZnO$ , and a flux and grinding the cooled product. A readily fusible glass may be used as flux.

**Enamel ware.** Oscar Hommel (to Enameters Guild, Inc.). U. S. 2,019,676, Nov. 5. Material such as a steel utensil for cooking is provided with an enamel coating of wavy surface and variegated color by covering the surface of the article with a coating of a plurality of vitrifiable preps., of which one is a feldspathic glass of sp. gr. of about 2.5 and one color and another consists essentially of a metallic salt, such as  $Pb$  borate or silicate, of a sp. gr. exceeding 3, and of another color (the coating preps. being applied in variegated distribution, and fired).

**Porcelain enameled articles of changeable color.** Richard H. Türk. (to Porcelain Enamel and Mfg. Co. of Baltimore). U. S. 2,021,819, Nov. 19. A coating of enamel is applied to a base, and enamel frit particles are applied to the enamel coating, in one direction against the facets of the enamel particles there is applied an enamel of one color stable at the firing temp., and there is then applied in a reverse direction against the reverse facets of the enamel particles an enamel of another color stable at the firing temp.; a light dusting of another enamel is applied,

and the article is fired so that it retains a rough surface and changeable color.

**Vitreous material.** Albert Wm. H. Wedlock and Mylex (Parent) Co. Ltd. Brit. 432,421, July 26, 1935. A vitreous material that adheres firmly to metal is prep'd by admixing a dried and finely ground ceramic material, which must contain a proportion of cryolite (I) and alkali carbonates, with mica and (or) asbestos. A preferred ceramic compn. is 1  $20$ ,  $B_2O_3$ , (or),  $K_2CO_3$ ,  $13$ ,  $Na_2CO_3$ ,  $0$ ,  $H_2BO_3$ ,  $45\%$ . This is dried at  $500^\circ$ , the resulting frit is ground to pass a 120 mesh per linear sieve, the ground frit is mixed with mica ground to the same fineness in the proportion  $40/60$ ,  $8\%$  of  $H_2O$  is added as a spray and the mass is pressed and heated slowly to about  $640^\circ$ , the temp. being kept below the dehydration point of the mica.

**Vitreous articles with a multi-color finish.** Franz Nowak (to Porcelain Enamel and Mfg. Co. of Baltimore). U. S. 2,021,820, Nov. 19. Fired vitreous enameled articles such as those of sheet iron having a multi-color effect are produced by applying to the article particles such as those of an enamel frit which are fusible at the firing temp. and on cooling form a vitreous coating, and applying to such particles, in different directions, coatings of different colors stable at the firing temp., then firing to effect fusion and produce a surface which on cooling has a multi-color effect. Various examples are given.

## 20—CEMENT AND OTHER BUILDING MATERIALS

J. C. WITT

**Cold application of asphalt roads.** J. Oberbach. *Bitumen* 5, 137-43 (1935).—Technical and economic advantages of cold asphaltic construction are discussed. Typical mixes and applications are also reviewed.

C. B. Jenni

**Methods for making bitumen dispersions for paving streets.** E. H. Kadmer. *Seifen- und Ztg.* 62, 813-14, 833-4 (1935); cf. C. A. 29, 8309<sup>2</sup>.

J. W. Perry

**Road emulsions and their new uses.** R. G. Smith. *J. Inst. Engrs. Australia* 7, 361-9 (1935).—An app. is described for measuring the amt. of stability against displacement resulting from various thicknesses of film of asphalt coating. The app. consists of 3 metal blocks and a base, as illustrated in the paper. The blocks are of metal with surfaces finished to true planes to eliminate mechanical and frictional resistance between adjacent surfaces. Two blocks correspond to coarse base stone and the third corresponds to the key stone between the other 2 pieces of base stone in a typical penetration pavement. The base plate corresponds to the subgrade. Details of operation are given. The use of emulsion asphalt, at greater costs than hot asphalt, is justified, for with it pavements are obtained having strengths obtainable in no other way. The emulsion has indisputable value in road building, whether penetration, road mix, or premixed types. Mixing or slow-setting emulsions are made with emulsions which mix with rock, sand or clay aggregates. The types of road built with these types of emulsions are: (1) treated type, (2) cold premix type and (3) hot premix type. The latest and most far-reaching development of emulsions is the stabilization of road bases. Soil stabilization refers to changes in soil characteristics in which the capillarity of the soil and its affinity for water are reduced to a point which will insure required bearing strength in the soil under actual conditions of exposure in the subgrade. The stabilization involves the uniform distribution of minute particles of asphalt throughout the entire clay mass. In soil stabilization the soil is simply rendered resistant to water from capillarity thereby retaining the bearing strength characteristic of the same soil when in a dry compacted condition. Expts. are described. W. H. B.

**Xyloth.** Heinrich Wiesenthal. *Kunststoffe* 25, 256-7 (1935).—Raw materials, production methods and final products are discussed.

J. W. Perry

**Testing of building lime.** Otto Graf. *Tomind.-Ztg.* 59, 1137-9 (1935).

P. S. Roller

**Rubber, bitumens and road surfaces.** (Pistor) 14. Corrosive effect of ground water on concrete (Karttunen) 14. Cements (Brit. pat. 432,493) 13. Preserving rubber latex [compn. for surfacing floors] (Brit. pat. 433,116) 30. Vacuum app. and method for impregnating timber, etc. (Brit. pat. 432,552) 1.

**Cement.** Charles H. Breerwood (to Valley Forge Cement Co.). U. S. 2,021,623, Nov. 19. A cement raw material mixt. of a desired analysis is prep'd. from inferior argillaceous limestones, deficient in  $CaCO_3$  and excessive in silica (some of the silica being in a form too coarse for favorable combination with the Ca in the process of burning to clinker), by crushing the stone to a size suitable for calcination, followed by calcining sufficiently to convert substantially all the  $CaCO_3$  to  $CaO$  but short of a degree causing C and silica reactions, converting the Ca compds. to powder without materially reducing the coarse silica, segg. the fine stone from the coarse silica, discarding the latter, recovering the fine constituents and proportioning them with other fines of different analysis to give a cement product of desired analysis. An arrangement of app. is described. Cf. C. A. 29, 5624<sup>1</sup>.

**Cement.** Bruno Neuhof. Ger. 595,924, June 18, 1935 (Cl. 80b 1 09). The setting of hydraulic cement is delayed by adding size or size-forming agents. A mixt. of potato meal and  $NaOH$  or sulfite waste lye may be used as size producers.

**Cement.** Fried. Krupp Grusonwerk A.-G. Ger. 616,754, Aug. 3, 1935 (Cl. 80c 14.10). A heat-exchanger for the manuf. of cement is described.

**Rotary cement kiln.** Mikael Vogel-Jørgensen. Fr. 784,084, July 22, 1935.

**Rotary kiln for making cement.** Johan S. Tasting. Fr. 783,488, Aug. 10, 1935.

**Shaft furnace for cement.** Fried. Krupp Grusonwerk A.-G. Fr. 785,813, Aug. 20, 1935. Clinker which been fired in amt. of at least  $1/4$ th of the total charge, is uniformly distributed in the charge.

**Cement, mortar, concrete, etc.** Wilhelm Klie. Ger. 610,407, Oct. 2, 1935 (Cl. 80b 1 06). Cements and c.

itself) in which has been incorporated halogen either as such or in the form of a hydracid is added before the hydrogenation. The absorbent material may be treated with the anhyd acid or with a soln. of the halogen in an org. solvent.

**Hydrogenating carbonaceous materials.** I. G. Farbenindustrie A-G. Brit. 432,638, July 25, 1935. In the production of valuable hydrocarbons by the heat-treatment of carbonaceous materials, particularly in presence of hydrogenating gases, and of finely divided catalysts or surface-active materials suspended in the reaction materials, a part of the materials contg. catalyst is returned to the preheater from a settling vessel that is arranged behind the preheater and before a reaction chamber or is connected with any place in the preheater behind the point of return. App. is described.

**Apparatus (with a hot air and steam supply) for the combustion of fuels such as coal in boiler furnaces.** Frank A. Chambers and Charles F. McGinley. U. S. 2,020,173, Nov. 5. Various structural and operative details.

**Fractionating coal by flotation.** Ernst Bierbrauer and Josef Popperle. Ger. 619,239, Sept. 26, 1935 (Cl. 1c 10.01). An aq. sludge of finely ground coal is treated with a water sol. oxidizing agent, e. g.,  $\text{HNO}_3$ ,  $\text{H}_2\text{O}_2$ , or  $\text{KMnO}_4$ . The humous and bituminous constituents, which are the reactive substances required for subsequent coking or hydrogenation, are thus caused to sink, together with the inorg. constituents, while the inert org. substances (mainly fibrous coal) remain in suspension; they can be sepd. in known manner by addn. of  $\text{PhOH}$ ,  $\text{PhNH}_2$ , or other foaming agent. The residue of reactive org. substances and inorg. constituents is treated with a coal-tar oil, whereby the reactive substances are brought into suspension, and sepn. is then effected by a second flotation treatment. The first flotation may be assisted by adding to the mixt. a hydrophilic aliphatic or aromatic compd., e. g., hydroquinone. Alternatively, humic acid, lignin-sulfonic acid or a deriv. thereof may be added.

**Apparatus for separating heavy impurities from coal sludges.** Hoesch-Köln Neussens A.-G. für Bergbau und Hüttenbetrieb. Ger. 619,451, Oct. 2, 1935 (Cl. 1a 10). The sludge traverses a channel, the bottom of which is formed by an endless rubber band having transverse projections for trapping the heavier impurities. The band is moved periodically in the opposite direction to the sludge.

**Screening devices, especially for coal.** Cwilym E. A. Thomas and Emlyn Anthracite Colliery Ltd. Brit. 432,240, July 23, 1935.

**Beater for use in apparatus such as that for pulverizing coal, etc.** James W. Armour (to Riley Stoker Corp.). U. S. 2,021,762, Nov. 19. Structural and mech. details.

**Briquetting coal.** Robert J. Piersol. U. S. 2,021,020, Nov. 12. Strong stable briquets are formed without the aid of added binders, from bituminous coal contg. considerable amounts of volatile matter, by heating the coal to remove a portion of the volatile matter, cooling to 150–350° and subjecting the coal, at such temp., in a die, to a single impact blow of about 1500–3000 foot pounds for each 0.1 lb. of coal compacted, to form a dense briquet. App. is described.

**Steam generator.** Wilson L. McLaughlin, Herrold L. McLaughlin and Virg. Ballou (H. L. McLaughlin and V. Ballou to National Pure Water Corp.). U. S. 2,020,369, Nov. 12. Structural features.

**Apparatus for purifying steam coming from a boiler.** Wilbur H. Armacost (to The Superheater Co.). Brit. 408,912, Apr. 19, 1934, 410,037, May 10, 1934, 431,037, June 28, 1935.

**Apparatus for deconcentrating or purifying boiler steam.** James A. Powell and Wilbur H. Armacost (to The Superheater Co.). Brit. 410,036, May 10, 1934.

**Treating exhaust gases from internal-combustion engines, etc.** Joseph S. Kaufman. U. S. 2,021,690, Nov. 19. Water vapor in exhaust gases is continuously condensed into liquid and the liquid is continuously subjected to contact with Fe heated to a temp. (suitably

about 200–500°) sufficient to vaporize instantly the liquid into wet steam in the presence of the gases. The steam reacts with CO to form  $\text{CO}_2$ . App. is described.

**Purifying gases.** Gordon Nonhebel, John L. Pearson and Imperial Chemical Industries Ltd. Brit. 433,039, July 29, 1935. In purifying gases from oxides of S and any smoke or ash present by scrubbing with a recirculated aq. washing medium that is continuously replenished with  $\text{CaO}$ , chalk or Mg limestone and removing the insol. solids that collect in the system by purging off a part only of the recirculating liquor without settling the bulk of the liquor, the liquor after leaving the scrubber is treated so as to remove practically the whole of the supersatn. due to  $\text{CaSO}_4$  and  $\text{CaSO}_3$  before recirculation by delaying the return of the liquor to the scrubber for a definite min. time sufficient to enable desupersatn. to be completed. Scaling of the scrubber surfaces is thus prevented. App. is described.

**Washing flue gases.** Gordon Nonhebel, John L. Pearson and Imperial Chemical Industries Ltd. Brit. 433,373, Aug. 6, 1935. In washing flue gases with a recirculated liquor to which  $\text{CaO}$  or chalk is added in amt. approx. equal to the S oxides and other strongly acid gases, e. g.,  $\text{HCl}$ , the addn. of  $\text{CaO}$  or chalk is regulated by detg. the  $p_{\text{H}}$  of the scrubber exit liquor continuously or at intervals and adding, at a point after that of the detn. of the  $p_{\text{H}}$ , sufficient  $\text{CaO}$  or chalk to keep the  $p_{\text{H}}$  within the range 6–6.4, preferably 6.2, with  $\text{CaO}$  neutralization, or 6–6.3, preferably 6.1, with chalk neutralization. App. is described.

**Water gas generators.** Norman F. Parker and John Strong. Fr. 785,471, Aug. 10, 1935. Regulating means. **Purifier for generator-gas plant.** Humboldt-Deut. Motoren A.-G. Ger. 616,908, Aug. 7, 1935 (Cl. 26d 1.20).

**Air gas producer.** Hermann Blau. Ger. 616,554, July 31, 1935 (Cl. 26c 1). App. for satg. air with the vapor of readily volatilized liquid hydrocarbons is described.

**Apparatus for producing gaseous mixture such as illuminating and heating gas from liquefied fuel gases, etc.** Roswell W. Thomas (to Phillips Petroleum Co.). U. S. 2,021,271, Nov. 19. Various structural and operative details.

**Apparatus for producing high- and low-grade gases from liquid and solid fuels such as for operating automotive vehicles.** Robert Falconer and Eduardo Collignon. U. S. 2,021,055, Nov. 12. Various structural and operative details.

**Removing weak acids from gases.** I. G. Farbenind. A.-G. Fr. 785,276, Aug. 6, 1935.  $\text{H}_2\text{S}$  and  $\text{CO}_2$  are removed from gases contg. them by washing the gases with bases, the mol. of which contains at least 2 atoms of N and is of the formula  $\text{A}_1(\text{A}_2)\text{N}_2\text{XN}(\text{A}_3)\text{A}_4$ , in which X is an aliphatic radical which may also contain one simple or substituted aminogen group, and at least 1 of the A groups denotes an alkyl or aryl group capable of contg. OH or a simple or substituted  $\text{NH}_2$ , or 2 of the A groups joined to different N atoms form an alkylene group, while each of the other A groups is H or alkyl or aryl. Examples of such compds. are  $\text{CH}_3\text{NHCH}_2\text{CH}_2\text{NHC}_2\text{H}_5$ ,  $\text{MeN-CH}_2\text{CH}_2\text{NHC}_2\text{H}_5$ ,  $\text{HOCH}_2\text{CH}_2\text{NHC}_2\text{H}_5$ ,  $\text{CH}_3\text{CH}_2\text{NHC}_2\text{H}_5$ ,  $\text{H}_2\text{NCH}_2\text{CH}_2\text{NMeC}_2\text{H}_5$ ,  $\text{HOCH}_2\text{CH}_2\text{NCH}_2\text{CH}_2\text{NH}_2$ , piperazine, pyrazine, and their derivs., etc.

**Removing hydrogen sulfide from gaseous mixtures.** Bert H. Lincoln (to Continental Oil Co.). U. S. 2,021,865, Nov. 19. The gas is brought into contact with sulfurous acid in an enlarged zone in the presence of activated C, so that a portion of the  $\text{H}_2\text{S}$  is converted into water and elemental S, the gas is then sepd. from the reaction products and is passed countercurrent to  $\text{H}_2\text{SO}_4$  to convert remaining  $\text{H}_2\text{S}$  into water and S, and the treated gas is washed. App. is described.

**Removing sulfur dioxide from flue gases.** Charles F. Goodeve. U. S. 2,021,548, Nov. 19. The gases are treated with an acid soln. of Fe sulfate and then with an alk. suspension of Fe hydrosulfide. An arrangement of app. is described.

Sulfur dioxide. Ingenium Hechenbleikner (to Chemical Construction Corp.). U. S. 2,021,725, Nov. 19. S or a sulfurous material is burned to produce high-temp. combustion gases contg.  $\text{SO}_2$  and heat is transferred from the gases, by direct contact, to a material such as acid sludge from petroleum refining from which  $\text{SO}_2$  is liberated by heating to below its temp. of combustion so that substantially all the  $\text{SO}_2$  is liberated from such material and mingled with the gaseous combustion products. App is described.

Means for compressing coal in discontinuously operated coke ovens. Gustav Hilger. Ger. 613,062, May 10, 1935 (Cl. 10a 15). Addn to 610,658 (C. A. 29, 5638<sup>1</sup>).

Refining benzene. Aktiebolaget Separator-Nobel

Fr. 785,078, Aug. 1, 1935. Benzene is mixed with  $\text{H}_2\text{SO}_4$  of 60–65°Bé and, after sufficient contact, the acid and resin are sepd from the benzene in a centrifuge without interruption.

Coking coal. Carl Stull G. m. b. H. Ger. 619,454, Oct. 1, 1935 (Cl. 10a. 19 01). Means is described for producing channels in the compressed charge and for withdrawing distn. products through the channels. Cl. C. A. 29, 3143<sup>4</sup>.

Coking-retort oven. Joseph Becker (to Koppers Co. of Del.) U. S. 2,019,483, Nov. 5. Various details.

Coking-retort oven. Joseph van Ackeren (to Koppers Co. of Del.) U. S. 2,020,919, Nov. 12. Various structural and operative details.

## 22—PETROLEUM, LUBRICANTS, ASPHALT AND WOOD PRODUCTS

W. F. PARAGHER

Petroleum-bearing rocks in France. lignites of Minervois. V. Charrin. *Mat. grasses* 27, 10029–31 (1935). — Analysis of 2 samples gave the following av. results:  $\text{H}_2\text{O}$  9.16, ash 8.00, volatile 47.85, fixed C 44.15, S 5.80, N 0.83%, and calorific value 6225 cal. Carbonization in a lab. Fischer retort gave anhyd. filtered tar (condensed at 15°) 14.75, semicoke 73.40,  $\text{H}_2\text{O}$ , gas and loss 11.91%, vol. of gas evolved 95 cu. m. per ton. The semicoke had the following compn: ash 21.50, volatile 21.00, fixed C 63.50, N 1.10, S (mostly in the form of  $\text{CaSO}_4$ ) 3.70%, calorific value 6000 cal. Com. scale carbonization tests carried out in a Salerni retort indicated that the following yields per metric ton could be obtained: semicoke 700 kg., gas 100 cu. m., anhyd. primary tar 140 kg. Fractionation of the tar yielded the following products: motor spirit ( $d_{40}$  0.825, flash pt. below atm. temp.) 7.60, solar oils ( $d_{40}$  0.871, flash pt. 71°, S 4.45%, calorific value 10,205) 11.76, gas oils ( $d_{40}$  0.913, flash pt. 130°, S 3.65%, calorific value 10,074) 10.70, light lubricating oils ( $d_{40}$  0.944, flash pt. 152°, burning pt. 183°, calorific value 9.978, S 3.37%, Engler viscosity at 20° 2.97, at 50° 1.44, solidifying pt. 4°) 6.85, heavy lubricating oils ( $d_{40}$  0.941, flash pt. 192°, burning pt. 233°, solidifying pt. 1°, Engler viscosity at 50° 3.60, at 100° 1.31) 6.00, hard paraffin (m 51.2°) 4.52, medium rectified cresols ( $d_{40}$  1.026, flash pt. 98°, burning pt. 110°, S 0.46%, calorific value 8307) 3.88, heavy cresols (m 21.7°, volatile 0.234, alk. ash 1.20, fixed C 6.50, S 3.07, petr. ether-sol 40.30,  $\text{C}_6\text{H}_6$ -sol 83.90, insol. 7.60%, calorific value 7182) 8.27, pyridine bases 2.74, asphalt ( $d_{40}$  1.172, m 119.4°, benzene-insol 32.85%, calorific value 8097) 20.50, loss 15.12%.

A. Papineau-Couture.

Aromatization by catalysis of fractions of Bakr (Sarak-hany) gasoline. N. D. Zelinski and N. I. Shulkin. *Bull. acad. sci. U. R. S. S.* 1935, 229–37 (in English 237). *Ind. Eng. Chem.* 27, 1209–11 (1935); cf. C. A. 29, 2721<sup>1</sup>. — Cyclohexane and its derivs. (about 50%) in gasoline fractions b. 75–125° and 118–140° are almost completely dehydrogenated to aromatic compds. by passage with H at 300° over Ni on  $\text{Al}_2\text{O}_3$  or Pt on charcoal. The catalysts remove the traces of S present, but are not poisoned thereby. The aromatic contents of mixts. detd. by the  $\text{H}_2\text{SO}_4$  and the PhNH<sub>2</sub> methods agree.

H. A. Beatty.

Viscosity of diluted oils and of oil mixtures. E. Erdheim. *Mat. grasses* 27, 10600–2 (1935). — See C. A. 29, 7630<sup>1</sup>.

A. Papineau-Couture.

Rational scale for classifying motor fuels. Max Seruys. *Compt. rend.* 201, 815–17 (1935). — Of 2 gasolines of the same octane no. but different calorific powers, the one of lower power gives rise to knock more readily than the other; the power obtainable, moreover, is not proportional to the calorific value. To compare the utilizable power of 2 gasolines, it is proposed to value them by their "synthetic indexes"; this index is the ratio of the max. power produced by the gasoline to that produced by a standard fuel in a standard motor under definite conditions of temp., compression, etc., both fuels being fed at

the knock limit and giving the same percentage of CO in the exhaust. C. A. Silberrad.

Fuels for aircraft engines. E. L. Bass. *J. Roy. Aeronaut. Soc.* 39, 879–962 (1935), cf. C. A. 29, 908<sup>1</sup>. — B discusses only volatile fuels. In his exptl. work on the E 35 engine, cylinder-head and exhaust-gas pyrometers were used in studying detonation. The effects of mixt. strength, fuel-antiknock characteristics, intake-mixt. temp. and ignition timing were observed. B also reviews the following fuel characteristics: boiling range and its relation to starting, distribution, and ice formation, vapor pressure, l. p.; sp. gr.; viscosity; calorific value; and impurities. The possible use of iso-octane as a blending agent in aviation gasolines is considered. M. W. Schwarz.

Influence of various additions to the fuel of injection motors. Marius Aubert, Pierre Clerget and Roger Duchêne. *Compt. rend.* 201, 879–81 (1935), cf. C. A. 29, 1615<sup>1</sup>. — The marked effect of  $\text{EtNO}_3$  in suppressing knock and diminishing ignition delay is demonstrated with 5% and larger addns. C. A. Silberrad.

Behavior in the engine, storage stability, and storage losses of gasoline. Nikolaus Mayer. *Chem.-Ztg.* 59, 843–5 (1935). — An elementary discussion is given of the factors affecting the behavior of gasoline in the engine, based chiefly on American literature references. Storage stability and losses occurring during storage in tanks are discussed, and definitions are given of the terms potential gum, inhibitor, induction time, gum-inhibiting index and soly index. D. F. Brown.

Gum in gasoline. A. A. Vuorela. *Suomen Kemistilehti* 8A, 115–18 (1935). — Peroxides, the first products of oxidation of unsatd. compds., form at rates that are greatly affected by small quantities of pos. and neg. catalysts and by the temp. The decompn. of peroxides is probably the source of gum in many instances. The end products are polymerized acids and higher aldehydes and ketones. Gum formation during storage can be prevented for long periods by the addn. of oxidation inhibitors. The deterioration of color can be retarded by some secondary anti-catalysts. E. E. Julkola.

Use of vegetable oils, and more particularly olive oil, for the lubrication of motors. Emile Robbe. *Mat. grasses* 27, 10588–8, 10615–17 (1935). — A discussion of the properties required in motor lubricants and of the suitability of various vegetable oils, especially olive oil, for use in motors. A. Papineau-Couture.

Gas in the "oil laundry." (Recovery of automotive lubricating oil.) L. H. Ronnick. *Industrial Gas* 14, No. 5, 20, 22 (1935). — A machine for refining used oil is made in 5-gallon units, with a batch process. The used oil is mixed with a special refining clay and then forced by air pressure into a gas-heated chamber. When the temp. reaches 618°F., the gas is shut off thermostatically. During the heating, the light ends are distd. and condensed. Steam is then passed through the oil until the temp. falls to 520°F. The oil is drained from the still into a lower chamber, from which it is forced by air at 15–20 lb. gauge pressure through two layers of heavy paper. A new filter



pad is used for each batch. The reclaimed oil has approx. the original viscosity and is claimed to be better than the original oil.

R. W. Ryan

Unusual cutaneous symptoms associated with retention of arsenic. Binford Thorne and C. N. Myers. *Arch. Dermatol. Syphilol.* 32, 181-95 (1935).—A case of As poisoning from handling clay in an oil-refining plant is included. The unburned clay contained 1.19 mg As per 100 g.

O. Hartley

Wood turpentine oil from *Pinus formosana* Hayata. I and II. Veiga Sebe. *J. Chem. Soc. Japan* 56, 1118-41 (1935).—The oil obtained by steam distn. showed  $d_4^{20}$  0.8636,  $n_D^{20}$  1.4708,  $[\alpha]_D^{20} = -33.80^\circ$ , ester value 11.8 (after acetylation 31.02) and acid no. 0.62. It contained 74-5% terpenes and 20% terpene alcs. and their esters. Presence of  $\alpha$ - and  $\beta$ -pinene, camphene, cineol,  $p$ -cymene,  $\gamma$ -terpinene,  $\alpha$ - and  $\beta$ -phellandrene, limonene, dipentene, borneol (I), camphor,  $\alpha$ -terpinol (II), fenchyl alc. (III) and acetates of I, II and III is shown. The oil contained no sylvesterene, terpinolene or fenchene. Diterpenes consisted mostly of *thumbeolene* (IV),  $C_{20}H_{32}$ ; slow oxidation of IV in air gave scale-like crystals,  $C_{20}H_{30}O$ , m. 131-2°. Distn. of wood grown at a high altitude gave an oil (0.05-0.06%), with  $d_4^{20}$  0.8824,  $n_D^{20}$  1.4700,  $[\alpha]_D^{20} = +6.23^\circ$ , acid no. 2.2, ester value 31.0 (after acetylation 96.1). It contained nearly the same constituents as the l-retatory oil, but a sesquiterpene alc. *sessugoyol* (V),  $C_{20}H_{32}O$ , m. 137-75°, b.p. 160-65°,  $b_{760}$  285-9°, with  $[\alpha]_D^{20} = +93.38^\circ$  was found. V is a dicyclic alc. and turns to dicyclic sesquiterpene by reduction with  $HCO_2H$  or  $H_2SO_4$ . Catalytic reduction of V gives *dihydrosessugoyol*,  $C_{20}H_{34}O$  (VI), m. 121°. Acetate of V b.p. 152-5°,  $d_4^{20}$  0.9922, has  $n_D^{20}$  1.4902,  $[\alpha]_D^{20} = +22.19^\circ$ , ester value 205. Oxidation of VI with  $CrO_3$  turns it to a viscous sirup,  $C_{20}H_{30}O_2$ , b.p. 158-60°, with  $d_4^{20}$  0.9627,  $n_D^{20}$  1.4807.

K. Kitsuata

The utilization of the larch tree in the U. S. S. R. I. S. Matyuk. *Bull. Applied Botany, Genetics Plant Breeding* (U. S. S. R.), Ser. A, No. 14, 185-94 (1934).—A compilation of data on the turpentine and cellulose content of the larch.

J. S. Joffe

Rubber and bitumens (Barron) 30. Mineral-oil sulfonic acids (Sceda, et al.) 10. Resin acid in the oleoresin of *Pinus sylvestris* (Pischchumuka) 10. App. for removing solid deposits from the mouth of conduits such as vapor outlets of oil-conversion app. (U. S. pat. 2,020,241) 1. Catalysts for hydrogenation (U. S. pat. 2,021,567) 10. Testing the adhesive properties of coatings such as asphalt (U. S. pat. 2,020,891) 13.

Refining petroleum products. Thomas T. Gray (to Gray Processes Corp.) U. S. 2,020,115, Nov. 5. For refining petroleum products such as cracked products contg. unstable hydrocarbon compds., the material in vapor phase is introduced into the lower portion of a treating zone, into the upper portion of which finely divided adsorptive material such as fuller's earth is introduced, and sufficient velocity is imparted to the vapor to retard the fall of the adsorptive material through the vapor. App. is described.

Removing elementary sulfur from petroleum oils. Walter A. Schulze and Lovell V. Chaney (to Philips Petroleum Co.) U. S. 2,020,661, Nov. 12. Oil contg. elementary S is brought into contact, at atm. temp., with an aq. soln. of a polysulfide lower than the tetrasulfide and which is one of the alkali metal or alk. earth metal polysulfides, and the resulting soln. is sep'd from the oil. Refining hydrocarbon vapors such as those of cracked products containing sulfur. Jacques C. Morrell and Gustav Eglöf (to Universal Oil Products Co.) U. S. 2,021,739, Nov. 19. The vapors are treated with an aq. soln. contg. free  $HOCl$  in the presence of a Zn halide such as  $ZnCl_2$  in an amt. greater than that which will dissolve in the acid so that some of the zinc halide remains in the solid phase. U. S. 2,021,740 relates to a process in which the hydrocarbon vapors are brought into contact with an aq. soln. contg. a free oxygenated halogen acid such as

$HOCl$ ,  $HOClO_2$ ,  $HBrO_3$ , or  $HIO_3$ , as the principal refining agent and in sufficient amt. to effect substantial desulfurization of the vapors.

Refining hydrocarbon oils with solvents. Louis A. Clarke (to Texas Co.) U. S. 2,020,290, Nov. 12. For refining a hydrocarbon oil such as a lubricating oil stock contg. relatively paraffinic and relatively nonparaffinic constituents including naphthenic, aromatic and unsat'd compds., to remove undesired relatively nonparaffinic constituents, the oil is ext'd with a relatively low-boiling aliphatic acyl deriv. of furan such as acetyl furan.

Purifying hydrocarbon oils. Anglo-Iranian Oil Co. Ltd. (formerly Anglo-Persian Oil Co. Ltd.), Ferdinand B. Thole and Stanley F. Burch. Brit. 433,197, Aug. 6, 1935. In the extn. of hydrocarbon oils with liquid  $SO_2$  and similar solvents to remove aromatic and unsat'd. substances, the ext. is cooled to 20-40°F. below the extn. temp., whereby paraffinic hydrocarbons are sep'd and removed. The resulting ext., rich in aromatic hydrocarbons, is freed from  $SO_2$  and mixed with motor fuels as an antiknock ingredient. Kerosene is treated in counter current at 16°F. with liquid  $SO_2$ ,  $NH_3$  or  $HCl$  and the resulting ext., mixed with add'l solvent, if necessary, is chilled to -20°F. by evap. part of the solvent, the upper paraffinic layer is drawn off and the ext., after removal of  $SO_2$ , is washed with  $NaOH$  soln. and then fractionated.

Hydrocarbon-oil conversion. Robert E. Wilson (to Standard Oil Co. of Ind.) U. S. 2,021,471, Nov. 19. For producing material within the gasoline b. p. range, an oil such as a crude or residuum oil is heated in a pipe coil or the like to a conversion temp. above 425° under a pressure above 50 atm., passed into an enlarged chamber maintained under a lower pressure but above 100 lb. per sq. in. and at a conversion temp. but not above that of the oil from the coil. Unvaporized products and vapors are separately removed and the pressure on them is separately reduced, and the unvaporized products, reduced to a temp. below that of the vapors, are passed into a reduced pressure chamber to which also pass vapors from the enlarged chamber which are introduced at a lower level for countercurrent contact. App. is described.

Hydrocarbon oils. Standard Oil Development Co. Fr. 785,348, Aug. 7, 1935. The coloration of kerosenes, lighting oils, etc., is prevented during storage by the add'n of a cyclic org. acid, such as salicylic, anthranilic, protocatechuic, gallic acid and anthracenecarboxylic acids contg.  $NH_3$  or  $OH$ .

Cracking hydrocarbon oils. Wm. M. Stratford (to Texas Co.) U. S. 2,020,086, Nov. 5. Hot residue is intermittently withdrawn from a pressure-cracking vessel maintained at cracking temps. and under superatm. pressure and the residue is directed into a surge chamber in which the pressure is materially lower than that of the cracking vessel, residue is transferred at a const. rate from the surge chamber to a stripping still, and vapors are removed from the surge chamber to prevent rise in pressure above a predet'd. point, the vapors are condensed to form a distillate, and vapors from the stripping still are conducted to a fractionating tower and the distillate mentioned is introduced into the tower as reflux condensate.

App. is described. Cracking hydrocarbon oils. Boyd W. Morgan (to Universal Oil Products Co.) U. S. 2,020,653, Nov. 12. Oil is heated to a cracking temp. under pressure while flowing in a restricted stream through a heating zone such as a pipe coil and is discharged into the upper portion of an enlarged vertical reaction zone maintained under cracking conditions of temp. and pressure and through which both vapors and unvaporized oil pass downwardly, the unvaporized oil and a portion of the vapors are removed as a mixt. from the bottom of the reaction zone at a rate adequate to prevent any appreciable accumulation of liquid in the reaction zone; the major portion of the vapors is separately removed, unadmixed with unvaporized oil, from the lower portion of the reaction zone but at a point above its bottom; the mixt. withdrawn from the bottom of the reaction zone is introduced into a sepy. zone main-

tained under lower pressure than the reaction zone and there sepd. into vapors and residue, and the said "major portion" of vapors is introduced into the sepg zone at a point above the point of introduction of the mixt mentioned so as to prevent contact of these vapors with said unvaporized oil while combining them with the vapors sepd. in the sepg. zone; the combined vapors are roughly fractionated in the upper portion of the sepg zone to remove high coke-forming components from them, the combined vapors are then removed from the sepg zone and further fractionated to condense insufficiently cracked fractions and resulting reflux condensate is subjected to further cracking treatment, fractionated vapors being finally condensed. App is described.

Cracking hydrocarbon oils. Andrew M Wood (to Universal Oil Products Co.) U S 2,021,761, Nov 19. Oil such as a fuel oil is passed through a heating coil in a furnace and vapors are sepd from the oil stream at spaced points along the coil, and incondensable hydrocarbon gas is injected into the oil stream at points immediately following the points of vapor sepn and in the direction of oil flow. App is described.

Cracking hydrocarbons. Holding Luxembourgaise pour produits liquides charbons-huiles Fr 784,801, July 25, 1935. Oils, tars, suspensions of coal in oil, etc., are passed under pressure through the annular space between 2 concentric vessels one of which is heated, the liquids being forced to move circularly as well as upwardly on the heated surface. App is described.

Cracking hydrocarbon materials. Harry S Reed and Ralph D Lame. U S 2,022,054, Nov 26. Material such as a high-boiling oil is subjected to a preliminary heating under pressure and at a temp below its cracking temp, it is passed through a small expansion valved aperture into a tumbling barrel-mill retort and there cracked by heating to a higher temp under a lower pressure while subjected to the combined action of angular grinding elements and of balls such as those of white iron which distribute heat and prevent carbon deposits adhering to the inner surface of the retort; powd material is discharged from the retort so that the process need not be interrupted for cleaning the retort. App. is described.

Cracking oils. Georg Zotos Brit 432,736, Aug 1, 1935. In processes for cracking oils wherein the oil is cracked by direct contact with superheated gases or vapors, the temp. of the reaction chamber is controlled by flowing the oil over a heat-exchanger through which the materials to be superheated are passed on their way to the superheater. App. is described.

Vessels such as those for oil cracking with linings of corrosion-resisting metal plates. Henry Anderson (to Universal Oil Products Co.). U S 2,020,630, Nov. 12. Structural details.

Dewaxing hydrocarbon oils. Robert E Manley and Wm P Gee (to Texas Co.). U S 2,020,693, Nov 12. In the dewaxing of a hydrocarbon oil by filtration in the presence of a comminuted solid filter-aid material such as fuller's earth with obtainment of a filter cake contg. wax hydrocarbons and the filter-aid, the filter-aid material is recovered from the filter cake and reconditioned for further use in treating wax-bearing oil by reducing the filter cake to a substantially fluid mass by admixture with a liquid to form a slurry; the substantially fluid mass is introduced into a vessel contg. a body of water at a temp. such that the waxy hydrocarbons are brought to a substantially liquid condition and form a layer on the surface of the water; steam is injected at substantially the surface of the body of water so that the filter-aid material is displaced from the hydrocarbons into the body of water and the filter-aid material is removed with assoc. water, and dried. App. is described. Cf. C. A. 29, 5260<sup>1</sup>.

Removing paraffin from oils. The Sharples Specialty Co. Fr. 784,513, July 23, 1935. The oil is dissd. with a mixt. of a chloro deriv. of a hydrocarbon, e. g., ethylene dichloride, about 75, and a hydrocarbon of a relatively low d. e. g.,  $C_{12}H_{26}$ , about 25%, and the paraffin is then

sepd. by centrifuging at a temp. sufficiently low to ppt. the greater part of the paraffin. App. is described.

Removing paraffin from oils. N V de Bataafsche Petroleum Maatschappij. Fr. 785,008, July 31, 1935. See Brit. 430,485 (C. A. 29, 8310<sup>1</sup>).

Removing paraffin from oils. The Sharples Specialty Co. Fr 785,229, Aug 5, 1935. Paraffin is sepd. from mineral oils by repeated coolings and centrifugings, the oil being heated to about 15° between each operation so as to redissolve at least part of the paraffin. By this means accumulation of small particles which cannot be removed easily by centrifuging is avoided.

Bubble tower apparatus suitable for use in oil refining. Charles H Leach U S 2,020,895, Nov. 12. Structural and operative details.

Treating mineral-oil distillates. Adrianus J van Peski (to Shell Development Co.) U S 2,019,772, Nov. 5.

A cracked mineral-oil distillate lighter than a lubricating oil and contg. unsatd. hydrocarbons and undesirable compds. tending to polymerize and condense with formation of readily oxidizable products is subjected to a light polymerizing treatment with use of a quantity of a normally solid polymerizing catalyst such as  $AlCl_3$ , which is not substantially greater than that necessary to remove undesirable compds. without substantially removing unsatd. hydrocarbons, and in the presence of a nitro hydrocarbon such as nitromethane and after removing resulting polymers, etc., the remaining oil is subjected to a further polymerizing treatment. App is described.

Refining mineral oils with selective solvents for naphthenic and paraffinic constituents. Malcolm H Tuttle (to Max B Miller & Co.) U S reissue 19,763, Nov 19. A reissue of original pat No 1,912,349 (C A 27, 4070).

Apparatus for placing explosive charges in oil wells or the like. Chester A Mathey and Paul F Lewis (to American Glycine Co.) U S 2,021,632, Nov. 19. Various mech. and operative details.

Plugging strata in wells such as oil wells. Harvey T Kennedy and Howard C Lawton (to Gulf Research & Development Corp.) U S 2,019,908, Nov. 5. There is injected into the strata a liquid halogen compd. of  $S_2$  or  $Te_2$  such as  $SiCl_4$  or  $TeCl_4$  to react with water and produce an insol ppt. in the strata. Cf. C. A. 29, 8317<sup>1</sup>.

Sulfonated oils, etc. Frich A Wermicke Brit. 432,797, Aug 2, 1935. See Fr. 771,401 (C. A. 29, 919<sup>1</sup>).

Highly viscous oil. I. G. Farbenind. A.-G. (Mathias Pier and August Fieschhut, inventors) Ger. 616,833, Aug. 6, 1935 (Cl. 23c. 1). Paraffins or waxes, etc., are subjected to a silent elec. discharge or are halogenated and condensed, or are dehydrogenated, split and condensed or polymerized. The products so obtained are treated with liquefied hydrocarbons with critical temps. below 250°, at high temps. and pressure, to give a viscous oil. Thus, the product obtained by volatilizing cerezin is dissolved in liquid propane and the soln. shaken at 90-95° to give a highly viscous oil useful as cylinder oil.

Oil-distilling, -rectifying and -reclaiming apparatus suitable for treating oil used in internal-combustion engines. Oliver W Pettit U S 2,020,570, Nov. 12. Various structural and operative details.

Motor fuel. Ernest F. Pevere (to Texas Co.). U S 2,021,088, Nov 12. The antiknock properties of hydrocarbon liquid motor fuels is improved by adding about 0.5% or more of ethylenediamine or of a hydrate of ethylenediamine.

Gasoline. Gasoline Products Co., Inc. Fr. 785,023, Aug. 22, 1935. Relatively heavy hydrocarbons of high b. p. are converted to relatively light hydrocarbons of low b. p. by bringing the former into direct contact, for heat exchange, with the decomposed vapors while preventing them from coming in contact with the residual parts of the decomposed products; causing the unvaporized parts of the heavy hydrocarbons, in admixt. with the heavy condensate formed by partial condensation of the decomposed vapors, to pass into a primary heating zone, in which they are brought to decompos. temp. and submitted to conversion; introducing the resulting decom-

posed products into a vaporization zone in which the vapors sep from a liquid residue and these vapors serve for the preliminary heating of the heavy hydrocarbons; causing the uncondensed vapors, after contact with the heavy hydrocarbons, to pass into a fractionating zone in which they sep into light vapors and a reflux condensate; removing and condensing the light vapors and causing the reflux condensate to pass to a 2nd heating zone in which it is brought to decomp. temp and submitted to conversion, and introducing the resulting decomposed products into the vaporization zone with the decomposed products from the primary heating zone. App. is described.

**Inhibiting gum formation in cracked gasoline.** Herbert G. M. Fischer and Clifford E. Gustafson (to Gasolene Antioxidant Co.) U. S. 2,019,899, Nov. 5. The gasoline is mixed with about 0.1% or less of an inhibitor, such as  $\alpha$ -naphthol or  $\alpha$ -naphthylamine, comprising a  $\alpha$ -substituted aromatic condensed ring hydrocarbons in which adjacent nuclei have 2 C atoms in common, some of the hydrocarbons have an amino substituent and others a hydroxy substituent. Cf. C. A. 29, 49337.

**Fluid conduit suitable for use with gasoline and oil.** Joseph J. Mascuch U. S. 2,019,540, Nov. 5. Structural details.

**Purifying aqueous sodium hydroxide solutions containing mercaptides, such as those used for washing gasoline.** Robert E. Burk and Everett C. Hughes (to Standard Oil Co. of Ohio). U. S. 2,020,932, Nov. 12. A mercaptide-contg. soln. of NaOH is washed with S and an org. solvent, such as naphtha, which is immiscible with NaOH soln. and in which both S and the reaction product of S with the mercaptide are sol. and the org. solvent soln. is sep. from the aq. NaOH soln.

**Electric system for controlling removal of water settings from oil tanks, etc.** James A. Jonas U. S. 2,021,655, Nov. 19. Various structural, elec. and operative details.

**Lubricating oils.** I. G. Farbenindustrie A.-G. Brit. 433,061, Aug. 8, 1935. Liquid hydrocarbons poor in H are treated with a silent elec. discharge and the resulting products that are still poor in H are hydrogenated under elevated temp. and pressure in the presence of a catalyst. Tar or mineral oils, or distillates thereof, or products obtained by destructive hydrogenation or extn. of solid carbonaceous materials may be treated. The initial materials are preferably freed from asphalt and readily polymerizable substances by distn. or pptn. with gas oil, paraffin oil, benzene or liquefied hydrocarbons, or by treatment with H at 150-300°. The content of O compds. should not exceed 5%. A lubricating oil distillate obtained by destructively hydrogenating brown coal and dewaxing and fractionating the product is treated at low pressure in an ozonization or "voltageing" tube at 2000-8000 v. and at a frequency of 500-10,000 Hertz. Diluents, e. g., illuminating oil, may be added. The resulting viscous oil is led at 250-500° over pellets of W sulfide with H under 230 atm pressure. The resulting oil has a low pour point and high viscosity index.

**Lubricating oil.** Standard Oil Development Co. Fr. 785,839, Aug. 20, 1935. Esters (1-4%) obtained from acids of low mol. wt. and alcs. of high mol. wt. are added to lubricating oils. The alcs. may be those obtained by reducing acids found in natural oils or by oxidizing waxes or paraffins.

**Refining petroleum lubricating oils.** John M. Musselman (to Standard Oil Co. of Ohio). U. S. 2,020,954, Nov. 12. A mid-continent lubricating stock of about 68 viscosity index is subjected to the simultaneous action of 10% of  $AlCl_3$  and 10% of fuller's earth for a half hr. at a temp. of about 175°.

**Lubricating oils and greases.** Standard Oil Development Co. Brit. 433,257, Aug. 12, 1935. Fr. 785,390, Aug. 8, 1935. Lubricating oils and greases especially for use at high temps. are mixed with an organo metallic compd. contg. a metal of the 2nd, 3rd or 5th group of the periodic system, particularly Hg, Zn, Cd, Al, Ti, Bi or Sb. The compds. contain alkyl, aryl or aralkyl groups which may also contain OH and amino groups. Suitable

compds. are  $Ph_3Bi$ , cresyl Ph Zn, ( $\alpha$ - $C_{10}H_7$ ) $SbPr$ , ditolyl Hg, diphenylchloroarsine,  $C_{10}H_7HgI$ ,  $Ph_3Sb$ , Mg phenylacetate, cresyl zinc chloride, bromide or acetate, or sulfides, polysulfides, selenides and tellurides.

**Hydrocarbon lubricating oils.** Curt Schumann and Hanns Ufer (to I. G. Farbenind. A.-G.). U. S. 2,020,703, Nov. 12. The setting point of material such as a hydrocarbon lubricating oil is lowered by adding about 10% (more or less) of a polymerization product of a vinyl ether of an unsat. alc. such as that of oleic alc. (the polymerization product used having a mol. wt. of at least 1000).

**Hydrocarbon polymerization products, lubricating oils.** Standard Oil Development Co. Brit. 432,310, July 24, 1935.

**Purifying and dewaxing oils.** Standard Oil Co. Brit. 432,478, July 22, 1935. Mineral oils, e. g., lubricating oils, are treated with 1 or more solvents that remove naphthenic constituents, e. g., phenols, xylidine, furfural, pyridine, Et chloroacetate, cellosolve acetate,  $\alpha$ -naphthol, ethylenediamine, glycol acetate, benzonitrile and also with a solvent for the paraffinic constituents, e. g.,  $C_4H_{10}$ ,  $C_5H_{12}$ ,  $C_6H_{14}$ ,  $C_7H_{16}$ , iso-butane, iso-pentane,  $C_8H_{18}$ ,  $C_9H_{20}$ ,  $C_{10}H_{22}$ ,  $MeCl$ ,  $CF_3Cl$ , petroleum ether, naphtha, gasoline, and the resulting paraffinic and naphthenic layers are sep. App. is described.

**Dewaxing lubricating oils to produce a low-pour test oil.** Merrill Kiser (to Indian Refining Co.) U. S. 2,020,066, Nov. 5. For dewaxing a hydrocarbon oil, acetone, toluene and  $C_4H_8$  are added in such proportions that on cooling to a temp. below that at which the wax is pptd.  $C_4H_8$  is pptd. in cryst. form; the must. is then cooled to effect successive pptn. of wax constituents and of  $C_4H_8$  and the pptd. materials are sep. by filtration of the oil.

**Lubricants.** The Atlantic Refining Co. Fr. 784,803, July 25, 1935. A lubricant contains a mineral oil and an org. compd. of P, which may be chlorinated, e. g., Pennsylvania lubricating oil 90 and  $Bu_3P_2O$  or chlorinated tri-cresyl phosphate 10%.

**Lubricants.** International Hydrogenation Patents Co. Ltd. Fr. 785,651, Aug. 16, 1935. Lubricating compds. comprise a mineral lubricating oil and a "hydroformed" naphtha prep. by destructive hydrogenation of carbonaceous material of high mol. wt. (petroleum ext.) at above 450°.

**Hydrocarbon lubricants.** Carl Wulff and Wilhelm Brenner (to I. G. Farbenind. A.-G.). U. S. 2,020,714, Nov. 12. A small proportion (suitably about 0.5-10%) of a polymerization product having a mol. wt. of at least 1000 and derived from a vinyl compd. contg. O, such as stearic acid vinyl ester, is used with a hydrocarbon lubricating oil or the like for increasing the viscosity and lowering the setting point.

**Lubricant suitable for use under high pressures.** Bruce B. Farrington and Robert L. Humphreys (to Standard Oil Co. of Calif.) U. S. 2,020,021, Nov. 5. About 1-5% of a thiocarbonate such as amyl benzyl dithiocarbonate or diphenyl monothiocarbonate is mixed with a lubricating oil.

**Apparatus for testing lubricants under high pressure and rubbing speed.** Gerrit D. Boerlage (to Shell Development Co.). U. S. 2,019,948, Nov. 5. Mech. and operative details.

**Apparatus for testing the frictional resistance of lubricants.** Geo. L. Neely and Eldred E. Edwards (to Standard Oil Co. of Calif.). U. S. 2,020,565, Nov. 12. Structural, mech. and operative details.

**Removing asphalt from oils.** Standard Oil Development Co. Fr. 785,349, Aug. 7, 1935. Asphalt is pptd. from oils by adding a primary, secondary or tertiary aliphatic alc. of 4 or more C atoms or a cyclic alc. of a light petroleum hydrocarbon, and a pptg. agent comprising practically anhyd.  $MeOH$ ,  $EtOH$ ,  $PrOH$ , iso- $PrOH$ , di- or tri-hydric aliphatic alcs. or ketones, sepp. the oil phase from the asphalt phase and eliminating the diluent and pptg. agent from the oil phase. A supplementary amt. of pptg. agent, such as  $MeOH$ , may be added to the sep.

oil phase to effect a further sepn. The oil may be finally treated with PbOH or other selective solvent to improve its lubricating properties.

Asphalt solutions Alexander Wacker Gesellschaft für elektrochemische Industrie G. m. b. H. Brit. 432,406, July 25, 1935. See Fr. 780,112 (C. A. 29, 60144)

Phosphatide preparations suitable for making asphalt emulsions or for use in the textile industries, etc. Albert

Schwieger (to American Lecithin Co.). U. S. 2,020,062, Nov. 12. Phosphatide prepn., such as those from soybeans or fish roes are produced in dry form with an admixt. of at least 10% NaOH.

Extracting rosin from resinous wood Leavitt N. Bent (to Hercules Powder Co.). U. S. reissue 19,749, Nov. 12. A reissue of original pat. No. 1,888,025 (C. A. 27, 1503)

## 23—CELLULOSE AND PAPER

CARLETON E. CURRAN

Sorption of water vapor on cellulosic materials Edgar Libby and O. Maass. Can. J. Research 13B, 1-10 (1935).—A direct method used for measuring the adsorption of water vapor on cellulosic materials and in which a no. of improvements have been made is described. It is ensured that no vapors other than water are present no matter how long a time is required for the establishment of equilibrium. The adsorption and desorption isotherms of standard cellulose, spruce wood, surgical cotton, Kodak rag cellulose and bleached sulfite have been measured, and the data which indicate the time required for the establishment of true equilibrium have been given. The data of Urech and Williams (C. A. 18, 1754) for standard cellulose determined by an indirect method have been shown to be correct.

J. W. Shupley  
Transparent containers from cellulose acetate Tr Baumgartel. Kunststoffe 25, 283-4 (1935).—Production methods are briefly reviewed.

J. W. Perry  
Effect of the hemicellulose content of caustic soda solutions used for soaking in the manufacture of viscose II. Soyer. Rusta 10, 499 (1935).—Exptl. data are given to show the disadvantages of the presence of hemicellulose in NaOH liquors used for soaking cellulose in viscose manuf. and the advisability of using liquors containing as little hemicellulose as possible.

A. Papineau-Couture  
The technique of coloring celluloid Willy Hacke. Kunststoffe 25, 257-8 (1935).—A discussion emphasizing surface treating and dyeing.

J. W. Perry  
Preparation of viscose crepe M. Coynaud and J. Constant. Rusta 8, 447, 523-7, 605-9, 683-7, 765-9, 853-9, 935-67, 1033-9 (1935); 9, 27-31, 115-19, 197, 199, 273, 275, 583-93, 669, 671, 737, 739, 833-9, 897, 899 (1934); 10, 101, 103, 315, 317, 333-7, 459, 491 (1935).—A monograph.

A. Papineau-Couture  
Specific gravity of pulpwood. Rudolf Sieber. Papier-Fabr. 33, Tech. Teil, 305-9, 313-19 (1935).—Using Niehammer's method (C. A. 26, 5093), S. in collaboration with a group of pulp and paper mills examined 12,204 samples of German (spruce) pulpwood and 8095 samples of spruce from other sources. In the following summary, the figures in parentheses give the no. of samples, while the others are the mean sp. grs. based on oven-dried wood: Central German woods (893) 0.447; South German woods (2054) 0.452; "Fichtelgebirge" and Bavarian forest woods (1074) 0.459; East Prussian woods (92) 0.42; Austrian woods (554) 0.457; Czechoslovakian woods (3187) 0.445; Roumanian woods (93) 0.394; Russian woods (3127) 0.447; Polish woods (586) 0.438; Baltic woods (430) 0.450; Finnish woods (368) 0.453; Swedish woods (450) 0.453. The mean sp. gr. for all spruce samples examined was 0.448. These figures are for representative boles used in the paper industry. A composite graph shows that the sp. gr. range varies from 0.30 to 0.62. In general when the sp. gr. is plotted against the width of the annual rings, smooth curves result, the d of the wood dropping as the ring width increases. However, the same ring width may lead to very different sp. grs. depending on the formation of the crown and the root system of an individual tree. The mean sp. gr. of 699 samples of Scots pine was 0.505. That of 608 samples of aspen was 0.454. S. gives 19 graphs.

A. Papineau-Couture  
A new control method for (following the course of) the digestion in sulfite pulp production. Celestin Haider,

Papier-Fabr. 33, Tech. Teil, 321-9, 332-5, 341-4, 347-51 (1935).—This method depends on a rapid, quant. sepn. of Ca lignosulfonate I from SO<sub>2</sub> and the sugars of sulfite liquor. After a series of orientating expts., which are fully described, the following technique was developed: the liquor drawn from the digester is treated very gradually with CaCO<sub>3</sub> in excess, heated to 80-100°, and shaken vigorously until no further CO<sub>2</sub> is evolved. The mixt. is then filtered or centrifuged until the liquor is clear. After cooling to 20°, 2-cc. portions of this soln. are pipetted off, and each is treated with 8 cc. 95% alc. (added in 0.5-cc. portions with const. stirring). When the pptn. of I is complete, the mixt. is centrifuged at 1000 r. p. m. for 5 min., and the alc. soln. decanted. The solid residue is stirred to a paste with 95% alc. (amts. not specified) and the decantation repeated. When I contains little residual sugar, the addn. of 0.01-0.02 g. Kieselsol may be required to effect complete flocculation. The combined mother liquors contain the sugars. The solid residue I, freed from alc. by heating *in vacuo* and finally at 100-5°, is dissolved in 4 cc. H<sub>2</sub>O at 20°, and the w of the soln. detd. by means of an immersion refractometer, each graduation of which corresponds to a definite wt. of I. A table shows the relation between g. I of I and the refractometer readings. Corrections for vol. changes and slight losses due to soly. in alc. are also noted. From these, a simple formula was devised permitting the computation of g. I per 0.1 l. of the original liquor. Known solns. of I (with and without sugar) indicate that errors range from -4.0 to +2.3% (until the I content drops to 0.625 g. per 100 cc. in the original liquor). By making w readings on the SO<sub>2</sub>-free liquor, and subsequently detg. I, H. was also able to est. the sugar content of such liquors. These agree (within ± 5%) with values found by Glassmann's Hg-(CN)<sub>2</sub> reduction method (cf. Ber. 39, 503 (1906)). The courses of 7 digestions (yielding pulps with Sieber no. ranging from 22 to 62) were followed by using the above procedure. The results are tabulated and expressed graphically. The percentage of I in the spent liquor is correlated with the percentage of lignin and the Sieber no. of the pulp, and, provided a graph has once been drawn for a certain digester and a definite set of cooking conditions (in which the Sieber no. has been plotted against the I content), the former may always thereafter be estd. at any desired stage of the digestion, by detg. the I in the sulfite liquor. Eighty references.

Louis E. Wise  
The German pulp and paper industry viewed from the standpoint of domestically available raw materials. Roll and Runkel. Zellstoff-Faser 32, 145-51 (1935).—At present about 3 million solid measures (fm.) of German wood are used as raw materials by the paper industry, whose requirements approximate 8 million fm. R. shows that this deficiency can be met largely in Germany by increasing pulp yields, utilizing other raw materials (like beech) and those wood wastes that are now used in mfg. building boards and for fuel, and by substituting Scots pine for spruce in mfg. mine timbers. These questions are discussed in some detail from the viewpoint of the forester and the industrialist. Thirty-eight references.

Louis E. Wise  
Paper industry of Japan. D. N. Tyagar. Bumazhaya Prom 14, No. 9, 69-73 (1935). Chas. Blanc  
Stock used for the manufacture of paper impregnated

with synthetic resin Fritz Ohl *Wochs. Papierfabr.* 66, Tech. Teil, 696-8(1935).—The best paper used in the manu. of *elec. insulating board* usually contains strong, carefully washed soda pulp. A typical paper showed the following properties (the first figures being those taken in the machine direction, while those in the cross direction are listed in parentheses): breaking length, 9600 m. (6500 m.), double folds, 3000 (2500), stretch 3% (6%); bursting strength 3 kg./sq. cm. A satisfactory sample of 50 g./sq. m. Manila paper (which is less frequently used) was 0.06 mm. thick, showed 6300 m. breaking length, 3% stretch, 3000 double folds and 2.8 kg./sq. cm. bursting strength. When plates or boards are formed, both sides of the sheet are impregnated and a series of stacked sheets are laminated together under heat and pressure. The paper should have a relatively high d. and absorptive capacity, and should be smooth on one side only. Rosin but not latex size may be used. When molded articles are to be formed, paper of lower absorptivity and wt./sq. m. may be employed and only one side is impregnated.

Louis E. Wise  
Theory and practice of sizing of paper stock with rosin size Ya G. Khinchin *Bumashnaya Prom.* 14, no. 7, 12-18(1935); cf. C. A. 29, 7073<sup>1</sup>. Chas. Blanc

Utilization of the larch tree [cellulose content] in the U. S. S. R. (Matyuk) 22 Pollution of the Volga River by the Pravdinsk paper combine (Pavlinova) 14 Chemistry of yute lignin (Sarkar) 10 Reaction of lignin and vanillin (Fourment, Roques) 10 Viscosity of solns. of substances of high mol. wt., especially cellulose (Marchlewski) 2 Fireproofing paper, etc. (Fr. pat. 785,915) 25 Elec. app. for detg. the temp. of calendar rollers (U. S. pat. 2,020,067) 4

Cellulose Max Baczewski Austrian 142,761, Sept. 10, 1935 (Cl. 55c). In the manu. of cellulose from wood, straw, peat, etc., by the sulfate, sulfite or soda process, a wetting agent which is inert to cellulose and stable under the reaction conditions, e. g., a sulfone ester of a higher polyhydric aliphatic alc., is added to the digesting liquid and (or) to any of the solns. ordinarily used for pretreating the cellulose material.

Alkali cellulose Oscar Kohorn (trading as Oscar Kohorn & Co. Maschinenfabrik) and Rudolf Brandes. Brit. 433,414, Aug. 14, 1935. See Fr. 782,396 (C. A. 29, 7073<sup>2</sup>).

Alkali cellulose I. G. Farben und A.-G. Fr. 784,955, July 30, 1935. The lye and moist cellulose are mixed in a fiber separator, mixer or sulfuration drum, the d. of the cellulose representing more than 25% of the mixt. The lye is finely divided as a fog and the mixt. is cooled below 20° during the mercerizing.

Bleaching cellulose materials. Imperial Chemical Industries Ltd. Fr. 785,002, July 31, 1935. Wood pulp, etc., is treated with a sulfonic acid or sulfonic acid ester, such as cetyl sulfonic acid, oleyl ester of  $\beta$  hydroxyethanesulfonic acid, and sulfonic esters of decyl, dodecyl and octadecyl alics., or their alkali or  $\text{NH}_4$  salts, before, during or after treatment with a bleaching agent.

Cellulose foils L'accessoire de precision Brit. 432,772, Aug. 1, 1935. Transparent nonmisting partitions suitable for gas-mask windows, optical instruments, etc., are prep'd by completely satg. a foil of regenerated cellulose with a liquid so that the foil cannot swell further and fastening it while said to a frame like structure so that contraction on drying is prevented or minimized. In a modification, the said foil is fastened over the whole of the contacting surface of a transparent rigid carrier such as a sheet of glass, safety glass, celluloid, etc., by means of an adhesive, e. g., a soln. of gelatin in dil.  $\text{AcOH}$  and alc. The foil is preferably satd. by immersion in  $\text{H}_2\text{O}$  at about 50° for 15 min.

Cellulose products Henry Dreyfus U. S. 2,019,598, Nov. 5. The cellulose and lignin components of material such as spruce chips are sep'd. by impregnating the material under pressure with an aq. soln. comprising bisulfites such

as those of an alkali, Turkey-red oil and free  $\text{SO}_2$ , in which the proportion of free  $\text{SO}_2$  to combined  $\text{SO}_2$  is less than 1:2, and cooking the impregnated material at temps. of 100-160°. Cf. C. A. 29, 589<sup>3</sup>.

Finishing and softening cellulose products Deutsche Hydrierwerke A.-G. Fr. 784,966, July 30, 1935. Ether or esters of glycerol or other polyhydric alics. or cyclic ethers which contain one or more atoms of O in the ring or aliphatic alics. of high mol. wt. or mixts. of more than one of these are used. Examples are Me, lauric, decetyl, diphenyl and triphenyl ethers of glycerol, acetate, laurate or adipic ester of glycerol, triacetin, acetone-glycerol, acetone-arabitol, acetophenone-glycerol, trihydrofurfuryl acetate, furfuryl benzoate and cyclohexanone-glycerol phthalate, cetyl, lauric, octadecyl and myristic alics.

Films of cellulose derivatives Kodak-Pathé. Fr. 785,639, Aug. 14, 1935. One of the faces of films made from org. esters of cellulose is superficially hydrolyzed to prevent curling. In photographic films the face other than that coated with emulsion is hydrolyzed.

Purifying cellulose ethers David Traill and Imperial Chemical Industries Ltd. Brit. 433,397, Aug. 14, 1935. Cellulose aralkyl ethers are purified by treatment, preferably at elevated temps. with a medium contg. an alkali metal bisulfite, said medium being capable of swelling but not dissolving the cellulose ether. In an example, benzylcellulose is treated with 2 consecutive portions of a mixt. of a satd. aq. soln. of  $\text{NaHSO}_3$ , alc. and  $\text{C}_6\text{H}_6$ , then washed until the pH of the washings is 8-10. The purified ether, when made into a plastic and tinted with titanox. and a mixt. of Rhodamine GS and Rhodamine 6GBS, may be used to manu. dental blanks.

Esterifying fibrous cellulose Carl J. Malm and Charles L. Fletcher (to Eastman Kodak Co.) U. S. 2,019,920, Nov. 5. Cellulose such as cotton linters is pretreated with a lower aliphatic acid (such as propionic and acetic acids in the production of cellulose acetate-propionate) and is then esterified and there is added, after the mixing of the esterifying materials but before soln., an unsubstituted aliphatic ether having a b. p. above 70° such as isomyl ether which serves to expedite the process. Cf. C. A. 29, 4174<sup>1</sup>.

Cellulose ester compositions for films or sheets Stewart J. Carroll and Henry B. Smith (to Eastman Kodak Co.) U. S. 2,019,892, Nov. 5. A cellulose ester such as cellulose acetate is plasticized with butyl or isomyl carbamate.

Stabilizing organic acid esters of cellulose Carl J. Malm and Charles L. Fletcher (to Eastman Kodak Co.) U. S. 2,019,921, Nov. 5. An ester such as cellulose acetate is treated, at a temp. of about 35-65°, in a bath essentially consisting of acetic, propionic or butyric acid and a liquid aliphatic hydrocarbon material such as a petroleum distillate having a b. p. of about 65-90°.

Cellulose acetate compositions Geo. W. Seymour (to Celanese Corp. of America) U. S. 2,020,338, Nov. 12. Ethylene formal is used as a volatile solvent with cellulose acetate having an acetyl value above 66% in forming products such as filaments or films, etc.

Precipitating cellulose acetate from "acid dope." Hobart O. Davidson (to Viscose Co.) U. S. 2,021,937, Nov. 19. An "acid dope" from acetylation of cellulose is extruded as a stream below the surface of a bath of dil. aq. pptg. liquid, and the ppt. is simultaneously sheared into pellets of uniform size. App. is described.

Decorative foil from "cellulose dope." Harold K. Haviland (to Celluloid Corp.) U. S. 2,020,684, Nov. 12. Differently colored "dopes" such as those of cellulose acetate are introduced at various points into various sections of a hopper from which the "dope" is cast.

Use of p-phenylacetophenone as a crystalline material with cellulose acetate or the like. Thomas F. Murray, Jr. (to Eastman Kodak Co.) U. S. 2,019,720, Nov. 5. About 20% of p-phenylacetophenone may be used with cellulose acetate in forming sheets, etc., of cryst. or nacreous appearance.

Apparatus (with adjustable rollers) for manufacture of

films and foils from cellulose acetate solutions, etc. Edward Kinella (to Celanese Corp. of America). U. S. 2,020,687, Nov. 12. Structural, mech. and operative details.

Cellulose triacetate. Ernst Berl. Fr 785,462, Aug. 10, 1925. A partial reformation of the acetyl index of cellulose triacetate is effected by heating it with an alc capable of causing it to swell or having a solvent action thereon in the presence of a small amt of esterification catalyst such as  $H_2PO_4$  or  $H_2SO_4$ . The alc is preferably a simple alc, such as  $EtOH$ ,  $PrOH$ ,  $iso-BuOH$  or  $AmOH$ , or a glycol or a cyclic alc such as benzyl alc or cyclohexanol.

Reupening cellulose triacetate. The Dretillers Co. Ltd., Harold A. Auden, Hanns P. Staudinger and Philip Eaglesfield. Brit 433,785, Aug. 16, 1935. Cellulose triacetate (I) is treated to modify its soly by subjecting it to regulated heating in solid form, i. e., without first dissolving or suspending in a liquid medium, with a substantial quantity of an alc having a swelling but no solvent action, e. g.,  $Et$ ,  $Pr$ ,  $iso-Bu$ ,  $Am$  alcs or ethyleneglycol, or of an alc having a solvent action, e. g., cyclohexanol, benzyl alc, to serve as a retro-acetylating agent thereof in the presence of a small amt of an esterifying agent, e. g.,  $H_2PO_4$ ,  $H_2SO_4$ . The extent of the de-esterification is controlled by the time of treatment, temp and nature of the alc and catalyst. Among examples, (1) I is treated 2.5 hrs at  $100^\circ$  with  $BuOH$  150 and 80%  $H_2PO_4$ , 15 parts, the sepd acetate is substantially unchanged in Ac value, is still insol in  $Me_2CO$  but the viscosity in methylene chloride is increased by about 20%, and (2) I is treated for 45 hrs at  $40^\circ$  with  $BuOH$  100 and 20%  $HClO_4$ , 2 parts, the acetate, after sepn, is completely sol in  $Me_2CO$ .

Benzylcellulose. Alfred S. Leveley and Frederick C. Randall (to Imperial Chemical Industries Ltd.) U. S. 2,019,529, Nov. 5. A water-sol, high mol wt alkylated naphthalenesulfonic acid or Turkey-red oil is incorporated with a mass resulting from benzoylation to facilitate washing and purification.

Benzylcellulose. Edward Ditt (to Hercules Powder Co.). U. S. 2,020,691, Nov. 12. In sepg benzylcellulose from a crude benzylcellulose reaction mass, a water-sol, soap is added and the materials are formed into a foamy mass by malaxation before extrn with org solvents such as  $MeOH$  and water.

Centrifugal machine for nitrating cellulose. C. G. Haubold A.-G. Ger 619,499, Oct. 1, 1935 (Cl 826.4).

Carrier supports for making films from cellulose derivatives, gelatin and other plastic substances. Gennady Frenkel. Brit. 432,846, Aug. 2, 1935.

Laminated opaque regenerated cellulose material. Roger N. Wallach (to Sylvania Industrial Corp.) U. S. 2,022,061, Nov. 26. A flexible material comprises a transparent sheet of regenerated cellulose having on each side a heat-fusible coating and laminated on each side with a sheet of regenerated cellulose impregnated with a substance rendering it opaque and giving it a mat appearance (the sheets being held together by the heat-fusible coating).

Viscose and cuprammonium cellulose spinning solutions and their products such as filaments or films. Rudolph S. Bley (to North American Rayon Corp.). U. S. 2,021,849, Nov. 19. An alkylated naphthalene such as an ethyl- or butyl-naphthalene is added, for the production of a soft-luster product. Various examples are given.

Clarifying viscose centrifugally. Henri Walch (to De Laval Separator Co.). U. S. 2,019,944, Nov. 5. The viscose is rapidly heated to a temp. below its point of instantaneous coagulation, and in this condition is clarified in a high-speed centrifugal purifier, and is subsequently cooled at a rate controlled according to the saline index which is to be given to the viscose. App. is described.

Spinning nozzles for supplying viscose for the manufacture of films. Ernest Bleibler. Brit. 432,880, Aug. 6, 1935.

Device for equalizing variations in the rate of flow of spinning solutions delivered by spinning pumps. Hans Dannenberg (to I. G. Farbenind. A.-G.). U. S. 2,020,

633, Nov. 12. A thick-walled rubber tube forms part of the duct conveying the soln. from the pump to the spinning nozzle, the tube having a wall thicker than its bore and its exterior being subject to atm. pressure.

Colored viscose articles. La soie artificielle de Valenciennes and Leonard M. Glazener. Fr. 785,752, Aug. 19, 1935. An azo component is added to the spinning soln. and the dye developed by coupling after spinning, or diazotization and coupling may take place after the spinning.

Desulfuring viscose rayon. Vereinigte Glanzstoff Fabriken A.-G. Brit. 432,992, July 31, 1935. The freshly spun filament from an acid spinning bath is washed, immediately after its pptn, with  $H_2O$ , and as far as possible, salt free  $H_2SO_4$  in individual filaments over a filament stretch of several meters, then washed preferably with hot  $H_2O$  and dried in known manner. Any S left may be easily removed, e. g., by a simple cleaning with soap such as is often involved in the dyeing or prepn of the products. App. is described.

System for drying rayon cakes. Algemeene Kunstzijde Unie N. V. Ger 617,958, Aug. 29, 1935 (Cl 29a 8 08). Addn to 562,001 (C 27, 843).

Artificial products such as rayon. Leon Lilienfeld U. S. 2,021,843, Nov. 19. A product obtained from treating viscose with a trithiocarbonic acid ester of a polyhydroxylic alc is formed, in soln in a solvent, into a shape such as a filament and then treated with a reagent which has both a coagulating and a plasticizing effect. Numerous examples and details are given. U. S. 2,021,864 relates to a process in which viscose is treated with at least one halogen-contg. org. reagent (other than a halohydrin or a halofatty alc) which reacts on viscose to cause substitution of a hydroxyl H atom in the cellulose radical by an org. radical, the resulting soln. being then shaped and treated with a coagulating and plasticizing material. Numerous examples are given.

Colored rayon. Courtaulds Ltd. Fr 785,343, Aug. 7, 1935. Colored rayon is produced by spinning viscose contg. a leuco deriv. of a vat dye, the dye being kept in soln in the viscose by the addn of a glycol ether, e. g.,  $Et$  or  $Me$  ether of ethylene- or diethylene-glycol.

Wet treatment of formed rayon filaments. August Hartmann, Walter Hoeslinghoff and Karl Meyer-Gaus (to American Bemberg Corp.). U. S. 2,020,657, Nov. 5. Various details of app. and operation.

Artificial materials such as threads or films. Leon Lilienfeld U. S. 2,021,861, Nov. 19. A soln of a hydroxyalkyl cellulose xanthate is formed into the desired shape and the shaped soln. is treated with a coagulating agent. Numerous examples are given. U. S. 2,021,862 relates to a process in which a product obtained from viscose and a halogen deriv. of a polyhydroxylic alc, such as a chlorohydrin is shaped and treated with a coagulating agent which has a plasticizing effect on the coagulated material. Numerous examples are given.

Artificial filaments, etc. British Celanese Ltd. and Percy I. C. Sower. Brit. 433,793, Aug. 21, 1935. Filaments, ribbons, etc., having a basis of cellulose acetate or other org. deriv. of cellulose are obtained by a wet spinning process in which the shaped materials are coagulated in a medium contg. a solvent or plasticizer for the cellulose deriv., and the wet materials are stretched and then allowed to shrink under the action of the solvent or plasticizer. In an example, cellulose acetate soln. is extruded into an glycol monoacetate and the coagulated filaments pass round a stretching roller and under a 2nd roller dipping into a bath of  $H_2O$ , then round a 3rd roller rotating more slowly than the 1st so as to allow shrinkage and finally to a take-up device.

Artificial filaments, yarns, films, etc. British Celanese Ltd. Brit. 433,219, Aug. 12, 1935. A soln. of an org. deriv. of cellulose contg. an oil, e. g., mineral oils, petroleum jelly, olive or castor oil and (or) a precipitant, e. g., xylene, gasoline,  $PhMe$ ,  $C_6H_6$ , kerosene, together with a soap and (or) tetrahydronaphthalene is spun. The soln. may contain also sulfonated oils or sulfonated aromatic

acids, fillers, pigments, dyes, lakes, fire retardants, plasticizers and sizes

Manufacture of artificial filaments such as those formed from cellulose acetate, etc. by the evaporative spinning method. Edward Kinsella and Reginald H. J. Riley (to Celanese Corp. of America) U. S. 2,019,609, Nov. 5 Various details of app. and operation

Apparatus (comprising a series of digesters) for the continuous manufacture of cellulose. Lubomir Lemberger Austrian 142,891, Sept. 25, 1935 (Cl. 55c)

Centrifugal apparatus for recovering fibers, etc., from waste waters from the manufacture of paper Firma J. M. Voith Austrian 142,807, Sept. 25, 1935 (Cl. 55a)

Apparatus for pulping wood Johannes Tamaschke Ger. 593,533, Sept. 17, 1935 (Cl. 55a. 1 01)

Apparatus for testing paper pulp. Alfred Schopper, U. S. 2,022,010, Nov. 20 Various structural, mech., and operative details of an app. with a strainer vessel.

Treating paper pulp for delignification before bleaching James S. Boone (to Hooker Electrochemical Co.) U. S. 2,021,612, Nov. 19 An aq. suspension of material such as wood pulp is treated with an elemental halogen such as Cl<sub>2</sub> in the presence of CaCO<sub>3</sub> and lime, the lime being proportioned to the halogen so as to maintain a nonalk. condition in the suspension. App. is described.

Apparatus for introducing reagents into liquid suspensions as in adding chlorine to aqueous paper pulp suspensions. Kenneth E. Stuart (to Hooker Electrochemical Co.) U. S. 2,021,616, Nov. 19 Various structural and operative details

Tubing of fibrous material such as sulfite screenings and ground wood pulp. Edmund Burke. U. S. 2,019,890, Nov. 5. A tube which is suitable for various purposes is constructed of a single band of felted fibrous material spirally wound upon itself and compacted into the form of a series of continuously overlapping convolutions. App. and various mfg. details are described.

Paper. Harold R. Rafton (to Raftold Process Corp.) U. S. 2,020,324, Nov. 12. In the manuf. of sized paper filled with alk. filler, there is applied to a fibrous web contg. alk. filler substantially water-insol. thermoplastic sizing material, such as paraffin in soln. or emulsion, in sufficient amt. to have a sizing effect on the paper.

Paper-making apparatus and sizing device Wm. P. McCorkindale (to McCorkindale Co.) U. S. 2,021,125, Nov. 12 Various structural and operative details

Device for use with paper making apparatus for indicating variations in weight of the paper. Ambrose Idall U. S. 2,020,001, Nov. 3 Various structural, mech. and operative details

Automatic control devices for paper-machine driers Norman B. Gibbs U. S. 2,020,056, Nov. 5 Various mech. and operative features.

Vertical type drier for paper webs. Carl E. Berry (to Reloit Iron Works). U. S. 2,019,649, Nov. 5 Structural, mech. and operative details

Apparatus for supplying web material as in making pasteboard box-blanks. Cutler D. Knowlton (to Hoag-Sprague Corp.) U. S. 2,020,419, Nov. 12. Structural and mech. features

Endless cloths or wire fabrics for use in paper making, etc. Hans Kurtz U. S. 2,020,894, Nov. 12 Various mfg. details are described.

Apparatus for testing the smoothness of paper or like sheet materials. Ralph A. Masten (to International Paper Co.) U. S. 2,019,541, Nov. 5 The material is held against a smooth surface between which and the sample to be tested air is passed at substantially const. pressure, and the time required for a unit quantity of air to pass is indicated

Hollander for comminuting scrap paper Maschinen-

bau-Werkstätte Niefen G. m. b. H. Ger. 619,102, Sept. 24, 1935 (Cl. 55c. 8 10).

Hollander knife rolls. Albert Elsenhans Ger. 619,427, Sept. 30, 1935 (Cl. 55c. 4 10) Structural features are described.

Sizing fibrous material for making paper, etc. Aktiebolaget Defibrator. Brit. 432,914, Aug. 6, 1935 See Fr. 768,181 (C. A. 29, 603).

Sizing paper. Louis L. Larson (to E. I. du Pont de Nemours & Co.) U. S. 2,022,004, Nov. 26 For increasing the water repellency of absorbent materials such as paper, they are treated with an aq. emulsion of polyhydric alc.-polybasic acid resin having an acid no. of 25 to 64 and contg. in combined form about 65-90% of a polyhydric alc. ester of an aliphatic monocarboxylic acid of the formula RCOOH, in which R is an alkyl radical contg. not less than 15 C atoms, such as a product from glycerol, stearic acid and phthalic anhydride

Apparatus for moistening paper bands Jagenbergwerke A.-G. Ger. 618,773, Aug. 10, 1935 (Cl. 55c. 3)

Watermarking composition. John C. Boyer (to National Liting Exchange). U. S. 2,021,141, Nov. 19 An emulsion of a suitable printing ink consistency comprises Canadian balsam 8-20, turpentine 5-17, finely divided substantially colorless mineral matter such as diatomaceous silica 8-25 and castor oil 12-30%, emulsified with a slightly alk. aq. soln. of borax and contains a substantially colorless indicator such as phenolphthalein reactive to both acids and alkalis, so that the compn. is suitable for "safety paper" for checks, etc.

Crinkled paper, etc. Rudolf Heilmann. U. S. 2,019,903, Nov. 5 Various mech. and mfg. details are described

Paper for checks, etc. Begolt Louis Leviet-Arnaud. Fr. 785,574, Aug. 13, 1935 The falsification of documents is prevented by introducing into the paper used or applying to its surface substances contg. one or more heterocyclic rings having one or more S atoms, in combination or not with oxidation catalysts or substances favoring oxidation. Thus, dehydrothio-p-toluidine, accompanied or not by alkali ferrocyanide and sulfite, may be used. These may be added to the ink used instead of applying them to the paper

Laminated printing paper. Carl Winkler. Ger. 619,052-3, Sept. 20, 1935 (Cl. 55f. 15 20). A paper sheet, supersized sufficiently to be impermeable to printing ink, is interposed between sheets of ordinary sized paper (619,052). According to Ger. 619,053, the supersized middle sheet may contain pond. cork.

Cigarette paper. La Cellophane, Soc. anon. Fr. 785,202, Aug. 3, 1935 A paper which burns without odor or smoke is made of viscose to which is added paraffin oil or liquid petrolatum oil

Wrapping paper for cigarettes, etc. Philip Hornstein U. S. 2,020,646, Nov. 12 A cigarette paper which will retain its shape during and after the burning of the cigarette filler and which is substantially impervious to nicotine is produced by adding finely ground asbestos to the pulp, boiling to produce a homogeneous mass and adding gum acacia during the boiling

Paper board with identification marks. Charles C. Colbert, Geo. E. Preston and Lloyd C. Daly U. S. 2,019,845, Nov. 5. Identification marks are placed under a clay coating and are adapted to be rendered apparent by wetting the clay coated side of the board.

Coating fibrous materials such as paper cartons to render them water repellent and antiseptic. Irwin Stone. U. S. 2,021,137, Nov. 10. A wax coating such as a paraffin and oil mixt. is used contg. a small proportion (suitably about 0.1%) of 8-hydroxyquinoline, its benzoate, palmitate or acetyl deriv. or the like

## 24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE AND C. G. STORM

A photographic investigation of flame movements in gaseous explosions. VII. The phenomenon of spin in

detonation. Wm. A. Bone, Reginald P. Fraser and Wm. H. Wheeler. Trans. Roy. Soc. (London) A253, 29-68

(1935); cf. C. A. 26, 1788—The phenomenon of spin in gaseous detonations previously described (C. A. 26, 1788) has been further investigated with an improved form of the Fraser high-speed camera which permits accurate measurement of flame speeds occurring in as short a time as 0.00001 sec. The expl. work has been done under a variety of conditions to study the influence of tube diam. and shapes, and of elec. and magnetic fields, upon detonation in a  $2\text{CO} + \text{O}_2$  medium. The explosive mixt. was investigated both moist and dry, and both with and without the successive adds. of electrolytic gas. The results of these recent expls. lead to the new conception of the detonation wave in gaseous explosions as a more or less stable assocn. of an intensely radiating flame-front with an invisible shock wave immediately ahead of it. The detonation wave is the propagation through the gas of an intense combustion set up in the shock wave by radiation from an assoc. flame front, spin occurring whenever the radiation from an attenuated flame-front causes localized intensive excitation of mols. in the shock wave. As a consequence the head of detonation begins to rotate and follow a spiral course along the walls of the explosion tube. All influences that destroy the spinning head (elec. and magnetic fields) cause the spin to cease and also the sep. of the flame-front from the assoc. shock wave so that the flame-speed falls and detonation ceases. The phenomenon is restored by reducing the distance between the still radiating flame-front and its formerly assoc. shock wave. Selected photographs illustrate the phenomena described.

C. C. Kieess

**Explosives, blasting cartridges** Soc. anon. d'Arendonck Brit. 432,850, Aug. 2, 1935 Potassic feldspar is used to prevent ignition of inflammable gases or materials in blasting. It is preferably mixed with a K salt or known ingredients, e. g.,  $\text{CaF}_2$ ,  $\text{NaCl}$ ,  $\text{CaCO}_3$ . A binder may also be added, e. g., clay, portland cement, plaster of Paris. One example contains orthoclase 55,  $\text{K}_2\text{SO}_4$  35 and plaster of Paris or clay 10%. The compn. is preferably made into a sleeve surrounding the explosive but may be incorporated, e. g., up to 30%, in the explosive which may be a mixt. of nitroglycerin 11, dinitrotoluene 1,  $\text{NaCl}$  22,  $\text{NH}_4\text{NO}_3$  59, cellulose 6.85 and lamp black 0.15 parts.

**Blasting cartridges** James Taylor and Imperial Chemical Industries Ltd. Brit. 431,935, July 15, 1935 In cartridges comprising a gas-generating charge in a pressure-resistant casing that vents the gases at a certain pressure, the charge consists of a mixt. of an alkali metal nitrite with 1 or more  $\text{NH}_4$  salts, which may be powd. or granular and may include small amts. of other ingredients, e. g., moisture, borax, mineral jelly. In Brit. 431,936, July 15, 1935, the charge consists of a liquid medium including constituents capable of exothermic gas-evolving decompn., e. g., a soln. of  $\text{NH}_4\text{NO}_3$  and (or)  $\text{NH}_4\text{NO}_2$ , or mixts. of alkali metal nitrates or nitrites with 1 or more  $\text{NH}_4$  salts and (or) urea, or a mixt. of  $\text{H}_2\text{O}_2$  and alc., together with means for initiating the decompn.

**Blasting cartridges** Alfred E. Lance Brit. 432,308, July 24, 1935 An absorbent for liquid O for blasting

cartridges comprises cellulose materials, e. g., sawdust, cork, peat, mixed with hydrocarbons having high b. ps. but of pasty consistency at ordinary temps., e. g., petroleum jelly, "Vaseline." Up to 20% hydrocarbon may be used and other ingredients may be added, e. g.,  $\text{MgCO}_3$ , kieselguhr, in amt. from 10 to 15% to prolong the life of the cartridge while reducing its strength, or Al or Fe-Si to increase its strength.  $\text{NaCl}$  and salts contg.  $\text{H}_2\text{O}$  of crystn. may be used as temp.-reducing ingredients.

**Blasting cartridges** The Lightfoot Refrigeration Co. Ltd. and Alfred E. Lance Brit. 432,338, July 24, 1935. An absorbent for liquid O for the cartridges comprises cellulose materials, e. g., sawdust, peat, cork, graded to pass through a 36-mesh screen but not through a 60-mesh screen. Metallic ingredients, e. g., Al, Fe-Si, when included, are similarly graded. Other ingredients, e. g.,  $\text{MgCO}_3$ , kieselguhr, may also be used to the extent of 10-15%.

**Priming compositions** Joseph D. McNutt (to Winchester Repeating Arms Co.) Brit. 432,096, July 19, 1935 Alkali or alk. earth salts of dinitrophenylazide are used in priming compns. instead of the Pb salt as described in Brit. 406,228 (C. A. 28, 4603). The K salt is prepd. by dissolving diazodinitrophenol in  $\text{Me}_2\text{CO}$  and adding an aq. soln. of  $\text{KN}_3$ , the temp. being maintained below  $50^\circ$ . The K salt is pptd. and filtered. Other salts are prepd. similarly. Cf. C. A. 29, 6274.

**Smoke-producing and wind-indicating cartridges** Francis Rankin and Imperial Chemical Industries Ltd. Brit. 432,438, July 20, 1935 A cartridge for discharge from a firearm from an aircraft toward the ground comprises, in addn. to the smoke-producing compn., a compn. that is ignited with the smoke-producing compn. when the cartridge is discharged and remains incandescent for a sufficient time to enable it to reignite the smoke-producing compn. if this is extinguished on landing. The incandescent compn. may be a mixt. of equal parts of Si, Fe-Si, Ca silicide or Zr with  $\text{KNO}_3$ ,  $\text{NaNO}_3$ ,  $\text{KClO}_4$ , or  $\text{KMnO}_4$ , baked hard.

**Safety detonating device for use in torpedoing oil wells** Haskell M. Greene U. S. 2,020,410, Nov. 12 Various structural details of a device including clockwork and an elec. control.

**Lead salts of dinitrosalicylic acid** Willi Brün (to Remington Arms Co.) U. S. 2,021,407, Nov. 19 Normal and acidic Pb salts of dinitrosalicylic acid are produced by adding a soln. of a Pb salt such as the nitrate, acetate or tartrate to a soln. formed from dinitrosalicylic acid and  $\text{NaOH}$ . The normal Pb dinitrosalicylate has a sensitiveness to shock substantially equal to that of Hg fulminate.

**Miners' safety lamps** Thomas A. Jones Brit. 433,577, Aug. 16, 1935

**Miners' and other safety lamps** Ernest A. Hailwood Brit. 430,721, June 24, 1935, divided on 432,962 and addn. to 419,594 (C. A. 29, 3164), 431,718, July 15, 1935, and 432,062, July 31, 1935

**Paper matches** Nels H. Nelson U. S. 2,022,088, Nov. 26 Various details of app. and operation

## 25—DYES AND TEXTILE CHEMISTRY

L. A. OLNEY

Further notes on the suitability of metals for dyeing machinery. J. G. Grundy. *J. Soc. Dyers Colourists* 51, 377-84 (1935); cf. C. A. 23, 2829—An investigation to det. the advantages of stainless steels for dyeing machinery as compared with wood, Fe, Cu, bronze, brass, monel metal, etc. Staybrite FDP differs in compn. from the original Staybrite in having a content of 0.50-1.0% Ti. This brand has no deleterious effect on dyes. Staybrite FMB differs from the FST and FDP brands in having a content of 2.3% Mo. Tables show the behavior of different metals under various treatments. W. H. Boynton

The dyeing of bathing suits O. Fuchs *Texts* 13,

559 (1935)—Attention is drawn to a no. of dyes of J. R. Geigy, Basel, which are suitable for this class of goods.

A. Papineau-Couture

**Effect of bleaching and dyeing conditions on the fastness to light of dyeing on albene** Jean Rolland. *Texts* 13, 401; *Rusta* 10, 447 (1935)—Dyeings on albene (lusterless acetate rayon) which has been bleached with  $\text{KMnO}_4$  and  $\text{Na}_2\text{SO}_3$  are affected by light, because of incomplete elimination of  $\text{Na}_2\text{SO}_3$ . This defect is overcome by using  $\text{H}_2\text{C}_2\text{O}_4$  instead of  $\text{Na}_2\text{SO}_3$ . This treatment is shown to have an adverse influence on the tensile strength and elongation of the fabric.

A. Papineau-Couture



The fading of dyed textiles—factors which influence it: instruments for testing fastness A. C. Goodings *Am. Dyestuff Repr.* 24, 663-5. *Am. Silk Rayon J.* 54, 33-4, 54-5 (Nov., 1935).—Sunlight, humidity and temp. are among factors influencing the fading of dyed textiles. Instruments for testing color fastness are discussed.

Notes on the inner and outer layers of silk cocoons. R. Trunokaye *J. Soc. Dyers Colourists* 51, 384-7 (1935).—It is shown that the outer layer filaments contain more sericin A than the inner ones, and less sericin B in the outer layer than in the inner. Several photomicrographs are shown.

Dull finishing of rayon knitted fabric F. P. W. Williams *Am. Dyestuff Repr.* 24, 643-4 (1935).

After-treatment of rayon crepe prints (drying) Allan P. Kirk *Am. Silk Rayon J.* 54, 29-31 (Nov., 1935), cf. *C. A.* 29, 6765<sup>4</sup>.—The 3 stages in the drying of any print are (1) the dripping stage, (2) the moist stage and (3) the completely dry stage. The first is represented by the goods immediately after washing, the second by the hydroxide or suction-dried state and the last by the stentered and widthed state. The tentering stage is the most important from the crepeing and widthing point of view.

Cotton filling materials David Hilfmarb *Am. Dyestuff Repr.* 24, 671-4 (1935); cf. *C. A.* 29, 6766<sup>4</sup>.—Cotton fibers for stuffing purposes include a great variety, but cotton furnishes the largest share. Cotton fibers for filling are classified as (1) linters, (2) new cotton mill processing wastes, (3) new reclaimed wastes from fabrics and (4) second hand reclaimed waste from fabrics, particularly shoddy. Phys. inspection and microscopic and chem. testing reveal the sources of the wastes.

Bleaching cotton piece goods with hydrogen peroxide R. E. Rupp *Cotton* 99, 45-8 (Nov., 1935).—The bleaching of cotton piece goods with  $H_2O_2$  is described. The preliminary alk. boil is shown to be of major importance. The bleaching depends further on the time, temp and concns of both  $H_2O_2$  and alkali.

Crease-resisting fabrics I. H. H. Chester *Am. Dyestuff Repr.* 24, 553-7, 622-4, 632-3, 645-50, 665 (1935).

Warp sizing I. Paul Seydel. *Cotton* 99, 43-4 (Nov., 1935).—Waxes, nitrogenous substances and mineral contents of cotton are discussed.

The use of methyl green for detecting traces of alkali in fibrous materials Ellis Clayton *J. Soc. Dyers Colourists* 51, 387-8 (1935).—A dil. soln of methyl green (0.1% No. 684), especially when used at a temp. of 80-100°, is a good reagent for the detection of minute traces of alkali in textile fibers. A standard 0.1% soln in distd. water was prepd. One cc. of this soln in 200 cc distd. water was practically unaffected when boiled in a Pt basin for 30 sec., with similar results when repeated in the presence of 0.25 g purified wool. One cc of a 0.1% soln of methyl green, 200 cc distd. water and 0.25 cc of 0.01 N NaOH became decolorized almost completely on boiling.

The arsenic problem. Report of a case of probable arsenic dermatitis from wearing apparel Maurice J. Reuter *Arch. Dermatol. Syphilis* 31, 811-18 (1935).—Analysis of the suit cloth causing the dermatitis showed 0.013 mg. As per g. material, apparently incorporated in the aniline black dye used.

Treatment of sponge towels P. Barsy *Tiba* 13, 403 (1935).—Brief practical operating directions.

Treatment of horsehair R. Monravel *Tiba* 13, 409, 411 (1935).—Practical operating directions on the bleaching of horsehair, based on 20 yrs' experience.

Chlorinated rubber in protective coatings (Redfarn) 30 Benzimidazole derivs (Fr. pat. 784,869) 10 Naphthalene derivs (Fr. pat. 785,588) 10 Impregnating

wooden spools, shuttles, etc., with resinous substances (U. S. pat. 2,020,172) 20 Phosphatide preps for use in the textile industries (U. S. pat. 2,020,662) 22 Preserving rubber latex [compn. for covering fabrics] (Brit. pat. 433, 116) 30

Dyes Société pour l'ind. chim. à Bâle. Brit. 433,090, Aug. 8, 1935. A negatively substituted monophthaloylcarbazole, e. g., nitro and halo derivs. of a phthaloylcarbazole, phthaloyl-naphthylcarbazole or phthaloyl-1,2,3,4-tetrahydrocarbazole, is caused to react with a nitrogenous compd. capable of being vatted and contg. at least 1 H attached to N, e. g., amino derivs. of anthraquinone, benzanthrone, mesobenzodanthrone, anthanthrone, dibenzopyrenequinone, anthraquinonecaradone and a pyranthrone, and the reaction product is treated with an acid condensing agent, e. g.,  $H_2SO_4$ ,  $ClSO_3H$ ,  $AlCl_3$  alone or with a tertiary base or  $NH_3$ . The products may be further halogenated, if desired. For an example, cf. Swiss 171,369 (C. A. 29, 5665<sup>9</sup>).

Dyes. I. G. Farbenind. A.-G. (Georg Kränzlein, Heinrich Greune and Branz Brunnerger, inventors). Ger. 616,661, Aug. 2, 1935 (Cl. 22e. 7 02). Water-sol. dyes are prepd. by heating compds. without sulfo groups of the general formula  $CO \text{ C(NHR) CX.CO C(NHR) CX}$

(benzene ring) with strong  $H_2SO_4$ ,  $ClSO_3H$  or oleum, in the presence or absence of diluent,  $H_2BO_3$  or an oxidation agent. In the above general formula X represents H or halogen, R a univalent aromatic residue of the benzene, naphthalene, anthracene, carbazole, or diphenylamine series, contg. no OH and COOH groups in the o-position to each other. In an example 2,5-diamino-3,6-dichloro-1,4-benzoquinone is heated to 95° with  $H_2SO_4/H_2O$  and  $K_2MnO_4$  to give a dye coloring animal, vegetable and artificial fibers in brown shades.

Dyes Chemische Fabrik vorm Sandoz Fr. 785,404, Aug. 10, 1935. Sol. dyes of the anthraquinone series are prepd. by condensing, in an appropriate medium, and by means of an oxidizing agent, a 1-arylaminoanthraquinone substituted in any manner but sulfonated at least once in the anthraquinone ring, and in which the position para to the imino group in the phenyl ring is free. Two mols combine forming a benzidine linkage between the anthraquinone radicals. The anthraquinone radical may be replaced by an anthrapyridone, anthrapyrmidone or anthrapyrmidine radical. Examples are given Cf. C. A. 20, 293<sup>9</sup>, 294<sup>9</sup>.

Mixtures of dyes I. G. Farbenind. A.-G. Fr. 785,269, Aug. 6, 1935. Mixts. of dyes contg. at least one dye of the azabenzanthrone series, the mol. of which contains also at least one acridine ring, with other dyes or dye constituents are used. Thus 8-azabenzanthrone- $\alpha$ -aminoanthraquinone is mixed with the dyes of U. S. 1,207,982 (C. A. 11, 332) or 1,452,505 (C. A. 17, 2196).

Azo dyes Herbert Kracker (to General Aniline Works). U. S. 2,019,914, Nov. 5. Dyes producing orange to red shades of good fastness to light are formed from components such as diazotized 1-amino-2-halobenzenes-6-carboxylic acid alkyl esters and  $\beta$ -naphthol, pyrazolone derivs., etc. Numerous examples are given.

Azo dyes I. G. Farbenindustrie A.-G. Brit. 432,599, July 30, 1935. Dyes are made by coupling diazotized aromatic amines not contg.  $SO_3H$  or COOH with N-aryl-amino nitriles not contg.  $SO_3H$  or COOH and of formula,  $X(Y)NRCN$ , where R is  $CH_3$  or a series of  $CH_2$  groups the H atoms of which may be substituted by alkyl, aryl or aralkyl, X is H or alkyl, hydroxyalkyl, aryl or aralkyl and Y is aryl, which may be substituted. Among examples, (1) diazotized 2-chloro-4-nitroaniline is coupled with N-ethyl-N-phenyl- $\beta$ -aminopropionitrile, and (2) diazotized 5-nitro-2-aminophenol is coupled with N-ethyl-N-methyl- $\beta$ -aminopropionitrile.

Azo dyes I. G. Farbenindustrie A.-G. Brit. 433,201, Aug. 6, 1935. See Fr. 708,913 (C. A. 29, 000<sup>9</sup>). The derivs. of 8-hydroxyquinoline should be free from COOH groups and the diazotized halo amino benzenes should also be free from COOH groups.

**Azo dyes.** I. G. Farbenind A.-G. (Richard Fleischhauer, inventor). Ger. 610,620, Aug. 1, 1935 (Cl. 22a.9). Addn to 615,799 (C. A. 29, 8352<sup>9</sup>). The method of 615,799 is modified by replacing one or both mols. of the diazotized *p*-nitro- or *p*-acylamino-arylamines by the corresponding *m*-comps. Thus, 1-amino-4-nitrobenzene is diazotized and coupled with 1-amino-8-naphthol-4,6-disulfonic acid. The resulting monoazo dye is coupled with the diazo compd from 4-nitro-2-amino-1-methoxybenzene in soda-alk. soln. The isolated diazo dye is reduced with Na<sub>2</sub>S and the product salted out, filtered and washed. The resulting dye is tetrazotized and treated with excess of Na<sub>2</sub>CO<sub>3</sub> and 1,3-diaminobenzene. The resulting dye colors vegetable fibers in black shades which are rendered wash-fast by treatment with CH<sub>3</sub>O and are rendered wash-fast and greenish by treatment with CH<sub>3</sub>O and a Cu salt. Other examples are given.

**Azo dyes** I. G. Farbenind A.-G. (Hans Krzizalla and Walter Lambacher, inventors). Ger. 617,848, Aug. 27, 1935 (Cl. 22a.1). Addn to 590,190 (C. A. 28, 2193<sup>9</sup>). 8-Hydroxyquinoline and its substitution products are converted into complex metal compds by the methods ordinarily used in making metal compds of azo dyes. The products are then coupled with diazo compds not contg a group capable of combining with metals. Metalliferous dyes identical with those obtainable by the process of Ger. 590,190 are produced. Sp. processes are described.

**Azo dyes** I. G. Farbenind A.-G. Fr. 784,909, July 29, 1935. Dyes are prepd by causing aromatic NO<sub>2</sub> compds to react with aromatic diamine or aminohydroxy compds in alk. medium, with the exclusion of NO<sub>2</sub> compds giving rise to the formation of stilbene, dinitrostilbene and dinitrodibenzyl compds. A reducing agent of weak action may be added. Thus, *p*-nitrobenzoic acid and *p*-phenylenediamine are heated in aq. NaOH. On cooling 4-aminoazobenzene-4'-carboxylic acid crystallizes. By acidification 4,4'-diaminoazobenzene seps and by reduction gives *p*-phenylenediamine again. Other examples are given.

**Azo dyes** I. G. Farbenind A.-G. Fr. 784,910, July 29, 1935. Dyes contg in the ortho position to the azo group (in the diazotization or coupling component) an aminoarylamino or aminoarylaminoarylamino group are prepd. by condensing azo dyes contg an amino group in the position ortho to the azo group with a nitroaryl halide, reducing the NO<sub>2</sub> to NH<sub>2</sub>, repeating if necessary the condensation and reduction and transforming if desired, the aminoazo dyes to other derivs. Thus, *o*-aminoazobenzene-3'-sulfonic acid is condensed in water with *p*-nitrobenzyl chloride in the presence of AcONa or NaOH, to *p*-nitrobenzyl-*o*-aminoazobenzene-3'-sulfonic acid which is reduced to the NH<sub>2</sub> compd. and this is treated with aminobenzoic chloride. The product dyes cotton yellow shades, and on diazotization and coupling with 1-phenyl-3-methyl-5-pyrazolone a yellow and with  $\beta$ -naphthol an orange shade is obtained. Other examples are given.

**Azo dyes.** I. G. Farbenind A.-G. Fr. 785,303, Aug. 7, 1935. Cellulosic fibers are dyed by treating them with a suspension of the coupling component in a fine state of division and afterward developing the dye in a bath contg a diazo compd. Between the 2 steps a drying or treatment in an alk. bath may take place. Thus, a paste of 1-(2',3'-hydroxynaphthylamino)-3,4,5-trichlorobenzene with the condensation product of  $\beta$ -naphthalenesulfonic acid and CH<sub>3</sub>O is dried, ground and dispersed in water. Cotton is impregnated with the dispersion, treated in a bath contg NaOH and dried. On developing with diazotized 2,6-dichloro-1-aminobenzene a scarlet dyeing is obtained.

**Azo dyes** I. G. Farbenind A.-G. Fr. 785,334, Aug. 7, 1935. Azo dyes insol in water are produced from diazoazo compds sol in water and suitable coupling components for ice dyes, the development of the dye being carried out in neutral or feebly alk. medium, provided that the coupling component is brought on to the fiber in the free OH form. The coupling component may be

applied in alk. medium and then treated with an acid or acid-forming substance in amt. sufficient to produce a neutral or feebly alk. reaction. The development is carried out at 80-100° in the presence of moisture.

**Azo dye.** I. G. Farbenind A.-G. Fr. 785,621, Aug. 14, 1935. A bright red fast dye is prepd. by diazotizing 1-amino-2-methoxy-5-trifluoromethylbenzene and coupling with 1-(2',3'-hydroxynaphthylamino)-3-methyl-4-methoxybenzene.

**Azo dyes** I. G. Farbenind A.-G. Fr. 785,692, Aug. 10, 1935. Diazotized aromatic amines contg. no SO<sub>2</sub>H or COOH groups are combined with (*N*-arylamino)-carboxylic nitriles also contg. no SO<sub>2</sub>H or COOH groups and of the formula X(Y)NRCN, where R is CH<sub>3</sub> or a chain of CH<sub>2</sub> groups, X is H, alkyl, hydroxyalkyl, aryl or aralkyl and Y is aryl. Thus, *p*-nitroaniline  $\rightarrow$  (*N*-phenyl- $\beta$ -amino)propionic nitrile dyes acetate silk orange-yellow. 1-Amino-2-chloro-4-nitrobenzene  $\rightarrow$  (*N*-ethyl-*N*-phenyl- $\beta$ -amino)propionic acid and *p*-nitroaniline  $\rightarrow$  (*N*-hydroxyethyl-*N*-(*m*-methylphenyl)amino)acetic nitrile dye acetate silk scarlet-red.

**Azo dyes** I. G. Farbenind A.-G. Fr. 785,742, Aug. 17, 1935. Diazotized *o*-amino-phenols contg. 1 or more NO<sub>2</sub> groups, one of which occupies the position para to the amino group and which may also contain any other substituents except SO<sub>2</sub>H and COOH groups are coupled with *N*-hydroxyalkyl derivs. of *m*-toluidine which contain also a supplementary alkyl or hydroxyalkyl group attached to the N. Thus, 5-nitro-2-aminophenol  $\rightarrow$  *N*-hydroxyethyl-*N*-methyl-*m*-toluidine dyes acetate silk an intense ruby red. Other examples are given.

**Azo dyes.** Soc. pour l'ind. chim. à Bâle. Fr. 784,570, July 27, 1935. Amino diphenylene dioxides are diazotized and coupled with components such as arylides of acetoacetic acid, or of  $\beta$ -ketonic acids, pyrazolones and their sulfonic acids,  $\beta$ -naphthol, sulfonic acids of  $\alpha$ - and  $\beta$ -naphthol, aminonaphthols, aminonaphtholsulfonic acids and arylides of 2,3-hydroxynaphthols.

**Azo dyes.** Soc. pour l'ind. chim. à Bâle. Fr. 785,294, Aug. 6, 1935. Compds. of the formula C(OR')CH C-

(NH<sub>2</sub>) C(OR') CH CN(X)SO<sub>2</sub>Y, (R' and R<sup>2</sup> are alkyl

groups, the same or different, X is H, an aryl, alkyl or aralkyl radical, Y is an aromatic radical which may be substituted) are diazotized and coupled with any coupling components. Thus, 4-*p*-toluenesulfonylethylmethylamino-1-amino-2,6-diethoxybenzene  $\rightarrow$  1-*p*-toluenesulfonamido-8-hydroxynaphthalene-3,6-disulfonic acid dyes wool fast violet shades in an acid bath. Other examples are given.

**Azo dyes.** Soc. pour l'ind. chim. à Bâle. Fr. 785,958, Aug. 23, 1935. Dyes are prepd. by coupling diazotized 4-nitro-2-cyano-1-aminobenzene, which may also contain other substituents, with components of the formula XN(R')R<sup>2</sup> (X is a C<sub>6</sub>H<sub>5</sub> or C<sub>6</sub>H<sub>4</sub> ring, substituted if desired, R' and R<sup>2</sup> are H, alkyl, aryl or aralkyl), the coupling taking place under such conditions that it is the N(R')R<sup>2</sup> group which provokes coupling, then alkylating or acylating the dyes obtained. Thus, 2-cyano-4-nitro-1-aminobenzene  $\rightarrow$  (*N*-ethyl-*N*-methoxyethylamino)benzene dyes acetate silk pure violet-red shades. Several examples are given.

Complex metal compounds of azo dyes. Hans Krzizalla and Karl Holzach (to General Aniline Works). U. S. 2,019,915, Nov. 5. Complex metal compds. of azo dyes are obtained by treating with agents supplying metals such azo dyes as are obtainable by coupling diazotized *o*-amino phenols or *o*-amino naphthols which contain at least a substituent selected from the class consisting of halogen, the nitro and the sulfo group, with a tetrahydro- $\beta$ -naphthol. Particularly suitable *o*-amino phenols are those contg. several of the substituents mentioned. Still further substituents may be present, if desired. Suitable coupling components are tetrahydro- $\beta$ -naphthol itself and derivs. thereof such as 2-hydroxytetrahydro- $\beta$ -naphthalene-3-sulfonic acid. Suitable agents supplying metals are those supplying Cr, Cu, Fe, Ni, Co, Mn, Ti and V.

The dyes may be employed for various purposes, e. g., for lacquering purposes, etc., and may be subjected to the said treatment in substance or on the fiber. Several examples are given.

**Disazo dyes containing copper.** I. G. Farbenund A.-G. (Karl Wiedemann and Heinrich Klingenstein, inventors). Ger. 616,676, Aug. 3, 1935 (Cl. 22a 6). A dye contg. Cu is prep'd by treating disazo compound from 1 mol of tetrazotized aromatic diamines, which contain an alkoxy group in the *o*-position to each amino group, and 2 mols. of a dihydroxynaphthalene-mono- or -disulfonic acid, with Cu-yeilding agents. Thus 4,4'-diamino-3,3'-dimethoxybiphenyl is tetrazotized and coupled with 1,8-dihydroxynaphthalene-3,6-disulfonic acid. The resulting dye is heated with CuSO<sub>4</sub> to give a dye coloring cotton and viscose in fast blue shades. Another example is given.

**Cupriferous disazo dyes.** Imperial Chemical Industries Ltd. and Mordecai Mendoza. Brit. 433,454, Aug. 12, 1935. A Cu-contg. dye is made by treating the dye hydrazide  $\rightleftharpoons$  1,8-aminonaphthol-2,4-disulfonic acid (Chlorazol sky blue FF) with a hot ammoniacal soln. of a Cu salt until the alkyl groups have been split off. It yields greenish blue shades on cellulose material, fast to light and soap.

**Vat dyes.** I. G. Farbenund A.-G. (Wilhelm Bauer, inventor). Ger. 619,080, Sept. 21, 1935 (Cl. 22b 3 08). New vat dyes (generally brown) are prep'd by the action of anhyd. acid-condensing agents, e. g., H<sub>2</sub>SO<sub>4</sub> or AlCl<sub>3</sub>, on the known dyes obtainable by introducing a 1-amino-4(or 5)-arylaminoanthraquinone residue into the benzene ring of an anthraquinone-2,1-(N)-benzotriene(or naphthalene)-acridone, which must be unsubstituted in the benzene ring in a position adjacent to that at which the aminoarylaminoanthraquinone residue is introduced. If the reaction is effected with H<sub>2</sub>SO<sub>4</sub> or like reagent at atm. temp., an intermediate product is obtained which must be oxidized to convert it into the dye. Reaction at a raised temp., e. g., 50-80°, yields the dye directly. Examples are given. The products are readily sol in alk. hyposulfite vats, and are particularly suitable for use in printing processes in assocn. with rongalite and potash.

**Vat dyes.** I. G. Farbenund A.-G. (Werner Zerweck and Ernst Honold, inventors). Ger. 619,403, Oct. 1, 1935 (Cl. 22b 3 03). Addn. to 602,336 (C. A. 29, 611). 600,481 (C. A. 29, 10509). New dyes, generally yellow or red, are obtained by forming amides from *o*-amino compds. of the anthraquinone series and arylene oxazole carboxylic acids substituted by an anthraquinonyl residue. The process may be effected by condensing *o*-aminoanthraquinones with the acid halides, or *o*-haloanthraquinones with the acid amines. The acids are obtainable by condensing anthraquinone aldehydes with *o*-aminohydroxyaryl carboxylic acids. Examples are given.

**Dyes of the anthraquinone series.** Joseph Deinet (to E. I. du Pont de Nemours & Co.). U. S. 2,010,846, Nov. 5, 1935. Dyes which give bright orange and other shades are formed by fusing a 2-methylantraquinone compd. with S monobromide at temps. above 150° or heating in an inert solvent such as C<sub>12</sub>H<sub>10</sub> at a higher temp.

**Anthraquinone dyes.** I. G. Farbenindustrie A.-G. Brit. 432,647, July 31, 1935. 1-Amino-4-haloanthraquinone-2-sulfonic acids contg. an acylamino group in 5- or 8-position are condensed with amines that contain at least 1 H atom attached to the amino N atom. Among examples, 1-amino-5-acetylamino-4-bromoanthraquinone-2-sulfonic acid is condensed with NH<sub>3</sub>, MeNH<sub>2</sub>, PhNH<sub>2</sub>, hexahydroaniline, *p*-aminoacetamide, 1,4-dimethoxy-2-amino-1,3-diaminobenzene and monoacetylbenzidine. The products dye wool from an acid bath blue to green shades.

**Dioxazine dyes.** I. G. Farbenund A.-G. Fr. 785,719, Aug. 17, 1935. Dyes are prep'd by condensing a diarylaminoquinone deriv. (by condensing a 1,4-benzoquinone with an aminoazo dye which may contain sulfonic groups in a high-boiling solvent) by heating it to a high temp. in a high-boiling solvent in the presence or not of an oxidizing agent, a metal chloride or acylating agent or more than one of these, and sulfonating the products obtained.

1 Thus, the product obtained by condensing *p*-aminoazobenzene and toluquinone is boiled in PhNO<sub>2</sub> with the addn. of sulfonyl chloride of *m*-nitrobenzene, and afterward sulfonated. The product dyes viscose silk violet shades. Other examples are given. Cf. C. A. 29, 1654<sup>1</sup>.

**Indigo.** Wm. R. Waldron (to E. I. du Pont de Nemours & Co.). U. S. 2,020,387, Nov. 12, 1935. Indigo which has especially good properties with respect to color and covering power is produced by subjecting alkali metal indoxyls to partial oxidation by means of alkali metal hypochlorites and completing the oxidation by means of air.

**Oxazine dyes.** I. G. Farbenindustrie A.-G. Brit. 433,215, Aug. 12, 1935. Dyes are prep'd by condensing a hydroxycarbazole having a free *o*-position to the OH with a nitrosobenzene having an alkylated amino group in *p*-position and a free *o*-position to the NO. In examples, (1) 2-hydroxycarbazole (I) is condensed with nitrosodimethylamine-HCl in boiling alc., the product dyes tanned cotton blue shades, and (2) *p*-nitrosodiethanolamine-HCl is condensed with I, 9-methyl-2-hydroxycarbazole and 2-hydroxy-3-carboxycarbazole. Cf. C. A. 29, 613<sup>2</sup>.

**Sulfur dyes.** Max Wyler (to Imperial Chemical Industries Ltd.). U. S. 2,020,345, Nov. 12, 1935. Anilinoacetamide, *o*-toluenopropionitrile or other suitable compd. of the general formula aryl-NHCH<sub>2</sub>CH<sub>2</sub>Y in which aryl represents an aryl radical of the benzene of naphthalene series having a free *p*-position, X represents H or a lower alkyl radical, and Y represents a -CN, -CONH<sub>2</sub>, or -CSNH<sub>2</sub> radical, is treated with nitrosophenol, quinone-chloramide or autophenol, and the resulting product is thionated, forming dyes which dye blue shades.

**Printing pastes.** I. G. Farbenund A.-G. Fr. 785,822, Aug. 20, 1935. Mercaptobenzothiazoles and mercaptobenzimidazoles are added to the pastes to increase the coloring power and accelerate the rapidity of fixation of vat and S dyes.

**Intermediates and dyes.** I. G. Farbenund A.-G. Fr. 784,606, July 22, 1935. Carboxylic acids of the formula HOOCR' (R' is alkylene, R' is alkyl, alkoxyalkyl, aralkyl or a hydroaromatic group) are condensed with *p*- or *m*-nitro amines of the C<sub>12</sub>H<sub>14</sub> series, the NO<sub>2</sub> is reduced to NH<sub>2</sub>, the NH<sub>2</sub> is diazotized and coupled with an arylamide of an aromatic *o*-hydroxy carboxylic acid coupling in the ortho position with respect to the OH group. Examples are given of the prep'n. of 5-nitro- (m. 147°) and 5-amino-4-methyl-2-methoxyacetylamino, m. 137°, 6-nitro- (m. 198°) and 6-amino-4-methoxyacetylamino-1,3-dimethoxybenzene, m. 155°, 6-nitro- (m. 78-9°) and 6-amino-3-methoxyethoxyacetylamino-2,5-diethoxybenzene, m. 58-9°, 6-nitro- (m. 87-8°) and 6-amino-3-benzyloxyacetylamino-2,5-diethoxybenzene, m. 98°, 6-nitro- (m. 102-3°) and 6-amino-3-cyclohexyloxyacetylamino-2,5-diethoxybenzene, and of dyes obtained therefrom.

**Dyeing.** Heinrich Karrer & Co. Ger. 547,532, July 31, 1935 (Cl. 8m 3 02). Textile goods are dyed in cops or on spools by aq. solns. of ester salts of vat dyes contg. oxidizing agents, neutral and alk. salts. The dye is developed by an acid bath. Thus, cotton yarn in cops or spools is treated with a bath contg. ester salts of indanthrene brilliant green B (Schultz Dye Table 7 1269), NaNO<sub>2</sub>, NaCl, Na<sub>2</sub>SO<sub>4</sub>, NH<sub>4</sub>OH and calcined soda. The yarn is washed and the dye developed with H<sub>2</sub>SO<sub>4</sub>.

**Substantive dyeings.** I. G. Farbenund A.-G. Fr. 785,043, July 31, 1935. Substantive dyeings on vegetable fibers are improved by using for the subsequent treatment aliphatic multivalent amines of high degree of polymerization, contg. trivalent N, using them in the form of salts of inorg. or org. acids. Thus cotton is dyed with "Sirus-blue BR," then treated with a soln. contg. a small amt. of a polyamine of high polymerization degree and HCOOH and rinsed. The dyeing does not exude or bleach in non-alk. baths.

**Dyeing animal fibers.** Société pour l'ind. chim. à Bâle. Brit. 433,230, Aug. 12, 1935. The fibers are dyed with Cr compds. of mordant-dyeing sulfonated azo dyes with an addn. to the bath of less than 1%, on the wt. of

the goods, of a quaternary  $\text{NH}_4$  compd. obtained by treating with an alkylating or aralkylating agent a *N*-alkyl or *N*-aralkyl benzimidazole substituted at the  $\mu$ -C atom by an aliphatic residue contg. at least 8 C atoms (cf. Brit. 419,010, *C. A.* 29, 22634). In an example, 100 parts of wool are dyed in an aq. bath contg. Neolan violet 5R,  $\text{H}_2\text{SO}_4$  and 0.02 part of  $\mu$ -heptadecyl-*N,N*-dibenzylbenzimidazolium chloride, the wool being introduced at 60° and the temp. being raised to the boil in 20 min. and so maintained for 45 min.

**Dyeing fibrous materials such as cotton.** Paul Bolen (to American Lecithin Co.) U. S. 2,020,496, Nov. 12. The material to be dyed is immersed in a reduced alk. soln. contg. lecithin and prep'd from a dye such as a carbazole or sulfur dye which is sol. only in an alk. medium with an alk. reducing agent such as Na hyposulfite. Various examples are given.

**Dyeing cellulose, cotton, etc.** I. G. Farberind A.-G. Fr. 783,489, Aug. 10, 1935. Sulfonic acids of fluorindines prep'd, e. g., by the process of Fr. 777,702 (*C. A.* 29, 41861) are substantive dyes for cotton, cellulose, etc. Examples are given of the prep'n of dyes by oxidizing azobenzene prep'd from (1) *p*-aminodiphenyl and *p*-nitrosophenol and (2) *p*-aminopyrene and *p*-quinone-diamine, and of the use of these dyes.

**Dyeing cellulosic material with acid wool or basic dyes.** Gustav Adolf Widmer and Edward Wallace Pierce (to Société pour l'industrie chimique à Bâle) Brit. 433,143, Aug. 6, 1935. Cotton, rayon, linen, jute, ramie, etc., as loose or woven material or as paper, is treated with an aq. soln. of a methylol compd. or compds obtained by condensing  $\text{CH}_2\text{O}$  with dicyandiamide and (or) guanidine and (or) heat-treated urea, the methylol product is then hardened by heat and the material is thereafter dyed or printed, e. g., with acid or basic dyes. The process of Brit. 433,210 (below) is excluded. An aq. mixt. of the components may be used instead of the methylol compd. or compds. Examples are given of the prep'n. of compds by (a) heating a mixt. of guanidine carbonate and aq.  $\text{CH}_2\text{O}$ , (b) heating urea at 160–170° for about 12 hrs. and heating the residue with aq.  $\text{CH}_2\text{O}$ , (c) heating a mixt. of dicyandiamide and aq.  $\text{CH}_2\text{O}$ , (d, e and f) adding an aq. soln. of dimethylolurea to the products of (a), (b) and (c), resp. Cotton is immersed in a dil. aq. soln. of the compd. or compds., pressed until double its original wt., heated 2 min. at 165°, soaked 5 min. at 55°, rinsed, dried and dyed with Benzyl green B from a  $\text{HCOOH}$  soln. at 55°. In 433,210, Aug. 9, 1935, the cellulosic material is treated before or during the application of the dyes with a carbamide- $\text{CH}_2\text{O}$  condensation product and with an org. base when acid dyes are used or with an org. acid, other than tannin or tannic acid, when basic dyes are used. Among examples, cotton fabric is impregnated by immersion 5–10 min. at room temp. in an aq. soln. contg. dimethylolurea and sufficient  $\text{AcOH}$  to give a  $\text{pH}$  of 4.5, pressed until double its original wt., dried, prep'd with a paste contg. British gum thickening and ethylenediamine, dried, heated by ironing with a hot iron for 2 min., dyed by treatment for 45 min. in a soln. contg.  $\text{HCOOH}$  and the dye obtained by methylating a tech. mixt. of the alkali salts of *p*-diaminoanthranquin-2-sulfonic acid and *p*-diaminocrysanthinesulfonic acid (cf. Brit. 308,713 (*C. A.* 24, 503)), rinsed at about 50° and dried, whereby the printed areas are colored a deep blue, fast to light.

**Dyeing and printing.** I. G. Farberind A.-G. Fr. 783,421, Aug. 9, 1935. Salts of sulfonic acid leuco-esters of vat dyes of the benzanthrone series are stabilized by water sol. salts of mercaptans, e. g., of mercaptobenzo-thiazole, 3-methoxyphenylmercaptan, thioglycolic acid, xanthogenic acid, thiosalicylic acid, dihydrocarbamic acid or its aryl derivs. (*C. A.* 29, 41881).

**Printing textiles.** Imperial Chemical Industries Ltd. Fr. 783,532, Aug. 12, 1935. White reserves are obtained using sulfonic esters of leuco vat dyes having an affinity for cotton, by incorporating in the printing paste a reserve formed of a salt of a primary, secondary or tertiary amine or a quaternary  $\text{NH}_4$  halide, and superprinting or overlaying and developing the dye. Examples of reserves

are benzylpyridinium chloride, dimethylbenzylphenylammonium chloride and cetyltrimethylammonium chloride.

**Printing rayon.** Société pour l'ind. chim. à Bâle. Brit. 433,100, Aug. 8, 1935. See Fr. 770,844; Swiss 173,042 (*C. A.* 29, 86744). The dyes must contain no  $\text{SO}_3\text{H}$  or  $\text{COOH}$  groups.

**Coloring textile materials containing organic cellulose derivatives such as cellulose acetate.** Henry C. Olpin and Geo. H. Ellis (to Celanese Corp. of America). U. S. 2,019,620, Nov. 5. The materials are treated with 4-chloro-2-nitro-1-ethanolaminobenzene or other suitable aromatic compds. free from azo groups and contg. as the sole chromophoric groupings nuclear nitro groups and also contg. hydroxyalkylamino groups. U. S. 2,019,627 relates to a process of dyeing material such as cellulose acetate with aromatic compds. such as a 1-amino-2-nitro-4-acylamino benzene or the like. Various examples are given.

**Fixing dyes on fibers.** The Calico Printers' Association Ltd. Ir. 784,692, July 22, 1935. Dyes are made fast to washing on silk, rayon, cotton, etc., by impregnating the dyed or printed fibers with the constituents of a synthetic resin, then drying them and heating to a relatively high temp. for a relatively short time.

**Apparatus for dyeing.** Hildred Charlesworth. Brit. 433,802, Aug. 21, 1935. An inner shell or roller is provided in a perforated roller for a fabric-steaming machine to cut down the internal capacity of the roller in order to save steam and also time in filling and emptying the roller during the operation of the machine.

**Machine for dyeing fabric in the piece.** Max Siegenheim and Richard Dirks. Ger. 616,542, July 31, 1935 (*Cl. 8a* 950).

**Apparatus for dyeing or bleaching textiles in lengths.** Gustave Cotel. Fr. 785,994, Aug. 23, 1935.

**Apparatus for dyeing and bleaching knitted goods.** Gustave Cotel. Fr. 784,985, July 30, 1935.

**Machine for printing fabrics.** Allgemeine Elektrizitäts-Gesellschaft (Herbert Stein, inventor). Ger. 616,804, Aug. 7, 1935 (*Cl. 8c* 703).

**Dyeing spool.** Thomas T. Davies. U. S. 2,021,050, Nov. 12. Structural details.

**Roller-reversing mechanism for dye-vat rollers.** Ernest Farrell. Brit. 433,739, Aug. 20, 1935.

**Apparatus for washing, impregnating, dyeing, etc., rayon.** Robert Jeanpierre. Fr. 785,182, Aug. 3, 1935.

**Toothed-drum machine for scutching leaves of Phormium tenax and other leaves and stems of bast plants.** Berthold Burlin. Brit. 432,467, July 26, 1935.

**Degumming ramie, etc.** Charles E. Gage. Brit. 433,205, Aug. 6, 1935. The plant stalks or semi-prep'd fibers of ramie, sisal, hemp, etc., are submitted successively to maceration, treatment with an aq. soln. of  $\text{NaOH}$ , linsed oil, turpentine and  $\text{NH}_3$  in the proportions of 5, 3, 3 and 4% by wt. and at 160–190°F., treatment with an aq. soln. of common soap 3 and linsed oil 3% at not less than 120°F., neutralization in  $\text{HCl}$  and, after drying, treatment in a softening machine comprising relatively reciprocating plates.

**Treatment of animal fibers.** Rosa Wiesner. Austrian 142,784, Sept. 10, 1935 (*Cl. 29b*). Hair and other animal fibers are prep'd. for felting by treatment in a bath contg.  $\text{H}_2\text{O}_2$ ,  $\text{HNO}_3$  and a metal compd. which does not act as a carrier of O, e. g.,  $\text{Zn}(\text{NO}_3)_2$ . The proportion of metal compd. in the bath must be more than about 10%. Cf. Ger. 527,012 (*C. A.* 25, 4719).

**Fulling wool.** Böhme Fettchemie-Ges. m. b. H. Ger. 619,182, Sept. 24, 1935 (*Cl. 8a* 2). Acid processes of fulling wool are improved by adding to the fulling liquid a sulfonic ester of a higher aliphatic alc., e. g., octadecyl alc.

**Treating filaments and yarns.** British Celanese Ltd. Brit. 433,110, Aug. 8, 1935. Filaments, etc., are treated by applying an antistatic dressing and spraying the dressed filaments or yarns with a coating of lubricant. App. is described. Cf. *C. A.* 29, 76734.

**Treating yarns with liquids.** British Celanese Ltd.

Nov. 5. The material is treated with a substratum of poly character composed of vulcanizable rubber material and coloring matter, and over this substratum there is applied a preservative and bonding emulsion composed of latex and a mordant comprising a vulcanizable oil and resin, and the assembled materials are together subjected to a vulcanizing operation.

Preserving fish nets. *Chemische Fabrik von I. E. Devrient A.-G.* Ger. 610,112, Sept. 21, 1935 (Cl. 8k 3). The known treatment with tanning baths is improved by adding to the baths an alk. inorg. compd. and, as a bactericide, a water-insol. compd. of Cu or Hg, with or without a dispersing agent, e. g., sulfite cellulose lye. Sp. baths are described.

Sizing fabrics. *Firma Louis Blumer* Ger. 616,805, Aug. 7, 1935 (Cl. 8k 1). Addn. to 605,573 (C. A. 29, 1939). The method of 605,573 for obtaining substances for finishing fabrics, especially rayon fabrics, by condensing polybasic acids, polyhydric alcs. and castor oil, is modified by wholly or partly replacing the later oil by other oils, fats, fatty acids or resin acids. Thus, phthalic acid anhydride, coconut oil fatty acid, stearic acid and glycerol, or maleic acid anhydride, colophony, glycerol and castor oil, are condensed to give a finishing agent.

Apparatus for drying and cleaning cotton, cottonseed, etc. *Jesse J. Fowler*. U. S. 2,019,608, Nov. 5. Various structural, mech. and operative details.

Apparatus for cleansing and extracting fabrics such as clothes by dry cleaning, etc. *James B. Kirby* (to Apex Electrical Mfg. Co.) U. S. 2,019,571, Nov. 5. Structural, mech. and operative details.

Dry cleaning. *Compagnie des produits chimiques et électrometallurgiques Alais, Froges et Camargue* Fr. 785,864, Aug. 21, 1935. Mixts. of dichloroethane (up to 75%) and trichloroethylene are used.

Phosphatide compositions. *Hansa-Mühle A.-G.* (Bruno Rewald, inventor). Ger. 619,235, Sept. 25, 1935 (Cl. 23c. 2). Crude phosphatides are partly or completely freed from oil by extr. with an org. solvent, e. g., acetone, which may also displace water (if present) from the phosphatides. The latter are then mixed with an emulsifying agent, e. g., sulfonated castor oil, and the residual solvent is evapd. The products are stable in air and are easily convertible into emulsions useful in the textile, leather and foodstuff industries.

Mats. *Franz Schürholz G. m. b. H.* Brit. 433,667, Aug. 19, 1935. Coconut fiber mats or matting are treated after weaving on their upper and (or) lower surfaces with rubber latex. Mats cut from such matting may have the cut edges treated with latex. The latex may contain fillers, e. g., chalk, talc, casein, powd. Al, Al<sub>2</sub>O<sub>3</sub>, and vulcanizing agents.

Temperature indicator suitable for contact with metal ironing or calendaring devices or the like to determine their temperature. *George H. Ellis and Alexander J. Wesson* (to Celanese Corp. of America) U. S. 2,020,676, Nov. 12. A device is used comprising a solid mass contg. a relatively infusible filling material such as chalk and a substance such as anthracene which is converted into a fluid form at a predet. temp. thus permitting transfer of the filling material from the mass to a surface having at least that temp. to form a visible but readily removable trace at the point of contact.

Fireproofing textiles, paper, etc. *Michel Marini* Fr. 785,915, Aug. 22, 1935. An insol. coating which prevents contact with the air is formed on the textile or paper by applying thereto a gel of colloidal oxides such as  $\alpha$ - and  $\beta$ -stannic acids, tungstic, molybdic or silicic acid and then drying the gel. A 2nd coating may be applied which liberates a gas preventing combustion, e. g., (NH<sub>4</sub>)<sub>2</sub>PO<sub>4</sub>.

## 26—PAINTS, VARNISHES AND NATURAL RESINS

A. H. SABIN AND CARLTON H. ROSE

Painting in Egypt. *C. T. Mason*. *Am. Paint J.* 20, 13-14 (Dec. 2, 1935).

A method of comparing the odor of drying paints. *Henry A. Gardner*. *Natl. Paint, Varnish & Lacquer Assoc., Circ.* No. 496, 388-9 (1935).—The odor of drying paint can be compared by painting the inside of an ordinary gallon can provided with a no. of small holes on the side, near the bottom, and a larger hole in the cover of the can. For masking the odor of drying paint, one part of industrial perfume to 2000 parts of paint is usually sufficient. Vanillin has been found useful for this purpose.

Applied paint and varnish chemistry for the chemist and the layman. *Benjamin Joachim*. *Am. Paint J.* 20, 45-7 (Dec. 2, 1935); cf. *C. A.* 29, 7675.—The use of synthetic phenolic resins in enamels is classed under the heads of architectural, marine and industrial. Each class is discussed.

Fluorescence analysis applied to paints and varnish. *A. Kufferath*. *Farbe u. Lack* 1935, 473-4.—A discussion of recent work on the use of luminescence analysis to investigate the various raw materials and finished products of the protective coating industry.

The manufacture of artists' colors. *D. H. S. Forbes*. *Paint Manuf.* 5, 352-4 (1935).—A brief review.

Black pigments in paint and ink manufacture. *H. M. Langton*. *Paint Manuf.* 5, 348-50 (1935).—A paper dealing with phys. properties, particle size, anticorrosive paints and inks.

Dehydration of pigments and pigment pastes by electro-osmosis. *J. F. Sacher*. *Farbe u. Lack* 1935, 543-4.—A review of German patents.

Color and constitution. *L. Dittmar*. *Farbe u. Lack* 1935, 471-2.—A brief discussion of the chem. constitution of a no. of inorg. pigments.

A new "laminal" reinforcing pigment. *F. C. Atwood*.

*Official Digest Fed. Paint & Varnish Production Clubs*, No. 148, 328-33 (1935), cf. *C. A.* 29, 5680.—Film strength of paints is attained by a judicious mixt. of pigments of various shapes and sizes. Large particles have relatively low hiding power, regardless of their n; hence they need not impart opacity. On the other hand, if colorless and transparent, they do not interfere with hiding and coloring pigments. Laminal pigments should be ideal for reinforcing purposes. Graphite and Al bronze have the disadvantages of color, leaching or reactivity. A specially prep'd mica and its use in different types of paint are described.

New York Production Club presents study of pigment wetting and dispersion. *Austin O. Allen, et al.* *Am. Paint J.* 19, *Convention Daily*, 14-15, 16, 17 (Oct. 31, 1935); *Paint Varnish Production Mgr.* 13, 40-5 (Nov., 1935), *Paint, Oil and Chem. Rev.* 97, No. 23, 81-6.—A preliminary paper. Sixty commercially available pigments were divided into 11 classes and representatives of each of these classes taken for the tests. Fifteen pigments from white to black in color were made into paint with 13 wetting agents. The procedure is outlined and the pigment vehicle ratios are tabulated. The wetting agents are evaluated and zinc naphthenate and lecithin are given the highest grading. The pigments are tested in 4 groups as shown by their wettability. Consistency setting, color, drying, odor and working properties must be considered in evaluation of the agents. The correct selection of the wetting agent will materially reduce the time factor in the expensive grinding operation. In addn. certain other properties of the pigmented product such as settling, brushing, leveling and flow are frequently improved by wetting agents.

A study of hydrogen-ion concentrations of some aqueous pigment extracts. *L. P. Hart*. *Natl. Paint, Varnish & Lacquer Assoc., Circ.* No. 492, 202-7 (1935).—The pH values of aq. suspensions of pigments may be of signifi-

cance in paint technology. The  $pH$  values of 80 representative pigments were detd. electrometrically on the supernatant liquids from suspensions of 5 g. of pigment in 100 g. of freshly distd. water. The glass electrode was used. G. G. Sward

Chrome yellow and its production by some new methods R. Heublyum *Peintures, Pigments, Vernis* 12, 132-3, 155-8 (1935)—A no. of newly developed methods are reviewed. J. W. Perry

Some physicochemical aspects of the iron blues A. Wooler *Faint Manuf.* 5, 328-31 (1935)—A brief discussion of reactions, reaction conditions, and varieties, raw materials and manuf. processes. Formulas are given for a medium bronze soda blue and a super-non-bronze blue. W. H. Boynton

Lithopone in white enamel manufacture Fritz Ohl *Paint Manuf.* 5, 302-4 (1935)—Lithopone is used as a white pigment in spirit and other acid varnishes. It prevents livering. A white enamel contg. 75% lithopone and 25% ZnO equals the best com. enamel. Active lithopone pigment, lithoxon, is used in oil enamel manuf. A peculiarity of lithoxon-pigmented enamels is the slight thickening up shortly after manuf. With 20% ZnO and a flow-improving agent such as benzoic acid or crotonic acid this difficulty is removed. W. H. Boynton

Zinc pigments in relation to southern climatic conditions S. Werthan *Official Digest Fed. Paint & Varnish Production Clubs* No 148, 321-8 (1935)—See C. A. 30, 305. G. G. Sward

Zinc oxide—its properties and uses in paint manufacture J. Stewart Remington *Paint Manuf.* 5, 202-5, 306-8, 355-7 (1935); cf. C. A. 29, 6775—Factors affecting the wetting of pigments, or oil absorption, are (a) viscosity of the vehicle, (b) pigment-vehicle ratio, and (c) force of flocculation. Three stages of flocculation of ZnO are illustrated by diagram. Gloss paints and oil absorption are discussed. Properties of a well-made enamel are tested, also recipes contg. synthetic varnishes. Yellowing is frequently caused by the mediums employed, and ZnO, if well made and of uniform particle size, is satisfactory. The time of wetting of pigments depends upon wetting forces, viscosity of vehicles, the amt of mech. work done upon the mixt. and the particle size; and the forces of wetting between pigment and vehicle have a decided influence on the true consistency. W. H. B.

Petroleum thinner vs turpentine J. W. Brock *Can Chem. Met.* 19, 297-8 (1935) W. H. Boynton

Future of natural resins T. Hedley Barry. *Oil Colour Trades J.* 88, 1625, 1627 (1935)—Congo copal, terpenes, dammars and kauri gum are discussed. W. H. Boynton

Paints and painting to protect [food] equipment and plant interior (Buhr) (Sheeler) 12. Dispersions such as those of pigments (U. S. pat. 2,021,143) 9. Protecting metals [with varnish] (Ir. pat. 784,998) 9. Spraying liquids in air [in paint spray booths] to effect its purification (U. S. pat. 2,021,281) 1. Testing the adhesive properties of coatings such as paint or lacquer (U. S. pat. 2,020,891) 13

Mixing device suitable for remixing settled pigments in cans of paint Joseph Taccogna U. S. 2,021,209, Nov. 19. Mech. details.

Lithopone. Gustave A. Kummer (to New Jersey Zinc Co.) U. S. 2,021,420, Nov. 19. For making high-strength lithopone, crude lithopone is pptd. by reaction of BaS with ZnSO<sub>4</sub> and is sepd. from assoc. soln. Crude ZnS is pptd. from a sepg. body of soln by reaction between ZnCl<sub>2</sub> and BaS, the crude ZnS is sepd. from assoc. liquor, and is mixed with the crude lithopone, and the resulting mixt. is calcined. Cf. C. A. 29, 8367.

Zinc sulfide pigment Leon S. Holstein and Geo. F. A. Stutz (to New Jersey Zinc Co.) U. S. 2,020,313, Nov. 12. Finely divided ZnS-bearing material such as zinc blende ore is heated and mixed with concd. H<sub>2</sub>SO<sub>4</sub> in excess of the chem. equiv. proportion for the ZnS and other metal sulfides present, under conditions which pro-

duce active evolution of H<sub>2</sub>S and formation of ZnSO<sub>4</sub>, without objectionable formation of elemental S, a purified aq. soln. of the ZnSO<sub>4</sub> is prepd. and the purified and concd. H<sub>2</sub>S from the process is used to produce a ZnS ppt. from the ZnSO<sub>4</sub>. An arrangement of app. is described.

Zinc sulfide pigment. Arne J. Myhren and Byron Marquis (to New Jersey Zinc Co.) U. S. 2,020,902, Nov. 12. See Can. 353,104 (C. A. 29, 8307).

Zinc sulfide pigment. Geo. F. A. Stutz and Arne J. Myhren (to New Jersey Zinc Co.) U. S. 2,020,918, Nov. 12. Bubbles of H<sub>2</sub>S gas are introduced into a body of mixed ZnSO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub> soln. to form a slurry of ZnS ppt. and the mixed liquor under treatment is moved concurrently to the bubbles of H<sub>2</sub>S so that the soln. is brought into contact with H<sub>2</sub>S of progressively increasing concn., the ZnS ppt. is aged in the resulting impoverished soln. and then removed from the soln.

Paint vehicle from mineral oil Giovanni Fiore U. S. 2,020,936, Nov. 12. For producing an oxidizable oil, a mineral oil is heated to a temp. not substantially above 100° (suitably about 100°), mixed with up to about 1/2 its quantity of a vegetable oil such as Sesamum Indicum oil and a current of O<sub>2</sub> is blown through the material for about an hr. while maintaining a temp. of about 100°, a mixt. of HNO<sub>3</sub> and alc. 2 parts is added in a quantity about 33-50% the quantity of the oils, and a drier such as spirits of turpentine is also added.

Suction filter for printing inks Paul Buschmann (Heinrich Zimmer, inventor) Ger. 602,503, July 15, 1935 (Cl. 12d 23).

Preventing offset in printing Jens A. Paasche. Fr. 784,826, July 25, 1935. Offset is prevented by atomizing a soln. or suspension of a solid substance in a volatile liquid onto the printed sheet. An example contains gum arabic 20, wood alc. 60 and CCl<sub>4</sub> 20%. App. is described.

Printing half tone designs on colored base material such as leather or fabrics Owen R. Huggins and Foster D. Snell (to Decorative Development, Inc.) U. S. 2,021,418, Nov. 19. Solid color areas are discharged from the base material, such areas being coincident with a half tone design to be applied to the material, and the discharged areas are printed with a half-tone design produced by photomechanical reproduction, both discharge and printing being effected under pressure.

Coating-compositions John P. Henbahren Brit 432,403, July 25, 1935. Rubber is dissolved, in the manuf. of paints, etc., by heating a mixt. of a neutral oil and rosin to 300°F., adding a drier, e. g., Co. linoleate, further heating to 540°F., adding rubber and heating until the rubber has dissolved.

Resistance coatings and deposits. Radioaktiengesellschaft D. S. Loewe Brit 433,790, Aug. 21, 1935 C, preferably as soot, is mixed, preferably in a ball mill, with a varnish base such as acetate or cellulose varnish or oil paint contg. Zn white, and the mixt. is applied to a support, e. g., of glass, hard paper, ceramic material or mica, by painting or spraying or by an inkling-wheel. The coating is then sintered at 300-400° by slow heating until the resistance becomes const.

Lacquers formed from a urea-formaldehyde reaction product Martin Luther, Wilhelm Pungs, Robert Gressbach and Claus Heuck (to Nynte Corp.) U. S. 2,019,865, Nov. 5. Compds. such as dimethylol urea, dimethylol thiourea, mono-methylol thiourea or their mixts. are condensed in the presence of a practically anhyd. org. solvent such as EtOH, etc., while heating until a clear soln. is formed and then heating the soln. at least until a resinous condensation product is formed which can be pptd. from the soln. by addition of ethyl ether.

Lacquers and plastic masses I. G. Farbenindustrie A. G. Brit 432,762, Aug. 1, 1935. These contain the usual fundamental substances for such materials, together with a resinous, from viscous liquid to solid, chlorination product of an aromatic hydrocarbon contg. at least 1 CH<sub>3</sub> group as a member of a ring. Hydrocarbons from which such products may be made are tetra- and deca-hydronaphthalene, acenaphthene, fluorene, benzanthrene and

indene, and the compns. may contain drying oils, chlorinated rubber and other solid conversion products of rubber, synthetic resins, cellulose esters and ethers and resins such as dammar, manila, congo, mastic, etc. Pigments and fillers may be present. Among examples, (1) a lacquer contains 1 t cellulose, PhMe, BuOH, alc. and chlorinated acenaphthene and (2) an impregnating liquid contains chlorinated indene or benzanthrene, polyvinyl chloride, Me<sub>2</sub>CO, PhMe and cyclohexane.

Cellulose acetate composition suitable for films or lacquers. Kennth M. Irey (to Commercial Solvents Corp.) U. S. 2,020,247, Nov. 6. Cellulose acetate is used with a plasticizing agent produced by the esterification of phthalic anhydride 1.5-2.0 mols with about 1 mol of ethylene glycol with addn. of a monohydric alc. such as BuOH before the esterification is complete and further heating with addn. glycol to effect condensation by reaction between the remaining free carboxyl and hydroxyl groups.

Coating composition containing synthetic resin, etc. Ernest G. Peterson (to Hercules Powder Co.). U. S. 2,022,149, Nov. 26. A compn. suitable for coating comprises raw linseed oil and a synthetic resin comprising the reaction product of a polyhydric alc. such as glycerol, terpinene-maleic anhydride and a fatty acid.

Two-tone metallic finish. Theodore A. Neuhaus (to Glidden Co.) U. S. 2,021,162, Nov. 10. A surface

presenting both porous and non-porous areas of a base coat such as an ordinary black and crackle lacquer is coated with a top-coat contg. a metallic powder such as Al and a dye so that the top-coat dries to the dyed metallic color over the non-porous areas and to the undyed metallic color over the porous areas.

Floor coverings. Johannes Ruder. Brit. 433,150, Aug. 9, 1935. A foundation material is impregnated from the underside with a moisture-repellent substance, e. g., stearin, a mixt. of stearin and boiled linseed oil, goudron, i. e., lubricating oil residues to which asphalt may be added, and from the upperside with boiled linseed oil to which a thickened oil, i. e., air-blown boiled linseed oil which may contain a drier or paint, may be added and the upper surface is then coated with a plurality of layers of boiled linseed oil and fillers, e. g., wood, cork, paper meal, straw meal, to which coloring materials may be added. To provide a brilliant surface, the product may be coated with a mixt. of copal or amber varnish, boiled linseed oil and turpentine.

Compositions for linoleum, etc. John P. Henharen. U. S. 2,021,063, Nov. 12. A material suitable for use in making linoleum, olecloth, floor and wall coverings, insulation and the like is obtained by heating common rosin to about 150°, adding a drier such as Co linoleate and heating to about 250°, adding rubber and stirring until the rubber is dissolved.

## 27—FATS, FATTY OILS, WAXES AND SOAPS

K. SCHIERUBEL

The interchange of ester radicals of fats. Hans G. Meyer. *Allgem. Oel- u. Fett-Ztg.* 32, 397-401 (1935).—The Bu and Me esters of fat acids and Ac esters of fatty acids were prep'd. from the acids and alcs. derived from several fats. The characteristics of these as compared with the original fat are tabulated. These esters are purer than the original fat and M. suggests they are convenient for many tech. applications. The prep'n of Me esters is more difficult than that of the Bu and Ac.

M. M. Piskur. Determining the free hydroxyl groups of fat acids and fatty alcohols. C. Steiner. *Chem.-Ztg.* 59, 795-6 (1935).—With the use of the Ac value and the sapon value of the acetylated fat acids and alcs., S. has formulated a series of equations for use as aids in detg. the av. no. of free OH groups per mol. of fat acid or fatty alc. in various fat-acid mixts. or fatty alc. mixts.

M. M. Piskur. Filter cloths for the oil and fat industries. Josef Heter. *Allgem. Oel- u. Fett-Ztg.* 32, 395-7 (1935).—Several com. filter cloths were tested with oils contg. bleaching earths. There was considerable variation in the durability of those made with cotton. The poorest camel-hair filter cloth was 4 times more durable than the best cotton one which was 1/2 cheaper. Since the material to be filtered is usually acid, H. recommends the use of animal-fiber filter cloths.

M. M. Piskur. Some African oil seeds. Anon. *Bull. Imp. Inst.* 33, 271-93 (1935).—Po-jok fruits from Sierra Leone. The tree was identified as *Afroliconia elaeagnifolia* Millbr. (Nat. Ord. Ro-aceae). Examn. of 6 samples of fruit gave the following results: av. wt. of a fruit 7.3-11.8 g., av. wt. of 1 kernel 4.6-7.2 g., shell in fruits 31.4-44.0, kernels 58.0-68.6, H<sub>2</sub>O in kernels 5.9-9.0, oil in kernels (as received) 41.7-68.3, oil on H<sub>2</sub>O-free kernels 45.8-63.8, oil on fruits (as received) 32.6-39.4%. The oils extd. from these samples, and also one prep'd. in Sierra Leone in 1917 had the following characteristics: d<sub>4</sub><sup>20</sup> 0.9335-0.9360, n<sub>D</sub><sup>20</sup> 1.5020-1.5110, acid value 0.4-10.3, sapon value 188.0-192.5, I value (Wys, 3 hrs.) 139.9-157.1, unsaponifiable matter 0.3-1.0%, solidifying pt. of fatty acids 30.0-50.3°, heating at 300° caused formation of jelly in 16-20 min. in some samples and no jelly at the end of 30 min. in others, I test (addn. of 5 cc. of satd. soln. of I in CHCl<sub>3</sub> and allowing to stand) jelly formed in a few min. in 1 sample, no gelation in 20 hrs. or more in the other

samples. The viscous, golden yellow oil, with odor resembling that of tung oil, deposits stearin on standing; it had marked drying properties, but its suitability in the manuf. of paints and varnishes could be det'd only by means of tech. tests. The small no. of trees and slow growth in Sierra Leone would preclude its com. utilization. *Balanites aegyptiaca* fruits from Uganda.—Fruits stated to have been obtained from the Madi area of the Gulu district of Uganda had an av. wt. of 5.7 g. and the kernels 0.7 g. The kernels contained 6.4% H<sub>2</sub>O, and yielded on extn. with light petroleum 46.8% of golden yellow oil, equiv. to 50% on the dry kernels and 5.9% on the whole fruits; the residual meal had a bitter unpleasant taste, contained a saponin-reacting principle, and a principle giving faint pos. reactions with the usual alkaloidal reagents. The oil had the following characteristics: d<sub>4</sub><sup>20</sup> 0.9220, n<sub>D</sub><sup>20</sup> 1.4640, acid value 0.0, sapon. value 191.6, I value (Wys, 3 hrs.) 98.0, unsaponifiable matter 0.3%, solidifying pt. of fatty acids 36.0°. The residual meal had the following compn. (figures in parentheses give the compn. calcd. to 7% fat): H<sub>2</sub>O 8.9 (8.3), crude proteins 48.8 (45.7), fat 0.6 (7.0), N-free ext. 30.3 (28.3), crude fiber 5.9 (5.5), ash 5.5 (5.2), nutrient ratio 1:0.65 (1:0.97), food units 154 (160). Owing to the difficulty of expg. the kernels from the shell it is doubtful whether the oil could be profitably produced commercially, though it could be employed for soap-making and probably also for edible purposes. The residual meal would be suitable only as fertilizer. *Ximenia americana* fruits from S. Africa.—The fruits had an av. wt. of 2.2 g. and consisted of 41.2% pulp and shell and 58.8% kernels which contained 5.1% H<sub>2</sub>O and yielded, on extn. with light petroleum, 60.6% of oil, equiv. to 63.9% on the dry basis and 35.6% on the whole fruits. Owing to its viscous character, the oil could probably not be successfully obtained by pressing. Oil extd. with petr. ether had a relatively high unsaponifiable content consisting mainly of a rubber-like substance, while that extd. with Me<sub>2</sub>CO had a considerably lower unsaponifiable content contg. a much lower proportion of rubber-like substances. The oils extd. with petr. ether and Me<sub>2</sub>CO, resp., had the following characteristics: d<sub>4</sub><sup>20</sup> 0.9362, 0.9227, n<sub>D</sub><sup>20</sup> 1.4700, 1.4688, acid value 2.6, 0.7, sapon. value 169.7, 173.4, I value (Wys, 3 hrs.) 93.7, 94.0, unsaponifiable matter 2.4, 0.6%. The residual meal after extn. with petr. ether had the following compn.:

H<sub>2</sub>O 10.6, crude proteins 44.6, oil 1.9, N-free ext. 33.0, crude fiber 5.2, ash 4.7%, cyanogenic glucosides none, nutrient ratio 1.08, food units 149. The Me<sub>2</sub>CO-ext. oil might be suitable for soap making, and possibly, after refining, for edible purposes. Under present conditions it is doubtful whether *Ximena* kernels could be profitably exploited as a source of oil, except possibly for local markets. *Sterculia foetida* seeds from the Gold Coast.—The so-called "fruits" of *Sterculia foetida* are in reality seeds possessing a 3 layered seed coat. The av. wt. per seed was 2 g. The seed consisted of: outer seed-coat layer (gray, thin papery) 3.6, middle layer (pinkish brown and pulpy) 16.2, inner layer (very dark reddish brown and horny) 26.2, kernel 54.0%. Both the middle layer of the seed coat and the kernel contained oil, the total yield, expressed on the entire seeds, being 34.2%. The middle layer of the seed coat contained 7.9% H<sub>2</sub>O and 43.2% (40.9% on the dry basis) of oil with the following characteristics: d<sub>4</sub><sup>20</sup> 0.8552, n<sub>D</sub> 1.4615, acid value 4.5, sapon value 198.1, I value (Wijs, 3 hrs) 84.5, unsaponifiable matter 0.7%. The kernel contained 8.0% H<sub>2</sub>O and 50.4% (53.6% on the dry basis) of oil with the following characteristics: d<sub>4</sub><sup>20</sup> 0.9281, n<sub>D</sub> 1.4650, acid value 0.6, sapon value 191.1, I value (Wijs, 3 hrs) 83.6, unsaponifiable matter 0.6%, solidifying point of fatty acids 30.8°. Both oils gave a pos. Helphen test, the kernel oil more strongly. The kernel oil polymerized in 6.5 min. at 245°; the seed-coat oil failed to polymerize in 15 min. at 290°, but thickened somewhat and darkened. The residual meals from the seed-coat layer and from the kernel, resp., had the following compn.: H<sub>2</sub>O 11.3, 9.9, crude proteins 9.5, 31.7; fat 4.4, 7.2, N-free ext. 49.3, 43.5, crude fiber 21.6, 2.6, ash 7.3, 5.1%; nutrient ratio 1:6.3, 1:1.9, food units, 84, 141. It is unlikely that, in working on a com. scale, the kernel meal could be obtained free from the horny inner layer of the seed coat, and the large percentage of indigestible fiber in the latter would preclude the use of such material as a stock food. Owing to the structure of the seeds, it is improbable that the middle layer of the seed coat and kernel could be effectively sepd. and the 2 different oils expressed from them. The oil from the total seed would be suitable for soap making, and possibly also for edible purposes. *Lophira alata* fruits from Uganda—Fruits of *Lophira alata* Banks (locally known as "Liku") from the Madi area of the Gulu district had an av. wt. of 0.76 g., and the kernels 0.42 g. The kernels contained 12.7% H<sub>2</sub>O, and on extn. with light petroleum yielded 36.9% (42.3% on dry kernels, 20.3% on entire fruits) of pale yellow, semi-solid fat having the following characteristics: d<sub>4</sub><sup>20</sup> 0.8604, m. p. (open-tube method) 24.5°, n<sub>D</sub> 1.4610, acid value 7.0, sapon value 187.9, I value (Wijs, 3 hrs) 73.2, unsaponifiable matter 1.3%, solidifying point of fatty acids 43.8°. The residual meal possessed a strong, bitter taste, contained 5.34% N, with the usual alkaloidal reagents gave faint pos. reactions which were not typical of alkaloids, and contained a saponin. The oil would be suitable for soap making, and possibly for edible purposes after refining. The meal would be suitable for use only as a fertilizer. *Ochna pulchra* fruits from South Africa.—The present sample contained less oil than the previously examd. one from Rhodesia (C. A. 19, 1957), but the oils and residual meals from the 2 samples were of similar compn. *Lulu* (shea) kernels and butter from the Sudan, cf. C. A. 28, 1561°. The butter prepd. in the Sudan, and also that prepd. at the Imp. Inst. from kernels obtained from the Sudan, had const. generally similar to those previously recorded, except that the butter prepd. in the Sudan had a somewhat low m. p.; the unsaponifiable matter contents in the samples were lower than those usually present in West African shea kernels. *Salvadora persica* fruits from the Sudan.—The fruit, weighing 7 g. per 100, were composed of 61.2% pulp and 38.8% seed, contained 12.3% H<sub>2</sub>O and 13.3% oil, equiv. to 15.2% on the dry basis and to 34.3% on the seeds. Extn. with light petroleum gave a hard, bright yellow fat having the following characteristics: d<sub>4</sub><sup>20</sup> 0.8669, n<sub>D</sub> 1.4500, m. p. (open-tube method) 34.3°, acid value 1.3, sapon value 247.5, I value (Wijs, 3 hrs)

17.6, unsaponifiable matter 0.9%, solidifying point of fatty acids 28.8°. The oil was similar to that of the seeds previously examd. (*Bull. Imp. Inst.* 11, 61(1913)); after refining it would be suitable for edible purposes, but it is doubtful whether, under present conditions, it could be profitably used. A Papineau-Couture.

The sulfonation of fish oil. Welwart. *Seifensieder-Zig.* 62, 707-8, 731-2(1935).—A review. Thirteen references. J. W. Perry.

African beeswax. Anon. *Bull. Imp. Inst.* 33, 294-303 (1935).—Two samples from the Gambia were of normal compn. and had the usual phys. and chem. characteristics of African beeswax. One sample from Tanganyika was of normal compn. and had the usual phys. and chem. characteristics of African beeswax except for a somewhat higher I value and lower clouding temp. (Salamon and Seaber test), but no great significance attaches to these exceptions. One sample from Kenya had analytical characteristics in agreement with those previously recorded for East African beeswax, with the exception of the d. (0.9707 as compared with 0.9489-0.9650), acid value (13.1 as compared with 17.3-21.6), ester value (87.1 as compared with 66.2-80.8) and ratio no. (6.6 as compared with 3.6-4.2). Comparison of a sample of crude and a sample of refined wax from the same source showed that the departures from generally accepted characteristics is due to the compn. of the crude wax, and not to a change in compn. on refining.

A Papineau-Couture. Historical (note on) the bleaching of beeswax. Georg Buchner. *Seifensieder-Zig.* 62, 831-2(1935).—The development of modern methods is traced. J. W. Perry.

The emulsifying properties of white wax and borax. Robert Bushby. *Pharm. J.* 135, 345(1935).—By the interaction of borax with fatty acid, whether cerotic or stearic acid, the resulting Na soap usually causes an oil-in-water emulsion. When 25% H<sub>2</sub>O (14.5% white wax, about 60% liquid paraffin) and not over 15% borax is used, a water-in-oil emulsion is formed which becomes less stable as the borax content approaches 1%. The change of phases is not dependent on the white wax; it takes place in precisely the same manner with a mixt. of paraffin hydrocarbons. S. Waldbott.

From the notebook of the soap manufacturer. Karl Pfaff. *Rieschloff Ind. u. Kosmetik* 10, 180-1(1935).—Palm-oil soaps, filled curd soaps and soaps with crude coconut or palm kernel oils are discussed. H. M. B.

The calculation of the amount of alkali required for saponification. G. Knigge. *Seifensieder-Zig.* 62, 941-2, 963-4(1935); cf. C. A. 29, 1670°.—Sample calcs. are given and a paper by Krings (C. A. 29, 6783°) is criticized. J. W. Perry.

Free and actual soap alkali. Th. Ruemele. *Allgem. Oel- u. Fett-Zig.* 32, 409-12(1935).—A review. M. M. Fiskur.

Starch as a filler for soap. Hans Nitschke. *Seifensieder-Zig.* 62, 840-1(1935), cf. C. A. 29, 6783°.—A Central American soap maker discusses his experiences. Up to 15% starch can be incorporated in soap without adversely affecting the quality. J. W. Perry.

Lobelia perfume compositions for soap. Fritz Schulz. *Seifensieder-Zig.* 62, 929-30(1935); cf. C. A. 29, 8233°.—Six recipes are given. J. W. Perry.

Shampoos. Ralph H. Auch. *Soap* 11, No. 11, 26-9, 53, No. 12, 32-5(1935).—The compn., clarification, perfuming, analysis and packaging of shampoos are discussed. E. Scherubel.

Vegetable oils for the lubrication of motors (Robbe) 22. Wetting, etc., agents—soaps (Brit. pat. 433,305) 13.

Oils and fats. Sidney Musher. *Fr.* 785,927, Aug. 22, 1935. These are preserved from rancidity by incorporating therewith or infusing thereto a vegetable material such as barley, oats, maize, soybean seed, cotton seed or tournesol. The vegetable material is removed after the treatment.

Steam-heated vessel for melting fats. Waagner-Burb. A.-G. Austrian 142,908, Oct. 10, 1935 (Cl. 23a).



**Fatty acids and their salts; soaps.** I. G. Farbenindustrie A.-G. Brit. 417,780, Aug. 14, 1935. The acids or salts thereof are obtained by heating oxidation products of liquid or solid nonaromatic hydrocarbons to above 200° in the presence of H<sub>2</sub> under pressure and in the presence of alk. substances, during or after the sapon, and then removing the unsaponifiable products. The acids may be obtained by acidifying with mineral acid. During the heating, any H<sub>2</sub>O present or formed may be removed. The oxidation products may be treated while dissolved in org. solvents. Among examples, paraffin oil or wax oxidation products or soaps therefrom are heated under pressure with NaOH in an atm. of H<sub>2</sub> produced in some cases by the addn of Zn dust, with or without a Ni catalyst and the unsaponifiable substances are removed by means of MeOH, iso-PrOH or benzene.

Removing the "catalyst taste" from hydrogenated oils. Dietrich Hildsch U. S. 2,021,552, Nov. 10 Hydrogenated oils such as those from fish oils and oils from other sea animals and from drying vegetable oils are treated with an alk. compd. such as NH<sub>3</sub>, carbonate or carbamate, alkali metal bicarbonate, diethanolamine or triethanolamine (suitably at a temp. of about 70-110°) until evolution of gas ceases. Cf. C. A. 29, 8130<sup>1</sup>

**Sulfonatable material suitable for making Turkey-red oil substitutes, etc.** Melvin De Groot and Bernhard Kelsor (to Tretolite Co.) U. S. 2,020,998, Nov. 12 An unsat., saponifiable, fatty material is produced, e. g., from sardine oil, by oxidizing with air at 150-250° until there is a drop in  $\eta$  to a value of not over two-thirds the original  $\eta$  and a corresponding increase in the acetyl value, and then subjecting the oxidized material to ther-

mal decompn. at 270-330° with a loss in wt. of not over 12% U. S. 2,020,999 relates to sulfo derivs. of material such as those produced and which may be neutralized with NH<sub>4</sub>OH.

**Alcohols.** Deutsche Hydrierwerke A.-G. (Walther Schrauth, inventor). Ger. 616,705, Aug. 3, 1935 (Cl. 12a, 6 02). High-mol. alcoh. are obtained by sapon. waxes, which may have been hydrogenated, with solid or highly coned alk. alkalis or alk. earths at 150-250°. The sapon. product may be subjected to distn. or extrn. processes. In an example, sperm oil is heated with Ca(OH)<sub>2</sub> at 220° for 6 hrs. The product is a 40% yield of alk. with a fish-like smell and a 60% yield of a valve-like odorless fatty acid.

**Soap.** Benjamin Clayton and Ralph E. Burns (to Refining, Inc.) U. S. 2,019,775, Nov. 6 In a continuous process, reactive soap-forming materials are mixed in proper proportions and sufficient pressure is exerted on the mixt. to cause it to flow through a restricted heating zone where it is heated to accelerate reaction, the soap escapes through a discharge orifice the frictional resistance of which tends to build up a considerable pressure in the heating zone; the soap is cooled, after it leaves the heating zone and while still under pressure, sufficiently partially to solidify it, and a modifier such as a filler or perfume is added during the cooling, and the finished soap is extruded and cut into bars. App. is described, and U. S. 2,019,776 relates especially to such app.

**Soap-frame stripper.** John Woesner (to Cincinnati Soap Co.) U. S. 2,020,755, Nov. 12 Structural, mech. and operative details.

## 28—SUGAR, STARCH AND GUMS

J. K. DALR

**Heat balance in Italian sugar factories.** Gino Carboni. *Ind. saccar. ital.* 28, 395-407 (1935).

**Utility of  $\eta$  determinations in the sugar industry.** Ph. Orth. *Bull. assoc. chim.* 52, 722-52 (1935).—Practical expts. have shown that in the control of factory operations where the  $\eta$  is below 6 or above 8.5, and in detg. of the alkyl, of highly buffered materials like molasses, the old titration method is preferable because of its greater sensitivity. However, in the sulfitation of beet juices or sirups to a  $\eta$  below 8, and in the control of cane-sugar manuf. and of refining the detn. of  $\eta$  is better. The colorimetric  $\eta$  method is preferable for sugar products, because certain nonsugars in them disturb measurements with both the H and the quinhydrone electrode, and because with unbuffered solns. the elec. method requires special equipment. But the elec. method should be used to check the buffer solns. required for the color comparisons. The  $\eta$  detns. must always be made under the same conditions of temp., diln., etc., in order that comparable results can be obtained. In the lab. test tubes should be used for the comparisons, but in the factory the spot-plate method is usually sufficient. F. W. Zerban

**The first "half-shadow" polarimeter.** Noël Deerr. *Intern. Sugar J.* 37, 421-2 (1935).—An historical note.

**Sucrose, beet pulp and adsorption.** I. Péron and Martraine. *Bull. assoc. chim.* 52, 775-8 (1935).—When sucrose is detd. in a beet pulp by the usual methods, without and with the addn. of a known quantity of sucrose, the difference between the 2 results is less than the quantity of sucrose added, and the difference becomes greater the more sucrose is added. H. P. Cazaubon. *Ibid.* 778-9. The above phenomenon can be explained by adsorption of sucrose on the beet pulp, and also by the retarding effect of the added sucrose on the diffusion from unruptured cells into the extn. liquid. F. W. Zerban

**Survey of the inhibiting properties of English beet sugars (on the acid corrosion of steel).** T. N. Morris. *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 183-5 (1935).—Samples of beet sugars, including

some brown unrefined sugars and molasses, were tested against a standard pure cane sugar and a specially pure com. cane sugar. It was shown that the inhibiting property is generally distributed in beet sugars and is particularly abundant in beet molasses and brown beet sugars. Various kinds of fruits were packed in double-lacquered cans with (a) a pure white sugar possessing neither inhibiting nor accelerating properties and (b) 3 parts of the same sugar mixed with 1 part of brown beet sugar possessing strong inhibiting properties. The cans were examd. periodically for H<sub>2</sub> swells and perforations. In every case (except 1, where the loss due to swelling had attained serious dimensions) there was an advantage in using the sugar which definitely contained an inhibitor to acid corrosion.

A. Papineau-Couture.  
**Lowered value of abnormally formed beets and losses resulting from injured roots.** Ferdinand Kryž. *Z. Zuckerind. Czechoslovak Rep.* 59, 454-6 (1935).—See C. A. 29, 6157<sup>1</sup>.

**The abnormal appearance of bloated beets during 1933.** Josef Páizer. *Z. Zuckerind. Czechoslovak Rep.* 60, 1-10 (1935).—See C. A. 29, 4121<sup>1</sup>.

**Effects of freezing temperatures on sugar cane in the Florida Everglades.** B. A. Bourne. *11a. Agr. Expt. Sta., Bull.* 278, 2-12 (1935).—Field samples of 75 sep. mid-stalk cores were extd. in a powerful hand press and preserved in glass jars with a little HgCl<sub>2</sub>. A special tool is required for making the so-called "core-punch" samplings. The hand press gave an extn. yield of 60% of the total juice. A small hand refractometer was found very useful in detg. the sol. solids in the cane juice. Frozen cane gave satisfactory yields of sugar since the deterioration is relatively slow. The absence of the common sugar-liquor difficulties usually caused by the decompn. of frozen cane and the formation of mannitol, dextran and AcOH was noted in connection with the handling of a cane crop over a 75-day period after freezing. C. R. Fellers

**An improved instrument for testing rind hardness in sugar cane.** K. L. Khanna. *Agr. Live-stock India* 5, 160-8 (1935).—The instrument consists of a measured

capacity spring incased in a cylindrical barrel and resting on a piston point which has an extended rod with a hole at its free end to hold the needle point. The pointer which indicates the pressure exerted is fixed to the piston and moves in a groove cut in the barrel. The diam. of the piercing point is 0.75 mm. The instrument is also very useful for studying the cohesion and penetrability of soils.

K. D. Jacob

Ash in raw cane sugars. Louis Sattler, John E. Mull and Irving Longe. *Facts about Sugar* 30, 377-8 (1935); cf. *C. A.* 21, 2244, 244, 1242.—The formulas previously given for the  $d_{20}^{25}$  of ash by measurements of elec. cond. apply only to filtered solids of raw sugars. If the solids are not filtered, the following formulas are to be used: percentage of total ash =  $0.001566 K - 0.0001954 K_1 + 0.4160$ , percentage of ash in the water-sol. portion of the sugar =  $0.001558 K - 0.0002024 K_1 + 0.4174$ . The results are within 0.05 of the chem. ash, in most cases within 0.03. If greater accuracy is desired, filtered solids should be used.

F. W. Zerban

Purity difference between first-expressed cane juice and sirup. Carlos L. Locust. *Intern. Sugar J.* 37, 431-3 (1935).—Two reasons are given for the difference between the crusher juice and sirup purities. One is the higher glucose ratio of the crusher juice than of the clarified juice or sirup. The other is the combined effect of the following factors: (1) double clarification of the impure fractions, (2) use of small resettlers, (3) application of hot mud for maceration on the mill, (4) quick removal of the light mill juices from the maceration system and (5) heating the combined primary mud and secondary juice to 215-220°F., the reaction of the secondary clarified when discharged being 6.7 to 6.8 pH.

D. P. Langlois

Refractometric estimation of dissolved solids in sugar sirups containing invert sugar. H. C. S. D. Whalley. *Intern. Sugar J.* 37, 353-5 (1935).—Solids by refractometer and solids by drying taken on sirups containing invert sugar show differences which are partly or wholly due to the assumption that dextrose and levulose have refractive indices identical with those of sucrose at the same concns. Pure sucrose sirups of known concn. and weight were used; refractive indices were taken before and after different degrees of inversion with 0.2 N HCl. The results showed that the refractometer solids read too low for the partially inverted sirups. The mean value detd. was 0.021% for each one % of invert sugar present. Inversion with invertase gave a value of 0.022% for each one % of invert sugar present. Synthetic mixts. of pure sucrose, pure dextrose and pure levulose gave a difference in refractometer solids of only 0.0009% for each one % of invert sugar present. This difference in behavior is assumed to be due to the fact that the sugars prepd. from starch and molasses are not identical with those from the inversion of cane sugar.

D. P. Langlois

[Report on] sugar technology. [Classification of re-factory processes.] W. L. McCleery. Hawaiian Sugar Planters' Assoc., *Proc. 54th Ann. Meeting (Rept. Comm. on Chem. Expt. Sta.)* 1934, 91-105, cf. *C. A.* 28, 7051.—In exp'l. runs on a factory scale, clarification was not markedly improved by the use of Na aluminate. With Zn hypophosphite there was marked decoloration but the juices darkened rapidly afterward; the best results were obtained in combination with double superphosphate. Promising results were obtained by treating the juice with more than the usual amt. of lime and pptg. the excess lime with  $CO_2$ . Treatment of the mixed juice with  $SO_2$  produced a marked increase in clarity, accompanied by a decrease in color, the juice settled rapidly and while there was a moderate increase in the vol. of settlings, the settlings filtered well with a firm washable cake. Mon-ammonium phosphate "A" was superior to double superphosphate in all respects except for the clarity of the juice and in this respect the improvement with the latter did not compensate for other disadvantages. Mon-ammonium phosphate "B" and  $(NH_4)_2HPO_4$  were not suited for clarification purposes. (The terms "mon-ammonium phosphate A" and "mon-ammonium phosphate B" apparently refer to the com-

materials sold under the names of Ammophos A and Ammophos B.)

K. D. Jacob

Reducing substances in diffusion juice. Renato Salam and Mario Testoni. *Ind. saccar. ital.* 28, 460-1 (1935).—The difference between the total reducing sugars, as invert, minus twice the levulose content (by reduction of Ost's soln.) varies with the quality and the state of preservation of the beets. The larger this difference for stored beets, the greater is the degradation of non-sugar substances, pectins, etc.

L. Cusachs

The  $pH$ -CaO diagram of juices. D. Meneghini and I. Sorago. *Ind. saccar. ital.* 28, 443-53 (1935).—Knowledge of the  $pH$ -CaO relation over a whole season for each beet factory juice and refinery liquor enables a more systematic control over mfg. operations. In the raw juice,  $pH$  and purity values run parallel. The Sb electrode was used.

L. Cusachs

The manufacture of "invert sirup" from surplus cane. Colin W. Waddell. *Intern. Sugar J.* 37, 392-3 (1935); cf. *C. A.* 29, 6785.—The process is to grind cane, partly clarify the juice, add acid, invert sufficient sucrose to prevent crystal and conc. to a sirup of about 86° Brix. The acid is added to the juice and inversion is carried out in the evaporators. The sirup is neutralized after leaving the evaporators and before going to the pans. A sirup having an invert/sucrose ratio of 210 or greater was found to be safe from crystal.

D. P. Langlois

Regenerative evaporation by thermocompressors. L. A. Tromp. *Intern. Sugar J.* 37, 386-9 (1935).—Several diagrams are given illustrating the use of thermocompressors in sugar-factory evapn.

D. P. Langlois

The optimal coagulation with lime: the influence of natural alkalinity. J. Dédek and J. Valášek. *Zucker-und. Cechoslovak Rep.* 59, 492-71 (1935).—See *C. A.* 29, 6479.

F. M.

Detection of adulteration in maple sirup. A simplified rapid method based on electrical conductivity. Augustus Conlin. *Ind. Eng. Chem., Anal. Ed.* 7, 429-7 (1935).—A modification of the method of Snell (cf. *C. A.* 7, 4021) was used with 7200 sirups. Values from 40 to 200 were observed. Sirups with values under 100 were considered adulterated. Canadian sirups showed higher values than American, and darker sirups higher values than light-colored sirups.

Amy Le Vesconte

Investigation of the aromatic materials in maple sugar. J. Risi and A. Labrie. *Can. J. Research* 13B, 175-84 (1935).—The aromatic substances occurring in maple sirup and sugar are partly solid and partly resinous, the former congs. vanillin and vanillic acid and the latter yielding guaiacol. The aromatic substances in the sap are increased by the process of boiling. The yellow or brown coloration of the sirup is not essentially due to the production of caramel but to the formation of phenols. The bark of the maple contains "acerose" which hydrolyzes coniferin and amygdalin. Maple sap contains an amylase type of enzyme which transforms starch into a disaccharide at relatively low temps. The wood of the maple contains very little coniferin and this is transformed into resinous substances of the nature of lignin early in Sept. Maple seeds do not contain coniferin but do contain the same aromatic substances as the sirup. It is suggested that coniferin may be the mother substance of the vanillin. The aroma observed during the boiling of maple sap is due principally to the formation of hadronal and partially to small quantities of other aromatic comds. Destructive sublimation of hadronal produces vanillin, vanillic acid and guaiacol just as does the aromatic ext. of maple sirup. Hadronal probably does not occur in the free state in the wood but is synthetically formed from lignin by catalytic action.

J. W. Shupley

Determination of fiber % cane by an indirect method. Saktrapda Sanjay. *Intern. Sugar J.* 37, 390-1 (1935).—The fiber % cane is at present calcd. from the formula:  $F = 101 - 101W/(100 - B)$ , where  $F$  = fiber % cane,  $W$  = water % cane and  $B$  = the Brix of the expressed juice. For more precise results S. proposes a different value for  $B$ . This value is the Brix of the abs. juice which is obtained by multiplying the Brix of the primary (or

crusher) juice by a dry milling factor. This factor is obtained by dividing the Brix of the mixed juice by that of the primary (or crusher) juice.

D. P. Langlois

The unfermentable sugar content of cane molasses F. Reindel and A. Frey. *Intern. Sugar J.* 37, 384-5 (1935).—Previous investigators have described a product of the reaction of bases on *d*-glucose and *d*-fructose to which they have given the name "glucose." They believed it to be identical with the unfermentable sugar residue of cane molasses. More recently it has been proposed that this residue is not a simple sugar but a mixt of sugars. The authors give explt evidence in support of the latter claim. The residue from completely fermented molasses was fractionated with abs alc and a fermentable sugar was sepd. Furthermore 2 osazones were sepd, one m 194.195° which suggested galactose, the other m 204-206° which points to glucose or fructose.

D. P. Langlois

Production of high-test molasses Victorino M. Kilayko. *Sugar News* 16, 386-8 (1935).—Data are given of heavy invert sugar from cane juice.

L. Cusachs

The molasses question and the saline coefficient R. Dutilloy. *Bull. Assoc. chim.* 52, 760-6 (1935).—The saline coeff is not a reliable criterion of the exhaustion of (beet) molasses. The latter should be judged by the ratio of the molasses wt to that of white sugar produced. This ratio should not exceed 0.33, except in abnormally dry yrs.

F. W. Zerbán

The amber sorghum from Minnesota as a source of sugar and alcohol. Cesare Grossi. *Ind. saccar. ital.* 28, 408-15 (1935).—The agr. results are promising, and the plant offers possibilities as a source of alc, acetone, Bu alc, and cellulose.

L. Cusachs

Juiciness and sweetness in sorghum stalks. G. N. Rangaswami Ayyangar. *Madras Agr J.* 23, 350-2 (1935).—The juice from sweet-stalked varieties was higher in Brix value and sucrose content and lower in Cl than was the juice from varieties which produced pithy and insipid stalks.

K. D. Jacob

Cane ecology (Hardy) 15. Effects of soil liming on sugar cane (Rodriguez) 15. Soil conditions detg. response of sugar cane to fertilization with potash (Turner) 15

7 Rept. on chemistry [growth-failure of cane—arsenic in refined sugar] (Hance) 15.

Sugar. Spreckels Sugar Co. Fr. 785,491, Aug. 10, 1935. App. is described for recovering sugar from soln. in which the rate of feed of CaO is regulated with respect to the rate of flow of the soln. itself. The proportion is such that after leaving the point of admission of the CaO the soln. contains practically the whole of the CaO necessary to ppt the sugar. The particles of CaO are broken up in the soln. Cf. C. A. 29, 83984.

Sugar syrup. Starkefabrik Kyritz G. m. b. H. Ger. 618,985, Aug. 9, 1935 (Cl. 89: 1 01). Freshly converted sugar syrup is refined by treatment with alkali to give a *pn* value of above 6. After removal of the pptd impurities, the syrup is acidified to a *pn* value of 4.5-6.7, filtered and coned. The syrup is preferably made alk by Ca(OH)<sub>2</sub> and acidified by (CO<sub>2</sub>H) or H<sub>2</sub>SO<sub>4</sub>.

Defecating diffusion juices. Rudolf Picka. Brit. 432,403, July 25, 1935. See Fr. 781,232 (C. A. 29, 64624).

Sugar recovery. Spreckels Sugar Co. Fr. 785,368, Aug. 8, 1935. Sugar is recovered from dild. molasses, etc., as Ca sucrate in increased amt by treating the soln. so as to reduce to a fine state the sucrate which it contains. By this means the insol. layer of Ca sucrate on the particles is broken and fresh ppts may be formed.

Recovery of nonsugars from molasses, etc. Gustave T. Reich. U. S. 2,022,093, Nov. 26. Materials such as molasses, malted grain products, sugar wastes, sweet waters and fruit juices, suitably dild., are treated with isopropyl alc until a sepn of the materials into layers (one of which contains nonsugars in sufficiently refined condition for processing) is effected.

Dextrose. International Patents Development Co. Fr. 785,057, Aug. 1, 1935. Very pure cryst dextrose is obtained in a vessel maintained under const. vacuum, fresh soln. being added to the vessel during the stage when the dextrose deposits from the soln. on the nuclear crystals at a speed greater than the speed of evapn of the water, so as to reduce gradually the b. p. At the end of the operation the soln. is dild. with water.

## 29—LEATHER AND GLUE

ALLEN ROGERS

Scientific development of the leather industry. Alphonse Fievez. *Bourse aux cours de Belg.* 1935, No. 10, *Chim. tech.* 24, 318-20 (1935).—An address.

H. B. M.

Corrosion of metals by contact with leather R. F. Innes. *J. Intern. Soc. Leather Trades Chem.* 19, 519-63 (1935).—Leathers tanned with single tanning materials (Cr, syntan sulfite cellulose and 8 vegetable tannins) were kept in contact with metal strips (steel, Cu, Zn, Sn, Duralumin, brass and bronze) for 38 weeks at relative humidities of 50, 70 and 90%, at room temp, and at 70% relative humidity at 37°. Corrosion was judged from appearance. Of the metals tested, Sn was most resistant and Cu and its alloys were the least. Of the leathers tested, that tanned with syntan caused greatest corrosion, probably because of an abnormally low *pn* value, followed by leathers tanned with sumac and myrobalans. Leathers tanned with oak bark, gambier, mimosa, quebracho and sulfite cellulose caused least corrosion. Aside from the syntan leather, there was no direct relationship between leather *pn* value and corrosion. The amt. of corrosion increased with increasing temp. or humidity. Corrosion was increased when leathers were oiled with cod-liver oil contg. about 12% free fatty acids, and decreased but not eliminated when leathers were oiled with petrolatum. Cf. Kubelka, et al., C. A. 30, 321<sup>8</sup>.

H. B. Merrill

Chlorinated rubber in leather finishing. M. C. Lamb and W. F. Chapman. *J. Intern. Soc. Leather Trades Chem.* 19, 563-9 (1935).—A satisfactory finish is prepd. from chlorinated rubber dissolved in xylene, plasticized

with either dibutyl phthalate or tricresyl phosphate, with the addn. of synthetic resins to give improved adhesion.

H. B. Merrill

7 Phosphatide compns. [products used in the leather industry] (Ger. pat. 619,235) 25. Vacuum app. and method for tanning, etc. (Brit. pat. 432,552) 1.

Preserving wet hides. Max Stern. Ger. 594,821, June 28, 1935 and Ger. 595,897, July 25, 1935 (Cl. 28a: 1). See Brit. 422,821 (C. A. 29, 4620<sup>9</sup>).

Degreasing skins. John H. Connor and Matthew M. Merritt (to The Tanning Process Co.). Brit. 432,631, July 22, 1935. Skins, hides and leather are degreased by treatment with a grease solvent which, after the treatment and while in contact with the skin, is caused to form an emulsion in H<sub>2</sub>O. A substance, e. g., a fat-liquoring oil, sulfonated or not, of animal or vegetable origin, may be added to the solvent, e. g., kerosene, to assist the subsequent formation of the emulsion. Cf. C. A. 28, 3938<sup>8</sup>.

Apparatus for drying furs and skins pinned to boards. Harry S. Wood. U. S. 2,020,392, Nov. 12. Various structural details.

Tanning. Herman A. Herzog (to A. C. Lawrence Leather Co.). U. S. 2,019,568, Nov. 5. Skins such as cowhides, etc., are tanned by treatment with a W compd. in acid soln. such as may be formed from Na tungstate and HOAc and then with Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>. Cf. C. A. 29, 636<sup>9</sup>.

Tanning. Franz Hassler. Ger. 588,074, Sept. 17,

1935 (Cl 12q 20 04) See U S 2,012,928 (C. A. 29, 17116\*).

Tanning agents I G Farber and A G Fr. 785,792, Aug 19, 1935 Sulfonation products of Ph, or its derivs, particularly OH derivs, are used. The derivs may be condensed by treatment with  $\text{CH}_3\text{O}$  before or after sulfonation.

Dyeing leather Soc pour l'ind chim à Bâle Fr. 784,829, July 25, 1935 Cu compds of azo dyes of the formula  $\text{R}_1 \rightarrow \text{R}_2 \rightarrow \text{R}_3 \rightarrow \text{R}_4$  ( $\text{R}_1$  is a tetrazo compd of a biphenyl radical contg at least one  $\text{SO}_2\text{H}$  group,  $\text{R}_2$  is an aryl group contg at least 2 OH groups capable of coupling twice,  $\text{R}_3$  a diazo compd contg groups capable

of forming lakes and  $\text{R}_4$  a coupling component) are used for dyeing leather. Thus, the Cu compd of 2-amino-1-hydroxybenzene-4-sulfonic acid  $\rightarrow$  1,3-dihydroxybenzene  $\rightarrow$  4,4'-diaminodiphenyl-2,2'-disulfonic acid  $\rightarrow$  1,3-dihydroxybenzene dyes leather a brown-yellow. Several examples are given.

Fat-liquoring leather Imperial Chemical Industries Ltd, Richard Greenhalgh and Geo S J. White Brit 432,636, July 22, 1935 Compos for fat-liquoring and oiling-off leather are prep'd by mixing fish or other marine animal oils and mineral and other oils with an unsatd fat acid and adding  $\text{NH}_3$ . The mixt is applied to leather, with or without  $\text{H}_2\text{O}$ , to form an emulsion.

### 30—RUBBER AND ALLIED SUBSTANCES

C C DAVIS

Possible developments in the rubber industry Alan Calver *India-Rubber J.* 90, No 17a(Intern No), 32-4(Oct 31, 1935)—A discussion, with particular reference to the plantation industry. C C Davis

Some incompletely solved rubber problems J. R. Scott *India-Rubber J.* 90, No 17a(Intern No), 2-5(Oct 31, 1935)—A crit discussion of the swelling of vulcanized rubber in certain org liquids and of the phenomena of plasticity. C C Davis

The training of young men in the rubber industry V H Wentworth *Trans Inst Rubber Ind.* 11, 241-50 (1935)—Particular attention is paid to chemists. C C Davis

The extraction of the rubber from rubber bearing plants by means of solvents V Provorov *Sovetskii Kauchuk* 1935, No 3, 26-34—A detailed description of the conditions of extn of rubber from chondrilla, guayule and kender by means of alc, benzene, toluene, etc. A Pestoff

The distribution of rubber in rubber-bearing plants A Prokof'ev *Sovetskii Kauchuk* 1935, No 1, 19-23—*Tau-saguz*—The main mass of rubber is in the roots. Plants one year old have 1-8% and 2-3 years old 8-30% of rubber in the roots. *Kok-saguz*—The roots are the source of the rubber. Plants one year old have 5% and a few 10% of rubber. *Krum-saguz*—The roots have the main mass of rubber. Wild plants have 5-6% rubber, cultivated plants one year old 0.7-0.8% and seldom 1.5-2% rubber. *Teks-saguz* (*Scorzonera acanthoclada*)—The roots accumulate the rubber to 5-6% *Guayule*—The roots and stem have the mass of rubber, leaves only traces. Plants one year or two years old have 5-10% rubber and 8-18% of tars. Cross sections of the plants are given. A Pestoff

The complete utilization of rubber bearing plants D Kraft *Sovetskii Kauchuk* 1935, No 4, 44—The guayule contains 6-8% rubber, 12% tars, terpenes and etheral oils. The roots of tau-saguz and kok-saguz contain 15% water, 10-14% rubber, 4-6% tars, 35-40% inulin, 3-4% sugars, 5-6% pectin and 15% lignin. A Pestoff

Obtaining natural latex from rubber-bearing plants. A Ignat'ev *Sovetskii Kauchuk* 1935, No 3, 22-6—The quantity of latex obtained from *Kok-saguz* was the equiv. of 30-40% of total rubber in the roots. The latex particles from tau-saguz and kok-saguz 3-4 months old are round and those from plants 7 months and older are oblong (11.5-4 microns long). The concn of the latex was 30-59%, with  $\text{pH}$  value 5-5.2. The mol. wt. of kok-saguz from latex is 200,000 (according to the formula of Staudinger). *Kok-saguz* contains 35-40% carbohydrates, mostly inulin. A Pestoff

The natural stabilizers of latex. W H Stevens *India-Rubber J.* 90, No 17a(Intern No), 36-7(Oct 31, 1935)—Since certain rubber latexes require no preservative, the identification of the natural stabilizing agent which they probably contain, and application of the latter to *Hevea* latex, represents an important tech problem. *Abarana* (gutta-percha) latex without preservative was shipped

from Brazil to England with no coagulation, and examn showed a  $\text{pH}$  value of 4.0. It was not coagulated by alkalis nor by acidification of such alk latex. Acidification of a mixt of *Abarana* latex and  $\text{NH}_3$ -preserved *Hevea* latex did not cause coagulation, indicating that the stabilizing power is transferable. In electrophoresis, deposition took place on the cathode. *Abarana* latex showed active Brownian movement, and in general resembled *Hevea* latex microscopically. It creamed rapidly, with a fairly clear serum with brownish sediment, and the system could be redispersed by agitation, before and after dialysis. It was coagulated by acetone or alc, and gave the following percentage values: total solids 37.1, dry crepe (by acetone) 33.6, diffusate (70 hrs) 3.4, 0.984, acetone ext. of dry crepe 2.2, N in dry crepe 0.20, gutta hydrocarbon (by difference) 31.2. The N was distributed thus (in percentage on the latex and dry material, resp): total solids 0.33, 0.89, crepe 0.20, 0.59, diffusate 0.17, 4.97. This indicates that some of the diffusible N is coagulated by the acetone and is not removed on washing the crepe. A solution of this problem might lead to the better production of acid *Hevea* latex, and its adaptation to useful ends. C C Davis

Investigation of the structure of rubber by means of electron diffraction K I Krulov. *J Exptl Theoret Phys.* (U S S R) 5, 524-33(1935)—Electronographs of rubber show much more detail than the corresponding x-ray pictures. A debyeograph of unstretched rubber gives 3 amorphous rings with identity periods of 4.15, 2.05 and 1.25 Å. With stretched rubber, interference points appear and the whole picture is that of a fiber diagram. The elementary cell of rubber is orthorhombic with  $a = 12.3$ ,  $b = 8.3$ ,  $c = 8.1$  Å. Chloroprene rubber, even when not stretched, gives a point-interference diagram indicating 8 chloroprene nuclei per elementary cell, with dimensions  $a = 10.93$  and  $b = 8.23$  Å and 3 amorphous rings with identity periods of 2.23, 1.47 and 1.18 Å. Duprene gave a multipoint electronograph. F H Rathmann

Rationalization of the hardness testing of rubber. J R Scott *Trans Inst Rubber Ind.* 11, 224-40(1935)—Testing rubber for hardness is at present in an unsatisfactory state because of the multiplicity of instruments which give different readings for the same sample. Attention is drawn to the need of standardizing this test. The force which must be applied to a ball to indent the surface of a large mass of rubber depends on the elastic modulus of the rubber (at small deformations), the radius of the ball and the ratio of the depth of indentation to this radius. The relation of the force to the modulus and the ball radius, with the 3rd factor const., is deduced theoretically. Expts to det the following relationships are described: (1) modulus and force to cause a given indentation, (2) force and depth of indentation, (3) ball radius and depth of indentation. The expts confirm relation (1) and that between force and modulus, and show how the force is related to the depth of indentation. By means of the relation between indentation, modulus, force and ball radius, the elastic modulus of rubber for small elongations or compressions can be calcd from hardness tests. Though

the method is only approx., it is precise enough for many practical purposes, and therefore forms a useful means of detg. moduli in cases where ordinary tensile or compression tests are impracticable. The results make it possible to interconvert the readings of any hardness instruments which use spherical indentors under definite loads, whether or not applied in addn. to a small "zero" load.

C. C. Davis

Modern developments in the softening of rubber  
T. L. Garner. *India-Rubber J.* 90, No. 17a (Intern No.), 6-8 (Oct. 31, 1935).—A review and discussion

C. C. Davis

Chlorinated rubber in protective coatings C A Redfern. *Silk and Rayon* 9, 492-3 (1935)—The advantages and limitations of chlorinated rubber in protective coatings are described.

M Harris

Crystals and fusion in stretched rubber Peter A Thiesen and Werner Wittstadt. *Z. physik. Chem.* B29, 359-62 (1935)—Upon stretching vulcanized rubber, the rearrangement of the mol. in the lattice does not stop after the stretching has ceased and the final orientation depends on the temp. Within a wide temp. range, there is a fixed ratio between crystals and a glassy enamel for each temp. The proportion of crystal material decreases with increase in temp. The change is reversible and the equl. depends on the pressure

G M Murphy

The changes in the process of contact transformation of ethyl alcohol into butadiene, in connection with fatigue of the catalyst Yu A Gorin, O M Nelmark and F N Kogan. *Sintet. Kauchuk* 1935, No 5, 6-10—Fatigue of the catalyst proceeded in 2 ways: (1) the C, formed by thermal decompn. of the org. mols., deposited on the catalyst in the form of a "carbon net," closing the dehydrating centers of the catalyst, and (2) regeneration of the catalyst, by means of burning this C with hot air (500°), changed the cryst. structure of the catalyst surface. These 2 phenomena resulted in an increase of H and decrease in ethylene in the gas products, an increase of unchanged EIOH, aldehydes, hydrocarbons and BuOH in the liquid products, and a lowered yield of butadiene. Tables and graphs are given

A Pestoff

Plastics based on rubber, with special reference to Rubbone. W. H. Stevens and H. P. Stevens. *Trans. Inst. Rubber Ind.* 11, 182-96 (1935).—Substantially the same as C. A. 29, 4625<sup>a</sup>, 8400<sup>a</sup>, with some minor additions.

C. C. Davis

Risks in the rubber industry E. R. A. Merewether. *Trans. Inst. Rubber Ind.* 11, 162-77 (1935)—The paper deals with risks associated with (1) machinery, electricity, fire and explosion, (2) raw materials (NH<sub>4</sub>, Pb, Hg and Sb compds., asphalt, C, inorg. fillers and dusting agents, org. accelerators, solvents), (3) finishing processes, e. g., sand-blasting; (4) radioactive substances, and (5) lab. work.

C. C. Davis

Fire risks and their prevention D W Wood. *Trans. Inst. Rubber Ind.* 11, 150-61 (1935).—The paper deals primarily with the rubber industry, and discusses building sites, plant design, construction, power and light, heating, raw materials, processes, reclaiming, solvent recovery, static electricity and fire-extinguishing methods.

C. C. Davis

Rubber, bitumens and road surfaces Harry Barron. *India-Rubber J.* 90, No. 17a (Intern No.), 21-31 (Oct. 31, 1935).—A crit. review and discussion, dealing with (1) bituminous materials (definitions, types, properties, tech. uses); (2) applications of bituminous materials to rubber manuf. (history, applications, elec. properties, hard rubber, aging, identification) and (3) rubber in bitumens, and road-surface problems (emulsions of asphaltic bitumens and tar, bituminous road surfaces, asphaltic macadam, rock asphalt, desirable features of road materials, the use of emulsions, tests for road emulsions, rubber-bitumen mixts., rubber solns., dispersion of rubber in molten bitumens, use of latex, latex-bitumen and emulsions). Sixty references.

C. C. Davis

Porosity in vulcanized rubber. Walter H. Bodger. *Trans. Inst. Rubber Ind.* 11, 197-223 (1935).—The work was undertaken to develop quant. methods for measuring

porosity in vulcanized rubber, to study the individual factors responsible, and to develop methods of overcoming it in practice. Of the methods developed for assessing the degree of porosity, which included visual examn., tensile strength, change in acetone ext. on aging, permeability and d, the percentage diminution in d. is the most satisfactory. The permeability to moisture, acetone ext. of the aged sample and visual examn. also give some indication of the degree of porosity. Severe over-mastication, particularly on cold rolls, increases the entrapment of gases during mixing. If the mech. pressure applied during vulcanization is low, the diminution in vol. which proceeds with vulcanization causes a further lowering of the pressure, some of the entrapped gases are liberated, the rubber is partially "set up," free escape is inhibited and pores develop. The effect of over-mastication can also be brought about by heat treatment or oxidation of raw rubber. Mech. work expended on a rubber mixt., such as sheeting at friction speed, calendaring thick, or extrusion of thick articles, may increase porosity under suitable conditions. Softeners increase porosity in the same way as does over-mastication, whereas stiffening fillers tend to prevent porosity. When no mech. pressure is applied during vulcanization, porosity is likely, but a slight pressure may cause even worse porosity by preventing the free escape of gases during the soft stage before vulcanization progresses. The degree of porosity depends on the conditions during the initial stages of vulcanization, and is a useful indication of setting-up when scorching tests are being carried out. It is not influenced by the action of heat, in the later stages of vulcanization, on the rubber-S or oxygenated rubber complexes. Moisture from the ingredients, processing or method of vulcanization increases porosity under appropriate conditions. Ultra-accelerators or high temps. of vulcanization tend to increase the porosity of mixts. which are sensitive, since the escaping gases are more completely entrapped by a rapid setting-up of the rubber. Some applications of the results of the methods developed for overcoming porosity in practice are given, including increase in mech. pressure, avoidance of soft mixts., drying of fillers, mastication at high temp., multiple-ply calendaring, etc.

C. C. Davis

Self-vulcanizing cements. B. Iabritziev, G. Buiko and E. Pakhomova. *India-Rubber J.* 90, No. 17a (Intern No.), 9-16 (Oct. 31, 1935); cf. C. A. 29, 2301<sup>a</sup>.—Self-vulcanizing cements are considered to be those which after evapn. become vulcanized within 1 day or 2 days at room temp. Tests of tetramethylthiuram disulfide, piperidinum pentamethylendithiocarbamate, Pipsoil X and Zn butylxanthate (I) showed that I has the lowest crit. temp., so it was used in all the expts. *p*-Toluidine (II) lowered its crit. temp. and was used as an activator. The technic was to prep. dry films 0.18-0.20 in. thick from C.I.I. cements, vulcanize these in water at 80° for various times and test the mech. properties of the vulcanizates. ZnO had a strong activating effect, the best mech. properties being obtained with 3% (based on the rubber). Zn stearate was without influence. Stearic acid diminished the tensile strength. The best percentage of S, in consideration of the rate of vulcanization and phys. properties, was 3 (based on the rubber). Over 4% of S accelerated only slightly more than did 4%, and impaired the mech. properties. II not only accelerated greatly, but improved, the mech. properties; 2% gave the optimum results. Aging tests in a Geer oven at 70° showed that II is an antioxidant. Tests of the adhesion of various films between rubber and leather and rubber and cotton duck under various conditions of temp., time and moisture, the object of which was to study the behavior of films in footwear manuf., are described. In general, lab. tests of the adhesion (judged by the force of sepn.) gave only an approx. indication of the behavior of cements in practice. Measurements of the changes in viscosity during storage of cements of different formulas showed that it is dangerous to keep mixed cements many hrs., and the 2-part method, with blending just before use, is highly recommended. Storage in 2 parts does not impair the subsequent adhesion. Above a certain concn., I and II

react, with liberation of S, so this must be avoided. The formula finally recommended is smoked sheet rubber (III) 100, pptd S 3, 13, II 2, ZnO 3; to be stored as 2 solas as follow: (1) III 100, S 8, II 2, ZnO 3, (2) III 100, I 6, II 2, ZnO 3. Cements of this type are of advantage in cementing power transmission belts, repairing automobile tubes, cementing soles to uppers in the shoe industry, other shoe construction problems and many repairing problems. C. C. Davis

Rubber products used in food manuf. (Juve) 12. Effect of rubber in the life of the rubber-bearing plant (Bosse) 11D. Chlorinated rubber in leather finishing (Lamb, Chapman) 29. Activated C (Sutcliffe) 18. Aralkyl triethoxycarbonates [for use as rubber-vulcanization accelerators] (U. S. pat. 2,021,726) 10. Elec. insulating compns (Ger. pat. 619,253) 13. Elastic fabric (U. S. pat. 2,021,352) 25. C black (U. S. pat. 19,750) 18. Preservative coatings on cloth, leather, etc. (U. S. pat. 2,019,531) 25. Polishing devices comprising resilient rubber (U. S. pat. 2,021,371) 13. Thermoplastic compns (Fr. pat. 785,641) 13. Active C (Austrian pat. 142,780) 18.

Concentrating latex. Rubber Producers Research Association, George Martin, Wilfrid S. Davey and Lionel Lindell-Stewart. Brit. 433,303, Aug. 8, 1935. Latex is concd by removing from the bulk thereof portions which are warmed and exposed to the open atm on heated surfaces to expel their H<sub>2</sub>O content before being returned to the bulk. App. is described.

Preserving rubber latex. Alfred T. B. Kell. Brit. 433,116, Aug. 12, 1935. The latex is preserved by adding to it in aq. soln. Na or K silicate and K<sub>2</sub>CO<sub>3</sub>. When certain fillers, e. g., compds. of Na, K and Zn, are compounded with the latex, HCl also may be added. In an example, K silicate 10 and K<sub>2</sub>CO<sub>3</sub> 2 are dissolved in H<sub>2</sub>O that is acidulated with 1% of HCl 4 parts. Then 5% of this soln., calcd. on the solid rubber content of the latex, is added to the latex. A cement-like filling compn. is made thus: K silicate 15, lithopone 5 and magnesite 0.5 parts are mixed and added to CaCO<sub>3</sub> 30, pptd. SiO<sub>2</sub> 10 and hydrated Mg silicate 5 parts and the whole is ground with 10 parts of the above stabilizing soln. This latex-cement compn., which may contain 20 parts latex to 80 cement, may be used for covering fabrics, surfacing floors, etc.

Latex and regenerated rubber. International Latex Processes Ltd., Arthur E. Barnard and William E. Messer. Fr. 785,751, Aug. 19, 1935. A stable compn. contg. natural rubber latex and a soap dispersion of regenerated rubber is obtained by making a dispersion having the same osmotic pressure as natural latex and mixing the 2.

Latex-treated tubing. Joseph A. Kennedy (to Anaconda Wire & Cable Co.). U. S. 2,019,709, Nov. 5. A tubing which resists oil, water, etc., is prep'd by spraying an *impermeable layer* such as one of latex or asphalt over the inner raceway of the tube (which has an inner tube of interconnected flexible strands), spraying an aq. dispersion of latex over the exterior of the inner tube and applying a protective covering such as lacquer over the outer latex coating. App. is described.

Rubber compositions. John Talbot. Brit. 432,551, July 30, 1935. Rubber, balata, etc., are rendered fire-proof by adding 5-16% of Ph<sub>3</sub>PO<sub>4</sub>, tricresyl phosphate, Ph<sub>3</sub>BO<sub>3</sub> or a mixt. of Ph<sub>3</sub>PO<sub>4</sub> and tricresyl phosphate. The esters are preferably added as emulsions at the time of masticating with the usual fillers, S, etc.

Rubber compositions. International Latex Processes, Ltd. Ger. 619,408, Oct. 1, 1935 (Cl. 39b 5). See Brit. 404,665 (C. A. 28, 3939i).

Halogenating rubber. Metallgesellschaft A.-G. Fr. 785,257, Aug. 6, 1935. A soln. of rubber is introduced slowly into a vessel contg. a halogenating agent in amt. sufficient to halogenate rapidly the rubber introduced above the middle halogenation stage which is the critical pt. for the stability of the soln. The solns. of the products

1 prep'd are stabilized by incorporating therewith min. proportions of a polyhydric phenol such as pyrogallol or resorcinol or an oil such as wood or linseed oil.

Chlorinating rubber. Thurm & Beschke Kommandit-Gesellschaft (personally responsible partner, Herbert Turnauer). Brit. 433,246, Aug. 12, 1935. See Fr. 777,865 (C. A. 29, 4631i).

Chlorinating and vulcanizing rubber. International Latex Processes Ltd. Brit. 433,252, Aug. 12, 1935. Cl. is introduced into a previously vulcanized latex at least until pptn. of discrete particles of chlorinated rubber takes place. Further chlorination may be effected in the liquid or the ppt. sep'd, e. g., by filtration or decantation, and further chlorinated in a closed chamber before or after drying.

Chlorinated rubber. Eugen Moloney (to the firm Chemische Fabrik Buckau). U. S. 2,020,076, Nov. 5. In the production of a rubber chloride soln. by chlorinating a rubber soln., there is added, prior to the final pptn. of the resulting rubber chloride, a small amt. of an alkali metal or alk. earth metal hypochlorite, which serves to lower the viscosity.

Chlorinated rubber. John McGavack (to United States Rubber Co.). U. S. 2,021,318, Nov. 19. Latex is vulcanized and Cl. is introduced into the vulcanized latex.

Chlorinated rubber. Frederick P. Leach, Wilfrid D. Spencer and Imperial Chemical Industries Ltd. Brit. 430,906, June 20, 1935. Solid chlorinated rubber is obtained from solns. thereof by bringing the soln. into contact with an excess of a liquid precipitant maintained in agitation by means of a stirring arrangement; the precipitant is without chem. action on chlorinated rubber and mixes with the solvent in the soln. to produce a mixt. having substantially no solvent power for the chlorinated rubber. The soln. of chlorinated rubber may be squirted as a thin stream or in finely divided form through nozzles onto the surface or below the surface of the precipitant. In an example, a soln. of chlorinated rubber in CCl<sub>4</sub> is brought into contact with a lower aliphatic alc., e. g., MeOH, the ppt. may be removed and washed with MeOH described in Brit. 433,196 (following abstr.). CCl<sub>4</sub> and MeOH may be recovered from their mixt. by distg. to obtain a const.-boiling mixt. and a residue of alc. and distg. the residue to obtain pure alc., the const.-boiling mixt. is treated with H<sub>2</sub>O to obtain a CCl<sub>4</sub> layer and an aq. alc. layer, which are sep'd., the aq. alc. layer being dist'd. to obtain pure alc.

Chlorinated rubber. Kenneth S. Jackson and Imperial Chemical Industries Ltd. Brit. 433,196, Aug. 12, 1935. Products of improved stability are obtained by pptg. solid chlorinated rubber from solns. thereof, e. g., in CCl<sub>4</sub>, by addn. of MeOH, sepg. the pptd. solid and bringing it into intimate contact with a further quantity of MeOH. Films of chlorinated rubber may be obtained by evapg. solns. of the product in solvents, e. g., C<sub>6</sub>H<sub>6</sub>, in which a plasticizing agent, e. g., up to 33% of di-Bu phthalate on the wt. of the chlorinated rubber, has been incorporated.

Chlorinated rubber. Rubber Producers Research Association, Geo. F. Bloomfield and Ernest H. Farmer. Brit. 433,082, Aug. 8, 1935. Org. hypochlorites, e. g., Et or Me hypochlorite, are added to rubber in soln. to obtain a product having some of the resilience of rubber and many of the properties of chlorinated rubber, e. g., low viscosity and chem. inertness. The process comprises (1) prep'n. of a dil. soln. of HClO by usual methods, (2) shaking this with half its vol. of an inert solvent, e. g., C<sub>6</sub>H<sub>6</sub>, xylene, CCl<sub>4</sub>, contg. 10-15% of alc., drying the solvent layer, e. g., with CaCl<sub>2</sub>, and adding to a rubber soln. and (3) concg. the soln. after at least 24 hrs. under reduced pressure, at preferably not exceeding 40°.

Chlorinated rubber. Terence N. Montgomery and Imperial Chemical Industries Ltd. Brit. 433,313, Aug. 13, 1935. Solid chlorinated rubber is produced directly by subjecting finely divided rubber in the absence of solvents simultaneously to an abrading action and to the action of Cl<sub>2</sub> gas. The fine powder of chlorinated rubber may be removed by sieving or air blowing or, in a continuous process, by the current of Cl.

**Films of chlorinated rubber.** Joseph G. Moore and Imperial Chemical Industries Ltd. Brit. 432,803, Aug. 2, 1935. In mfg. films by running a soln. of chlorinated rubber onto a supporting surface, evap. the solvent and stripping the film, the removal of the film is facilitated by precoating the surface with a substratum insol in the solvent for the chlorinated rubber but sol. in a liquid having no action on the chlorinated rubber. Glue, gelatin and Na silicate are suitable.

**Preserving chlorinated rubber.** Joseph G. Moore and Imperial Chemical Industries Ltd. Brit. 432,805, Aug. 10, 1935. Chlorinated rubber is preserved by adding an org. peroxide sol. therein, e. g., H<sub>2</sub> peroxide or hydroperoxide.

**Preserving rubber.** Albert M. Clifford (to Wingfoot Corp.). U. S. 2,020,291, Nov. 12. About 0.1-5.0% of a  $\beta$ -hydroxyphenyl naphthylamine or other suitable aryl naphthylamine having at least one hydroxy group substituted on an aromatic nucleus is added to a rubber compn. as a preservative.

**Preserving rubber.** The Rubber Service Laboratories Co. Ir. 785,783, Aug. 19, 1935. See Can. 131,115 (C. A. 29, 81059).

**Apparatus for plastifying crude rubber and mixtures thereof.** Società Italiana Pirelli. Ir. 785,420, Aug. 9, and 785,481, Aug. 10, 1935.

**Sponge rubber.** Charles I. Lemming (to Roth Rubber Co.). U. S. 2,019,489, Nov. 5. Rubber is mixed with a "blowing agent" such as NaHCO<sub>3</sub> or (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> and with a vulkanizing agent including an accelerator, the compn. is shaped without curing, and the shaped mass is submerged in a heated liquid bath such as water at a temp. (suitably about 100°) and for a time sufficient to vulkanize quickly the surface and then to blow and complete the vulkanization while retaining the desired shape of the product.

**Sponge like or cellular rubber products.** Wilfred H. Chapman, Eric W. B. Owen and Donald W. Pounder (to Dunlop Rubber Co. Ltd.). U. S. 2,020,094, Nov. 12. A foam is formed from an aq. rubber dispersion and the pressure around the foam is decreased so as to expand the cells, and the expanded material is vulkanized.

**Dipped rubber articles.** John R. Gimmeter. U. S. 2,021,289, Nov. 19. Various details of app. and operation.

**Molding hollow rubber dolls, toy animals, etc.** Iordyee C. Jones. Brit. 432,371, July 25, 1935.

**Rubber threads.** International Latex Processes Ltd. and Ugo Pestalozza. Ir. 785,012, July 31, 1935. An aq. dispersion of rubber is disposed in grooves and caused to solidify by heating.

**Cutting rubber threads from sheet rubber.** Dunlop Rubber Co. Ltd. and John Healey. Brit. 432,801, Aug. 2, 1935. The sheet is subjected to pressure adjacent the knife. The process is applicable to both the intermittent and continuous spiral cut methods in which the sheet is wrapped around a rotating drum.

**Jet box and associated apparatus for forming threads or filaments of rubber from aqueous dispersions.** Edward A. Murphy (to Dunlop Rubber Co. Ltd.). U. S. 2,019,543, Nov. 5. Structural and operative details.

**Apparatus for making rubber strip.** International Latex Processes Ltd. Ger. 619,266, Sept. 26, 1935 (Cl. 30a 10 03). See Brit. 400,147 (C. A. 28, 2210\*).

**Rubber conversion products.** I. G. Farbenind. A.-G. (Julius Söll and Albert Koch, inventors). Ger. 619,211, Sept. 25, 1935 (Cl. 30b. 3). Addn. to 615,050 (C. A. 29, 6471\*). Rubber is treated with H<sub>2</sub> under conditions which are mild in comparison with those recommended in Ger. 615,050. Thus, a benzene soln. of rubber may be warmed for 14 days with an ethereal soln. of H<sub>2</sub>, or rubber sheets may be kneaded with aq. H<sub>2</sub> and then heated in an autoclave to 100° for 16 hrs. The products, unlike those described in Ger. 615,050, are sol. in org. solvents, and their solns. may be used as leathers or as adhesives for uniting rubber sheets to metal surfaces.

**Carboy cushions of reclaimed rubber.** Richard W. Lahey and Harry A. Kast (to American Cyanamid & Chemical Corp.). U. S. 2,021,878, Nov. 19. Carboy cushions are formed of reclaimed rubber so as to have speci-

fied desirable and durable cushioning properties at temps. of about -18° to 34° (various details of manuf. being given).

**Rubber mat for running boards or the like.** William F. Schacht. U. S. 2,021,623, Nov. 19. A body portion of completely vulcanized rubber carrying a substantially unvulcanized facing contg. sufficient compounding ingredients such as S, etc., to prevent "creeping" of the mat on the board, the exposed surface of the facing being readily sol. by rubber solvent, and a protective layer covers the facing.

**Vehicle bodies.** Società Italiana Pirelli. Brit. 432,699, July 31, 1935. A vehicle body, or parts thereof, is constructed of a rigid stratified material composed of layers of hard rubber- or carbonite-treated fabric united by vulkanization.

**Metal coats on rubber.** Max Ow-Ischlungen. Brit. 432,242, July 23, 1935. See Austrian 140,200 (C. A. 29, 3190\*).

**Fireproofing rubber.** H. & L. de Poux & Cie. Fr. 785,108, Aug. 22, 1935. A product capable of fixing, under the effect of heat of combustion, on to the free bonds of rubber mols. so as to form an incombustible compd., and finally liberate gases or vapors which prevent combustion, is incorporated with rubber during its prepn. Comps. of Cl. I or preferably Br, e. g., NH<sub>4</sub>Br, may be used, the proportion of NH<sub>4</sub>Br for spongy rubber being 30-50%.

**Fireproofing rubber, etc.** John Latbot. Ir. 785,548, Aug. 12, 1935. Rubber, etc., is treated chemically by a neutral ester of an inorg. acid, e. g., triphenyl- or tri-*tert*-butyl-phosphate or triphenyl borate.

**Vulcanization accelerators.** Wingfoot Corp. Ir. 785,101, Aug. 2, 1935. Thiaryl esters of secondary aliphatic dithiocarbamic acids, made by causing a salt of the acid to react with a halogenated aryl thiazole, are used. Ex. samples are given of the prepn. and use of 5-nitrobenzothiazole-1-dicyclohexyl dithiocarbamate, m. 188.9°, and 5-nitrobenzothiazole-1-*N*-ethyl cyclohexyl dithiocarbamate, m. 146.7°.

**Furoyl-substituted mercaptobenzothiazoles (rubber vulkanization accelerators).** Sylvester M. Evans (to Rubber Service Laboratories Co.). U. S. 2,020,051, Nov. 5. A furoyl halide such as the chloride and a mercaptobenzothiazole are caused to react in an aq. medium contg. a small amount of an inert org. solvent such as C<sub>6</sub>H<sub>6</sub> at a temp. below the b. p. but above the freezing point of the solvent (suitably about 10-15°). Several examples are given.

**Vulcanizing rubber.** The B. F. Goodrich Co. Ir. 785,805, Aug. 20, 1935. Rubber is vulkanized in the presence of a thiuram sulfide in which 1 aryl radicals have been introduced, e. g., diphenyl-di- $\beta$ -naphthyl disulfide, m. 217°.

**Molding and vulkanizing ebonite articles.** Società Italiana Pirelli. Brit. 432,800, July 31, 1935. Glossy ebonite articles are produced by molding and vulkanizing a mixt. of crude ebonite stock in a specular surfaced mold, the operation being effected in stages by successively reducing the vol. of the mold to that of the finished article.

**Apparatus for vulkanizing inner tubes, etc.** Thomas H. Williams (to National Rubber Machinery Co.). U. S. 2,020,169, Nov. 12. Mech. features.

**Molding press suitable for vulkanizing rubber articles.** Henry C. Hostwick (to Akron Standard Mold Co.). U. S. 2,019,888, Nov. 5. Structural, mech. and operative details.

**Vulcanizing mold for rubber tubes.** Charles E. Maynard (to Fisk Rubber Corp.). U. S. 2,021,373, Nov. 19. Structural, mech. and operative details.

**Vulcanizing long lengths of material, etc.** Kooperativa Lörbundet, Lörerning U. P. A. Brit. 432,675, July 31, 1935. In the manuf. of floor or like covering material as slabs, long lengths or mats from vulkanizable material, this is advanced between and by means of 2 endless bands driven at the same or approx. the same speed to a vulkanizing app. to be shaped between the bands under pressure and at the temp. necessary for vulkanization; the bands are guided over drums, guide rollers and (or)

other guiding members bearing on the material, whereby the shaped material is cooled between the bands while the same or approx the same pressure is maintained on the material as that exerted during the vulcanization process and while the bands are guided over sep cooling drums, guide rollers or other cooled guiding members. App is described.

Vulcanizing long lengths electrically. Aktieselskapet den Norske Remfabrik Brit 432,943, Aug. 6, 1935. Rubber belts, etc., are vulcanized by wrapping them on a drum with an interposed preheated steel ribbon under controlled tension and maintaining the temp of the ribbon by the passage of an elec current App is described

1 Process for forming and vulcanizing marbled erasers and other rubber goods from a plurality of one-color rubber sheets Josef Anton Talalay Brit. 432,884, Aug. 6, 1935

2 Devulcanizing rubber. Charles H Campbell U S 2,021,046, Nov. 12 In devulcanizing ground vulcanized rubber scrap, an oil including essentially benzene, toluene, xylene and a heavy solvent such as a heavy naphtha solvent is introduced into a digester in the presence of a desulfurizing agent such as NaOH which upon the application of heat, moisture and pressure causes the rubber particles to swell and soften and thus permits the desulfurizing agent used to react upon the S present in the vulcanized rubber.



# CHEMICAL ABSTRACTS

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## I—APPARATUS, PLANT EQUIPMENT AND UNIT OPERATIONS

W. L. BADGER

Aluminum, a German material for construction of apparatus H. Röhring *Chem. Fabrik* 1935, 487 b.—A discussion of the suitability of Al for various app., of methods to increase its resistance to corrosion and to facilitate welding, with 7 cuts and 10 references.

Ebonite as a construction material in chemical industry Alfred Salmons-Karsten *Quim. e ind.* 12, 188-90 (1935).—Descriptive.

Usefulness of some Ukrainian granites for acid resisting construction A. I. Kogan and L. E. Shvartzburg *Khim. i tekhnol.* 7, 429-31 (1935). cf. *C. A.* 29, 5007.—Various tests of the granites from different quarries showed that they can be successfully used for acid-resisting construction in place of andesite.

The Dräger carbon monoxide filter III Gerhard Stampe and Otto Sommer *Dräger-Mitteil.* No. 180, 2987-9 (1935).—Improvements in the CO-gas mask filter are described.

New method of quantitative extraction A. Kulman, *Mashinnoye Zhivoe Delo* 11, 498-502 (1935), cf. *Pishch. chernaya Prom.* No. 8 (1929).—Several lab. app. for extrn. of solids are illustrated and described.

Removing glass stoppers F. Bentley. *Pharm. J.* 135, 237 (1935).—A single or repeated application of a mix. of chloral hydrate 10, glycerol 5,  $H_2O$  5 and HCl (25%) 3 g. is recommended.

A modern dust collector A study of the principles governing its design and operation Herman van Tongeren. *Mech. Eng.* 57, 753-9 (1935).

A simple thermoregulator. H. P. Bloxam. *J. Sci. Instruments* 22, 361-3 (1935).—A portable and a built-in bimetallic type of app. are described, with 2 cuts. They are suitable for temps. up to 300° and are accurate to  $\pm 2-3^\circ$  in long runs, but closer control is possible in short runs.

Kjeldahl flasks and other vessels from arsenic free glass Georg Lockemann *Z. anal. Chem.* 103, 81-2 (1935); cf. *C. A.* 29, 2990, 3767.—A new glass made by the Jena Glaswerke Schott u. Gen. and marked "arsenic-free" has proved suitable for all digestions with  $H_2SO_4$  or solns. of bases without danger of getting appreciable As into soln. The new glass, because no  $As_2O_3$  was used in its manuf., contains air bubbles but is not fragile and no trouble results from heating over a free flame.

An ionization gage for the detection of molecular rays M. J. Copley, T. E. Phipps and Julian Glasser. *Rev. Sci. Instruments* 6, 371 (1935).

Wetted surface in ring-packed towers F. Mayo, T. G. Hunter and A. W. Nash. *J. Soc. Chem. Ind.* 54, 375-837 (1935).—The degree of wetting of rings made of paper ( $1/2$ " and 1" size) was detd. by estg. the area colored when a dye soln. was run over the packing; the wetting of the tower wall (2", 3" and 6" diams.) was similarly detd. The liquid flow was found to tend toward the walls in such towers. The wetting increased with increasing liquid flow, but was not affected by gas velocity (up to the flooding point). The liquid film thickness was estd. to be independent of the liquor rate. Cf. Baker, Chilton and Vernon, *C. A.* 29, 3877.

Crystallization of melts. III Julius Meyer and Willy Pfaff *Z. anorg. allgem. Chem.* 224, 305-14 (1935); cf. *C. A.* 29, 5727.—The crystn. of supercooled liquids

depends upon the no. of crystn. seeds and crystn. velocity. This is studied for  $H_2O$  and single crystals of ice are obtained by crystn. at about  $-29^\circ$ . Benzene is studied in a similar manner.

Superiority of a Knauden type vacuum gage for large metal systems with organic vapor pumps, its design and operation Jesse W. M. DuMond and W. M. Pickels, Jr. *Rev. Sci. Instruments* 6, 362-70 (1935).

Bureau of Mines multiple-diaphragm recording sub-surface-pressure gage W. B. Berwald, H. A. Buss and C. E. Kestle, Jr. *Bur. Mines, Rept. of Investigations* No. 3291, 19 pp (1935).

New device for the toxicological determination of arsenic E. Kohn-Abrest. *Ann. f. d. 28*, 537-9 (1935).—The app. includes various simplifications but its chief feature resides in the use of a quartz tube heated electrically to  $850^\circ$  (by means of a Lhomme and Darcy element) which ensures complete decompn. of the  $AsH_3$  and formation of remarkably uniform rings. As little as 0.01 mg. As can be detected, and formation of the ring is complete in 30 min. A series of standard rings can be formed on the same tube by successively displacing the heating element along the quartz tube. The rings can be identified by dissolving in  $HNO_3$ , evapng the  $HNO_3$  completely and treating for 10 min. on the water bath with a HCl soln. of Na hypophosphite.

Drying methyl chloride E. W. McGovern. *Power* 79, 579-80 (1935).—Water in  $MeCl$  in a refrigeration system causes freezing of lines and corrosion. The system can be dried by flushing with  $MeOH$ , blowing out with dry air or  $MeCl$  gas, followed by chem. drying after charging. The drying agent should be coarse granules and used with an efficient filter such as felt or mineral wool to prevent passage of finely divided or disintegrated material into the refrigeration system. It is not practical to reactivate any of the drying agents; contamination by oil makes reactivation impractical.  $CaCl_2$  has been used efficiently, but it induces corrosion especially in the expansion valve; it should not be allowed to absorb more than 16.2% by wt. of  $H_2O$ .  $CaO$  is satisfactory; its max.  $H_2O$ -absorbing capacity is 32.1%; it prevents corrosion by neutralizing acid, its only disadvantage is its tendency to disintegrate on hydrating. Soda lime and activated  $Al_2O_3$  are satisfactory.  $Mg(ClO)_2$  or  $Ba(ClO)_2$  should not be used.

The drying of solids A review of plant and theory. Alfred H. Loveless. *Ind. Chem.* 11, 427-33 (1935).

Heat transfer coefficients on inclined tubes. D. F. Jurgensen, Jr., and G. H. Montillon. *Ind. Eng. Chem.* 27, 1466-75 (1935).—An extn. single-tube condenser was used to study liquid and condensing film coeffs. The app. was arranged to permit ready change of angle with the horizontal. There are significant differences in liquid film coeffs. for liquid flowing upward and liquid flowing downward through the heating tube when the value of Reynold's no. was below about 20,000. For higher values the differences are not noticeable. This is ascribed to the lessening of the natural convection effect as the liquid velocity is increased by forces other than convection. The results are shown graphically. Steam-film coeffs. obtained by several investigators have been correlated with that of the authors and included in a comparison with theoretical coeffs. predicted from the equation of Parr

(*The Engineer* 131, 559(1921)). Deviations from theoretical become larger as the angle of inclination approaches 90° M. C. Rogers

Velocity of bubbles in vertical tubes Morrough P. O'Brien and James E. Gosline. *Ind. Eng. Chem.* 27, 1436-40(1935)—Information about flow of bubbles through liquids in vertical tubes is necessary for consideration of gas-oil or air water lifts. Exptl work has been carried out in glass tubes of 1.18, 2.24 and 6.0 in internal diam and lengths from 5 to 26 ft. Water and two colorless petroleum oils were used. Bubbles were of sizes above those for which Stokes' law is valid. When compared with other work these results show fair agreement for bubbles of radius of about 3 mm. The disagreement for bubbles of smaller sizes is attributed to a condition similar to that of the transition from laminar to turbulent flow in pipes. When the bubble radius is increased above this value, the tube walls have a noticeable effect. In general, the increase in size of bubbles increases their rates of flow until a certain max is reached, beyond which no change in size will produce an increase in velocity. The data obtained indicate that the Gibson (*Phil. Mag.* [6], 26, 932-65(1913)) formula for this limiting velocity is high for small tubes and low for large tubes M. C. Rogers

Experiments on the vertical flow of gas-liquid mixtures in glass pipes J. E. Gosline *Am Inst Mining Met Engrs Contribution No* 90, 15 pp (1935). A H. E.

Flow of water in piping studied with movie camera Edgar C. Ambrosius and John C. Reed. *Heating, Piping, Air Conditioning* 7, 578-80(1935)—A thin flat sheet of very intense light was projected through the horizontal diametral plane of a glass pipe. The light was reflected from small globules of an insol mixt having the same d as that of the H<sub>2</sub>O M. W. Schwartz

Further notes on power used in crushing John S. Owens *Bull Inst Mining Met* No 357, 1-21(1935); cf C. A. 29, 3950—Discussion Alden H. Emery

A new type of rotary furnace for fusion methods F. J. Kohlmeier *Mettall u Erz* 32, 511-19, 543(1935).

Fume exhaust systems B. F. Postman *Heating, Piping, Air Conditioning* 7, 572-4(1935) M. W. S.

Thermostat Mme S. Lallemand *Bull soc chim biol* 17, 1470-1(1935)—A thermostat intended to operate at a temp below room temp, c. g., 18°, is supplied with a slow stream of water from a source colder than the temp to be maintained. Before entering the bath the water passes through a small chamber where it is warmed by an immersed lamp bulb which is switched on and off by the thermoregulator. One cut L. E. Gilson

Apparatus for carrying out chemical gas reactions Metalgesellschaft A-G (Ludolf Plass and Walther Deutsch, inventors) *Ger* 617,878, Aug 27, 1935 (Cl 124 4). The reactions are carried out by the aid of an elec. field.

Fractionations in column stills. Gustav A. Kramer (to Shell Development Co.) U. S. 2,022,803, Dec. 3 For the automatic control of a fractionating column to obtain a product of predetd purity and definite const. compn, vapor components are sep'd from liquid within the column and the vapor and liquid are separately withdrawn from the column, the withdrawn vapors are substantially totally condensed and a portion of the condensate is returned to the column as reflux in a controlled amt. in accordance with the temp. difference gradient of the high part of the column. App. is described.

Still Coyac fils & Cie *Fr.* 788,630, Sept. 6, 1935. Openings are made at the bends of the coil for cleaning, the coil being in the form of a series of straight tubes joined at alternate ends.

Balance with a weighing chain Robert G. Ainsworth and Alfred W. Ainsworth (to William Ainsworth & Sons, Inc.) U. S. 2,022,306, Nov. 20 Structural details.

Apparatus for sampling heaped materials Franz Schröder (Julius Hillmann, inventor) *Ger* 620,425, Oct. 21, 1935 (Cl. 422, 17).

Cooling-towers Stewart C. Coey. *Brit.* 433,113, Aug 8, 1935

Liquid treating apparatus The Pfaudder Co. *Brit* 433,559, Aug 16, 1935 The app comprises a tank having an opening in its wall for an agitator shaft which is supported by means of bearings, adjustable transversely of the tank opening to align the shaft therewith, and also by means of a bushing, adjustable transversely of the shaft to align the bushing with the shaft.

Apparatus for homogenizing liquids John A. Clark (to Mantion-Gaulin Mfg Co.) U. S. 2,023,043, Dec. 3 Various structural, mech. and operative details.

Arrangement for indicating liquid levels Electrical Improvements Ltd and Charles L. Blackburn *Brit* 433,123, Aug 9, 1935

Electrical indicating instrument suitable for showing liquid levels in tanks Theodore J. Smulski (to Anderson Co.) U. S. 2,022,441, Nov. 20 Various elec., structural and operative details.

Portable tank valve suitable for use with combustible or other liquids or vapors under pressure. Harrison V. Snodgrass U. S. 2,021,969, Nov. 20 Various structural details.

Liquid seal for apparatus such as chemical mixing and agitating apparatus Fred W. Paffen (to Celanese Corp of America) U. S. 2,022,384, Nov. 20 Structural details.

Apparatus for proportioned supply of reagents as in supplying chemicals to water mains Henry J. Savage U. S. 2,023,532, Dec. 10 Structural and operative details.

Vacuum-jacketed vessels Jesse T. Littleton (to Corning Glass Works) *Brit* 433,588, Aug. 16, 1935. A vessel of glass is formed of sep. walls, the glass of the inner wall having a lower coeff. of expansion than that of the outer wall, whereby stresses induced by the annealing operation after the mouths have been joined by fusion are counteracted by the evacuation of the jacket. Cl. C. A. 29, 3489.

Self-starting siphoning device suitable for use with bottles Theodore J. Simpson U. S. 2,022,927, Dec. 3 Structural details.

Viscometer with a diaphragm and friction tube, etc. Moses E. Cheney and Aleck W. Meston (to Moto Meter Gauge & Equipment Corp.) U. S. 2,023,309, Dec. 3 Various structural details.

Electric relay system for controlling automatically the composition of baths to which reagents are continuously fed Siemens-Schuckertwerke A-G *Ger* 620,325, Oct. 19, 1935 (Cl. 12f. 4).

Temperature regulators Karl Baumann and Associated Electrical Industries Ltd. *Brit* 433,149, Aug. 9, 1935. In a fluid heater, e.g., steam, superheater, the temp. at the outlet is automatically controlled by a control member at the outlet and by a control member at an intermediate point in the fluid path, the effect of the 1st member being 3 times as great as that of the latter for equal changes in the temp. of the 2 members.

Thermocouple pyrometer John L. Orchard (to Cambridge Instrument Co., Ltd.) U. S. 2,022,515, Nov. 20 Structural details.

Air-filtering and -conditioning apparatus Charles P. Erschen and Richard C. Jeperington (to Air Conditioning Equipment Corp.) U. S. 2,022,463, Nov. 20 Various structural and operative details.

Apparatus for filtering and conditioning air. John W. Manley. *Brit* 434,674, Sept. 6, 1935

Filter for arsenics, toxic fumes, etc. Riccardo Spasiani *Fr.* 788,646, Sept. 6, 1935. A filter suitable for gas masks contains feathers, down or finely cut natural sponge in combination with microscopical grains of amber, lacquer or resins.

Rotary-filter apparatus for cleaning, moistening and cooling air. Eugen Steiner *Ger* 620,409, Oct. 22, 1935 (Cl. 36d, 1.30).

Rotary leaf apparatus suitable for filtering materials under pressure Wm L. Chapman (to Vallec Rotary

Filters Co.). U. S. 2,022,403, Nov. 26. Various structural, mech. and operative details.

Rotating filter for liquids. Schlichtermann & Kremer-Baum A.-G. fur Aufbereitung. Ger. 617,783, Aug. 20, 1935 (Cl. 12d. 15 02).

Apparatus with sponge-packed cells suitable for filtering liquids. Manolo Cordoro Santiago. U. S. 2,022,521, Nov. 26. Various structural and operative details.

Filtering device for separating solids from liquids. Mag. Muehlenbau und Industrie A.-G. Ger. 617,970, Aug. 30, 1935 (Cl. 12d. 13).

Apparatus for filtering rapidly settling sludges. Maschinenfabrik Buckau R. Wolf A.-G. Ger. 617,977, Sept. 18, 1935 (Cl. 12d. 30).

Funnel for filtering precipitates, etc., from liquids. Jenaer Glaswerk Schott & Gen. Ger. 617,783, Aug. 27, 1935 (Cl. 12d. 21). The funnel has a capillary delivery tube and three raised portions on its inner face spaced at 120° from each other.

Filter press. Herbert T. Leo. U. S. 2,022,670, Dec. 3.

Filter leaf. Wm. H. Bachelder (to Standard Oil Co. of Ind.). U. S. 2,023,010, Dec. 10.

Fractionating columns. Henry W. Howes. Brit. 433,770, Aug. 20, 1935. A Young and Thomas fractionating column comprises a top section, a bottom section and 1 or more standard center sections, adjacent sections being connected by interchangeable ground joints.

Centrifugal separators. Ange Nyrop and Koefoed, Hauberg, Marstrand & Helweg, Aktieselskabet Titan. Brit. 431,203, Aug. 28, 1935. The cream of a sap, e. g., rubber latex, is produced in a separator where the relation of the feed of the sap to the discharge of the heavy slum can be regulated and held.

Centrifugal separators. Gutehoffnungshütte Oberhausen A.-G. Brit. 434,693, Sept. 6, 1935. Liquid from wet or sludgy material in a centrifugal separator is discharged through a hollow body tapering toward the drum wall and the peripheral opening.

Centrifugal separators having tightly connected inlets and outlets and with apparatus, such as pumps, for raising the pressure, provided at the inlet and outlets. Aktiebolaget Separator. Brit. 434,006, Aug. 23, 1935.

Arrangement for the closed supply of liquid to a centrifugal separator through the hollow spindle of the bowl. Aktiebolaget Separator. Brit. 434,008, Aug. 23, 1935.

Centrifugal bowls. Aktiebolaget Separator-Nobel. Brit. 433,608, Aug. 19, 1935.

Centrifugal bowls for separating heavy sludge and solids from lighter liquids. Geo. J. Strezyński (to De Laval Separator Co.). U. S. 2,022,814-15-16-17, Dec. 3. Various details of app. suitable for sepg. foets and wax from oil, etc.

Magnetic separators. Electromagnets Ltd and Wm. E. Box. Brit. 433,615, Aug. 9, 1935. Supported above electromagnets mounted on a base plate secured to a frame which is pivoted at its upper end to standards and bolted at its lower end to standards is a trough provided with transversely arranged armatures which cooperate with the pole pieces of the magnets. The trough is oscillated transversely by eccentrics.

Magnetic separators. Electromagnets Ltd and Wm. E. Box. Brit. 433,616, Aug. 9, 1935. A rotary magnetic separator drum comprises a central core having radial arms forming the cores of electromagnets and being of greater thickness than intermediate arms, the windings for the electromagnets being arranged in the spaces between the arms.

Magnetic separator. Robert C. Forrer and Mines domaniales de potasse d'Alsace. Ger. 620,391, Oct. 21, 1935 (Cl. 15. 3). Addn. to 602,170 (C. A. 29, 3869).

Magnetic separators of the trough or tray type. Electromagnets Ltd and Wm. E. Box. Brit. 433,458, Aug. 9, 1935. Inclined flaps or baffles of para- or dia-magnetic materials are arranged above pole pieces of electromagnets so as to intercept pieces of material that may ricochet from 1 of the pole pieces.

Pneumatic separators. Albin B. Helbig and Victor B. Reichwald. Brit. 434,467, Sept. 2, 1935.

Apparatus for separating mixtures of liquids of different specific weights. I. G. Farbenind. A.-G. Fr. 786,930, Sept. 14, 1935.

Apparatus for sitting or straining materials suspended in liquid, such as wood pulp. Walther Voith, Hermann Voith and Hanns Voith (trading as a firm of) J. M. Voith). Brit. 434,000, Aug. 26, 1935.

Apparatus for mixing gases such as butane and air, etc. John K. Heller (to Utility Development Co.). U. S. 2,023,809, Dec. 10. Various structural and operative details.

Gas-analysis apparatus. Oliver W. Johnson and Philip S. Williams. U. S. 2,023,731, Dec. 10. Gas such as that contg. hydrocarbons is passed through an app. contg. combustion filaments. Various structural and operative details are described.

Apparatus for gas analysis. The Asiatic Petroleum Co. Ltd and John K. Joyce. Brit. 434,970, Aug. 23, 1935.

The app. comprises a chamber contg. a solid reagent for removing a component from the gas, internally actuated means for passing a given vol. of gas through the chamber, and a vacuum gage for measuring the reduction in pressure.

Centrifugal gas-cleaners. Charles H. W. Cheltnam and Cyril H. Cheltnam. Brit. 433,647, Aug. 19, 1935. To prevent turbulence in a separator of the cyclone type having a tangential entry, a rotary motion is imparted to the dust-laden gas before its entry so that it rotates in the same direction as the vortex ring it joins.

Method and means for gas scrubbing. Dana W. Flowers. Brit. 433,977, Aug. 23, 1935. In treating gas with liquid, the gas is passed through a radially bladed high-speed centrifugal blower through the hub of which liquid is introduced so as to be projected in sheet form along the blade surfaces and to traverse a space between the blade ends and the blower wall in the form of large drops.

Method and apparatus for conditioning gases and vapors. Babcock & Wilcox Co. Brit. 431,457, Sept. 2, 1935. Gas is passed into contact with a no. of perforate ring or wall-like masses presenting progressively decreasing flow areas to the gas, films of liquid being formed on the solid surfaces of the masses and flowing down to the lower level of each mass, whence accretions of liquid are removed.

Continuous system for adsorbing gases or vapors and regenerating the adsorbent. Carbo-Norit-Union-Verwaltungs-G m b H. Ger. 620,388, Oct. 21, 1935 (Cl. 12c. 3 02).

Apparatus for the physical or chemical wet treatment of gases. Jules M. Revy. Fr. 786,804, Sept. 11, 1935.

Drying agents for gases. Soc. de recherches et d'exploitations pétrolières. Ger. 620,397, Oct. 21, 1935 (Cl. 12c. 3 01). See Fr. 700,511 (C. A. 25, 3781).

System for fractionating natural gas and other gas mixtures by liquefaction. Ges. fur Linde's Eismischungen A.-G. Ger. 620,077, Oct. 14, 1933 (Cl. 17c. 2 02).

Effecting reactions between solids and gases such as pyrites and sulfur chloride. Ernest W. Wescott (to Sulphide Corp.). U. S. 2,023,942, Dec. 10. A comminuted solid material is fed into one end of a rotating horizontal drum contg. a loosely packed body of pebbles and is treated countercurrentwise with a reactive gas. Various details of app. are described.

Apparatus for drying volatile solids. Hermann Sandkuhl. Ger. 620,227, Oct. 17, 1935 (Cl. 12a. 6). The app. comprises a closed inclined tube which is heated in sections and up which the solid is moved. A condenser is attached to the lower end of the tube. Specified applications of the app. include (1) the recovery of isoborneol from a crude product contg. camphene 4 and water 10%, (2) treatment of salicylic acid, after it has been crystd. from alc., to recover dry acid and alc., (3) the drying of boric acid.

Apparatus for aerating liquids. J. Stone & Co. Ltd and Donald Elder. Brit. 433,857, Aug. 19, 1935. The app. comprises a tower in which the liquid is aerated, a supplementary chamber to which the liquid is supplied under pressure and in which the liquid collects under pressure.

sure of a gas cushion, and spraying or atomizing devices connecting the chamber and tower and to which gas is supplied under pressure

**Rotary and bling furnaces** Metallgesellschaft A-G Brit 433,669, Aug 19, 1935 A rotary or rocking furnace, particularly for refining Cu, is mounted on rollers secured to a turntable so that it can be rotated at least 180° about a vertical axis to enable it to be charged and (or) fired at both ends

**Furnace (with conveyor rollers)** John Fallon U.S. 2,021,913, Nov 26 Structural and mech. features of a "roller hearth" type furnace are described

**Fusion furnaces** Akt-Ges Brown, Boveri & Cie Fr 788,983, Sept 14, 1935 Method of charging is described

**Muffle furnace** Gibbons Brothers Ltd and Thomas E Bridgford Ger 620,090, Oct 14, 1935 (Cl 24c 9). This corresponds to Brit 384,788 (C. A. 27, 5588)

**Apparatus for regulated combustion of atomized oil in furnaces** Aldo Macchi (to General Elec Co.). U.S. 2,022,512, Nov 26 Various structural and operative details U.S. 2,022,513 also relates to oil-burning app

**Furnace floors** Rolfe Shellenberger (to Babcock & Wilcox Co Ltd). Brit 431,518, Sept 3, 1935 In a furnace adapted to retain at the bottom a pool of molten slag, the furnace floor comprises metallic blocks secured to spaced cooling tubes and means positioned externally of the blocks and co-operating with the tubes to form narrow slag-solidifying passages communicating with the joints between the blocks

**Furnace wall cooled by tubes through which water or mercury vapor, etc., may be passed** Joseph S Bennett (to American Engineering Co.) U.S. 2,023,302, Dec 3 Various structural details

**Furnace-charging truck** Edgar E Brosius Ger 620,408, Oct 21, 1935 (Cl 18c 11.20) See Brit 370,764 (C. A. 27, 2353)

**Conveyor rolls for charging and discharging annealing furnaces** Siemens-Schuckertwerke A-G (Johann Schnepf, Otto Gunther, and Alois Schmitt, inventors). Ger 620,452, Oct 21, 1935 (Cl 18c 11.20)

**Apparatus for removing dust from furnace gases** Axel Larsen Fr 787,028, Sept 16, 1935

**Plate heat-exchangers** Søren Jørgen Jensen Brit 433,604, Aug 7, 1935

**Tubular heat-exchange apparatus suitable for water-tube boilers or heaters** Henri A Richard U.S. 2,023,646, Dec 10 Structural features

**Concentric-tube heat exchangers** Michael Fröh Brit 433,847, Aug 21, 1935

**Straight-tube heat-exchangers** Thomas G. Thomas. Brit 433,743, Aug 13, 1935

**U-tube heat exchangers** Wm Y. Lewis Brit. 474,023, Aug 21, 1935

**Heat transfer apparatus suitable for heating air by furnace gases** Ali Lysholm (to Aktiebolaget Ljungströms Angturbin). U.S. 2,023,965, Dec. 10 Various structural details

**Air-conditioning apparatus** Edward W. Roessler (to General Elec Co.) U.S. 2,022,523, Nov. 26. Structural and operative details.

**Air-conditioning apparatus** Ira W. Rowell. U.S. 2,022,740, Dec 3 Various structural and operative details

**Air-conditioning apparatus** Leonard C Smith (to Heating Ventilating & Air Conditioning Co.). U.S. 2,023,492, Dec 10 Various structural and operative details

**Air-conditioning apparatus** Leopold Lithman U.S. 2,023,602, Dec 10. Various structural and operative details

**Air-conditioning apparatus** John H Textorius and Joseph F. Broderick. U.S. 2,023,623, Dec 10 Various structural and operative details

**Air heating and -conditioning apparatus** Geo Herberster (to Cleveland Cooperative Stove Co.). U.S. 2,023,130, Dec 3 Various structural features

**Air heating and -conditioning apparatus** Clarence M

Woolley (to American Radiator Co.). U.S. 2,022,332-3, Nov 26 Structural, mech and operative details  
**Apparatus for humidifying air** Wm Feldermann U.S. 2,022,415, Nov. 26 Various structural, mech and operative details.

**Apparatus for humidifying air** Albert J. Loepsinger (to American Moistening Co.). U.S. 2,022,680, Dec 3 Structural and operative details

**Apparatus and process for drying and conditioning air** for supply to a living room, or for drying purposes or for the evaporative cooling of water Edmund Altenkirch Brit 433,639, Aug 19, 1935

**Apparatus for purifying and regulating the humidity of air** Ludwig Matthaei and Josef O Zeuzern. Fr. 786,647, Sept 6, 1935 App is described in which air is treated with short-wave rays such as ultraviolet rays and with a pulverized liquid Means is provided for cooling the air

**Humidifier for air suitable for use with warm-air furnaces** Pierce A. Weyl. U.S. 2,022,394, Nov. 26 Structural and operative details.

**Apparatus for incorporating a liquid with a gas as in air conditioning** Pierre G Vicard U.S. 2,023,061, Dec 3 Structural and operative details are described of an app having a rotary device carrying a series of blades with sharp cutting edges

**Apparatus for heating, cooling, drying or moistening air** Frigidaire Ltd and Arthur T. Hawkins Brit. 434,281, Aug 29, 1935

**Valve and method for releasing the pressure on mixtures containing liquid and solid substances under high pressure, such as occurs in autoclaves, etc., where the parts are subjected to considerable corrosion and abrasion** Friedrich Uhde and Kurt Bredtschneider. Brit 433,496, Aug 16, 1935

**Apparatus for making dispersions** Wm. H Ashton Brit 433,583, Aug 16, 1935 Dispersions of 2 or more substances are made by subjecting them to compressional wave vibrations of sonic frequencies, e.g., 8000 cycles, and sufficient amplitude to cause cavitation in the mixt. App is described

**Apparatus for cleaning liquid conduits such as beer pipes by use of cleansing solutions** Hans M Erdmann (to Saronia, Inc.). U.S. 2,022,882, Dec 3 Various structural and operative details

**Apparatus for treating molten material as with fluids in a multi-chambered ladle** Oskar Blume U.S. 2,022,755, Dec 3 Numerous structural, mech and operative details

**Apparatus for drying and grinding materials such as blood or fruit pulps** Wm. B Senseman (to Raymond Bros Impact Pulverizer Co.). U.S. 2,023,247, Dec 3 Structural, mech and operative details employing heated gases

**Pneumatic conveyor for transferring material in bulk from a hopper through a pipe by means of an air jet** Eusebe J L F Constantin Brit. 434,408, Aug. 30, 1935

**Apparatus for condensing mercury vapor** Joseph F Tapie U.S. 2,021,903, Nov 26 Structural and operative details are described of an app cooled by water and subjected to a partial vacuum

**Mixing or grinding rolls, with heating or cooling means** Gebrüder Buhler. Ger. 620,040, Nov 7, 1935 (Cl 50b 4) Structural features are described

**Crushing mills** Thomas A Long and Wm W Potts Brit 433,765, Aug 20, 1935

**Rotary-drum ball or rod mill** Carl G H Löwenhielm U.S. 2,023,131, Dec 3 Structural and mech details

**Control of the combustion of oil atomized with steam** John J Harman (to Walworth Patents Inc.). U.S. 2,023,074, Dec 3 Various details of app and operation

**Apparatus for treatment of substantially immiscible liquids as in treating oil fractions with sulfuric acid** Gustav A Kramer (to Shell Development Co.) U.S. 2,022,205, Nov 26 Various structural and operative details

**Electric system for remote control of pressure such as that in gas mains** Philip B R. Baas (to Brooklyn

Borough Gas Co.). U. S. 2,023,503, Dec. 10. Various elec. and operative details.

Apparatus for testing the permeability of porous materials such as paper by air suction. Rudolf Schopper. U. S. 2,021,948, Nov. 26. Various structural and operative details.

Liquid seal and vent for tanks, etc., containing volatile solvents such as those used for "dry cleaning." John Mahoney (to American Laundry Machinery Co.) U. S. 2,023,634, Dec. 10. Details are described of an app. which automatically drains condensate back to a tank

or the like after a predetd. amount of the condensate has collected when formed from escaped vapors.

Apparatus for producing solid carbon dioxide. Harlon A. Bullock. U. S. 2,023,703, Dec. 3. Structural and operative details.

Liquefier suitable for use with solid carbon dioxide. Frank C. Seck. U. S. 2,021,997, Nov. 26. Various structural details.

Apparatus for separating oil from refrigerants such as sulfur dioxide. Albert A. Ralston (to General Elec. Co.). U. S. 2,023,241, Dec. 3. Structural details.

## 2—GENERAL AND PHYSICAL CHEMISTRY

FREDERICK L. BROWNE

Niels Bohr's fiftieth birthday W. Heisenberg *Naturwissenschaften* 23, 679-81(1935) B. J. C. v. d. H.

Adolf v. Baeyer, the hundredth anniversary of his birth Friedrich Richter *Ber* 68A, 175-80(1935) G. G.

To the memory of George v. Georgievics Eduard J. Juchacz *Meliland Textilber* 16, 777-8(1935) C. H.

Alexis Vasilievitch Saposchnukoff H. Brunswick Z. ges. Schess-Sprengstoffw. 30, 329-32(1935) —Obituary C. G. Storm

P. J. Tarbouniech, 1871-1935 A. Astruc *J. pharm. chim.* 21, 479-81(1935) —Obituary S. Waldbott

Dr. Hans Tropsch Anon. *Mon. pitrole roumain* 36, No. 21, 1581(1935) —Obituary with portrait J. R. S.

A sixteenth-century German treatise, von Stahel und Eysen, translated with explanatory notes Hermann W. Williams, Jr. *Tech. Studies Field Fine Arts* 4, 63-92(1935) E. H.

Organization of secondary-school chemistry according to unitarian principles Robert P. Wray *Science Education* 19, 142-9(1935) W. H. Boynton

Major aims in science teaching W. C. Croxson *Science Education* 19, 149-52(1935) W. H. Boynton

Criteria for the selection of science teaching materials Cyrus W. Barnes *Science Education* 19, 152-7(1935) W. H. Boynton

Orientation in chemistry. L. F. Bate *Science Education* 19, 170-2(1935) W. H. Boynton

The approach to the absolute zero of temperature F. Simon. *Sci. Monthly* 1935, 530-41 —See C. A. 29, 2795<sup>4</sup> E. H.

The atomic weight of hydrogen E. Moles *Annales soc. chim. phys. chim.* 33, 721-8(1935) —The value 1.00778, derived by M. in 1925, according to recent results of investigations of isotopes, corresponds not to normal H, but to H electrolytically produced H low in deuterium.

If normal H is regarded as the isotope mixt. in ordinary, pure, distd. water, the ratio is D/H 1.5550 = 110, according to recent detns. by others. However, almost simultaneously and independently, Bethe (C. A. 29, 3908<sup>9</sup>) and Oliphant, Kempton and Rutherford (C. A. 29, 4668<sup>9</sup>) concluded that for B and Be nuclei, in comparison with mass spectroscopic detns., the mass ratio He/O = 3 × 10<sup>-4</sup> (approx.) is not true, and consequently in both publications the isotope mass of the light elements must be recalcd. on O = 16. H then = 1.0081, D = 2.0142. Recently Aston, by new, mass-spectroscopic detns., found H = 1.0081, D = 2.0148. From these concordant results, and with the above D:H ratio, the phys. at. wt. of normal H is 1.00828, which in a chem. conversion scale corresponds to a factor of 1.00022, H = 1.00906 = 0.00035. This value, considerably higher than that assumed today, requires a chem. revision, now in progress. The old literature value, about corresponding to normal H, leads to a higher value. E. M. S.

Revision of the atomic weight of germanium I. Analysis of germanium tetrabromide. O. Hönigsmid, K. Wintersberger and F. Wittner. *Z. anorg. allgem. Chem.* 225, 81-9(1935) —GeBr<sub>4</sub> was prepd. from spectroscopically pure Ge and fractionated in a high vacuum in an app. previously described (C. A. 28, 7085<sup>7</sup>). Detn. of

the ratio GeBr<sub>4</sub>:4Ag:4AgBr gave Ge = 72.59

L. R. Rushton

Revision of the atomic weight of tantalum. Analysis of tantalum pentachloride O. Hönigsmid and R. Schlee *Z. anorg. allgem. Chem.* 225, 64-8(1935); cf. C. A. 29, 2037<sup>1</sup> —Tantalite was extd. with strong HCl to remove most of the Fe, then dissolved in HF, K<sub>2</sub>TaF<sub>7</sub> prepd. and recrystd. 5 times. Ta<sub>2</sub>O<sub>5</sub> was then pptd. and ignited. Spark and x-ray spectra showed that it was free of Co, Th, Zr, etc., but contained Fe. It was fused with soda, dissolved in H<sub>2</sub>O and pptd. with (NH<sub>4</sub>)<sub>2</sub>S. TaCl<sub>5</sub> was prepd. and distd. 3 times in Cl<sub>2</sub>, then sublimed *in vacuo*. The pure TaCl<sub>5</sub> was dissolved in EtOH and pptd. with NH<sub>4</sub>OH. The ratio TaCl<sub>5</sub>:5Ag was detd. in the soln. in which the ppt. remained suspended. The av. of 6 detns. gave Ta = 180.88 ± 0.01. E. R. Rushton

The group relation between the Mulliken and Slater-Pauling theories of valence J. H. Van Vleck. *J. Chem. Physics* 3, 803-6(1935) —By means of the group theory of characters, an intimate relation is shown between Mulliken's (I) mol. orbitals and the Slater-Pauling (II) directed wave functions. A simple transformation from an irreducible to a reducible representation makes possible a transition from (I) to (II). Consequently the same formal valence rules are usually given by either method, and wave functions of the central atom which are nonbonding in Mulliken's procedure are likewise never employed in constructing Pauling's "hybridized" linear combinations. G. M. P.

A summary of experimental activation energies of elementary reactions between hydrogen and the halogens. J. Carrell Morris and Robert N. Pease. *J. Chem. Physics* 3, 796-802(1935) —Exptl. data on reactions between H and the halogens are analyzed to give values of the activation energies of the intermediate reactions involved. These are compared with values calcd. by Eyring and Wheeler. G. M. P.

Effect of thermal agitation on atomic arrangement in alloys. H. W. L. Bragg and E. J. Williams. *Proc. Roy. Soc. (London)* A151, 540-66(1935); cf. C. A. 28, 5749<sup>4</sup> —Treatment and discussion of the previous paper are compared with the treatments of Bethe, Borelius, Gorsky and Dehlinger. Changes in internal energy due to transformation in the alloys CuZn and Cu<sub>3</sub>Au were estd. by sp.-heat measurements. These measurements are compared with theoretical results. Expressions are given for the dependence of crit. temp. upon compn. C. B. Jenni

Effect of thermal agitation on atomic arrangement in alloys III. C. J. Williams. *Proc. Roy. Soc. (London)* A152, 231-52(1935), cf. preceding abstr. —The equl. equation for the general case is derived in detail from the standpoint of the Boltzmann distribution formula and from that of the principle of min. free energy. This assumes that the energy assocd. with the at. arrangement is uniquely detd. by the superlattice order. The relation between entropy and the internal energy is developed. When f(S) is plotted against S, curves are obtained from which the existence of stable and metastable arrangements can be predicted. The entropy and energy corresponding to the solution given by Bethe are discussed (it is assumed

the energy of the at. arrangement is detd by the order of nearest neighbors), and a method of estg the quant. relation of Bethe's treatment to that of Bragg and Williams is given. With  $\text{CuZn}$  as an example it is shown that both types of treatment lead to very nearly the same result, which is in accord with exptl observations.

Howard A. Smith

The molecular structure of sulfur dioxide, carbon disulfide and carbonyl sulfide. Paul C. Cross and L. O. Brockway. *J Chem Physics* 3, 821-4 (1935).—By use of the electron diffraction method (C. A. 29, 677) the following interat distances were found:  $\text{SO}_2$ ,  $\text{C—S} = 1.40 \pm 0.02$  Å,  $\text{CS}_2$ ,  $\text{C—S} = 1.54 \pm 0.03$  Å;  $\text{COS}$ ,  $\text{C—O} = 1.16 \pm 0.02$  Å,  $\text{C—S} = 1.56 \pm 0.03$  Å. The types of bond arrangement compatible with these interat distances are discussed. The  $\text{SO}_2$  mol resonates between the structures having single-double and double-single bonds between the S and the O atoms, with a bond angle of  $122^\circ \pm 5^\circ$ .  $\text{CS}_2$  is a linear mol with the structure having the 2 double bonds predominating over those having a single and a triple bond. In  $\text{COS}$  the double-double bond arrangement and the structure having the triple C—O bond predominate.

G M P

Thermodynamic properties of sulfur compounds. II. Sulfur dioxide, carbon disulfide and carbonyl sulfide. Paul C. Cross. *J Chem Physics* 3, 825-7 (1935), cf C A 29, 2832; and preceding abstr.—The thermodynamic properties of  $\text{SO}_2$ ,  $\text{CS}_2$  and  $\text{COS}$  are calcd from the mol consts obtained by electron diffraction and from Raman and infrared spectra.  $-(F^\circ - E^\circ)/T_{298.15} = 50.95$ , 48.28, 47.39;  $S^\circ_{298.15} = 59.40$ , 56.84 (gas), 55.34, resp. The results are applied to several reactions involving these compds. The free energies of formation of  $\text{CS}_2(l)$  and  $\text{COS}$  at 298.1°K are +15.24 and -40.48 kg.-cal, resp.

G M P

The molecular structure of nickel carbonyl. L. O. Brockway and Paul C. Cross. *J Chem Physics* 3, 828-33 (1935).—Electron diffraction by the vapor of  $\text{Ni(CO)}_4$  indicates a mol model in which the CO groups have a tetrahedral arrangement about the Ni atom with the distances  $\text{Ni—C} = 1.82 \pm 0.03$  Å and  $\text{C—O} = 1.15$  Å. These distances are compatible with resonance between 2 electronic structures in which the C—O bond resonates between triple and double electron pair bonds and the Ni—C bond between single and double electron pair bonds.  $\text{Ni(CO)}_4$  is the first quadrivalent compd of neutral Ni whose structure has been detd, and its tetrahedral configuration is contrasted with the square arrangement of bonds in the quadrivalent compds of  $\text{Ni}^{2+}$ .

G M P

Electron diffraction by chloroform and its molecular structure. Urban and tetrahedral models. Charles Degard. *Compt rend* 201, 951-2 (1935), cf C A 29, 6501.—The mol structure of  $\text{CHCl}_3$  was reexamined by the method of electron diffraction, and the results deduced therefrom were shown to be consistent with both the ordinary tetrahedral formula, and Urban's coordinatc formula,  $\text{CCl}_2\text{HCl}$ , within the limits of error.

C A Silberrad

Magnetic birefringence of nitric oxide. Henri Buzette and Tsai Belling. *Compt rend* 201, 955-6 (1935).—The Cotton-Mouton const,  $C_m$ , of NO for  $\lambda$  5780 is  $456 \times 10^{-14}$  at  $15^\circ$  and  $143 \text{ kg./sq cm}$  pressure.  $C_m/d$  is const, and the birefringence varies inversely as  $\lambda$ .

C A Silberrad

Magneto-optical rotation of uranyl salts. S. S. Bhatnagar, P. L. Kapur and N. R. Verma. *J. Indian Chem Soc* 12, 514-18 (1935).—The mol rotations of uranyl chloride, nitrate and sulfate were found to be  $1.22 \pm 0.6$ ,  $-5.85 \pm 0.4$ , and  $-8.5 \pm 0.5$ , resp, giving a value of  $-8.0 \pm 0.6$  for the uranyl ion in the first 2 cases and  $-11.46 \pm 0.5$  with the sulfate ion. Complete analysis showed the formulas of the salts to be  $\text{UO}_2\text{Cl}_2$ ,  $\text{UO}_2(\text{NO}_3)_2$  and  $2\text{UO}_2\text{SO}_4$ ,  $\text{H}_2\text{SO}_4$ ,  $6\text{H}_2\text{O}$ .

H E Phipps

A modified photographic method for substances of small rotatory dispersion. R. Padmanabhan. *J. Indian Chem Soc* 12, 559-63 (1935); cf preceding abstr.—The optical rotatory dispersion of a substance whose optical

activity does not change much with wave length was detd in conjunction with a substance (like quartz) whose rotatory and dispersive power is more nearly normal. By using the difference between this value and that of the latter substance alone, fairly accurate results could be obtained on the former type of compd. Some data are given for  $\beta$ -pinene.

H E Phipps

Valence strength and the magnetism of complex salts. J. H. Van Vleck. *J. Chem. Physics* 3, 807-13 (1935).—Certain complex salts, notably the iron cyanides and the cobaltammines, have magnetic susceptibilities much lower than those predicted by the Bose-Stoner "spin only" formula. This was interpreted by Pauling on the basis of directed wave function (C. A. 25, 2910, 28, 3629). Alternative explanations are possible with the crystal potential model of Schlapp and Penney (C. A. 26, 5233, 27, 1804) or with Mulliken's method of mol orbitals (C. A. 26, 4237). The 3 theories give results so similar that no preference can be established between them. Covalent bonds, as in cyanides, seem to be more effective in suppressing magnetism than are ionic ones, as in fluorides, but so far the evidence is empirical rather than theoretical.

G M P

The principal magnetic susceptibilities of  $\text{K}_2\text{Fe(CN)}_6$ . J. B. Howard. *J Chem Physics* 3, 813-17 (1935).—The magnitudes, anisotropy and temp dependence of the principal magnetic susceptibilities of  $\text{K}_2\text{Fe(CN)}_6$  are calcd by the method of crystal potentials, and agree well with exptl data (cf Jackson, C. A. 27, 4143-4).

G M P

Magnetic moment of the manganese ion. L. C. Jackson. *Proc Phys Soc (London)* 47, 1029-31 (1935).—The  $\mu$  susceptibility of carefully purified manganic acetylacetonate was detd by the Faraday method at 12 temps from  $292^\circ$  down to  $17^\circ\text{K}$ . When corrected for the diamagnetism of the org groups in the mol, the mol susceptibility obeys the Weiss law,  $\chi(T + 55) = \text{const}$ , down to about  $70^\circ\text{K}$ , below which temp  $\chi$  increases somewhat more rapidly than is indicated by this relation. These results give 4.98 as the magneton no for  $\text{Mn}^{4+}$ , which agrees excellently with the requirements of the Bose-Stoner theory.

W. W. Stiller

Diamagnetic study of structure by a new plan. Francis W. Gray and James H. Cruickshank. *Trans Faraday Soc* 31, 1491-1510 (1935).—When the results of Pauling and of Pascal are corrected according to the most recent values for the fundamental phys consts, they furnish a theoretical and an exptl standard, resp, for diamagnetism. These 2 standards can be reconciled and applied to the study of mol structure. Calcn of the "depression" of diamagnetism due to bonds of different types is essential in this method. Frequently fractional values are assigned to the charge on an atom. This is merely statistical and implies that the atom has one integral charge for a definite fraction of the time and a different integral charge for the remainder. A comparison between theoretical diamagnetisms calcd. for different possible structures for a compd and the exptl value indicates which of the suggested structures is the correct one. This method is illustrated by consideration of the structures of benzene, naphthalene, acid carbonyl group,  $\text{H}_2\text{O}$ , and  $\text{H}_2\text{O}_2$ . With benzene, some properties seem to require the double-bonded Kekule structure while others are best explained by the internal-ionic. The diamagnetic analysis indicates that the mol exists in each of these states for half the time. Resonance phenomena are also explained satisfactorily. The theoretical significance is given for Pascal's empirical rules. Tables of screening nos, ionic diamagnetisms and residual charges are given.

W. W. Stiller

Diamagnetic susceptibilities of salts forming ions with inert-gas configurations. II. The alkali halides. G. W. Brindley and P. E. Hoare. *Proc. Roy. Soc. (London)* A152, 342-53 (1935), cf C. A. 29, 1299.—Values of the mol diamagnetic susceptibilities are given for the halides of Li, Rb and Cs. These new data, combined with results previously reported for the halides of Na and K, are used to test the additivity of diamagnetic susceptibilities. This law fails for  $\text{LiCl}$ ,  $\text{LiBr}$ ,  $\text{LiI}$ ,  $\text{CsCl}$ ,  $\text{CsBr}$  and  $\text{CsI}$ .

but holds within exptl. error for the remaining compds. Possible causes for this are discussed; the discrepancies may be due to deformation of the ions, produced by unusually close approach of the neg. ions in the Li salts, and by change of crystal structure in the Cs salts. By assuming  $0.7 \times 10^{-4}$  as the ionic susceptibility of  $\text{Li}^+$  in  $\text{LiI}$ , the following ionic susceptibilities in crystals of the rock salt type are calculated:  $\text{Na}^+ = 6.1$ ,  $\text{K}^+ = 14.6$ ,  $\text{Rb}^+ = 22.0$ ,  $\text{Cs}^+ = 35.1$ ,  $\text{I}^- = 9.4$ ,  $\text{Cl}^- = 24.2$ ,  $\text{Br}^- = 34.5$ ,  $\text{I}^- = 50.6$ , all  $\times 10^{-4}$ . W. W. Stiefel

**Diamagnetism of the trivalent bismuth ion** S. S. Bhatnagar and Bhim Sam Bahl *Current Sci.* 4, 234 (1935).—In an earlier calcn. (cf. *C. A.* 30, 339) based on a modified Slater method,  $-x \times 10^4 = 43.80$  was obtained for the trivalent Bi ion. By use of the orthodox Slater formula 42.23 is now obtained and by a modification suggested by Angus, 42.00. The agreement with the exptl. value, 41.24, is considered satisfactory. The difference between the calcd values for  $\text{Bi}^{3+}$  and  $\text{Bi}^{5+}$  is of the same order of magnitude as that found by Kido for a no. of other ions. W. W. Stiefel

**The diamagnetism of ions in the solid state and in solution** Hermann Lahlénbrach *Amer. Physik.* 24, 453-8 (1935).—Irvold and Sogn (*C. A.* 29, 7137) found that the diamagnetic susceptibilities of certain salts calcd by Wiedemann's addn rule from measurements on aq. solns are greater than those calcd for the same salts when  $\text{LiOH}$  is the solvent. Irvold and Sogn (*C. A.* 29, 7137) observed also that in general the susceptibility of a salt measured in the solid state is less than that calcd by the addn rule from measurements on aq. solns. The work of Cabrera and  $\delta$  demonstrated that, when used as a solvent,  $\text{H}_2\text{O}$  is in a different state of polymerization and consequently its diamagnetism is not the same as that of pure  $\text{H}_2\text{O}$ . These seeming failures of the addn rule are attributed to failure to allow for this change in diamagnetism of  $\text{H}_2\text{O}$  when used as solvent. W. W. Stiefel

**The dipole moment of tetralin.** M. A. Govinda Rao and S. Satyanarayana Rao. *Proc. Indian Acad. Sci.* 2A, 232-5 (1935).—The moment of tetralin as measured in  $\text{C}_6\text{H}_6$  is only  $0.4$  to  $0.5 \times 10^{-18}$ , and not  $1.66 \times 10^{-18}$ , as previously believed. The origin of this small but finite moment is discussed. Harold Gershinowitz

**The discontinuity in the dielectric constant of liquids and their saturated vapors at the critical temperature** J. Maréchal and O. Maass. *Can. J. Research* 13B, 290-7 (1935).—A dielec. cell which was specially adapted to a study of the dielec. const. of liquid and gas over a large temp. range, including the crit. temp. region, is described. The dielec. const. of  $\text{Me}_2\text{O}$  (liquid and satd. vapor) and propylene (liquid and satd. vapor), from room temp. to  $8^\circ$  above the crit. temp., are given. The dielec. const. of liquid and gas are not identical at the crit. temp. These results with previous work in this lab on the discontinuity in properties of the medium above and below the point of disappearance of the meniscus. J. W. S.

**The motion of a heavy drop in an acoustic field** S. V. Gorbachev and A. B. Severny. *Kolloid-Z.* 73, 140-54 (1935).—The distance between centers of 2 liquid drops (5% aq.  $\text{H}_2\text{SO}_4$  soln.) suspended from thin glass threads was studied as a function of (1) the frequency of an acoustic field which was applied both parallel and perpendicular to their line of centers and (2) the velocity and direction of a stream of air. From theoretical as well as exptl. considerations it is concluded that the complicated motion executed by a drop in an acoustic field may be described as similar to that of a uniformly progressing overtone subject to a ponderomotive effect. Two drops in the plane of the sound waves attract each other and may coalesce at sufficiently high (ultra-sonic) frequencies. O. T. Q.

**Density and compressibility of subcane and silicoethane.** K. L. Ramaswamy and G. Gundu Rao. *Proc. Indian Acad. Sci.* 2A, 213-20 (1935).—An app. for simultaneous measurements of  $d$  and approx. compressibilities at ordinary and low temp. is described. Density and compressibility of mono- $\alpha$ -lactone at different temps.

and of diluante at the ordinary temp. were measured. The  $\alpha$ ,  $\alpha$ , of Si (23.10  $\pm$  0.03) obtained from the corrected mol. wts. is compared with the existing values. Harold Gershinowitz

**Effect of molecular association on infrared absorption.** Alfredo Maione *Nuovo cimento* 12, 441-3 (1935); cf. *C. A.* 30, 237.—The absorption coeff. of the  $\text{C}_6\text{H}_5\text{OH}$  band at  $3 \mu$  is increased by adding  $\text{C}_6\text{H}_6$ . Conclusion: Absorption is diminished by mol. assocn. J. B. A.

**Variation of the molecular refraction of ethyl alcohol in mixtures with benzene** Paolo Trautteur. *Nuovo cimento* 12, 444-7 (1935).—The  $n$ ,  $d$ , and mol. refraction of mixts. of  $\text{C}_6\text{H}_5\text{OH}$  and  $\text{C}_6\text{H}_6$  are linear with compn. No evidence of assocn. is found (cf. preceding abstract). J. B. Austin

**Evaporation as a mechanical division to molecules.** P. A. Zolotarev. *Trans. Inst. Chem. Tech. Ivanovo (U. S. S. R.)* 1, 50-3 (1935).—A discussion with math. treatment of the energy consumption in the evapn. of  $\text{H}_2\text{O}$ ,  $\text{N}_2\text{O}$ ,  $\text{Me}_2\text{CO}$  and  $\text{EtOH}$  is based on the Rittenger formula. Chas. Blanc

**Variation in the viscosity of liquid methane and ethylene with temperature** N. S. Rudenko and L. V. Shubnikov. *Fizik. Z. Sovyetunion* 8, 170-84 (1935).—The viscosity of liquid  $\text{CH}_4$  was measured over the temp. range  $90^\circ$  to  $111^\circ\text{K}$ , and that of liquid ethylene over the range  $110^\circ$  to  $169^\circ\text{K}$ . Helen S. Hopfield

**A capillary viscometer for anomalous liquids** Vladislav L. Daneš. *Kolloid-Z.* 73, 174-81 (1935).—D describes an overflow viscometer designed for measuring the viscosity of anomalous liquids. It has the special advantage of being applicable over a million-fold range of streaming velocities. The movement of liquid is followed by weighing the overflow in a definite interval of time. Tables and graphs of data are given for water, 0.0024 N and 0.048 N  $\text{NH}_4$  oleate sol. and 0.3% sinus yellow GG sol. at  $18^\circ$  and the dil. oleate sol., the log of the vol. transferred per sec. was approx. a linear function of the log of the applied pressure, the line being approx. parallel to that for pure water. The more concd. oleate and the sinus yellow sols gave a line concave to the log  $P$  axis. O. T. Q.

**Metal-cuprous oxide contact resistance.** Jean Rouleau. *Compt. rend.* 201, 947-8 (1935).—By a special arrangement the Cu-Cu<sub>2</sub>O contact resistance is shown to depend on (a) the age of the surface, (b) temp., (c) sp. resistance of the Cu<sub>2</sub>O, and (d) treatment of the surface. With (a), (b), and (d) const. it is proportional to (c); variation in (c), e. g., attack by dil. acid, alkali or salts, affects only the factor of proportionality. For high resistance the surface must be cryst. C. A. Silberrad

**The conductance of salt crystals.** W. H. Rodenbush and T. G. Cooke. *J. Chem. Physics* 3, 834 (1935).—An expression is derived for the cond. of salt crystals. Jost's equation (*C. A.* 27, 4457) is considered inadequate to account for the behavior of various salts at different temps. G. M. P.

**The use of direct current in the measurement of electrolytic conductance.** J. N. Brønsted and Ralph F. Nielsen. *Trans. Faraday Soc.* 31, 1478-81 (1935).—Polarization of electrodes in the d. c. method is eliminated by the use of platinized  $\text{H}_2$  electrodes. The cell consists of a U-tube of 6 mm. bore, 50 cm. long and provided with wide-bore electrode vessels at the ends. Potentials of 10-50 v. were used for resistances of the order of 10,000 ohms, the heating effect being minimized by the short time of current flow during setting of the bridge. An accuracy of 1 part in 10,000 was observed in measurements of the equiv. cond. of 0.001-0.1 N  $\text{HCl}$  and 0.002-0.1 N  $\text{H}_2\text{SO}_4$  at  $25^\circ$ . The method is recommended for simplicity of app. and procedure, especially for solns. of low cond. L. W. Elder

**Superconductivity** H. Grayson Smith and J. O. Wilhelm. *Rev. Modern Physics* 7, 237-71 (1935). E. H.

**New method of measuring the elastic constants of transparent isotropic solids.** E. Hiedemann and K. H. Hoersch. *Naturwissenschaften* 23, 577-8 (1935).—Elastic const. of glass are detd. by observation of the interference

bands caused by ultrasonic waves in polarized light

B. J. C. van der Hoeven  
Thermal studies of aluminum hydroxide—aluminum oxide by the emanation method Robert Jagtisch *Z. physik. Chem.* A174, 49-59 (1935)—Various produced samples of Al hydroxide were studied by the emanation method in their behavior on being heated from 20° to 1300° and the change in the preps was deduced from their change in power of emanation. The greatest surface development was reached in the range of stability of  $\gamma$ -AlOOH (200-300°). The  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, occurring at 300° showed a gradual decrease in activity corresponding to the gradual decrease in surface. At about 1100° to 1200° the emanation shows a rapidly proceeding change in structure which by means of Röntgen studies was shown to be due to change of  $\gamma$ -into  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>.

R. H. Baechler  
Inhibition of hydrosols and dye solutions by porous substances I General considerations and experimental methods Augustin Bontaric. *Bull. soc. chim. bel.* 17, 1522-33 (1935)—See C. A. 28, 53104.

L. E. G.  
The polarization effect in thallium fluoride Erich Hayek *Z. anorg. allgem. Chem.* 225, 47-8 (1935)—The prepn and purification are described. TlF melts at 327° to a yellow liquid. The crystals are foliated and birefringent and can be regarded as contg a polarizing anion layer of small F ions surrounded by 2 polarizing layers of large, unsymmetrical Tl ions. TlF dissolves in water with absorption of heat. The soly. at 20° is 78.8%, and the solid phase is anhyd. TlF. It is only slightly sol in EtOH. The concd aq soln is strongly alk. The b p is 653°  $\pm$  10° and  $d_4^{20}$  is 8.36.

E. R. Rushton  
New problems of theoretical crystallography. Willi Kleber. *Naturwissenschaften* 23, 608-8 (1935)—A short review of theoretical aspects of crystal structure, particularly crystal defects and inhomogeneities.

B. J. C. van der Hoeven  
The possibility of a metallic modification of hydrogen E. Wigner and H. B. Huntington. *J. Chem. Physics* 3, 764-70 (1935)—Math-theoretical. Any lattice in which the H atoms would be translationally identical (Bravais lattice) would have metallic properties. The energy of a body-centered lattice of H is calcd as a function of the lattice const. This energy attains a min for a lattice const. that corresponds to a  $d$  of 0.59—much higher than that of the usual mol lattice of solid H<sub>2</sub>. This min., though neg., is much higher than that of the mol form. Neither the body-centered nor other simple metallic lattices are attainable with pressures now available. The chances are perhaps better for intermediate, layer-like lattices.

G. M. P.  
Crystallography of the copper-pyridine saccharin complex CuPy(H<sub>2</sub>O)<sub>2</sub>SA. J. Benjamins, P. Terpstra and J. J. de Vries. *Pharm. Weekblad* 72, 1287-94 (1935)—Optical and x ray measurements are reported on the rhombic bipyramidal crystals (8  $\times$  5  $\times$  3 mm.) obtained by slow evapn of dil solns contg CuSO<sub>4</sub>, pyridine and Na saccharin.

A. W. Dox  
Crystal structure of ammonium cupric bromide Alfred Silberstein. *Compt. rend.* 201, 970-1 (1935)—(NH<sub>4</sub>)<sub>2</sub>CuBr<sub>2</sub>·2H<sub>2</sub>O,  $d$  2.82, is isomorphous with (NH<sub>4</sub>)<sub>2</sub>CuCl<sub>2</sub>·2H<sub>2</sub>O (cf. Hendricks, et al., C. A. 21, 3500), and has  $a$  7.98,  $c$  8.41, space group  $D_{2h}^{14}$  ( $Pr/mnm$ ). Intensity depts show that its structure is identical with that of (NH<sub>4</sub>)<sub>2</sub>CuCl<sub>2</sub>·2H<sub>2</sub>O, i. e., the Cu atom is surrounded octahedrally by Br and (NH<sub>4</sub>)<sub>2</sub>.

C. A. Silberrad  
X-ray characteristics of various forms of dicalcium silicate E. Brandenberger. *Schweiz. mineral. petrogr. Mitt.* 14, 473-7 (1935)—X-ray examn of  $\alpha$ -,  $\beta$ - and  $\gamma$ -Ca<sub>2</sub>SiO<sub>4</sub> shows them to be independent phases, but owing to the general weakness of the lines and their similarity to those of Ca<sub>2</sub>SiO<sub>3</sub> the min. amt detectable in a mixt is rather high (cf. Sandus, C. A. 27, 3267).

C. A. Silberrad  
Sorption of hydrogen by tungsten W. Frankenburg and A. Hodler. *Naturwissenschaften* 23, 609 (1935)—Absorption of H<sub>2</sub> by W surfaces shows in 2 temp. ranges (—80 to —10°, 60° to 180°) the characteristics of activated adsorption (Taylor, C. A. 25, 1422). Thorough cleaning

of both H<sub>2</sub> and W, however, changes the picture completely. It is evidently due to minute traces of org vapors (cock grease, etc.) in the presence of small amts of O<sub>2</sub>. At certain temps a fractional self-cleaning occurs. Pure W and H<sub>2</sub> give a max. of adsorption which covers 1/4 of the surface and decreases with temp.

B. J. C. van der Hoeven  
Adsorption of zinc sulfide by copper sulfide A. del Campo and A. Hoyo de Castro. *Andalus. soc. esp. fis. quim.* 33, 754-64 (1935)—Strong agitation of the soln during pptn avoids entrainment and supports the hypothesis of surface phenomena. Similar entrainment of Pb in 2 N HCl soln at 80° is appreciably reduced by agitation, although entrainment cannot here be wholly prevented.

E. M. Symmes  
Adsorption theory of the electrokinetic potential. A. March. *Trans. Faraday Soc.* 31, 1468-78 (1935), cf. Rabinovich, C. A. 28, 17°—Math development of the postulate that the  $\psi$  and  $\phi$  potentials of colloidal particles are independent quantities, the latter being related to the adsorption potential,  $\psi$ . It is shown that the principle of pos-neg equivalency does not hold for the process of exchange adsorption.

L. W. Elder  
The viscosity of titanium dioxide sol in the presence of electrolytes S. M. Mehta and Olive Joseph. *J. Indian Chem. Soc.* 12, 552-9 (1935); cf. C. A. 27, 4996.—The viscosity of the sol increased with time when KCl, MgCl<sub>2</sub> or AlCl<sub>3</sub> was added as a coagulant. Periodicity was observed in the slow pptn region. The autocatalytic nature of the coagulation process disappeared on progressive dialysis.

H. E. Phipps  
The acid-combining capacity of wool J. B. Speakman and E. Stott. *Trans. Faraday Soc.* 31, 1425-32 (1935), cf. C. A. 28, 6009°—The titration curves of wool with HCl, H<sub>2</sub>SO<sub>4</sub>, ClCH<sub>2</sub>COOH, (COOH)<sub>2</sub>, and H<sub>3</sub>PO<sub>4</sub> were determined by the glass electrode and show that (COOH)<sub>2</sub> and H<sub>3</sub>PO<sub>4</sub> are monobasic in reaction with wool. At high  $p_H$  H<sub>2</sub>SO<sub>4</sub> is more nearly monobasic than dibasic. At low  $p_H$  ClCH<sub>2</sub>COOH combines to a far greater extent than HCl at the same  $p_H$  and also exhibits a greater heat of reaction and greater swelling of the wool fiber. The hypothesis is advanced that concd solns of weak acids, owing to the higher swelling pressure developed, can separate the sheets of covalently linked peptide chains and liberate NH groups for combination with acid. At the surface of the fiber some NH groups are always available, hence cataphoretic detns of the isoelec. point of wool are not valid for the fiber as a whole.

L. W. Elder  
The double refraction of chitin tendons J. M. Drell and G. van Iterson, Jr. *Kolloid-Z.* 73, 1425-6 (1935)—The double refraction observed by the method of Moehring (cf. C. A. 20, 3634), varied with medium bathing the chitin layer. The min. double refraction occurred at a  $n$  of 1.475 in glycerol-quinoline mixts but at  $n$  = 1.60-1.62 in aq. solns of K<sub>2</sub>HgI<sub>4</sub>. The difference is attributed to salt adsorption, for chitin decreased the  $n$  of K<sub>2</sub>HgI<sub>4</sub> solns, but had no effect on the  $n$  of glycerol-quinoline mixts. Plant and animal chitin show the same optical behavior.

Oscar T. Quimby  
X-ray investigations of elastic fibers with special consideration of stretching and shrinking Heinz Köppl. *Kolloid-Z.* 73, 129-42 (1935)—Rod shaped pieces 1-1.5 mm. thick were cut from the ligamentum nuchae of fresh beef and examd both moist and dry under various tensions by means of x-ray beams (X<sub>u</sub> and X<sub>u</sub> lines of Cu) at various angles. With the x-ray beam inclined at right angles to the fiber axis, the most unstretched fiber gave 2 halos, the inner of which showed 4 weak max while the dry fiber gave 2 interference rings in addition to the 2 halos. These halos were present in all of the photographs. At an extension of 75%, both moist and dry fibers gave a typical fiber diagram. Up to the max. extension (200%) the fiber diagram remained the same except that the interference rings increased in intensity. With the x-ray beam parallel to the fiber axis, the dry fiber gave 2 interference rings at low tension, the no. increasing to 5 at high tensions. No further interferences were found at other angles of inclination of the x-ray beam. In the unstretched fiber



the primary-valence complexes can be oriented in any direction with a slight preference for the fiber axis; when extended 75% or more all these complexes lie in planes parallel to the fiber axis. Shrinkage in absence of applied pressure tends to orient the primary-valence complexes in the direction of the fiber axis, but shrinkage under tension does not change the regularities along the fiber axis. The intramolecular water amounts to perhaps one-fourth of the total water in the tissue. Data from the analysis of the x-ray photographs and photometric curves are tabulated.

Oscar T. Quimby

The solubility of hydrogen in molten aluminum L. L. Bircumshaw *Trans. Faraday Soc.* 31, 1439-43 (1935).—Solubilities were determined by a method similar to that of Röntgen and Braun (C. A. 26, 5478) in an apparatus which provides for an atmosphere of  $H_2$  outside the  $SiO_2$  furnace tube to prevent loss of  $H_2$  by diffusion through the latter. The results, in cc  $H_2$  at normal temp and pressure per 100 g. Al are 0.23, 0.89, 1.87 and 3.80 at 700°, 800°, 900° and 1000°, resp. These values agree well with those of R. and B. Equilibrium is reached almost instantaneously at 1000° and after 15 to 20 min at 800°. The "saturated" heat of soln. calculated on the assumption that  $H_2$  dissolves as atoms, is 43,400 cal/g. mol.

L. W. Elder

The solubility of lime in water G. L. Larocque and O. Maass *Can. J. Research* 13B, 276-90 (1935), cf. C. A. 27, 5231.—The solubility of lime was determined accurately over the temperature range 0° to 30° by a conductometric method. The degree of dissociation of lime over this temperature range and over the concentration range 0 to 1.64 g. of CaO per l. is calculated.

J. W. S.

Interrelation between the volumes of substances in free and solvated state I. I. Zaslavskii, A. A. Astasheva and I. A. Sakov *Trans. Inst. Chem. Tech. Ivanovo (U. S. S. R.)* 1, 47-90 (1935).—The relation between the volumes of 1 N solutions of fluorides and 2 N solutions of chlorides, bromides and iodides, determined at 15-20°, and the volumes of the same compounds in the solid state is graphed and tabulated.

Chas. Blanc

Measurements of the surface tension of solutions of potassium and lithium salts of the higher fatty acids by the ring method with exclusion of carbon dioxide and with special consideration of other disturbing effects I. A. Lottermoser and E. Giese *Kolloid-Z.* 73, 155-70 (1935).—By use of an improved form of the apparatus of Lottermoser and Baumgärtel (C. A. 29, 3890), the surface tension,  $S$ , of K laurate, K myristate and Na dodecyl sulfate solutions was studied at low concentrations (0.001-0.1%). As a function of time and temperature. Exclusion of  $CO_2$  is a necessary but not sufficient condition for reproducibility in the soap solutions. Three adsorption effects were discovered, all leading to high values of  $S$ . (1) Adsorption at the air-soln. interface was sometimes so great that crystals took place; even when surface crystals did not take place, erroneously high results might be obtained if the soln. was transferred to the measuring dish from a large flask without stirring (e. g., pipetting) because of the concentration decrease in the bulk of the soln. (2) A more important source of error was found in the wetted glass surface just above the liquid surface. This error could be avoided either by filling the measuring dish to the brim or by using a non-wetted dish (e. g., paraffin coated). (3) Because of their practice of cleaning the Pt ring by heating to redness between  $S$  measurements, L. and G. found that adsorption on the Pt ring may cause appreciable error in  $S$ . The first two adsorption effects were observed with the soaps as well as the alkyl sulfate, the third only with the alkyl sulfate. It was further demonstrated that the static value of  $S$  is attained very slowly at such low concentrations of Na dodecyl sulfate.

Oscar T. Quimby

The surface tensions of ternary solutions I. Aqueous solutions of (a) NaCl and KCl, (b) NaCl and HCl J. W. Belton *Trans. Faraday Soc.* 31, 1413-19 (1935).—Measurements were made at 25° by the method of maximum bubble pressure in an apparatus similar to that used by Brown (C. A. 26, 3425). For binary solutions the simple relation  $(1/\gamma) - (1/\gamma_0) = km$  holds, in which  $\gamma$  and  $\gamma_0$  are the surface tensions (dynes/cm) of soln. and of water, resp., and  $m$  is concentration in mols. per 1000 g.  $H_2O$ . Values of  $k$  at 25°

are for NaCl, 1.7; KCl, 1.6; and HCl, -0.12 over the range  $m = 1$  to 5. In mixtures of KCl and NaCl, where  $\gamma_{12} - \gamma_0 = k_1m_1 + k_2m_2$  (II),  $k_1$  (NaCl) = 1.6 and  $k_2$  (KCl) = 1.5 over the range  $m = 1$  to 3. For mixtures of NaCl and HCl ( $m = 1$  to 5), the observed relation is  $\gamma_{12} - \gamma_0 = k_1m_1 + k_2m_2 + k_3m_1m_2^{1/2}$ , where  $k_1$  and  $k_2$  refer to HCl and  $k_3 = -0.5$ . Equations (I) and (II) are special forms of the general equation derived from the Gibbs equation. The negative surface adsorptions of KCl and NaCl in aqueous solutions are additive in the mixed solutions. At high concentrations the thickness of the unimolecular surface layer, calculated from surface concentrations, tends to diminish. In the NaCl-HCl mixtures the HCl is more effective than NaCl in reducing the thickness of the surface layer. (a) Ethyl alcohol-water-salt mixtures. (b) Acetic acid-water-salt mixtures. *Ibid.* 1420-51.—Solutions of LiCl, NH<sub>4</sub>Cl, KBr, KI, KCNS, KNO<sub>3</sub>, KCl and NaCl from 1 to 5 molar were investigated in a 0.03% EtOH soln. The data agree with the equation  $(\partial\gamma/\partial m_1)m_1 = k_1(1/\gamma_1) - RTT_1(\partial \log f_1/\partial m_1)m_1$ , where  $m_1$  = bulk concn,  $\gamma_1$  = surface concn,  $f_1$  = activity coefficient of EtOH, and the subscripts 1 and 2 refer to salt and EtOH, resp.,  $\gamma_1$  is the surface concentration of salt in pure  $H_2O$  at the same value of  $m_1$ . For all salts at low concentration  $(\partial\gamma/\partial m_1)m_1$  is negative, approaching 0 at concentration  $m_1 > 3$  except for NaCl. LiCl and NH<sub>4</sub>Cl show definite minima in  $\gamma$  at high concentrations. On the assumption that  $1/\gamma$  is independent of  $m_1$  and by using values of  $f_1$  calculated from published data,  $1/\gamma_1$  can be calculated, from which it is shown that the amount of  $H_2O$  adsorbed is in some cases greater, in others less than for an aqueous solution at the same concentration  $(\partial\gamma/\partial m_1)m_1$  is positive for solutions of KCl, NaCl, KBr and KNO<sub>3</sub> in 0.153 mol % AcOH and constant for the first three. For the AcOH solutions,  $1/\gamma_1$  is less than unity and less variable with concentration of salt than in the EtOH solutions.

L. W. Elder

The viscosity of sucrose and dextrin solutions containing salts J. H. C. Merckel *Kolloid-Z.* 73, 171-4 (1935), cf. C. A. 30, 12.—Relative viscosities at 25° are given for (1) 2% sucrose solutions 0.25 and 0.5 N with respect to Na salts of univalent anions and (2) 2.5% dextrin solutions 0.25 N with respect to the same salts. The viscosity of these solutions can be expressed quantitatively as a parabolic function of the lyotropic numbers.

Oscar T. Quimby

Diffusion potentials II V. Čupr *Fak. Facult. Sci. Univ. Masaryk* No. 197, 1-17 (in English 13-14) (1934), cf. C. A. 28, 4291.—Mathematical conditions under which Planck's equation for diffusion potentials satisfies the relation  $II_1 + II_2 - II = 0$  or  $II_1 + II_2 - II = 0.5$  millivolt are sought.

V. D. Karpenko

Mass action N. A. Brunst *Chem. Weekblad* 32, 446-8 (1935).—Polemical (cf. Koltthoff, C. A. 29, 5722\*).

Reply I. M. Koltthoff *Ibid.* 448-9.

B. J. C. van der Hoeven

The action of alkaline solutions on ultramarine blue. Kurt Leschewski and Ernst Podschus *Z. anorg. allgem. Chem.* 225, 43-6 (1935), cf. C. A. 29, 6858\*.—Ultramarine is decomposed by strong, boiling alkali solutions into colorless alkali aluminosilicates. KOH acts very slowly. LiOH and KOH give compounds containing no S, but NaOH forms a Na aluminosilicate containing S as thionate. K and Li will replace Na in the compound. The blue color disappears when the ultramarine lattice is altered or destroyed.

E. R. Rushton

Dissociation of ferrous carbonate J. Kristinsson *Z. anorg. allgem. Chem.* 225, 93-6 (1935).— $FeCO_3$  was prepared by heating  $aq. FeCl_3$  with  $CaCO_3$  at 130-80° for 20 hrs. in an evacuated sealed tube, and dried at 120° for 47 min. It contained 92.07%  $FeCO_3$  and 2.22%  $CaCO_3$ . Dissociation pressures were measured at 258.5-285.5°, in a compensating apparatus (C. A. 25, 447). The pressure is 760 mm. at 282°. The heat of dissociation, calculated from the Nerst approximation formula, is 20,900 cal. Dissociation takes place in steps, with the formation of intermediate compounds. Complete decomposition takes place at 430-450°, leaving a residue of black  $FeO$ .

E. R. Rushton

Equilibrium of the system: calcium cyanamide-sodium chloride. M. S. Maksimenko and Ya. I. Izraelovich. *Khim. i Tekhnol.* 7, 411-13 (1935); cf. Franck and Burg, C. A.

29, 1348<sup>2</sup>—The isothermic equilibria of the reactions.  $\text{CaCN}_2 + \text{C} \rightleftharpoons \text{Ca}(\text{CN})_2$ ,  $\text{Ca}(\text{CN})_2 + 2\text{NaCl} \rightleftharpoons \text{CaCl}_2 + 2\text{NaCN}$ , were studied with com. "black cyanide" in the interval 490–1520° for various periods of time. The product was heated in a crucible in an elec. furnace. The graphs and tables disclose that the cyanide is completely decomposed at 490–850°. This shows that the cooling within this temp. must be reduced to a min. of time. The reaction of decomposition is  $\text{Ca}(\text{CN})_2 \rightarrow \text{CaCN}_2 + \text{C}$ , followed on further heating by a partial irreversible reaction  $\text{CaCN}_2 + \text{C} \rightarrow \text{CaC}_2 + \text{N}_2$ . The diagram of decomposition shows that the formation of cyanide begins at 900–100°, whereby the concn. of NaCN equiv. rises from 0% at 850° to 13–3.5% at 950°. At 1400–1520° the reaction is energetically and completely shifted toward the cyanide formation. At 1490° the max. is reached in 5 min. and at 1520° in 2 min. Thereafter the irreversible decomposition of cyanides into the elements begins, the process is accelerated by increasing temp. Chas. Blanc

Pressure oxidation of ferrous sulfate in neutral and in acid solutions. G. Agde and F. Schummel. *Z. anorg. allgem. Chem.* 225, 29–32 (1935).—An autoclave with a capacity of 400 cc. was used. Air or  $\text{O}_2$  was introduced at the bottom of the tube through a lead pipe, and the exit was provided with a reflux condenser. The rate of oxidation decreased with increasing concn. of  $\text{FeSO}_4$ , increased with increasing  $\text{O}_2$  pressure. Curves are given showing the percentage oxidation in relation to the vol. of  $\text{O}_2$  passed through at different concns. and temps. In neutral solns., a basic salt, contg. only ferric iron, is pptd. When enough  $\text{H}_2\text{SO}_4$  was added to form  $\text{Fe}_2(\text{SO}_4)_3$ , the reaction went more nearly to completion. When air at 105 atm. was substituted for  $\text{O}_2$  at 29 atm., more rapid oxidation took place, because a greater surface was offered to the  $\text{FeSO}_4$  soln. E. R. Rushton

Hydrolysis of the aluminum benzenesulfonates. V. Čupr and B. Šliva. *Publ. Facult. Sci. Univ. Masaryk No. 200*, 1–11 (in English 12–13) (1935).—An investigation into hydrolysis of 0.01–0.5 N solns. of aluminum benzenesulfonate and Al *p*-hydroxybenzenesulfonate is described. V. D. Karpenko

Hydrolysis of phenylalanine. Emil Baur and G. Schindler. *Biochem. Z.* 281, 238–48 (1935), cf. C. A. 29, 190<sup>1</sup>.—Hydrolysis of phenylalanine at 110° follows the mass-action law, the velocity of the reaction being of the first order. S. Morgulis

The absolute rate of homogeneous atomic reactions. Henry Eyring, Harold Gershinowitz and Cheng E. Sun. *J. Chem. Physics* 3, 789–96 (1935).—Math-theoretical. The abs. rate of the recombination of 3 H atoms is calculated. The manner in which rotation affects the dimensions of the activated complex in cases having little or no activation energy is discussed. The calcs. agree well with the exptl. rates of Steiner (C. A. 29, 4265<sup>4</sup>) and of Amdur (C. A. 29, 4223<sup>3</sup>). An immediate consequence of the theory is that energy transfer occurs most effectively among particles that can react with each other, free atoms being more effective than molts. A qual. application of potential surfaces to the problem of energy transfer as met in velocity-of-sound expts. and in expts. on maintenance of high-pressure rates of unimol. reactions is made. G. M. P.

Effect of gases adsorbed by the walls of the container on the chain reaction of oxy hydrogen mixtures. Marcel Prettre. *Compt. rend.* 201, 96–24 (1935); cf. C. A. 29, 3909<sup>1</sup>.—The change in rate of contraction with time,  $R_c$ , of a mixt. of 21%  $\text{H}_2$  and  $\text{O}_2$  introduced at 600 mm. and 540° into vessels whose walls were in various states as regards adsorbed  $\text{H}_2\text{O}$  or  $\text{H}_2$  were detd.  $R_c$  increases rapidly with the amt. of  $\text{H}_2\text{O}$  adsorbed, but decreases with that of  $\text{H}_2$ , showing, however, in this case little or no induction. In all cases the reaction is a characteristically chain one. C. A. Silberrad

The speed of thermal decomposition of chloropercs. Dan Rădulescu and G. Zamfirescu. *Bul. soc. chim. Romania* 17, 87–92 (1935).—The rate of decompos. of chloropercs (I) to phosgene and nitrosyl chloride (II) was followed by heating in sealed bulbs and detg. the amt. of II produced by breaking under 0.1 N  $\text{KMnO}_4$  at 45–50° and

1 titrating the excess  $\text{KMnO}_4$ , not reduced by the nascent  $\text{HNO}_3$  from the hydrolysis of II, with KI and  $\text{Na}_2\text{S}_2\text{O}_3$ . The reaction is unimol. with const.  $K_{\text{H}_2\text{O}}$   $0.11 \times 10^{-3}$ ,  $K_{\text{H}_2\text{O}_2}$   $0.22 \times 10^{-3}$ ;  $K_{\text{H}_2\text{O}_2}$   $0.97 \times 10^{-3}$ ,  $K_{\text{H}_2\text{O}_2}$   $3.27 \times 10^{-3}$ . The reaction to form the 2 poisonous gases would be almost instantaneous at the temp. of explosion of munitions. I is decomposed photochemically by violet or ultraviolet light but the reaction though rapid at first soon stops because the product II absorbs the violet light and a small concn. of it protects the rest of I. This explains the relative stability of I in daylight. Janet E. Austin

The kinetics of heterogeneous organic reactions.—The reaction between benzyl chloride and solid silver nitrate. M. V. Nabar and T. S. Wheeler. *Proc. Indian Acad. Sci. A*, 265–78 (1935).—The kinetics of the reaction between benzyl chloride and solid  $\text{AgNO}_3$  were studied in the absence of solvents and diluents. The reaction is independent of the amt. of benzyl chloride, but is proportional to the surface of  $\text{AgNO}_3$  present. The reaction was studied with particles of 3 different sizes. All the exptl. results can be reproduced by the kinetic equation derived on the assumption that the rate of reaction depends only on the surface of  $\text{AgNO}_3$  present. The velocity of reaction is independent of the speed of shaking. Water inhibits the reaction; the effect of 0.18% by weight of benzyl chloride taken is marked and this effect increases with the amt. of water added. Harold Gershinowitz

The transition point of hexachloroethane. E. H. Wieberga. *Z. anorg. allgem. Chem.* 225, 38–42 (1935).— $\text{C}_2\text{Cl}_6$  was sublimed and the middle fraction used. This had a triple point of 189.8°, in agreement with that detd. by van der Lee (C. A. 29, 6118<sup>1</sup>) from vapor-pressure measurements. For the rhombic  $\rightarrow$  triclinic transition, 48.3–48.7° was found on the heating curve and 34–35.6° on the cooling curve. For the triclinic  $\rightarrow$  cubic transition, these temps. were 71–72.2° and 70.6°, resp. Dilatometer measurements narrowed these hysteresis ranges to 40–44.6° and to 70.83–71.15°, resp. The velocity of transition was then measured as a function of temp. by observing the rate of change in the level of the dilatometer liquid when approx. equal quantities of the 2 modifications were in contact, and the data were plotted to find the points of zero velocity. Since, in true hysteresis, all points inside of the hysteresis range can be detd., true hysteresis cannot be present in this case, within the limits of exptl. error. C. I. C. A. 28, 27<sup>1</sup>. E. R. Rushton

Numerical relation in binary metallic systems. D. Stockdale. *Proc. Roy. Soc. (London)* A152, 81–104 (1935).—During the last 7 years the 6 systems, Cd–Zn, Cu–Ag, Pb–Sn, Cu–Al, Sb–Ag and Cd–Sn were studied exhaustively. This work was originally undertaken to obtain data to test the validity of the assumptions that (1) in all eutectic mixts. the atoms are present in a simple ratio and (2) at crit. points on the equl. diagram the solute and solvent atoms are present in a simple ratio. The results do not seem to support these assumptions. However, it is the purpose of this contribution to show that when combined with the recent work of Hume-Rothery and coworkers (C. A. 28, 5723<sup>1</sup>) some simplifying relation may be stated as a step toward the solution of a larger problem. With 20 alloy systems of Cu and Ag the at. relations at the crit. points B (limit of solid soly. at the eutectic temp.) are discussed. In addn. with 9 alloy systems of Cu and Ag similar relations at the crit. points C (limit of solid soly. at the temp. of transformation of B) are considered. It is concluded that (a) bivalent solutes are generally most sol., tri- and quadri-valent atoms being less so, (b) where the C point is that of max. soly. for trivalent solutes the atom-electron ratio is always almost exactly 5.7, and (c) there is a simple relation between the dissolving powers of a solvent for one solute at the B and C points. The soly. at C can be calcd. from  $\frac{\text{Dissolving power at B}}{\text{Dissolving power at C}} = (N - 1)/N$ , where  $N$  is 4, 5, 6 or 7. Dissolving power is the increase of the no. of solvent atoms required to dissolve one solute atom. The compn. of the eutectic and of the solid solns. at the

ends of the eutectic horizontal were carefully correlated graphically for the systems Ag-Cu, Cd-Sn, Cd-Zn, Pb-Sn, Ag-Sb, Al-Cu and Cu-Mg. Evidence for the following rule is put forward: (1) the solute and solvent atoms are present in a simple integral ratio in a solid solid soln at the temp. of the eutectic or peritectic horizontal, (2) in a eutectic mixt. the elements are present in a simple integral at. ratio; and (3) in a eutectic mixt. the ratio of the number of atoms irrespective of their kind in the phases is simple. These 3 roles in the strictest sense are incompatible, and it is felt that (3) is surely true because the exptl work on which it is based is best. H. A. Smith

Habits and laws of decomposition of supercooled solutions, with special regard to austenite. G. B. Lpton Penna. *State Coll. (Mineral Ind. Expt. Sta.) Bull.* No. 18, 24-58 (1935), cf. C. A. 28, 1674. H. A. Smith

Molecular clustering in binary liquid mixtures. Variation with composition and temperature. R. S. Krishnan. *Proc. Indian Acad. Sci.* 2A, 221-31 (1935), cf. C. A. 29, 7732. —A comparative study is made of the intensity and depolarization of the light scattered transversely by mixts of phenol and water in different proportions and at different temps. The lowest values for the depolarizations  $\mu_v$ ,  $\rho_v$  and  $\rho_h$  are obtained with the crit. compn. mixt. (in this case 34% phenol mixt.) at the crit. soln temp. The photoelectric method was employed for the comparison of intensities of scattering by these mixts at various temps. above the crit. soln temp. The intensity of scattering increases considerably for the 34% phenol mixt. as the crit. soln temp. is approached. The tendency for the formation of clusters and the size of the clusters formed depend on the temp. and compn. of the mixt. These phenomena are most pronounced for the crit. compn. mixt. at the crit. soln temp. The bearing of these results on the anomalies of viscosity, magnetic birefringence, etc., noticed in the vicinity of the crit. conditions is also pointed out. Harold Gerharowitz

Thermal analysis of the system argon-boron trifluoride. Compounds with the inert gases of the atmosphere. Harold S. Booth and Karl S. Willson. *J. Am. Chem. Soc.* 57, 2273-85 (1935). —Study of the system A-BF<sub>3</sub> showed that the f. p. of any mixt. of the 2 increases with increase in pressure up to a certain value and in general is little affected by any further pressure increase, indicating an equl., either A + xBF<sub>3</sub> = A xBF<sub>3</sub> or A xBF<sub>3</sub> + yBF<sub>3</sub> = (A + y)BF<sub>3</sub>. A graph of the f. ps vs. compn. shows max. and min., the latter corresponding to the ratios A-BF<sub>3</sub>, A-2BF<sub>3</sub>, A-3BF<sub>3</sub>, A-4BF<sub>3</sub>, A-5BF<sub>3</sub>, and A-6BF<sub>3</sub>, indicating compd. formation. From the shape of the curve the ratio A-2BF<sub>3</sub> appears to be the most stable. The f. ps of the A-BF<sub>3</sub> mixts of compns. approximating those of the max. and min. were sharp, while mixts. of intermediate compn. melted over a temp. range. At pressures near 35 atm., a 2nd liquid layer appears, which is probably best explained as due to retrograde immiscibility. L. K.

Critical phenomena of the system argon-boron trifluoride. Harold S. Booth and Karl S. Willson. *J. Am. Chem. Soc.* 57, 2290-4 (1935). —The crit. phenomena of the system BF<sub>3</sub>-A exhibited the usual retrograde condensation. A new phenomenon, best described as retrograde immiscibility, appeared at the low-temp., high-pressure range. The compds. formed by the gases at their m. ps. are practically completely dissocd. at the "crit. temp. region," which is about 100° higher, and so affect the curve of the max. temp. of liquefaction only very slightly. Louise Kelley

New composition diagrams for the systems lead thallium and lead-thallium-cadmium. Correction. Ernst Jänecke. *Z. Metallkunde* 27, 141 (1935); cf. C. A. 29, 201. —New exptl. work of A. Ölander (C. A. 28, 5743) indicates that the two-phase field between the Pb and PbTl solid solns. should extend from 49 to 55% Pb, and that the PbTl field should end at about 10 Pb on the Tl side. No changes are required for the Pb-Tl-Cd system. G. Derge

The boiling points and vapor-phase composition of the three-component mixture ethyl acetate-ethyl alcohol-water. V. A. Kureev, I. Ya. Klinov and A. N. Grigorovich

*J. Chem. Ind. (Moscow)* 12, 936-40 (1935). —Orienting but not exact data are presented. The solubility of the boiling points at a given ratio of EtOAc to EtOH show a max. and then a min. as H<sub>2</sub>O is added. The effect is most pronounced at about 20% EtOAc and 80% EtOH.

H. M. Leicester

The system calcium oxide-sulfur dioxide-water. II. Calculation of ionic concentrations. P. E. Gubler and O. Maass. *Can. J. Research* 13B, 308-22 (1935); cf. C. A. 30, 3677. —The cond. and vapor pressure data of the system CaO-SO<sub>2</sub>-H<sub>2</sub>O presented in an earlier paper were used to calc. ionic concns. existing in this system, in the temp. pressure range investigated. Two methods of calcn. were employed, one depending on the combination of vapor pressure and cond. data, and the other, on cond. data. For both, recourse was had to the data of Campbell and Maass (C. A. 24, 1796) on the system SO<sub>2</sub>-H<sub>2</sub>O. These methods gave results in good agreement with each other. The existence of a complex CaSO<sub>3</sub>·Ca(HSO<sub>3</sub>)<sub>2</sub> was deduced. H. ion concns. over the concn. and temp. range investigated were calcd. and extrapolations made to higher temps. These are tabulated. Apart from the theoretical interest, these results are of value in connection with studies of the delignification of wood by CaO-SO<sub>2</sub>-H<sub>2</sub>O solns. J. W. Shipley

Intensity parameters and stable thermodynamic equilibrium. T. Ehrenfest-Afanasyeva and G. L. de Haas-Lorentz. *Physica* 2, 743-52 (1935). B. J. C. v. d. H.

The heats of solution and specific heats of rhombic sulfur in carbon disulfide. The surface energy of solid rhombic sulfur. A. R. Williams, F. M. G. Johnson and O. Maass. *Can. J. Research* 13B, 240-8 (1935). —The heats of soln. of rhombic S in CS<sub>2</sub> were measured over the concn. range 6 to 17% S and at 20° and 25°, and the sp. heats of these solns. calcd. The app. designed for the measurements is described. By measuring the heat of soln. of finely divided S and its particle size, the surface energy of solid rhombic S was estd. J. W. Shipley

Energy of dissociation of carbon monoxide. Paul Goldfinger, Wladimir Lasareff and Boris Rosen. *Compt. rend.* 201, 928-69 (1935), cf. C. A. 29, 6730. —The various methods of deducing D<sub>CO</sub> (the energy of disson. of CO into normal atoms), and hence L<sub>C</sub> (the heat of sublimation of C at 0° abs.) are discussed, and it is shown that D<sub>CO</sub> = 9.10 e. v. with L<sub>C</sub> = 123.6 kg.-cal., are the only values consistent with those deduced from them for D<sub>CO</sub>\* (6.1 e. v.), D<sub>CO</sub> (5.9 e. v.) and the mean energy of each C-H link in CH<sub>4</sub> (110 kg.-cal.) (cf. C. A. 29, 6133).

C. A. Suberrad

Heats of transition of triglycerides. M. M. Ram Rao and S. K. Kulkarni. *J. Indian Chem. Soc.* 12, 574-81 (1935). —The work was based on the assumption that the triglycerides existed in only 2 forms and that the metastable solid obtained on sudden cooling was a pure phase. The heats of soln. of the 2 forms of tristearin, trilaurin and tripalmitin were detd. in different solvents by means of a double-calorimeter method. The difference in each case was 15.4 cal./g. and was the same as the difference between the latent heats. H. E. Phipps

Heat of combustion of isobutane. Frederick D. Rossini. *J. Research Natl. Bur. Standards* 15, 357-61 (1935) (Research Paper No. 833); cf. C. A. 29, 6730. —Calorimetric data on the reaction, iso-C<sub>4</sub>H<sub>10</sub>(g) + 13/2 O<sub>2</sub>(g) = 4 CO<sub>2</sub>(g) + 5 H<sub>2</sub>O(l), at 25° and a const. pressure of 1 atm., yield ΔH = -2871.06 ± 0.53 international kilojoules per mol. (measured as 5 × 18.0156 g. H<sub>2</sub>O). With the factor 1/4 1833, the value is -695.31 ± 0.13 kg.-cal. per mol. The isobutane used was estd. to be of such purity that its heat of combustion would differ from that of an absolutely pure sample by less than 0.01%. F. D. R.

Determining molecular weights of gases and vapors. Hamilton P. Cady. U. S. 2,023,164 Dec. 3. A method of detg. the d., sp. gr. or mol. wt. of a gaseous material includes the step of varying the temp. of the gaseous material being observed responsive to atm. pressure while ascertaining the d. App. and various operative details are described.

## 3—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

W. ALBERT NOYFS, JR

Present-day views on the constitution of matter. Max Cosyns *Bull soc Belge ing ind* 1935, 754-64.

E. H.

Can quantum mechanical description of physical reality be considered complete? N Bohr *Phys Rev* 48, 696-702(1935).—The "criterion of physical reality" formulated by Einstein, Podolsky and Rosen (*C. A.* 29, 5007) contains an essential ambiguity when it is applied to quantum phenomena. This ambiguity is illustrated for several special problems, including the one considered by E. P and R. A viewpoint termed "complementarity" is explained from which quantum mechanical description of physical phenomena would seem to fulfill, within its scope, all rational demands of completeness. L S K.

The energy levels of inert gas configurations. C L. Barthelet *Phys Rev* 48, 682-3(1935).—Math

L S Kassel

The value of the electronic charge. Raymond T. Birge *Phys Rev* 48, 918(1935), cf *C A* 29, 4664.—A recent redetermination of the viscosity of air brings the oil-drop and grating values of  $e$  [ $(4.816 \pm 0.013) \times 10^{-10}$  e s u,  $(4.8036 \pm 0.0005) \times 10^{-10}$  e s u], resp] into essential agreement. Probable values of  $e/m$ ,  $h/e$  and  $1/e$  are discussed. G M P

Determination of  $h$  (Planck's constant). G. Schatzberger. *Ann Physik* 24, 84-88(1935).—Disagreement between the results of Duane, Palmer and Yeh (*C A* 10, 2806) and Feder (*Ann Physik* 1, 497(1929)) occasioned a new determination of  $h$ . The so-called isochromatic method was applied. This consists in standardizing the crystal and ionization chamber in a Bragg spectrograph on a definite wave length and measuring the reflected radiation at different potentials in the region of the short-wave limit. The short-wave limit is obtained by interpolation. Apparatus and method are described. Values for  $h$  at different wave lengths are tabulated, the av was 6.553;  $= 0.002 \times 10^{-10}$  erg/sec.  $h$  was also calculated from the relation  $h/e = V/\nu$ . These values are compared with previous ones and errors are discussed. Allen S. Smith

Symmetry considerations concerning the splitting of vibration-rotation levels in polyatomic molecules. E. Bright Wilson, Jr. *J Chem Physics* 3, 18-21(1935); cf *C A* 29, 4262.—The interaction of rotation and vibration and perhaps other effects may split degenerate vibration-rotation energy levels of sym polyat mols into a no of components. The permutation symmetry of mols containing several identical atoms provides certain restrictions on this splitting. This paper discusses the maximum no of fine-structure components, their quantum numbers when nuclear spins are taken into account, and the selection rule for transitions. All arguments are based solely on symmetry considerations so that no use of the magnitude of the splitting is given. G M P.

Electronic states of the carbon atom and calculation of the bond energies. Vladimir Lasareff, *Physica* 2, 737-42(1935).—By combining results of Edlen (*Z Physik* 84, 746(1933)) and of Bacher and Goudsmid (*Phys Rev* 46, 948(1934)) the energy difference  $\Delta E$  between  $^3P$  and  $^1S$  for C is taken to be  $4.2 \pm 0.2$  e v. From this is calculated for CN  $CN(^3\Sigma) = C(^1S) + N(^1S) - 11.9 \pm 0.7$  e v. This is higher than the spectroscopic values of 7.0 and 9.7 e v, hence  $CN(^3\Sigma)$  dissociates into  $C(^1P) + N(^1S)$  with energy  $7.7 \pm 0.5$  e v (cf Mulliken, *C A* 26, 3182). It is assumed that in diamond C is in the quadrivalent  $^1S$  form and the calculated heat sublimation of  $C(^1S)$  into the bivalent vapor  $C(^1P)$  is  $7.20 \pm 0.1$  e v, formation of quadrivalent vapor  $C(^3P)$  requires  $11.46 \pm 0.3$  e v. From this is concluded that the energy of the bond C—C in diamond is  $5.73 \pm 0.15$  e v. ( $132 \pm 3.5$  kcal), in aliphatic chains  $5.56$  e v ( $128$  kcal). B J C van der Hoeven

Electric discharges in vacuum and in gases at low pressures. I Langmuir. *Gen Elec. Rev.* 38, 514-20(1935).—A detailed review. C. G. P.

Discharge processes in gases prior to breakdown. E. Hegler and H. Raether. *Naturwissenschaften* 23, 501(1935).—The ionization process in an impulse discharge ( $10^{-7}$  sec. period) was studied by cloud chamber at different pressures with point or flat electrodes. Channeling always precedes the spark. The no of channels increases with potential and duration of impulse, the channels widen for lower pressure. B J C. v. d. H.

The mechanism of unimolecular electron capture. Felix Bloch and Norris E. Bradbury *Phys Rev* 48, 689-95(1935).—The formation of negative ions by electron capture in gases in which a dissociation process does not occur is explained by a unimolecular process involving excitation of molecular vibration levels and subsequent energy loss by collision or resonance. The quantum-mechanical theory for such a process is developed. Comparison with experiment then shows that the vibrational quantum number changes by only 1 unit, this in turn sets an upper limit of  $0.17$  v for the electron affinity of the O molecule. The theory also gives a dependence of calculated capture cross section on average electron energy which agrees with the experimental results. L S Kassel

The role of space charge in the study of the Townsend ionization coefficients and the mechanism of static spark breakdown. R. N. Varney, H. J. White, L. B. Loeb and D. Q. Posin *Phys Rev* 48, 818-22(1935). L S K.

Photoionization in gases. R. N. Varney and L. B. Loeb *Phys Rev* 48, 822-4(1935).—No photoionization of N, A, Xe or air could be produced by radiation passing a fluorite window. In the absence of any window A and Xe are photoionized by their own radiation, but N and air are not. L S Kassel

The energy distribution of electrons in the photoelectric effect. Erik Rudberg *Phys Rev* 48, 811-17(1935).—The results of various theoretical calculations of the energy distribution of photoelectrons from metals are compared with experiment. The predicted proportion of low-energy electrons is always too high. This low-energy region, where the Fermi factor is nearly unity, is the important one for studies of the electronic structure of metals. L S Kassel

Photoelectromotive forces and currents in single crystals of selenium. R. M. Holmes *J Optical Soc. Am.* 25, 326-9(1935).—Lamellar crystals grown from Se vapor, having a surface area of about  $75$  sq mm and a thickness of about  $0.12$  mm, were used. Translucent Pt films sputtered on portions of the flat surfaces were the electrodes. When there was no source of e m f in the external circuit, light on one of the Pt films caused an electron current to flow from Se to Pt at the illuminated surface. Curves are given showing the variation of current, e m f and resistance of this photoelectric cell with illumination, also the variation of current and e m f with wave length of the light used. The photo-current and photo-e m f decrease to zero near the red end of the visible spectrum. An interpretation of the results is given. H. S. H.

Theory of some photoelectric and photomagneto-electric phenomena in semiconductors. Ya. Frenkel *Physik Z. Sowjetunion* 8, 183-203(1935), cf *C A* 28, 5326.—Equations are derived for the no of free electrons and positrons in an intrinsic semiconductor (such as Se), and applied to the theory of the Dember effect for strong and for weak illuminations, and also to the photomagneto-electric effect of Kikoin and Noskov (*C A* 28, 5326).

Helen S. Hopfield

Action of ultraviolet light on electric conductivity of quartz. Reza Radmanesh. *Compt rend* 201, 1021-2(1935), cf *C A* 29, 6810.—A current was passed through a plate of quartz  $20 \times 20 \times 2$  mm enclosed in a dry atm when the quartz was and was not illuminated by light from a Hg lamp, the contacts between quartz and electrodes being protected from the light. With potentials of 180, 90 and 60 v between the electrodes the current steadily increased on illumination, attaining a maximum of

approx. 10, 8 and 3-fold after about an hr.'s exposure.

C. A. Silberad

**Diffraction of electrons by metal crystals and by mica.** J. A. Darbyshire and E. R. Cooper. *Proc. Roy. Soc. (London)* A152, 104-23 (1935); cf. C. A. 28, 2260<sup>3</sup>, 29, 993<sup>1</sup>.—The object of the research was to exam the spot patterns obtained when an electron beam of 30 kv velocity is transmitted through a cryst. film. The first section of the paper is a brief review of the work relating to such patterns, the reciprocal lattice picture of electron diffraction is presented in a form suitable for discussing the results. Films of the oxides of the metals Zn, Cd, Bi and Al were prep'd by removing the surface skin from the melt by means of a Cu wire loop whose diam was 0.5 cm. The films were formed in an atm having an excess of N<sub>2</sub> and the temp of the metal was held just above the m p. Debye-Scherrer oxide patterns were always obtained and occasionally spot patterns due to metal crystals caught up on the oxide film. Such spot patterns from Zn, Cd and Bi seem to be due to an aggregate of crystallites varying very little in orientation and presumably arising from the distortion of single crystals. The spot patterns from Al seem to be due to aggregates. Forbidden spectra are observed whose source is most plausibly explained by successive reflections by different crystallites. Spot patterns from curved sheets of mica support the conclusion that such patterns in metals are due chiefly to distortion of the diffracting crystal.

H. A. Smith

**Inhomogeneous fields for mass spectroscopy** S. H. Bauer. *Phys. Rev.* 48, 917 (1935).

G. M. P.

**Proton-proton forces in anomalous scattering and in nuclear binding.** R. D. Present. *Phys. Rev.* 48, 919-20 (1935).—White's data (C. A. 29, 3592<sup>4</sup>) on the scattering of 600-750 kv protons in H are incompatible with data calcd. from the Feenberg-Knipp nuclear model.

G. M. P.

**The effect of primary cosmic ray energy upon burst production.** W. F. G. Swann and D. B. Cowie. *Phys. Rev.* 48, 649-52 (1935).—The usual coincidence technic was used to det the direction of the cosmic-ray assoc'd with a given burst. The numbers of cosmic-ray counts obtained by the vertical and 45° counters were in the ratio of 1.5:1, the associated bursts in the ratio 10:1. This result, as well as the relatively greater burst production previously found on Pike's Peak, suggests that the efficiency of burst production increases rapidly with the energy of the primary particle.

L. S. Kassel

**The showers of rays which produce bursts of cosmic-ray ionization.** C. G. Montgomery and D. D. Montgomery. *Phys. Rev.* 48, 786-9 (1935).—By the application of statistical methods it is shown that there is no sharp distinction between the sprays of small nos of cosmic rays usually observed by several counters out of line and referred to as showers, and the larger groups found with ionization chambers and called bursts or Stöses.

L. S. Kassel

**The corpuscular theory of the primary cosmic radiation.** W. F. G. Swann. *Phys. Rev.* 48, 641-8 (1935), cf. C. A. 29, 4667<sup>2</sup>.—S. proposes a theory in which the primaries are not directly observed by any ionization, but only through secondaries produced with a linear d proportional to the primary energy. A list of 11 facts concerned with cosmic-ray absorption, latitude and directional effects, burst and shower production is made; this list includes all the important facts known about cosmic rays, and it is shown that the proposed theory can account for all of them.

L. S. Kassel

**The radioactivity of potassium.** G. v. Hevesy. *Naturwissenschaften* 23, 583-5 (1935); cf. C. A. 29, 2439<sup>1</sup>.—The  $\beta$ -radioactivity of K is reviewed in the light of artificial radioactivity. In the preterrestrial era radioactive K<sup>40</sup> and Rb<sup>87</sup> were formed, both of very long life period (order of 10<sup>8</sup> to 10<sup>10</sup> yrs). The traces remaining at present cause the above effect.

B. J. C. van der Hoeven

**Scattering of slow neutrons.** Allan C. G. Mitchell and Edgar J. Murphy. *Phys. Rev.* 48, 653-6 (1935); cf. C. A. 29, 5343<sup>1</sup>.—With the radioactivity produced in Ag foil as a neutron detector, the scattering of slow neutrons by Fe, Cu, Pb, Sn and Hg is det'd. Curves of

percentage scattering against thickness of scatterer are given, and the relative scattering cross-sections calcd.

L. S. Kassel

**Scattering of slow neutrons by iron and other substances.** D. Budnitskii and I. Kurchatov. *Physik. Z. Sowjetunion* 8, 170-8 (1935).—The length of the free path of slow neutrons in C, Fe, Cu and Pb was measured by using their reflection and was found smaller than the free path of fast neutrons. The values found agree with those of Dunning and others except for graphite. In calog the free path, it is necessary to take into account the possible selective sensitivity of the indicator used and the absorption of neutrons in water.

Helen S. Hopfield

**Disintegration of lithium by lithium ions.** V. Petuhov, K. D. Sinelnikov and A. Val'ter. *Physik. Z. Sowjetunion* 8, 212-14 (1935).—A Li oxide target was bombarded with Li ions accelerated to an energy of 1.2 million e. v. A homogeneous group of particles of range 8.5 cm. was emitted by the target. However, it is thought that these are due to protons in the ionic beam, and not to Li ions.

Helen S. Hopfield

**The radioactivity induced in oxygen by deuterium bombardment.** Henry W. Newson. *Phys. Rev.* 48, 790-6 (1935).—Bombardment of O by 3-m e. v. deuterons gave a F isotope with positron radioactivity of half-life 1.16 min. The probable reactions are O<sup>16</sup> + D<sup>2</sup> = F<sup>17</sup> + n<sup>1</sup>, F<sup>17</sup> = O<sup>17</sup> + e<sup>+</sup>. The same active substance has previously been produced by  $\alpha$ -particle bombardment of N. The excitation efficiency is det'd for O, it drops sharply to zero at 2 m. e. v. This is explained by assuming a heat of reaction of -1.8 m e. v. This estimate is confirmed by detg. the max angle between the paths of the deuterium beam and the radioactive recoil atom. This method was checked by application to deuterium bombardment of C, the heat of reaction of -0.1 m. e. v. for C<sup>12</sup> + D<sup>2</sup> = N<sup>14</sup> + n<sup>1</sup> agrees well with values found in other ways.

L. S. Kassel

**Absorption of slow neutrons in iron.** I. Kara, L. Rosenkevich, K. D. Sinelnikov and A. Val'ter. *Physik. Z. Sowjetunion* 8, 215-18 (1935).—Radon with Be powder was used as a source of neutrons, and the measurements were made with very slow neutrons scattered back from a paraffin block. The induced radioactivity of the Ag detector was measured with a Geiger-Müller counter with mech registration. The absorption of slow neutrons by Fe and Cu was found to be very small.

H. S. H.

**Selective absorption of neutrons.** I. Kara, L. Rosenkevich, K. D. Sinelnikov and A. Val'ter. *Physik. Z. Sowjetunion* 8, 219-22 (1935), cf. preceding abstr.—Since Fe and Cu absorb slow neutrons much less than neutrons with medium velocities, the problem of selective absorption was investigated. Ra with Be was used as a source, and slow neutrons with different velocities were obtained by dissolving boric acid in water. A table and a graph show the results. Apparently the av. values of the sensitivity for B, Ag and Cu be consecutively one above the other, B being the lowest. The optimum absorption value for B is probably very near to the zero velocity of neutrons, and the max for Ag may be near zero also. The effective cross-section for absorption decreases rapidly with increasing velocity of the neutrons, which is to be expected on theoretical grounds.

Helen S. Hopfield

**After-effect of aluminum bombarded by electrons.** Masamichi Tanaka. *Phys. Rev.* 48, 916 (1935).—Al was bombarded by 250-300 kv. electrons. It gave off an electron radiation, with half-value periods of 7 sec., 40 sec. and 10 min. The 40-sec group had a max. energy of 2.5 kilo-e. v.

G. M. P.

**X-ray light source in single crystals.** G. Bornmann. *Naturwissenschaften* 23, 591-2 (1935).—A monocryst. ball of Cu at the proper distance (several cm.) from the photographic film gives a Laue x-ray diagram by reflection of the Cu K $\alpha$  radiation excited by x-rays in the crystal interior on the various crystal faces.

B. J. C. v. d. H.

**A two-crystal spectrometer for x-rays of wave length 0.030 <  $\lambda$  < 0.215 Å.** T. R. Cuykendall and M. T. Jones. *Rev. Sci. Instruments* 6, 356-61 (1935). E. H.

Energy bands in copper. Harry M Krutter. *Phys. Rev.* 48, 664-71 (1935).—Math. The electronic energy levels in the Cu lattice are calcd by use of a corrected Hartree field. The model used accounts theoretically for the high cond of copper, but cannot give quant. results.

The computation of spectral intensities for hydrogen. Philip Rudnick. *Phys. Rev.* 48, 807-11 (1935).—Math.

Hyperfine structure in selenium, palladium and gold. L Sibuya. *Proc. Indian Acad. Sci. A*, 313-10 (1935).—Hyperfine structure analysis of some Se and Pd lines shows that none of the levels examd reveals any even isotope displacement. The nuclei of Se 77 and Pd 105 have very small magnetic moments and their spin moment is probably  $(1/2)h/2\pi$ . The doublet structure ( $\Delta\epsilon = 0.224 \text{ cm}^{-1}$ ) observed by Ritschl in the resonance lines of Au has been confirmed by the redoubling of each component due to self-reversal in the source, this test proves that the originally observed doublet structure does not arise from self-reversal as the earlier results of Frisch would suggest. While in the isoelectronic spectrum of Hg II the  $5d^9 6s^2 D_{3/2}$  level exhibits isotope displacement, arc lines of Au involving this level point definitely to the existence of a single isotope of mass 197; the accepted chem. at wt is therefore considered to be too high. The nuclear spin moment of Au is  $(1/2)h/2\pi$  and the  $g(I)$  factor comes out as 0.130, agreeing with Landé's theoretical value.

The hyperfine structure of europium. H Casimir. *Physica* 2, 719-23 (1935).—The irregularities in the Lu hyperfine spectrum explained by Schuler and Schmidt (*C. A.* 29, 5346) from nonspherical symmetry of the nucleus are further worked out on this basis.

Doubly excited states of helium—a correction. Ta-You Wu and S T Ma. *Phys. Rev.* 48, 917 (1935), cf. *C. A.* 28, 6629.

Ultraviolet resonance series of sodium. A Seidel. *Physik. Z. Sowjetunion* 8, 201-5 (1935).—On excitation of Na vapor heated to 500-50° by different metal lines (Ag, Cu and Zn), resonance spectra of the mol. Na<sub>2</sub> are observed near the second member of the principal series of Na.

The spectrum of trebly ionized cerium. R. J. Lang. *Can. J. Research* 13A, 1-4 (1935).—The spectrum of Ce has been photographed from 3300 to 500 Å by means of a two-meter grating of 30,000 lines per in. mounted in a vacuum spectrograph, with the vacuum spark and the spark in N as sources. An analysis of the spectrum of Ce IV results in the location of the 6S-6P, 6P-6D, 6P-7S, 6D-6P, 6P-8S and possibly the 6P-7D multiplets. The ionization potential is approx. 33.3 v.

Deepest terms in ions of the isoelectronic sequence A I—Mn VIII. P Gerald Krüger and S G Weissberg. *Phys. Rev.* 48, 659-63 (1935).—A number of new terms in this sequence are detd. and correlated by means of Moseley diagrams.

The spectrum of the zinc arc in vacuum. Charles William Hetzler, Robert W Boreman and Kevin Burns. *Phys. Rev.* 48, 656-0 (1935).—Sixty lines in the spectrum of the vacuum Zn arc were observed, including all known solar lines of Zn and the stronger lines of Pb, Cu, Cd, Ag, Sn, Na, K, Rb, Cs, Sr and Be. A source for obtaining weak lines in vacuum is described.

The spectrum of doubly ionized zinc. Sudhendu Basu. *Indian J. Physics* 9, 537-44 (1935).—This spectrum was analyzed in the region 5313-2387 Å. Twenty-three new terms were obtained, mostly from the configuration  $3d^4 4d$  and over 100 lines were accounted for.

Suggested new interpretation of the structure of band spectra. S. Bhagavantam. *Proc. Indian Acad. Sci. A*, 32, 102-100 (1935).—It is assumed that in the process of electronic excitation the atoms become elec. doublets whose axes can become either parallel or perpendicular to the nuclear axis. In the former case the doublets attract each other with consequent diminution of the nuclear sepn and in the latter the nuclear sepn is increased. By

adding the mutual potential energy of the doublets to the nuclear potential energy function it is shown that the creation of doublets may be regarded as equiv. to an increase in the vibrational frequency of the mol. if they are of the attractive type and a decrease if they are of the repellent type. An independent method of calcg the strength of the induced elec. moments is given on the assumption that the energy of electronic excitation is all spent in polarizing atoms.

Continuous spectrum of deuterium. Adolphe Tournaire and Etienne Vassy. *Compt. rend.* 201, 957-8 (1935).—The spectrum of D<sub>2</sub> has been examd for  $\lambda\lambda$  4861-2300 Å. For  $\lambda$  greater than 4000 Å it is practically identical with that of H<sub>2</sub>. For shorter  $\lambda$  there is a slight difference increasing as  $\lambda$  diminishes, the continuous spectrum of D<sub>2</sub> being slightly more intense than that of H<sub>2</sub>.

Rotational structure of the Schumann-Runge bands of oxygen in the vacuum region. Harold P. Knaus and Stanley S. Ballard. *Phys. Rev.* 48, 706-9 (1935).—Rotational analysis of these bands photographed with a 3-meter grating gives new upper state consts. for the range  $v' = 8$  to 15. The energy of disson. of the normal O mol. to normal atoms, D<sub>2</sub>, is 0.05 v.

The absorption of liquid oxygen. P. Tulipano. *Nuovo cimento* 12, 418-22 (1935).—The visible absorption spectrum of liquid O<sub>2</sub> is as follows, where the first figure is the wave no. (in  $\text{cm}^{-1}$ ) of the max. and the second is the absorption coeff. 15,858, 0.228, 17,296, 0.274, 18,813, 0.074, 20,827, 0.084, 22,425, 0.028, 26,259, 0.054, 27,685, 0.214. A Carrelli. *Ibid.* 423-5.—The data of Tulipano (cf. above) indicate the existence of mols. assocd. as a result of polarization.

The energy of formation of negative ions in oxygen. Leonard B. Loeb. *Phys. Rev.* 48, 684-9 (1935).—Electrons and neg. ions from an arc in purified O were driven by a steady d.c. potential through parallel wire grids S<sub>1</sub>, S<sub>2</sub> to a collector plate P. Transverse, high-frequency a-c potentials at the grids caused capture of most of the electrons reaching them. The currents reaching S<sub>1</sub> and P were detd. for various gas pressures and a-c frequencies. Increase in the current to S<sub>2</sub> and simultaneous decrease in that to P represents break-up of the neg. ions in the d-c. field between S<sub>1</sub> and S<sub>2</sub>. The actual curves are very complex, but the break-up seems to occur when the ratio of field strength in v./cm. to gas pressure in mm. is approx. 90. This value corresponds to an ion energy of not over 0.68 v. Since not over half of this is available for electron detachment, the probable upper limit for the energy of formation of the neg. ion is 0.34 v., which might correspond to a wave length of 36,000 Å.

The electron affinity of iodine from space-charge effects. Geo. Glocker and Melvin Calvin. *J. Chem. Physics* 3, 711-7 (1935).—The electron affinity of I atoms was detd. by a direct method in which only 1 atom and electrons were involved. The value obtained, 74.6 kcal., is in good agreement with values obtained by other methods. The consens. of the several ions of different masses ( $I^-$  and  $E^-$ ) were calcd. from their effects on space charge. I has no effect on the thermionic emission of W.

Potential-energy curves and structure of the alkaline earth oxides. P. C. Mahanti. *Indian J. Physics* 9, 517-36 (1935).—Curves for different electronic states of BeO, MgO, BaO, SrO and CaO are drawn by the method of Morse. The electronic configurations and disson. products are discussed in each case.

A new afterglow spectrum in nitrogen. Joseph Kaplan. *Phys. Rev.* 48, 800-1 (1935).—See *C. A.* 29, 6141.

Predissociation in the sulfur bands. Maurice Déchant and Jules Duchene. *Phys. Rev.* 48, 916 (1935).—Absorption spectra of sulfur vapor. Louis D. Or. *Compt. rend.* 201, 1023-8 (1935).—The spectrum of S vapor in the visible and near ultraviolet has been examd under conditions in which the concn. does, and does not vary with temp. Previous results are not confirmed. The spectrum consisting of bands without fine structure between 3600

and 4200 Å., attributed by Graham (C. A. 5, 422) to  $S_2$  and  $S_3$ , is definitely not due to these mols., but to some intermediate mol. probably  $S_4$ , or perhaps  $S_5$  (cf. Dobbie and Fox, C. A. 13, 1965). C. A. Silberrad

Rotation structure of band systems  $D$  and  $E$  of cuprous chloride. Jean Ternier. *Compt. rend.* 201, 1029-33 (1935).—The band spectrum of  $\text{CuCl}$  is conveniently obtained by passing a c. at 2000 v through  $\text{CuCl}$  vaporized in an evacuated Pyrex tube. A detailed account of systems  $D$  and  $E$ , examd. by means of a grating giving dispersion of 1 Å./mm. is given, showing clearly the isotopic effect in conformity with theory (cf. Ritschl, C. A. 21, 1931). C. A. Silberrad

Ultraviolet absorption of mixtures of  $\text{NO}$ ,  $\text{NO}_2$  and  $\text{H}_2\text{O}$ . Eugene H. Melvin and Oliver R. Wulf. *J. Chem. Phys.* 3, 755-9 (1935); cf. C. A. 26, 1514.—In  $\text{NO}$  contg. small amts. of  $\text{NO}_2$  a continuous absorption occurs in the ultraviolet, which is due to  $\text{N}_2\text{O}_2$ . This obscures the absorption of  $\text{NO}$  and that part of the absorption of  $\text{NO}_2$  which lies below 2500 Å. With small amts. of  $\text{H}_2\text{O}$ , a group of bands occurs in the near ultraviolet, extending from 3850 Å. to shorter wave lengths. They are diffuse, but possess an ordered arrangement, and decrease in intensity with increasing temp. The first members are broader and more diffuse than those that follow, indicating a predissociation process in the carrier, which is probably  $\text{HNO}$ . G. M. P.

Normal vibrations of heavy acetylene and of the ethylene halides. Yonezo Morino and San-churo Murakami. *Physik. Z.* 36, 600-2 (1935).—Valence-force calcs. are given for  $\text{C}_2\text{D}_2$ ,  $\text{C}_2\text{H}_2$ ,  $\text{C}_2\text{H}_2\text{Cl}_2$  and  $\text{C}_2\text{H}_2\text{Br}_2$ . The agreement with expt. (Raman spectra) is good in the first two cases. The calcs. in the case of the last two mols. are used as a basis of assignment of some observed Raman lines. A. B. F. Duncan

Hindered rotation and oscillation of molecules in liquids and in crystals. S. Bhagavantam. *Proc. Indian Acad. Sci.* 2A, 65-6 (1935).—Wings on either side of Rayleigh lines are interpreted as Raman radiations somewhat analogous to rotational Raman spectra. The extended portions of the rotational wings in liquids are due to small oscillatory motions of the mols. about their equil. positions. In liquids there is only an imperfect quantization on account of a quasicrystalline arrangement. J. H. H.

Vibrations of benzene and Raman spectra of benzene- $d_6$  and benzene- $d_5$ . O. Redlich and W. Stricks. *J. Chem. Phys.* 3, 824 (1935). G. M. P.

The photographic infrared spectrum of methylacetylene and the distance between centers in the C-C bond. G. Herzberg, F. Patat and H. Verleger. *Physik. Z.* 36, 625 (1935).—An absorption band at  $1.03 \mu$  was analyzed, single P, Q and R branches were found. The mol. is a symmetrical top with the CC-CH group linear. The sep. of centers in the C-C bond is  $1.525 \pm 0.003$  Å. A. B. F. Duncan

Doppler effect in light scattering in liquids. II. Polarization of the transversely scattered radiations. B. V. Raghavendra Rao. *Proc. Indian Acad. Sci.* 2A, 236-41 (1935).—A study has been made of the state of polarization of the 3 components observed in the spectrum of the scattered light when examd. with a Fabry-Perot etalon. Three typical liquids,  $\text{CCl}_4$ ,  $\text{PhCH}_3$  and  $\text{CS}_2$ , have been examd. The results indicate that the 2 Doppler components are completely polarized, as is to be expected, and that the central component is practically completely polarized. The significance of the latter result, which appears surprising at first sight, is discussed in some detail. Harold Gershonowitz

The Raman spectra of the isotopic molecules  $\text{H}_2$ , HD and  $\text{D}_2$ . Gordon K. Teal and Geo. E. MacWood. *J. Chem. Phys.* 3, 780-4 (1935).—Raman spectra are reported for  $\text{H}_2$ , HD and  $\text{D}_2$  at 3 atm. and 32°. The positions of the pure rotation lines for  $\text{H}_2$  agree with Rasetti's measurements (C. A. 24, 789); the lines in the Q vibration band do not. Calcd. and observed results are in good agreement. Contrary to theoretical predictions, the 0,0 line of the Q vibrational band of HD was observed. A change in the external pressure from 505 to 745 mm. caused

Raman spectrum of carbon disulfide. A. Veerabhadra Rao *Proc. Indian Acad. Sci. 2A*, 46-53 (1935).—The intensity and polarization of Raman lines of  $CS_2$  were detd. The Q branch of  $\Delta\nu=636$  cannot be sep'd from the PP and RR branches. The depolarization is 0.15 and is increased to 0.2 by progressing from  $\lambda$  4358 to  $\lambda$  4946.

J. H. Hibben

The Raman spectra of dioxane and tetralin. C. S. Venkateswaran *Proc. Indian Acad. Sci. 2A*, 279-99 (1935).—The Raman spectra of dioxane and tetralin have been obtained with the filter technique. The spectrum of dioxane consists of 24 lines, of which 14 are reported for the first time, and resembles closely that of cyclohexane. Tetralin has also given 11 new lines. The results are discussed with reference to the structure of the mols.

Harold Gershinowitz

The Raman spectra of cis- and trans-decalins. S. K. Kulkarni *Jatkar Indian J. Physics* 9, 545-551 (1935).—The Raman spectra of cis- and trans-decalin mixts show that the cyclohexane ring is not in one plane. The trans form is considered more sym. because of its smaller number of frequencies. The shift  $750\text{ cm}^{-1}$  is broad in a mixt of the two forms, showing that the characteristic frequency of the fused rings is slightly different in the two forms.

A. B. F. Duncan

Fluorescence in cyclohexane. R. Padmanabhan. *Proc. Indian Acad. Sci. 2A*, 209-12 (1935).—An app. for continuous distn. has been described which works satisfactorily and is free from the defects of the arrangement described by Pal and Sen Gupta (*C. A.* 25, 1159). With the help of this app. pure cyclohexane is shown to have no fluorescence, that reported by Haberl (*C. A.* 29, 1711\*) has been proved to be due to products of photochem. decompn. The wave lengths in the region  $\lambda = 2490-2600\text{ Å}$  have been found to be active in the above reaction.

Harold Gershinowitz

A blue fluorescing substance from yellow carrots. F. H. Cohen *Chem. Weekblad* 32, 441 2 (1935).—Aq. ext. from carrots, with 3% HCl, twice adsorbed on frankonite, extd. the second time with  $H_2O$ -EtOH-pyridine (4:1:3), has a strong blue fluorescence. The substance is  $CHCl_3$ -sol but does not fluoresce in this solvent, it can be extd. therefrom by soda soln. After 48 hrs in daylight the fluorescence decreases to one-half the intensity; it increases by addn. of dil. alkali,  $NH_3$  or  $Na_2CO_3$ , and does not change with AcOH. In 0.5 N HCl the shade is dark blue and the intensity decreased. The fluorescence disappears irreversibly in 24 hrs. in the presence of 0.1 N NaOH, reversibly by Na hyposulfite treatment, is not affected by heating in neutral soln. to  $60^\circ$ .  $KH_2I_2$ , lead acetate or basic lead acetate does not give a ppt. and

$FeCl_3$  gives no violet color. The substance is not a decompn. product of lactoflavin. It has not been identified as yet. B. J. C. van der Hoeven

The fluorescence of pyrimidine, purine and pyrimidine derivatives. H. v. Eijler, K. M. Brandt and G. Neumüller. *Biochem. Z.* 281, 206-14 (1935). S. M.

Ultraviolet absorption and color-center formation in alkali halide crystals. Ernst Rexer *Physik. Z.* 35, 602 (1935).—The absorption coeffs. in the main absorption band of KBr increase with pressures of 800 g/sq. mm. At the same time the intensities of the satellite bands at longer wave lengths are increased. This latter absorption is connected with the color centers formed, which are probably atoms. A. B. F. Duncan

The inversion of cane-sugar solutions in tropical sunlight. N. A. Yajnik, D. N. Goyle and M. L. Wadhwa. *Z. anorg. allgem. Chem.* 225, 24-8 (1935).—The sugar solns. were placed in a Jena-glass flask in a glass beaker contg. water inside another beaker filled with water at the same temp. Const. temp. was maintained by passing cold water through the outer beaker. The entire app. was placed in direct sunlight, and it was shown that the light alone would cause the reaction to take place but the velocity const. is small. In the control expt. the flask was blackened. The effects of HCl and  $H_2SO_4$  on the velocity const. in the dark and in the sunlight were measured and the light was found to increase the rate slightly. Aq. solns. of inorg. salts and dyestuffs were used as light filters in the inner beaker and ranges found for the max. inversion of the soln. These varied with the catalyst used.

E. R. Rushton

The presence of metastable active oxygen molecules in sensitized photooxidations. Joseph Weiss. *Naturwissenschaften* 23, 610 (1935).—Results of Gaffner (*C. A.* 29, 7813\*) and Frauck (*Z. physik. Chem.* B27, 409 (1935), cf. *C. A.* 29, 5021\*) were confirmed by expts. on quenching of the fluorescence of dye solns. (eosin, uranin) by sulfite or arsenite. The dye absorbs  $h\nu$ , the excited dye mol. can either give off light or transfer the quantum to the sulfite substrate or liberate photochem. H atoms and form  $HO_2$ ; this formation explains certain phases of the expts. of Kautsky (*C. A.* 27, 1023). The  $HO_2$  radical thereby fulfills the role of "active" O. B. J. C. van der Hoeven

New investigations on the photoresistance of metals. Quirino Mayrona. *Nuovo cimento* 12, 4/9-17 (1935), cf. *C. A.* 27, 2620.—Further expts. confirm the existence of the effect and show that it is not merely a thermal action. J. B. Austin

Speed of thermal decompn. of chloropicroin (Räddlesén, Zamfirescu) 2

#### 4-ELECTROCHEMISTRY

COLIN G. FINE

The influence of molten aluminum on firebrick lining in an electric furnace. A. N. Lusenko. *Legkie Metal* 4, No 5, 8-13 (1935).—In remelting Al in an elec. resistance furnace with a firebrick lining, the Al penetrated into the porous brick, reduced the  $SiO_2$  and  $Fe_2O_3$  and made the brick a conductor of electricity. The slagging of the walls progressed upward from the level of the metal to the top of the furnace where it short-circuited the Ni-Cr resistor units.

H. W. Rathmann

Electrically heated industrial furnaces. R. Boye. *Eng. Progress* 16, 265-72 (1935).—A review. E. H.

Operation of fused-electrolyte cells with an increased charge of alumina. A. I. Zhelezov and B. N. Maksimenko. *Legkie Metal* 4, No 5, 4-8 (1935).—The amt. of alumina charged at one time into 2 Hall cells at the Dnepropetrovsk plant was increased to 120-140 kg. (about 50-75%) for a 4-mo. period. As a result the output increased 1% and the consumption of cryolite decreased 10%, that of anodes 15%.

H. W. Rathmann

Production of titanium-aluminum alloys in fused-electrolyte cells. A. I. Zhelezov and B. N. Maksimenko

*Legkie Metal* 4, No 4, 24-22 (1935).—Alloys contg. up to 1.53% Ti were made by dissolving and electrolyzing a mixt. of  $Al_2O_3$  and  $TiO_2$ . A total of 2800 kg. of alloy was obtained with an av. daily output of 141.3 kg. per cell and a current efficiency of 73.5%. Ti segregated somewhat because of the large difference in sp. gr. of Ti and Al, this can be prevented by frequent stirring. H. W. R.

Oxidation of foam carbon with air (aluminum). B. I. Ivanov and B. N. Maksimenko. *Legkie Metal* 4, No 5, 14-18 (1935).—The foam formed in the electrolysis of the fused Al salt bath was crushed and heated in a muffle furnace to 700-900° to oxidize the C and recover the electrolyte. At 780° the C content was reduced from 12% to 0.84% in 1 hr. and to 0.24% in 2 hrs. The loss of electrolyte was insignificant. H. W. Rathmann

The best concentration of sodium chloride for the alkali-chlorine cell. D. V. Adamovich. *Khimiya* 7, 481-6 (1935).—A discussion with math. treatment of the advantages and disadvantages of the electrolysis of NaCl solns. of higher than the usual concn. of 300 g/l.

Chas. Blake



The electrolytic preparation of magnesium persulfate. Lin-Yü Li and Kin-Tchouan Pei. *Contrib. Inst. Chem., Natl. Acad. Peiping* 2, 1-20(1935).—An aq. soln. of about 60%  $MgSO_4$  was prep'd. by electrolyzing a  $MgSO_4$  soln. with Pt electrodes. Attempts to isolate the persulfate in solid condition were not successful. The best anodic current density was 70 amp./sq. dm., at a current efficiency of about 50%. It is essential to sep. the anolyte from the catholyte by an unglazed porcelain diaphragm. Production of Caro's acid is negligible. C. L. Tseng

Electrodeposition of tin alloys from alkaline stannate baths. R. G. Monk and H. J. T. Flingham. *Trans. Faraday Soc.* 31, 1460-8(1935), cf. Hothersall, Clarke and Macnaughtan, C. A. 28, 5344.—Satisfactory deposits of Sn-Ni alloys contg. up to about 25% Ni are obtainable at high current efficiencies from an alk. bath at 70-5° contg. 90-100 g. Sn as Na stannate, 2 g. Ni as K nickelocyanide, and 5 g. of KCN per l. with Ni anodes. Deposits contg. 18-25% Ni, obtained at about 0.15-0.5 amp./sq. dm., remain bright up to about 0.0005 in., then become mat. Their hardness is about 7 times that of electrodeposited Sn. Deposits of higher Ni content, obtainable only at lower current efficiencies, are much harder, but more brittle and less wear-resistant. Alloys contg. Sn and Sb in almost any proportion can be electrodeposited at practically 100% current efficiency from an alk. stannate-thioantimonate bath at 70-75° by currents from 0.1 to 10 amp./sq. dm. Only those contg. less than 50% Sb form satisfactory deposits as thick as 0.0005 in. and even these are brittle. L. W. Elder

Bright zinc. C. M. Hoff. *Metal Cleaning and Finishing* 7, 483-6, 490(1935).—In view of the present shortage of Cd and the resulting increase in its price Zn is pointed to as a logical substitute. The phys., chem. and rust-protective properties of Zn and Cd are compared and the factors pertaining to the electrodeposition of these 2 metals are discussed. D. Thuesen

Electrolytic lead plating of chemical apparatus. P. P. Belyaev and Ya. N. Birman. *Khimistrol* 17, 426-8(1935).—Various Fe objects can be directly electroplated with Pb by the use of  $H_2SiF_6$  electrolyte on addn. of ice,  $Be(OH)_2$ , and some  $CaCO_3$  to neutralize any  $H_2SO_4$ . In prep. the bath  $H_2SiF_6$  is neutralized with  $Pb$  carbonate or oxide, and the filtrate, dild. to a definite concn. and treated with carpenter's glue, is used in the electrolysis at room temp. Good results are obtained with the following: 87-291 g./l. of 0.5-1.5 N  $PbSiF_6$ , 18-72 g./l. of 0.25-1 N  $H_2SiF_6$ , 5 g./l. of 0.5%  $Be(OH)_2$ , and 1-2 g./l. of 0.1-0.2% glue.  $CaH_2(SO_4)_2$ ,  $HOC_2H_4SO_3H$  and  $MeC_2H_4(OH)SO_3H$  can be substituted for  $H_2SiF_6$ . Many tech. and economic advantages are claimed for the electrodeposition of Pb on Fe as compared with the hot-dipping process. Among them is that the Pb-electroplated objects can be stamped, machined and formed without distortion. The method, however, requires further development and refinement. Chas. Blanc

Almost instantaneous action of some colloids on electrolytic deposits of copper. Pierre A. Jacquet. *Compt. rend.* 201, 953-5(1935); cf. C. A. 29, 2093.—The distinction between the behavior in an electrolytic bath of pectones and protein on the one hand, and of gum arabic, gum tragacanth and dextrin on the other, is confirmed by expts. with Marie and Thon's contractometer (C. A. 25, 5100) with aq.  $CuSO_4$ . These show that only the pectones and proteins have immediate action on the deposition of Cu, the gums acting much more slowly and less powerfully, while dextrin has scarcely any effect at all. C. A. S.

The copper voltammeter (coulometer). F. E. L. Parsons. *School. Sci. Rev.* 17, 171-80(1935).—App. for demonstration and student work. O. Reimnuth

Alkaline plating baths containing ethanalamines. I. Copper plating from triethanolamine solutions. C. J. Brockman and A. L. Brewer. *Trans. Electrochem. Soc.* 69, 6 pp. (preprint)(1935).—The production of strike plates from solns. of Cu salts contg. triethanolamine is compared with those from other solns. A new soln. was developed which will not plate Cu by replacement when brought in contact with Fe. This soln. will in 70 sec.

produce a bright adherent Cu plate at 0.4 amp./sq. dm. capable of taking a subsequent good, adherent Cu plate from the com. acid  $CuSO_4$  bath. The soln. contains 15 g./l.  $CuSO_4$ , 51H<sub>2</sub>O, 10 g./l. Na oxalate and 22 cc./l. triethanolamine. C. G. F.

Vacuum discharge with cold cathode as the cause of delayed ignition of certain high-tension rectifier tubes. F. M. Penning and J. G. W. Mulder. *Physica* 2, 724-30 (1935).—A time lag in the starting of anode current in certain high-voltage Hg-arc rectifiers is explained from a negative charge of the glass wall around the anode. This charge is attributed to a vacuum discharge during the negative phase with anode as cathode. These discharges were studied in a special tube, they have characteristics of auto-electronic emission. B. J. C. v. d. H.

Electrotypers' waxes. Charles F. Mason. *Chem. Industries* 37, 443-4(1935). E. H.

Electrochem. methods in varnish and pigment industry (Parnilox) 26.  $H_2SO_4$  (Brit. pat. 433,676) 18. Coated ferrous wire, etc. (U. S. pat. 2,023,564) 9. Flue furnace for baking porcelain (Fr. pat. 757,009) 19.

Electric battery. Gustav Willmck. U. S. 2,023,717, Dec. 10. An anode of  $PbO_2$  and a cathode of an alloy contg. Sn 96, Pb 2 and Sb 2% is used with an electrolyte of 24%  $B_4H_4$ .  $H_2SO_4$  to each l. of which is added Cr sulfate 3 g., Na-Cr<sub>2</sub>O<sub>7</sub> 1 g. and  $SnSO_4$  30 g.

Primary electrolytic battery. Martin L. Martus and Edmund H. Becker. U. S. 2,023,515, Dec. 10.  $ZrO_2$  0.1-0.8% is used in the electrolyte of a primary cell of the type using a depolarizer and a caustic alkali soln. as electrolyte, and serves to improve cell operation. Some  $TiO_2$  also may be used.

Electric battery with zinc and carbon electrodes. Charles K. Bamber (to Bamberite Battery Co., Ltd.) U. S. 2,022,998, Dec. 3. Various structural details.

Dry cell. Bruto Laurenti. Ger. 617,785, Aug. 26, 1935 (Cl. 21b 7.01). A dry or semi-dry cell has a cylindrical Zn cathode, forming also the cell container, coated on its inner side with insol. animal gelatin. This forms an insulation from the anode rod which may be made of C.

Lead storage-battery plates. Charles F. Haunz (one-third each to Alexander McGary and George R. Berger). U. S. 2,023,170, Dec. 3. Structural features.

Active material for lead storage-battery plates. Alexander Stewart (to National Lead Co.). U. S. 2,022,482, Nov. 20. Pb oxide is used with a small proportion of solid org. material pptd. from waste sulfite liquor by the action of a water-sol. Pb compd. such as Pb acetate, etc.

Filling device for storage batteries. Asa H. Mosher. U. S. 2,022,380, Nov. 26. Structural and operative details.

Electrolytic condensers. Frank W. Godsey, Jr. (to Sprague Specialties Co.). U. S. 2,023,522, Dec. 10. A viscous electrolyte is used comprising a substance such as gluconic, mannonic or gulonic acid which acts both as a weak acid and as a viscous ionizing solvent of the electrolyte which also may contain an  $NH_4$  salt of the acid and water.

Electroplating apparatus suitable for plating various articles. Burton G. Daw. U. S. 2,023,668, Dec. 10. Structural and mech. details.

Electrolytic capacitors, etc. Frank M. Clark and John H. Koenig (to General Elec. Co.). U. S. 2,022,500, Nov. 26. For impregnating capacitor spacers, etc., a mixt. of salts is used which is noncryst. in the working range of temps. of the capacitor, such as a mixt. of  $NH_4$  borate and acetate.

Electric current rectifier filled with a rarefied gaseous medium such as mercury vapor. Walter Dillenbach. U. S. 2,023,166, Dec. 3. Various structural and elec. details.

Electric rectifier unit for electrolysis control. Edgar A. Hartv (to General Elec. Co.). U. S. 2,023,226, Dec. 3. Structural details of an app. suitable for control of electrolysis in pipe lines, etc.

Electrolytic production of alcohols and hydrocarbons. Walter H. McAllister (to Procter & Gamble Co.). U. S. 2,022,894, Dec. 3 For making alcohols or unsaturated hydrocarbons having one less C atom per mol. than the salt used as raw material, graphitic anodes are used in electrolyzing a soln. contg. a sol salt of an aliphatic acid having at least 6 C atoms per mol. such as K coconut oil soap and also contg. about 10-30% of a water-sol. aliphatic monohydric alk. such as EtOH and about 3.0-8.5% of an alkali metal chlorate, perchlorate, bicarbonate or sulfate. Numerous examples are given.

Electrolytic apparatus for making sheet metal. Allgemeine Elektrizitäts-Ges. Ger. 620,209, Oct. 16, 1935 (Cl. 48a. 3).

Electrolytic manufacture of hollow metal tanks, etc. Blasius Bart. U. S. 2,023,192, Dec. 3. Various operative details.

Coated aluminum articles. Helmer Bengtson (to Aluminum Colors Inc.). U. S. 2,022,798, Dec. 3. An article of Al to be decorated and protected from undesired change is made anode in a bath contg. H<sub>2</sub>SO<sub>4</sub> until a substantial porous coating is formed (the bath having an acid concn. of 60-77%) and the coating formed is impregnated with chemicals such as FeSO<sub>4</sub>, etc., this produces a light-fast inorg. pigment, which is pptd. in the pores of the coating, e. g., by use of a ferricyanide soln.

Separating copper and zinc from their alloys. Frederick Laist (to Anaconda Copper Mining Co.). U. S. 2,023,424, Dec. 10. An alloy contg. Cu and Zn is melted and blown with air to recover a portion of the Zn content as ZnO; the modified alloy is subjected to electrolytic treatment in an electrolyte contg. H<sub>2</sub>SO<sub>4</sub> and sulfates of Zn and Cu to convert the Zn into ZnSO<sub>4</sub>, which dissolves in the electrolyte, acid in the electrolyte is neutralized with material including ZnO, effecting pptn. of Cu and forming a substantially Cu-free soln. of ZnSO<sub>4</sub>; metallic Zn is electrolytically produced from this soln., and remaining soln. is used for producing electrolyte for treating the alloy.

Bessemer steel. Hauts Fourneaux & Acieries de Differdange-St. Ingbert-Rume-Lange and Jules Welter Fr. 789,735, Sept. 9, 1935. The converter is provided with adjustable electrodes so that an elec. arc may be produced above the bath of steel, the converter being in the turned-down position.

Treatment of cast iron or steel. Vereinigte Stahlwerke A.-G. Ger. 620,210, Oct. 16, 1935 (Cl. 48a. 14). A process is described for increasing the resistance to corrosion of cast iron or steel contg. more than 0.1% Cu and small proportions of constituents which tend to form gelatinous compounds by combination with OH ions in the corrosion medium, e. g., Al, Si, Ni or Sn. The process consists in immersing the iron or steel in a corrosion medium, e. g., sea water, together with a more electropos. metal, and connecting the metals so as to produce an electrolytic circuit. A coherent Cu coating is thus produced on the iron or steel. The dimensions of the cathode should be such as to yield a current of  $0.5-1.5 \times 10^{-4}$  amps. Exptl. results are given of the treatment of a steel contg. C 0.33, Si 0.02, Mn 0.29, P 0.037, S 0.031, Cu 0.20 and Al 0.18%.

Electrolytic cleaning of metals such as iron and steel and deposition of tin on the cleaned metal. Thomas E. Dunn (to Bullard Co.). U. S. reissue 19,773, Dec. 3. A reissue of original pat. No. 1,898,765 (C. A. 27, 2637).

Beryllium. Harry C. Clafin (to Beryllium Corp.). U. S. 2,022,494, Nov. 26. In the electrolytic production of Be, NH<sub>4</sub> Be fluoride is added to a fusion of the fluorides of one or more metals more electropos. than Be, such as those of Na and other alkali metals and the material is electrolyzed at a temp. below the m. p. of Be.

Hydrogen peroxide. Lynn H. Dawsey. U. S. 2,022,650, Dec. 3. H and O are introduced at high velocity into a silent elec. discharge using high-frequency power of 500 to 3000 cycles per sec. App. is described.

Hydrogen peroxide. Ferdinand Krauss. Brit. 434,488, Aug. 26, 1935. See Fr. 781,506 (C. A. 29, 6156).

Electric furnaces. Siemens-Schuckertwerke A.-G.

(Carl T. Buff, inventor). Ger. 620,000, Oct. 11, 1935 (Cl. 21a. 15 03). In furnaces having heating elements made of materials which are conductors only at high temps., e. g., metal oxides, rare earths, or silicates or other salts, the heating of the materials to a temp. at which they become conductive is facilitated by mixing the materials with a material having a higher cond. at atm. temp. The latter may be a material which is vaporized when the required temp. has been attained, e. g., Pb or Sn.

Electric furnaces. Siemens-Schuckertwerke A.-G. (Johann Schnepf and Rudolf Grundmann, inventors). Ger. 620,185, Oct. 19, 1935 (Cl. 21a. 15.50). Means for supporting heating units from the furnace cover is described.

Coreless induction furnace for melting metals in vacuo or in an inert gas. Fried Krupp A.-G. Ger. 620,458, Oct. 21, 1935 (Cl. 21a. 18 01).

Electric furnace for making sheets from quartz or like refractory material. Soc. anon. des manufactures de glaces et produits chim. de St. Gobain, Chauny & Cirey. Ger. 620,191, Oct. 16, 1935 (Cl. 32a. 35). See Brit. 400,020 (C. A. 28, 1937).

Electric furnaces for annealing metal strip in a protective atmosphere. Heraeus-Vacuumschmelze A.-G. Brit. 433,918, Aug. 22, 1935.

Electric resistance furnace suitable for annealing metal articles. Willard Roth and Frank X. Kerin (to Westinghouse Elec. & Mfg. Co.). U. S. 2,023,101, Dec. 3.

Electric resistance heater. Albert H. Heyroth and Walter E. Schildhauer (to Globar Corp.). U. S. 2,022,314, Nov. 26. A nonconducting slab which may be formed of unglazed ceramic material and which has a rounded end carries a conductive layer of Si and bentonite baked on the sides and curved end of the slab (various structural details being described).

Glowing electrode. Patent-Treuhand-Gesellschaft für elektrische Glühlampen m. b. H. Ger. 617,548, Aug. 21, 1935 (Cl. 21f. 82 03). The electrodes for gas filled discharge tubes, especially electric light tubes, contain alk. earth metal silicate or a mixt. of such silicates as the sole electron-emitting substances.

Treating graphite electrodes. The British Thomson-Houston Co. Ltd. Brit. 434,230, Aug. 29, 1935. Graphite electrodes for discharge devices are freed from dust by heating to 1000° and quenching, the quenching liquid is preferably H<sub>2</sub>O, heated to 90°, but CCl<sub>4</sub>, a 28% soln. of AcOH or 25% soln. of glycerol may be used. After quenching, the electrode is dried at 1200° in H<sub>2</sub> or in vacuo.

Carbon electrodes for primary cells. Siemens-Planawerke A.-G. für Kohlefabrikate (Oswald von Wartburg, inventor). Ger. 620,455, Oct. 22, 1935 (Cl. 21b. 7.01).

A core of coarse porous C prep. by a high temp. coking process is coated with a layer of fine active C which has not been subjected to a high temp. The layer may be produced *in situ* by coating the core with a paste of a material such as peat or wood, and carbonizing the coating at a relatively low temp. Constructions of electrode are described.

Electrostatic filters. Siemens-Lurgi Cottrell-Elektrofilter-G. m. b. H. für Forschung und Patentverwertung. Fr. 787,043, Sept. 16, 1935. Means for cleaning the electrodes.

Neon tube. Allen P. Tallman. U. S. 2,023,558, Dec. 10. The rear half of a Ne tube is provided with a reflecting coat contg. Ti oxide.

Emissive coatings on electron tube filaments. Hans J. Spanner and Ulrich Doering (to Electrons, Inc.). U. S. 2,023,707, Dec. 10. For alloying an alk. earth metal such as Ba with a core metal, the latter is heated in an atm. contg. a gaseous compd. of an alk. earth metal, such as a Ba alkyl compd., and the compd. is d-compd. at such a temp. as to effect alloying of the alk. earth metal with the core. App. is described.

Electrical glow discharge for effecting reactions such as production of biphenyl from benzene. Robert V. Kleinschmidt (to Arthur D. Little, Inc.). U. S. 2,023,667.

Dec. 10. A hydrocarbon, such as  $C_4H_{10}$  for the production of biphenyl, is subjected, in a dispersed state, to an electric glow discharge between electrodes in a reaction chamber; free access of current is permitted from one electrode to the other through the material, while the pressure in the chamber is maintained at a value corresponding to the max. elec. cond. of the dispersion. App. is described.

Electrical gaseous discharge vacuum pump Clarence W. Havell (to Radio Corp. of America) U. S. 2,192,465, Nov. 26. Structural and elec. details.

Discharge lamps N. V. Philips' Gloeilampenfabrieken Brit. 433,671, Aug. 19, 1935. In a lamp contg. a gas, e. g., Ne, and a metal volatilizable with comparative difficulty,

e. g., Na, Cd, Mg, Ti, K or Li, means are provided whereby the metal condensing at the coldest part of the tube either flows to another part where it is evaporated by a heating device or is evaporated by heat transmitted from the anode, the temp. of which rises because of the increase in operating voltage resulting from the decrease in vapor pressure.

Discharge lamps. The British Thomson-Houston Co. Ltd. and Wm. J. Scott. Brit. 434,633, Sept. 5, 1935. A high-pressure lamp contains a mixt. of Hg and another vaporizable material, e. g., Cd, the Hg only being entirely vaporized and the unevaporated portion of the other material acting as the cathode.

## 5—PHOTOGRAPHY

E. R. BULLOCK

Advances in photography. G. Heymer *Z. Ver. deut. Ing.* 79, 1295-1301 (1935). I. II

Photographic process in variable-density sound recordings F. v. Orban *Filmtechnik* 11, 8-10 (1935).—Development of the negative up to a  $\gamma$  of 1.1 gave very little increase in distortion. Beyond this point, further gain in modulation is accompanied by a sharp increase in the percentage of distortion for high modulation in the recording.

O. E. Miller

Present position of the theories of the latent photographic image Hans Arens and Fritz Luft. *Veroffentlich. voss. Zentral-Lab. phot. Abt. AGFA* 4, 1-14 (1935).—The 3 most important theories of the latent image at the present time, namely, the Ag germ theory, the surface discharge theory (of G. Schwarz) and the micelle theory, are considered. According to the Ag germ theory, the developability of the grains depends on the development nuclei. The genesis of these nuclei by the deposition of "photo-Ag" on the centers produced by ripening is discussed, and the reason why only certain of the grains on which photo-Ag could be formed become developable is investigated on the basis of probability. The fundamental effects which have led to the advancing of the surface-discharge and micelle theories are described, and some consequences of these theories are discussed. Authors.

Spectro-sensitometer for the determination of a color-sensitivity curve Kurt Looge. *Atelier Phot.* 42, 170-3 (1935); cf. C. A. 29, 1337.—A new spectral sensitometer uses a step diaphragm, with steps in geometrical progression, moved in front of the exposure plane by a synchronous motor, to give a time-scale stepped exposure. The light source is a W lamp, operated at 2300° and screened with a Davis-Gilson filter, to give mean sunlight quality.

H. Parker

A new method for the determination of the resolving power of photographic emulsions A. Narath *Kino-technik* 17, 91-5, 107-10 (1935).—Resolving-power measurements on a no. of Agfa emulsions are given. Resolving power is nearly inversely proportional to grain size. Expressed in linear size of the smallest structure resolved, the resolution of most emulsions lies between 10 and 30  $\mu$ .

O. E. Miller

Light filters of cyanine dyes W. Dieterle *Veroffentlich. voss. Zentral-Lab. phot. Abt. AGFA* 4, 95-7 (1935).—Gelatin filters contg. benzothiazine, benzothiazine-pentacarbocyanine, and naphthothiazine-pentacarbocyanine, resp., have been prep'd. and tested. They all have very sharp extinction curves, and as regards stability in light are not greatly inferior to ordinary light filters. E. R. Bullock.

Bromoll prints on ordinary enlargement paper. P. L. English. *Am. Phot.* 29, 290-0 (1935).—Am. papers are not satisfactory for bromoll when used in the usual way, since the emulsion is already hardened. The following technique is suggested: Use a paper of low contrast and smooth surface. Develop with amidol 7.5 g.,  $Na_2SO_3$  30 g.,  $H_2O$  to 1 l. dild. 1:3 and treat with 4 cc. of 10% KBr soln. per l. of dild. developer. Expose so that development will be completed in not less than 2

min. Fix for 15-20 min. in Eastman F-1 formula with alum omitted. Bleaching and tanning may be combined. Bleach by immersing in  $CuSO_4$ , 5H $_2O$  53 g., KBr 28 g.,  $H_2O$  500 cc. until gray disappears, drain 15 sec., and transfer to the tanning soln.,  $K_2Cr_2O_7$  2.5 g., KBr 50 g.,  $H_2O$  500 cc. for an 11  $\times$  14-in. print, dil. 30 cc. to 150 cc. and rock in this for 2 min. Redevelop for 4-5 min. in the developer described above, from which the bromide may be omitted. Bleach and tan again in the same way as described above. Fix as above, but less time is required. Washing must be thorough after initial fixing, while between other operations, except where otherwise specified, 5 min. is sufficient, wash finally 30 min. After complete drying, swell in  $H_2O$  at various temps. depending upon the consistency of ink to be used, or in  $NH_4OH$  soln. at room temp., the concn. depending upon the ink. For inking are suggested rosin ink, and professional hard, medium and soft. The first is described in detail, together with fine-grain inking technique. I. P. Wightman.

Marking, ornamenting or coloring Al and its alloys by photographic processes (Ger. pat. 620,664) 9

Photographic plates. Anton Jasmatzki. Ger. 617,714 Aug. 21, 1935 (Cl. 57b. 18 02). The strengths of negatives with differing Ag halide contents, e. g., of tripack negatives, are equalized by coloring the washed relief from the Ag negative with a dye of suitable actinic value.

Apparatus and method for drying coated plates for photolithographic and other purposes. Pictorial Machinery Ltd., Leslie Linzell and John P. L. Corkett. Brit. 433,718, Aug. 10, 1935.

Photographic paper. I. G. Farhennd A.-G. Ger. 617,712, Aug. 21, 1935 (Cl. 57b. 9). Halogen-Ag-gelatin printing-out paper is made by adding org. comp'ds. which form Ag salts the soly of which is not materially greater than that of AgCl. Thus, a soln. of nitroindazole is added to the paper. Such paper is free from gray or yellow fog on developing. Other suitable substances are benzotriazole, nitrobenzotriazole and nitrobenzimidazole.

Printing apparatus for transferring dichrome or trichrome pictures to lenticulated films Armand Rodde, Albert H. Herault, Victor Hudeley and Jean Lagrave. U. S. 2,022,432, Nov. 26. Various optical, mech. and operative details.

Pentacarbocyanine dyes. I. G. Farhenndindustrie A.-G. Brit. 434,211, Aug. 28, 1935. See Fr. 774,028 (C. A. 29, 21034). Corresponding propene comp'ds., wherein 1 or more of the H atoms of the trimethylene chain are replaced by an alkyl group or groups may be used.

Photostats Conzett & Huber. Ger. 617,614, Aug. 28, 1935 (Cl. 57d. 1.01). In copying by photography, the sensitive layer consists of a substance bleached by exposure to light. Thus, the sensitive layer consists of distd. water,  $KMnO_4$ , lactic acid and  $AcOK$ . After exposure, the print is fixed by a 5% soln. of the  $HCl$  salt of *p*-aminophenol. Other examples give  $MnO_2$ - $H_2PO_4$  and tartaric acid and primuline as the light-sensitive agents.

## 6—INORGANIC CHEMISTRY

A R MIDDLETON

The direct formation of bromides and the distance of the closest approach of atoms of bromine. Bimayendra Nath Sen *Proc Acad Sci United Provinces Agra Oudh, India* 4, 316-18(1935), cf *C A* 27, 4476—All the available data indicate that only those elements combine directly with Br whose distance of the closest approach of atoms is greater than about 2 Å. From this S concludes that the distance of closest approach of Br atoms is about 2 Å, that is, less than that of any of the elements with which Br combines directly. This is supported by the value (1.73 Å) which S obtains by calcg the closest at approach of Br by his formula. If this is true, then the unknown values for the closest at approach of S, Se, Te, Be, P, As, Ti and Sr must all be greater than 1.73 Å since these elements combine directly with Br. On the other hand, the distances of closest approach for Pt, Os, Ir, Ru and Pd are all over 1.73 Å yet none of these elements combines directly with Br. John E. Milbrey

Double chlorides and bromides of nickel and organic bases. Jean Amiel *Compt rend* 201, 1198-1200(1935), cf *C A* 30, 4064—It was not possible to prep.  $[NiCl_2 \cdot (C_6H_5NH_2)_2]$  by the previous method, but violet-brown crystals of approx  $[NiBr_2 \cdot (C_6H_5NH_2)_2]$  were obtained by evapng mixed solns of the constituent chlorides (or bromides) under reduced pressure, crystg near 100°, redissolving in MeOH and pptg by gaseous HCl (or HBr) the following have been prepd:  $NiX_2 \cdot B$  HCl and  $2NiX_2 \cdot B'$  2HCl, X = Cl or Br, B = MeNH<sub>2</sub>, EtNH<sub>2</sub>, PrNH<sub>2</sub>, C<sub>2</sub>H<sub>5</sub>N, C<sub>3</sub>H<sub>7</sub>N and C<sub>4</sub>H<sub>9</sub>N, B' = C<sub>2</sub>H<sub>5</sub>(NH<sub>2</sub>)<sub>2</sub> and C<sub>4</sub>H<sub>9</sub>N<sub>2</sub>, and also  $2NiCl_2 \cdot C_6H_5(NH_2)_2$ , 2HCl, and, at a lower temp.,  $2NiX_2 \cdot C_6H_5N_2$ , 2HCl 6H<sub>2</sub>O, of which last d<sup>10</sup> (X = Cl) is 2.04, and (X = Br) 2.58. The Cl compds are yellow and moderately deliquescent, the Br compds. are dark yellow or brown and very deliquescent. All are sol in MeOH and EtOH, but only slightly or not sol in other org solvents. On heating they decompose without melting. Duval's method (*C A* 29, 5373) shows no cation except  $Ni^{++}$ . C A Silberrad

Comparative oxidizing actions of corresponding oxychloro and oxybromo compounds on sodium thiosulfate. Emile Carnère and Lucy Faysse *Compt rend* 201, 1036-7 (1935).—The oxidizing actions of solns of HClO<sub>2</sub> and HIO<sub>2</sub> and of HClO and HIO on Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> compared under similar conditions show the action of the I compd to be greater than that of the Cl compd, contrary to the view sometimes held that the reverse should be the case on the assumption that O is less firmly attached in the Cl compds. C A Silberrad

The carbides of lead and of silver. E Montgnie, *Bull soc chim* [5], 2, 1807-9(1935)—Unsuccessful attempts were made to prep PbC<sub>2</sub> by several procedures. In attempting to prep PbC<sub>2</sub> by heating Pb(CN)<sub>2</sub>, it was discovered that Pb(CN)<sub>2</sub> does not exist. A MeOH soln of Pb(OAc)<sub>2</sub> dropped on CaC<sub>2</sub> gave a mixt of PbC<sub>2</sub> with several other compds. PbC<sub>2</sub> is stable in air, but is readily hydrolyzed by acid or alk solns, yielding C<sub>2</sub>H<sub>2</sub> (cf Durand, *C A* 18, 657; Heuz and Neukirch, *C A* 18, 799, Gründt, *C A* 21, 2858). Uniformly unsuccessful attempts were made to prep AgC<sub>2</sub> by calcining org Ag salts the oxalate, benzoate, salicylate, tartrate and oleate were used, the residue in each case was Ag (cf Sabatier, *Bull soc chim* [4], 29, 791(1921); Durand and Banos (*C A* 22, 715)). G M. P.

Action of oxidizing agents on sodium hypoferrite. Xavier Thesse *Compt rend* 201, 1135-7(1935); cf *C A* 29, 1349—When Cl is passed into a satd soln of Na<sub>2</sub>FeO<sub>2</sub> contg 35% NaOH at 120° Fe(OH)<sub>3</sub> is pptd, with 40% NaOH some Fe<sub>2</sub>O<sub>3</sub> is formed, with 50% at 130-40° yellow-green crystals of (probably) Na<sub>2</sub>FeO<sub>4</sub> · 10H<sub>2</sub>O; and with 60-70% the metaferrite Na<sub>2</sub>FeO<sub>3</sub> · Br acts similarly. Solns contg up to 4 g/l of Fe(OH)<sub>3</sub> in 40% NaOH are obtainable by oxidizing solns of Na<sub>2</sub>FeO<sub>2</sub> of suitable strength. On adding Pb(OH)<sub>2</sub> to a hot soln of Na<sub>2</sub>FeO<sub>2</sub> (50% NaOH) there is a transitory formation

of fine black needles (Pb or Pb<sub>2</sub>O), which quickly redissolve with evolution of gas 2Pb(OH)<sub>2</sub> + 2Na<sub>2</sub>FeO<sub>2</sub> + 2NaOH = 2Na<sub>2</sub>FeO<sub>3</sub> + Pb<sub>2</sub>O + 3H<sub>2</sub>O; PbO + 4NaOH = 2Na<sub>2</sub>PbO<sub>2</sub> + H<sub>2</sub>O + H<sub>2</sub>, while some Pb(OH)<sub>2</sub> is merely dehydrated. On adding a soln of Na<sub>2</sub>FeO<sub>2</sub> to hot aq NaOCl contg 10-15% NaOH, the soln turns purple through formation of Na<sub>2</sub>FeO<sub>4</sub>. C A Silberrad

Action of perchloric acid on iodine and iodo derivatives. Determination of iodine in organic substances. Ernest Kahane and T. Tomesco, *Compt rend* 201, 1195-8 (1935)—HClO<sub>4</sub> and KI interact in 2 stages, first at about 135° with liberation of I, and then at 200-10° with disappearance of the I, the final result (in a sealed tube) being 7HClO<sub>4</sub> + 4KI = 4HIO<sub>4</sub> + 3HCl + 4KClO<sub>4</sub>, with no formation of free O<sub>2</sub>. Other inorg iodides react similarly. Organic iodo compds are as a rule completely decompd into H<sub>2</sub>O, CO<sub>2</sub> and HIO<sub>4</sub>. There is some evidence that at about 210° HClO<sub>4</sub> (72%) undergoes some reversible disson. into 2 products both of which react with the I to form only HIO<sub>4</sub>, HCl and H<sub>2</sub>O. Iodine can be detd by an adaptation of K's method (cf. *C A* 26, 940) by passing the product formed by heating the substance under examn. with the sulfo-nitro perchloric mixt into Br H<sub>2</sub>O, which converts any I passing over into HIO<sub>4</sub>, or better by adding a large excess of As<sub>2</sub>O<sub>3</sub> to the original mixt. when all the I thus passes over. C A Silberrad

Action of heat on ortho-, pyro- and meta-arsenates of calcium and strontium. Henri Guérin, *Compt rend* 201, 1133-5(1935).—The ortho-arsenates of Ca and Sr are stable in a vacuum up to 1200°, any excess of As<sub>2</sub>O<sub>3</sub> left in prepn passing off as As<sub>2</sub>O<sub>3</sub> + O<sub>2</sub>. The pyro- and meta-arsenates pass into the ortho salt on heating above about 800° and 500°, resp., in the latter case with intermediate formation of pyroarsenate. C A Silberrad

Stabilized cubic ferric oxide. André Michel and Georges Chaudron *Compt rend* 201, 1181-3(1935).—Solid solns of varying amts of NaFeO<sub>3</sub> in Fe<sub>2</sub>O<sub>3</sub> were prepd by adding various amts of aq NaOH to pptd Fe(OH)<sub>3</sub>, the product being reduced to Fe<sub>3</sub>O<sub>4</sub>, oxidized at 300° and then heated to 650°. The Curie point decreased regularly with increasing content of Na<sub>2</sub>O, while the side of the unit cube (a) increased with equal regularity, the figures for an oxide contg about 5% Na<sub>2</sub>O being about 400° and 8.52 Å, and (by extrapolation) 675° and 8.32 Å. for pure Fe<sub>3</sub>O<sub>4</sub>. The dissociability of Fe<sub>3</sub>O<sub>4</sub> (into Fe<sub>2</sub>O<sub>3</sub> and O) decreases rapidly with increase in Na<sub>2</sub>O, and with 5% thereof it is practically undissociable at 300°. Though the content of Na<sub>2</sub>O is reduced by hydrolysis in hot H<sub>2</sub>O or by AcOH, no change in Curie point or a occurs until after reheating to 650°. Solid solns of other ferrites behave similarly, the limit solns of those of Be, Ag and K having Curie points at 220°, 270° and 290°, resp. C A Silberrad

Potentiometric investigations of ammonium mercury sulfites. G Spacu and C Drăgulescu, *Z. anorg allgem. Chem* 224, 273-9(1935), cf *C A* 29, 5353<sup>1</sup>.—By potentiometric titration of HgCl<sub>2</sub> solns with (NH<sub>4</sub>)<sub>2</sub>SO<sub>3</sub> under various conditions, 2 compds. are indicated [HgCl(SO<sub>3</sub>)](NH<sub>4</sub>) and [Hg(SO<sub>3</sub>)<sub>2</sub>](NH<sub>4</sub>)<sub>2</sub>. These are well-crystallized stable compds. H. Stoertz

Decomposition of cupric sulfate pentahydrate by heat. Osias Binder *Compt rend* 201, 1035-6(1935), cf *C A* 30, 3631—On heating CuSO<sub>4</sub> · 5H<sub>2</sub>O in an elec. furnace at 650° 2CuO SO<sub>3</sub> is obtained as a homogeneous Indian red powder with distinctive x-ray spectrum. It is stable in air; treated with H<sub>2</sub>O it gives 4CuO SO<sub>3</sub> · 4H<sub>2</sub>O or 3CuO SO<sub>3</sub> · 2H<sub>2</sub>O according as the H<sub>2</sub>O is cold (20°) or boiling. C A Silberrad

Reactions of sulfuryl diamide (sulfamide). F. C. Wood *Nature* 136, 837(1935).—When a soln of xanthidrol in I:OH is added to a soln of sulfamide in AcOH and H<sub>2</sub>O, after some time, crystals of dioxanthylsulfamide, m 182-4°, are obtained. Condensation products have also been obtained with xanthidrol and *p*-aminobenzenesulfonamide,

m. 209°; *m*-benzenedisulfonamide, m. 170°; *p*-toluenedisulfonamide, m. 198°. The reaction between sulfamides and sulfonamides in some cases may conveniently be used for their estimation. G. M. P.

The dimer of carbon chlorosulfide. The chlorosulfide  $C_2S_2OCl_2$  and a new chlorosulfide  $C_2S_2Cl_4$  Marcel Delépine, Louis Labro and Frederique Lange. *Bull. soc. chim.* [5], 2, 1969-80(1935) —  $C_2S_2Cl_4$  was prep'd by heating 10 g.  $CSCl_2$  dimer with 15 cc. of abs.  $FeOH$  on the water bath; 1.6 g. of  $C_2S_2Cl_4$  was obtained. It crystallizes in small yellow plates, m. 58-9°, is sol in most org. solvents, but not in  $H_2O$ , and is stable at ordinary temps. Some reactions, chiefly condensations with secondary bases, are described.  $C_2S_2OCl_2$  was prep'd from 1 l.  $H_2O$  and 46 g. of  $CSCl_2$  dimer, with repeated shaking. The yield was 36 g. of crude crystals, 26.50 g. remained after purification. The properties are given by Schönberg and Stephenson (*C. A.* 27, 3197). Several reactions, chiefly condensations with secondary bases, are described. Several structural formulas each are proposed for  $C_2S_2OCl_2$  and for  $C_2S_2Cl_4$ . G. M. P.

System lithium nitrate-water Jean Bureau. *Compt. rend.* 201, 1197-5(1935) — The hydrate  $LiNO_3 \cdot 1.5H_2O$  (I) is stable below -7.95° at which temp it undergoes a peritectic transformation into  $LiNO_3 \cdot H_2O$ , which similarly passes into  $LiNO_3 \cdot 0.5H_2O$  at 50.9°, and this again peritectically without fusion into  $LiNO_3$  at 94°. Iodine and  $H_2O$  form a eutectic at -38.7°. Solns. show no hydrolysis below 100°. The anhyd salt (99.5%) decomposes to a slight extent on fusion, the fused product solidifying at 210° and contg. 95.5%  $LiNO_3$ . C. A. Silberrad.

Alkaline oxide systems  $CaO-Al_2O_3-SiO_2-CO_2$ , IV. The  $CO_2$  pressure of the silice acid-rich part of the system  $Li_2O-SiO_2-CO_2$  and the action of  $Al_2O_3$  upon  $Li_2CO_3$ . C. Kröger and E. Pingas. *Z. anorg. allgem. Chem.* 224, 289-304(1935), cf. *C. A.* 29, 7210°. — The action of  $Li_2CO_3$  upon  $Li_2O-xSiO_2$  was studied, with  $x = 3, 5$  and 7. Action of quartz upon  $Li_2CO_3$  leads to metasilicate. The last equal which is established leads to orthosilicate formation as follows:  $[Li_2SiO_3] + Li_2CO_3 = [Li_2SiO_4] + (CO_2)$ . At higher temps. the orthosilicate reacts with  $Li_2CO_3$  thus:  $[Li_2SiO_3] + [Li_2SiO_4] = [Li_2SiO_5] + (CO_2)$ , and again  $[Li_2SiO_4] + Li_2CO_3 = [Li_2SiO_5] + (CO_2)$ . In the action of  $Al_2O_3$  upon  $Li_2CO_3$ , the following reactions occur:  $[Li_2CO_3] + [Al_2O_3] = [Li_2O \cdot Al_2O_3] + (CO_2)$  and  $3-[Li_2CO_3] + [Al_2O_3] = [3Li_2O \cdot Al_2O_3] + 3CO_2$ . Above 750°, the system is bivalent by the occurrence of  $Li_2O-Al_2O_3$  solid melts, until with further decompn. the disocn. pressure of  $Li_2CO_3$  is reached. H. Stoertz.

Investigations of alkali aluminum silicates. IX. The silicate part of ultramarines. Erhard Gruner. *Z. anorg. allgem. Chem.* 224, 351-68(1935); cf. *C. A.* 29, 5329°, 7210°. — By repeated fusion of ultramarine with alkali cyanides the ultramarine is completely desulfured and forms a colorless silicate similar to nephelite or kaliolephite, having the compn.  $Al_2Si_4R_{10}O_{38}$  or  $3(R_2O \cdot Al_2O_3 \cdot 2SiO_2)$ . In the desulfurization the polysulfidic S is first given up and forms thiocyanate with the cyanide melt. Gradually monosulfidic S is also given up, but this is so firmly attached to lattice alkali that it tears it apart and seps. in the form of  $Na_2S$ , leaving the ultramarine lattice resembling those ultramarines constructed on the basis  $Al_2Si_4$ . The lattice consists of the cubic elementary cell, green,  $a = 9.14$  Å.; blue,  $a = 9.16$  Å.; violet,  $a = 9.08$  Å.; red,  $a = 9.19$  Å.; yellow,  $a = 9.09$  Å. The lattice const. of S-free ultramarine residue from green is  $a = 9.19$  Å.; a change in the ultramarine lattice by alkali sepn. has therefore not occurred. For the cubic crystal, ultramarine residue of the compn. of nephelite (NaCN melt) and kaliolephite (KCN melt) the names pseudonephelite and pseudokaliolephite are used. X. The system ultramarine silicate-sulfur. Erhard Gruner and Johannes Förster. *Ibid.* 367-57. — Blue and green ultramarine are subjected to isothermal S sepn. and give the same form of curve as the decompn. of substances contg. zeolitic  $H_2O$ . From the point where the basic substance corresponds to a pure monosulfidic ultramarine, the lattice constituents experience the same decompn. as occurs in the treatment of ultra-

marine with fused alk. cyanides. The ultramarine gives up alkali simultaneously with monosulfidic S. While S is present in the basic substance all the intermediate substances are blue or green. When about all of the S has been sepd., the residue sinters at about 1030° to a hard stony mass which gives the x ray spectrum of nephelite. Neither blue nor green ultramarine will take up S, nor will pseudonephelite or nephelite, but pseudonephelite will form ultramarines with alk. sulfides. Only by the presence of alkalis, S and the ultramarine lattice can stable ultramarine be formed. H. Stoertz.

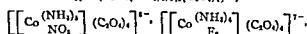
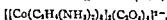
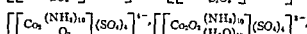
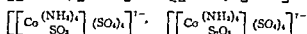
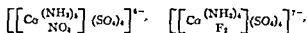
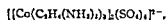
The transformation of sodium nitroferrocyanide to blue ferrocyanide. Ed. Justin-Mueller. *Bull. soc. chim.* [5], 2, 1932-6(1935) — According to Overbeck (*Pogg. Ann.* 87, 110),  $Na_2Fe(CN)_6 \cdot NO$  decomposes slowly in sunlight, yielding  $Fe(CN)_6$  and NO. A partial yield of blue ferrocyanide was likewise observed in a study of the reaction of  $H_2S$  with  $Na_2Fe(CN)_6 \cdot NO$  in NaOH soln. These and other reactions are used to suggest that the structural

formula is either  $Na_2(CN)_5Fe^{III}[(CN)_6NO]$   $[(CN)_5NO]Fe^{III}(CN)_5$  or  $Na_2[(CN)_5NO]Fe^{III}(CN)_5$  or  $Na_2[(CN)_5NO]Fe^{III}(CN)_5$ . The tricyanide and

nitrosobicyanide groups have cyclic structures  $\begin{array}{c} \text{C} \quad \text{N} \quad \text{C} \\ \diagup \quad \diagdown \quad \diagup \\ \text{N} \quad \text{C} \quad \text{N} \end{array}$  and  $\begin{array}{c} \text{C} \quad \text{N} \quad \text{O} \quad \text{N} \quad \text{C} \quad \text{N} \end{array}$ . This compd. splits to form

the colorless salt  $Na_2Fe_2(CN)_8 \cdot Na_2Fe(CN)_6[(CN)_5NO]_2 \cdot 2H_2O \rightarrow Na_2Fe_2(CN)_8 + 4HCN + 2NaNO_2$ . This is followed by a slower reaction  $4Na_2Fe_2(CN)_8 \rightarrow Na_2Fe_2[Fe_2(CN)_8]_2 + 6NaCN$ . This, in the presence of NaOH is transformed to Prussian blue (1)  $NaOH + CO_2 \rightarrow NaHCO_3$ , (2)  $Na_2Fe_2[Fe_2(CN)_8]_2 + 2NaHCO_3 + O_2 \rightarrow [Fe^{II}(CN)_6]Fe^{III}Fe^{III}(OH)_2 + 2Na_2CO_3$ . The NaOH is a carrier for  $CO_2$ . The same reaction occurs in the presence of  $H_2SO_4$ , yielding  $Na_2SO_4$  instead of  $2Na_2CO_3$ . In a closed flask, the reaction is  $Na_2Fe_2(CN)_8 + 6HCN + 4NaNO_2 \rightarrow Fe_2(CN)_8 + Fe(OH)_2 + 6NaCN + 4NO + 2H_2O$ . This reaction, in the presence of  $FeCl_3$  and sunlight, gives Prussian blue directly, without the formation of the colorless salt, and the  $NO$  is liberated directly, without the formation of  $NaNO_2$ .  $3Na_2Fe_2(CN)_8(NO)_2 + 2FeCl_3 + 9H_2O \rightarrow Fe_2(CN)_8 + Fe(OH)_2 + 12NaCl + 9HCN + 3HCNO + 6NO$ . G. M. P.

A new group of complex compounds. — Complex compounds whose central ion is a complex cation. II. Complex sulfato- and oxalato-compounds with complex cobalt cations as central ions. H. Brintzinger and H. Osswald. *Z. anorg. allgem. Chem.* 224, 283-8(1935), cf. *C. A.* 29, 7211°. — Certain complex Co cations when dissolved in  $(NH_4)_2SO_4$  or  $K_2C_2O_4$  soln. combine with sulfate and oxalate radicals to form new complex anions. The following anions were prep'd. and their ion wt. was det'd by dialysis:



are given to prove that the chromate method (cf. Karasch and Sugarschew, *C. A.* 24, 5234) serves for e.g. Pb from Cu, Ag, Ni, Ca, Ba, Sr, Mn, Zn, Cd, Al and Fe. It is necessary to ppt. the Pb with  $(\text{NH}_4)_2\text{CO}_3$  during 10 min. by adding the reagent slowly, with stirring, to the boiling soln. contg. free  $\text{HNO}_3$ . The ppt. is filtered after standing a few hrs., washed with water, dried at  $140^\circ$  and weighed as  $\text{PbCrO}_4$ . In the filtrate the Cu can be detd. as  $\text{CuCN}_2$ , the Ag as  $\text{AgCl}$ , the Ni as salt of dimethylglyoxime, the Ca as  $\text{CaO}$  and Ba as  $\text{BaSO}_4$ . The results are excellent. W. T. H.

**Determination of magnesium in Duralumin.** G. Stanley Smith. *Analyst* 60, 812-14 (1935).—Dissolve 2 g. of the alloy in 60 ml. of 10%  $\text{NaOH}$  soln. Filter, wash the residue with hot water and transfer it back to the original beaker. Add 5 ml. of  $\text{H}_2\text{SO}_4$  (d. 1.2), boil 1 min., filter and wash the residual Cu with hot water. To the last filtrate contg. the Mg, add  $\text{NaOH}$  soln. until a faint permanent ppt. is formed. Clear the soln with a drop of dil.  $\text{H}_2\text{SO}_4$ , and add 0.1 N  $\text{KMnO}_4$  dropwise to oxidize Fe. Boil and add  $\text{ZnO}$  suspension in slight excess. Continue adding  $\text{KMnO}_4$  until the liquid is colored pink on boiling and adding more  $\text{ZnO}$ . Filter, wash with hot water. The filtrate contains Mg, sometimes a little Cu and also Ni besides Zn. Add about 1 g. of KCN and 10 ml. of 10%  $\text{NaOH}$  to ppt.  $\text{Mg}(\text{OH})_2$ . Filter with the aid of paper pulp and wash with 1%  $\text{NaOH}$ . Transfer the pulp and ppt. to a beaker and add a little more than sufficient  $\text{H}_2\text{SO}_4$  to give an acid reaction to methyl orange. Filter through the same filter and wash with hot water. Add a little  $\text{NH}_4\text{Cl}$  and make the soln. slightly alk. The absence of a ppt. shows the absence of Al and Fe. Make the soln. slightly acid and ppt. the Mg as  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$  with suitable precautions. Or, suitable results can be obtained by adding a measured vol. of standard  $\text{NaOH}$  to the neutral soln., filtering and titrating the excess  $\text{NaOH}$  with acid. The results obtained in 6 test analyses were very satisfactory. W. T. H.

**Determination of small quantities of mercury with dithizone.** Hellmut Fischer and Grete Leopoldi. *Z. anorg. Chem.* 103, 241-57 (1935).—F. has already shown (*C. A.* 20, 5600) that small quantities of Hg can be detected by means of dithizone dissolved in  $\text{CCl}_4$ . By colorimetry, about 0.01 mg. of  $\text{Hg}^{++}$  can be detd. The detn. can also be accomplished, and with somewhat greater accuracy, by extg. the Hg with a measured vol. of the reagent, adding an excess of standard  $\text{AgNO}_3$  soln. and titrating the excess Ag with a very dil. soln. of dithizone. Quantities of Hg lying between 0.3  $\gamma$  and 10  $\gamma$  can be detd. by the color tint formed between the orange-colored Hg compd. and the green of the reagent in  $\text{CCl}_4$ . A definite vol. of the green soln. obtained by dissolving 1-3 mg. of dithizone in 100 cc. of  $\text{CCl}_4$  is added to the Hg soln. and the resulting color is matched by adding known quantities of Hg soln. to an equal vol. of the reagent. The detn. of Hg in this way is not prevented by the presence of  $\text{Cl}^-$ . The only metals that interfere with the procedure recommended are Ag, Au, Pd and Pt. W. T. H.

**Photometric silicon determination in presence of iron and accompanying substances.** Hans Pansl. *Arch. Eisenhüttenw.* 9, 225-30 (1935).—The known methods for colorimetric and photometric detns. of Si in Fe require an intermediary phosphate pptn. and filtration. A new simpler method is described by which the same end can be obtained by addn. of NaF after the Mo reaction. The procedure without compensating measurement requires 0.1 g. of the sample which is dissolved in a 100-cc. Erlenmeyer flask in 15 cc.  $\text{HNO}_3$  and 1 cc.  $\text{H}_2\text{O}_2$  at not more than  $50^\circ$ ; this takes about 5 min. To the hot soln. so much permanganate soln. is added, drop by drop, until the red color remains even under strong shaking when 2 cc.  $\text{HCl}$  is added and permitted to act about 10 sec. under heat. After cooling to  $20^\circ$  and filling up to the mark, the soln. is divided in 2 parts of 50 cc. each. One part is mixed with 5 cc. cobaltate soln. and, after 6 min. reaction, with 20 cc. NaI soln.; the other serves as compensating soln. and is mixed with 20 cc. NaI and 5 cc. distd.  $\text{H}_2\text{O}$ . The 2 solns. are compared photometrically with one an-

other, preferably with Hg light and a special Hg filter. The amts. of Si present can be obtained from a calibration curve. The method is applicable to alloy and nonalloy steels which dissolve without residue. For high-Cr and W steels the procedure is somewhat modified and for V steels a correction of -0.02% Si must be made. It cannot be applied to steels with more than 0.6% Ti. Eight references. M. Hartenhein.

**Colorimetric method for the determination of small amounts of silver by the use of p-dimethylaminobenzalrhodanine.** Irl C. Schoonover. *J. Research Natl. Bur. Standards* 15, 377-84 (1935) (Research Paper No. 636).—The procedure recommended is similar to that suggested by Feigl (*C. A.* 22, 4089) and later used by Kolthoff (*C. A.* 24, 3720) with some modification. It was found that soft glass was absolutely unsuitable, Jena glass better but fused silica best for the detn. Appreciable quantities of Ag are likely to be adsorbed by the contg. vessel. The adsorption is negligible with  $\text{SiO}_2$  vessels. W. T. H.

**Iodometric titration of tin.** F. L. Okell. *Analyst* 60, 813-11 (1935).—Low results in the iodometric titration of stannous solns. with  $\text{I}_2$  are often due to  $\text{O}_2$  dissolved in the  $\text{I}_2$  soln. A method for prep. air-free  $\text{I}_2$  soln. is described. The various methods adopted by previous investigators for the improvement of the method are for the most part methods of preventing the oxidation of the  $\text{Sn}^{++}$  by dissolved  $\text{O}_2$ . The assumption that the O interference can be allowed for by standardizing the  $\text{I}_2$  soln. against pure Sn is not justified in the assay of Sn ores, because Ti is the main cause of the inconstancy of the O effect and it is rather troublesome to remove all Ti from the ore. W. T. H.

**A colorimetric method for determining nitrate nitrogen in grass.** F. L. Ashton. *J. Soc. Chem. Ind.* 54, 389-90 (1935).—Finally ground grass is boiled with  $\text{MgO}$ , and solns. of potash alum, Pb subacetate and  $\text{Ag}_2\text{SO}_4$  are added. The mixt. is filtered and a small amt. of  $\text{H}_2\text{O}_2$  added to the filtrate. The soln. is evapd. to dryness and the residue heated on the water bath. Phenoldisulfonic acid,  $\text{H}_2\text{O}$  and  $\text{NH}_4\text{OH}$  are added and the nitrate N is detd. colorimetrically. R. R. Rushton.

**Determination of iodine in iodized salt.** R. L. Andrew and J. L. Mandeno. *Analyst* 60, 801-3 (1935).—The procedure recommended is based upon that of von Fellenberg (*C. A.* 24, 1817-18, 5322), which depends upon the conversion of the iodide to iodate by  $\text{Cl}_2$  water and titration of the  $\text{I}$  formed by the interaction of  $\text{I}^-$  and  $\text{IO}_3^-$  in the presence of acid. It was found more convenient to use Br water for the oxidation and 0.02 N  $\text{Na}_2\text{S}_2\text{O}_3$  for the titration. The conversion of the iodide into iodate is complete if the soln. is nearly neutral but as  $\text{HCl}$  is added the results are progressively low. W. T. H.

**Determination of the solubility of potassium fluosilicate,  $\text{K}_2\text{SiF}_6$ , under different conditions.** A. A. Vas'lev and Nina N. Mart'yanova. *Z. anorg. Chem.* 103, 103-6 (1935).—Inasmuch as F is often pptd. as  $\text{K}_2\text{SiF}_6$ , it is desirable to know how sol. the ppt. is in liquids which are likely to be present during an analysis. The following values were obtained by shaking with 150 cc. of liquid and allowing to stand overnight. In 100 cc. of soln. there was found of  $\text{K}_2\text{SiF}_6$  0.115 g. in pure water at room temp., 4.0 mg. in 50%  $\text{EtOH}$ , 5.0 mg. in satd.  $\text{KNO}_3$  soln., 5.0 mg. in satd.  $\text{KCl}$  soln., 2.2 mg. in 50% alc. +  $\text{KCl}$  soln. mixt., 2.0 mg. in 50% alc. contg. 2%  $\text{KCl}$  + 0.5 cc.  $\text{N HCl}$  in 100 cc. and 0.9 mg. dissolved by washing on the filter with 50%  $\text{EtOH}$  contg. 2%  $\text{KCl}$ . Determination of fluorine as potassium fluosilicate with subsequent titration of the complex salt. *ibid.* 107-13.—Dissolve a sample equiv. to 0.2 g. NaF in 15 cc. of water in a 200-cc. beaker. Add 15 cc. of water-glass soln. contg. 10 mg. dissolved  $\text{SiO}_2$  per cc. add 2-3 drops of methyl orange indicator soln., 1 g. of  $\text{KCl}$  and sufficient 6 N  $\text{HCl}$  to make the indicator assume a red color and provide an excess of 2-3 drops of the acid. Add sufficient alc. to make the content 50% by wt. After an hr. filter through a moderately hard filter and transfer the ppt. to the filter with the aid of portions of the mother liquor. Wash out the beaker twice with the

wash liquid (50% alc. contg. 2 g KCl per 100 ml) and wash the ppt. on the filter 3 times more with the same soln. The vol. of the mother liquor and that of the wash liquor must be subsequently measured. Transfer the ppt. and filter to the same vessel in which the pptn. took place, add 100 cc of CO-free water and titrate with NaOH to a phenolphthalein end point. Toward the end of the titration heat the soln. nearly to boiling. One cc of 0.1 N NaOH = 6.3 mg NaF. To find the wt. of NaF in mg to be added in order to correct for the soly. of the ppt., take the vol. of the mother liquor in cc. and multiply by 0.023 and the vol. of the wash liquid by 0.10. To det. F in insol. fluorides, fuse 0.5 g of the sample with 1.25 g quartz sand and 6 g NaKCO<sub>3</sub>. Introduce the hot crucible into cold water, remove the melt and heat it with 200 cc of water. Filter into a 300-cc measuring flask, add 20 g conc. (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, dil. to 200 cc and keep at 40° for an hr. Cool to room temp., dil. to exactly 300 cc and allow to stand overnight, filter and take 200 cc of the filtrate for the further analysis. Evap. thus on the water bath to 30-40 cc., cool, add 2 g KCl, etc., as in the analysis of a sol. fluoride. The results agree fairly well with those obtained by the much longer procedure of Starck (cf. Hawley, C. A. 29, 2471) or that recommended by Hoffman and Lundell (cf. C. A. 24, 1192).

Qualitative test for bromate. I. M. Korenman, Z. anal. Chem. 103, 269-71 (1935).—See C. A. 29, 7866.

The action of charcoal on aqueous solutions of silver nitrate. T. R. Bolam and W. A. Phillips. Trans. Faraday Soc. 31, 1443-52 (1935).—The action of sugar charcoal on solns. of AgNO<sub>3</sub> was examined by titrating the Ag ion and free acid in the filtrate, pptg. the adsorbed Ag ion as chloride, extg. the reduced Ag by HNO<sub>3</sub> for direct titration and estg. the adsorbed Ag ion by difference. The reaction occurs in 2 steps: (1) rapid adsorption of Ag ion and (2) slower reduction of adsorbed Ag ion to metallic Ag which aggregates to form sep. particles. The ratio of reduced Ag to (adsorbed Ag ion + adsorbed acid) is const. and less than unity at fixed concns. of AgNO<sub>3</sub> on charcoals subjected to a variety of pretreatments. It is concluded that only an amt. of Ag ion corresponding to a unimol. layer of adsorbed AgNO<sub>3</sub> undergoes reduction and that the C atoms which are oxidized by the Ag ion retain their capacity of adsorbing AgNO<sub>3</sub>. At high concns. of AgNO<sub>3</sub> the adsorbed layer of salt appears to be multimol. The suppression of reduction by added acid appears to be due to selective adsorption of acid on the reducing surface. Exposure to air at room temp. decreases the reducing power of the charcoal. Variation of the amt. of reduction with degree of activation and temp. of treatment of charcoal appears to be due to variation in sp. adsorptive surface. The quantity, adsorbed Ag ion + adsorbed acid is independent of degree of aging of charcoal or time of contact with the soln.

Conductometric titration of molybdate with silver nitrate. C. Candea and I. G. Murgulescu. Bul. soc. chim. Romania 17, 183-5 (1935).—Solns. which are 0.005-0.05 N in Na<sub>2</sub>MoO<sub>4</sub> can be titrated conductometrically with 0.05-0.5 N AgNO<sub>3</sub>. The greatest error in 8 expts. was 0.3 cc. when 16 cc. of reagent was used.

Determination of small quantities of water by the method of Crismer. Lucia de Brouckere and Albert Gillet. Bull. soc. chim. Belg. 44, 473-503 (1935).—Instead of attempting to det. H<sub>2</sub>O directly, it is sometimes better to take advantage of the change in the phys. properties caused by the presence of a little H<sub>2</sub>O. Thus Crismer (Bull. soc. chim. Belg. 18, 4 (1904)) detd. H<sub>2</sub>O accurately by noting the temp. at which a solvent became turbid. By means of methanol-C<sub>2</sub>H<sub>5</sub>I, or methanol-C<sub>2</sub>H<sub>5</sub>I it is possible to det. H<sub>2</sub>O accurately by detg. the temp. at which congelation takes place. The method is described.

Determination of carbon monoxide in mixtures with hydrogen and methane. G. Meyer and A. Stoeff. Rec. trav. chim. 54, 800-3 (1935).—Scheffer (C. A. 24, 4436, 26, 1874, 27, 5051) and his students have studied methods of analyzing gases by combustion over CuO. When a mixt. of CO, CH<sub>4</sub>, and H<sub>2</sub> is passed through a combustion tube

contg. CuO, the CO<sub>2</sub> formed is a measure of the total C content and the water formed gives the total H. By passing the original mixt. over I<sub>2</sub>O<sub>5</sub> at 120-130° the CO is oxidized to CO<sub>2</sub>, which, together with a little CH<sub>4</sub>, can be condensed by passing through a tube chilled with liquid air. By proper fractional distn. of the condensate, the CO<sub>2</sub> can be volatilized and measured. The moisture formed will remain with the I<sub>2</sub>O<sub>5</sub> but can be obtained by heating to 180°.

Dependence of the palladium salt test for carbon monoxide upon the presence of other substances. W. Daller. Z. anal. Chem. 103, 83-8 (1935).—The importance of the test for CO, because of its poisonous property, makes it desirable to know whether the presence of other substances may affect the delicacy of the test. PdCl<sub>2</sub> dissolves to form a clear soln. in dil. HCl or in solns. contg. sol. chlorides, double salts contg. the PdCl<sub>2</sub> anion are formed. PdCl<sub>2</sub> solns. add largely give a ppt. of basic salt and a little free HCl is formed. When dissolved in a little water and a little HCl, a nearly clear soln. is obtained which, when filtered, can be held without the formation of a turbidity. The test for CO depends on the reaction Pd<sup>++</sup> + CO + H<sub>2</sub>O = Pd + CO<sub>2</sub> + 2H<sup>+</sup>. According to the mass law, excess of acid retards pptn. of Pd and the presence of NaOAc or other buffer salt is helpful. An excess of Cl<sup>-</sup> makes pptn. of Pd more difficult because it lowers the concn. of Pd<sup>++</sup>. The correctness of these conclusions was proved by many expts. Sometimes the first effect of the CO introduced into PdCl<sub>2</sub> in dil. HCl is to form a white adsorption compd. of CO and PdCl<sub>2</sub> which, however, is not very stable, and soon breaks down in the desired direction. Na<sub>2</sub>PdCl<sub>4</sub> solns. contg. suitable quantities of Na<sub>2</sub>SO<sub>4</sub> can be used to detect CO in the presence of considerable H<sub>2</sub>.

Determination of arsenic acid, phosphoric acid and iron in the presence of one another. Gr. Balanescu and V. Ionescu. Bul. soc. chim. Romania 17, 93-102 (1935).—The results of over 100 analyses are given and the following conclusions drawn: (1) H<sub>3</sub>AsO<sub>4</sub> in the presence of H<sub>3</sub>PO<sub>4</sub> can be detd. iodometrically by adding KI to the soln. contg. 16% HCl and titrating the liberated I<sub>2</sub> with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. (2) To det. H<sub>3</sub>PO<sub>4</sub> in the presence of H<sub>3</sub>AsO<sub>4</sub>, good result can be obtained by the usual molybdate method after adding (NH<sub>4</sub>)<sub>2</sub>S to the acid soln. to ppt. As<sub>2</sub>S<sub>3</sub>, evapg. the filtrate to dryness and carrying out the molybdate pptn. of H<sub>3</sub>PO<sub>4</sub> in the usual way, preferably measuring the vol. of NaOH soln. required to dissolve the ppt. (3) To det. As in a soln. contg. H<sub>3</sub>PO<sub>4</sub>, H<sub>3</sub>AsO<sub>4</sub>, and Fe salt, the As can be detd. directly as As<sub>2</sub>S<sub>3</sub> and the Fe in the filtrate as in (4) or, as is better, the Fe and As can be detd. together iodometrically as in (1) and the Fe in a sep. portion as in (4). (4) To det. Fe in the presence of H<sub>3</sub>AsO<sub>4</sub>, it is best to ppt. the As as pentasulfide from a strongly acid soln., evapg. the filtrate to dryness with HNO<sub>3</sub>, and finally det. the Fe<sup>+++</sup> iodometrically. (5) To det. H<sub>3</sub>PO<sub>4</sub> in the presence of Fe and As, proceed as in (1).

Colorimetric determination of phosphoric acid in grass and similar materials by the Fiske and Subbarow method. A. W. Greenhill and N. Pollard. J. Soc. Chem. Ind. 54, 404-6T (1935), cf. C. A. 20, 1092-3.—The dried and finely ground material is mixed with a dil. soln. of Mg(NO<sub>3</sub>)<sub>2</sub>, evapg. to dryness and ignited. The residue is dissolved in 10 N H<sub>2</sub>SO<sub>4</sub> and phosphate detd. colorimetrically in an aliquot with the (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub>-ammoniasulphuric acid reagent.

Mixed perchloric and sulfuric acids. III. Determination of chromium in chromic oxide. G. Frederick Smith, L. D. McVickers and V. R. Sullivan. J. Soc. Chem. Ind. 54, 369-72T (1935).—Further expts. are described to show that Cr in chromite or in Cr<sub>2</sub>O<sub>3</sub> can be detd. rapidly and accurately by dissolving the sample in a mixt. of 1 part 72% HClO<sub>4</sub> and 2 parts H<sub>2</sub>SO<sub>4</sub> at 190°, cooling rapidly in ice water, dilg., boiling off Cl<sub>2</sub> and titrating with FeSO<sub>4</sub> soln. with o-phenanthroline-ferrous complex as indicator. Cf. C. A. 29, 6174, 6793, 7838.

System of qualitative analysis for the anions. J. T.

Dobbins and H. A. Ljung *J. Chem. Education* 12, 580-8 (1935).—Anions such as are likely to be present in salts given to students in elementary qual. analysis are divided into 5 groups as follows: Group I,  $\text{CO}_3^{--}$ ,  $\text{F}^-$ ,  $\text{C}_2\text{O}_4^{--}$ ,  $\text{AsO}_4^{--}$ ,  $\text{AsO}_3^{--}$ ,  $\text{PO}_4^{--}$  and  $\text{C}_2\text{H}_3\text{O}_2^{--}$ , pptd. by  $\text{Ca}(\text{NO}_3)_2$  in slightly basic soln. Group II,  $\text{SO}_4^{--}$  and  $\text{CrO}_4^{--}$ , pptd. by  $\text{Ba}(\text{NO}_3)_2$  in slightly basic soln. Group III,  $\text{CN}^-$ ,  $\text{BO}_3^{--}$ ,  $\text{I}^-$ ,  $\text{Br}^-$  and  $\text{Cl}^-$  pptd. by  $\text{AgNO}_3$  in dil acid soln. Group IV,  $\text{ClO}_4^-$ ,  $\text{NO}_3^-$  and  $\text{C}_2\text{H}_3\text{O}_2^-$  detected in the filtrate from the above group tests. Group V,  $\text{NO}_2^-$ , detected in the original substance.

New device for the rapid determination of certain gases or vapors in the atmosphere Kohn-Abrest *Ann. f. d. 28*, 547-61 (1935).—This is an improvement on the previously described portable app. (*C. A.* 13, 1938, 28, 63 (1932)) which is suitable for the detn. of  $\text{CO}_2$  and halogen derivs. in the atm. or in viscera. The air is drawn through a soln. of  $\text{Ba}(\text{OH})_2$  (absorbing  $\text{CO}_2$  which can be detd. if required), through a 25-cm. quartz tube of 5-7 mm. bore heated electrically to 850-900°, and through a 2nd  $\text{Ba}(\text{OH})_2$  washer which absorbs the products of combustion.  $\text{CO}_2$  is most conveniently detd. in each of the  $\text{Ba}(\text{OH})_2$  washers by adding 2 drops of a 1 l. mixt. of 1% phenolphthalein and 1% lueanthin in alc. and titrating with 0.125 N  $\text{HNO}_3$ , the 1st end point (red to light yellow) indicates neutralization of the excess  $\text{Ba}(\text{OH})_2$ , and the 2nd end point (yellow to pink) indicates decompn. of the  $\text{BaCO}_3$ . Halogens and  $\text{HCN}$  are detd. in the usual way in the titrated soln. When relatively high boiling alkyl halides are suspected, the first washer should preferably be maintained at 60-70°.

A Papineau-Couture  
The detection of respiratory poisons Gerhard Stampe *Drugs-Heft* No. 180, 2904-6 (Sept.-Oct., 1935).—A description of the app. and methods used in detecting the presence of poisonous gases. Of special interest is the description of a new specific test for mustard gas which is based on a reaction with certain gold salts.

A. L. K.  
Method for making latent fingerprints visible M. Wogenarr. *Pharm. Weekblad* 72, 1265-71 (1935).—The object bearing the latent fingerprint is placed under a Petri dish cover on the inner side of which a few I crystals have been fused. The I vaporizes and is absorbed by the fingerprint. The image thus obtained is evanescent, but

a permanent copy can be made by covering with a sheet of slightly moist paper contg. a paste of rice starch and KI, and finally varnishing the paper with a 3% soln. of dammar resin in  $\text{C}_2\text{H}_5$ . Several copies of the same fingerprint can thus be made. The color of the print is bluish brown.

A. W. Dux  
Colorimetric determination of salicylic acid. J. L. Heesterman. *Chem. Weekblad* 32, 403 (1935); cf. *C. A.* 29, 42914.—The violet color of  $\text{FeCl}_3$ -salicylic acid is stable in the presence of  $\text{AcOH}$ . To 100 cc. of soln. add 2 drops  $\text{FeCl}_3$  soln. (1%  $\text{M}$ ) and 1 cc. 4 N  $\text{AcOH}$ ; less than 1 mg. salicylic acid can be detd.

B. J. C. v. d. H.  
The potentiometric determination of oxalate with silver nitrate P. Sprau *Z. anal. Chem.* 103, 272-4 (1935).—E. Müller (*Die elektrometrische Massanalyse*, *C. A.* 21, 39) states that the potentiometric titration of  $\text{C}_2\text{O}_4^{--}$  with  $\text{Ag}^+$  is impossible, but if to approx. 2.55 ml. of 0.25 N  $\text{Na}_2\text{C}_2\text{O}_4$  soln. there is added 80 cc. of 60% alc. a good potentiometric end point can be obtained in titrating with  $\text{AgNO}_3$  soln. As electrodes a wire of Ag and a calomel half cell were used.

W. T. H.  
Rapid identification of methyl anthranilate. Sébastien Sabatay *Ann. f. d.* 28, 478-9 (1935).—Heat 4 drops of methyl anthranilate and 12 drops of a 1.2  $\text{Ac}_2\text{O}$ - $\text{C}_2\text{H}_5\text{N}$  mixt. in a test tube for 5 min. in a boiling water bath, add a few cc. of  $\text{H}_2\text{O}$ , heat a few min. longer, cool; on rubbing with a glass rod, a cryst. mass of methyl *N*-acetyl anthranilate seps out which, after recrystn. from aq. alc., in 90-100°. For the detn. of methyl anthranilate in neroli, the generally used Hesse and Zeitschell method (*Ber.* 34, 296 (1901)), based on the quant. sepn. of the sulfate from an  $\text{Et}_2\text{O}$  soln. of the oil, is rather long and requires a large sample, the Zeisel method gives equally accurate results (in absence of  $\text{EtOH}$ ) on a 0.5-2-g. sample.

A. P.-C.  
Levulose reaction with dinitrobenzenes. Ladislav v. Székényi-Nagy. *Biochem. Z.* 281, 175-7 (1935).—The color reaction with levulose ascribed to *m*-dinitrobenzene is due to *o*-dinitrobenzene present as an impurity. Samples of pure *m*-dinitrobenzene gave no color reaction. Only *o*-dinitrobenzene (m. 115.9°) is therefore a suitable color reagent for levulose.

S. Morgulis  
Detn. of  $\text{P}_2\text{O}_5$  by pptn. as  $\text{NH}_4$  phosphomolybdate (Terlet, Briaux) 15. Detn. of I in org. substances (Kahane, Tomesco) 6

## 8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND J. F. SCHLAIRER

Some properties of opal. N. L. Tahaferro. *Am. J. Sci.* 30, 450-74 (1935).—Natural opals and pure cherts are hardened, greatly hydrated silica gels. In nature they dehydrate and contract in a manner similar to the dehydration of such gels in the lab. As a gel, opal or chert dehydrates there is a steady and const. loss of vol. and a const. structural modification which causes a progressive increase in both  $n$  and sp. gr. with loss of  $\text{H}_2\text{O}$ . When  $n$  and  $d$  are plotted vs.  $\text{H}_2\text{O}$  content the points lie scattered between the theoretical amorphous  $\text{SiO}_2$ - $\text{H}_2\text{O}$  curve and that of the theoretical system cristobalite- $\text{H}_2\text{O}$ . The measured values are always higher than the computed theoretical values. X-ray patterns of a no. of opals, cherts and  $\text{SiO}_2$  gels show faint, but unmistakable  $\beta$ -cristobalite patterns, even though the substances have never been heated above 100°. The departure of  $n$  and  $d$  from the theoretical amorphous  $\text{SiO}_2$ - $\text{H}_2\text{O}$  curve is due to the tendency toward a denser atomic arrangement in the direction of the  $\beta$ -cristobalite modification. This arrangement of the atoms is not a function of the aging of the gel, opal or chert, but appears to take place at an early stage in their formation while there is yet a great deal more  $\text{H}_2\text{O}$  than  $\text{SiO}_2$  present.

Alden H. Emery  
Morphological reference directions of plagioclases and their suitability for determining the anorthite content. G. Manolescu. *Schweiz. mineral. petrog. Mit.* 14, 452-63

(1935).—The chief cause of uncertainty in the compn. of a plagioclase as deduced by the Fodorov method is that the twinning plane is often a vicinal face, though there is also frequently a small residual variation not so explicable.

C. A. Silberrad  
Bronzite rock from Loderio (Blenio tal, Ticino). F. de Quervain. *Schweiz. mineral. petrog. Mit.* 14, 447-9 (1935).—Near Loderio (Blenio valley, Ticino) in the peridotite occur blocks of bronzite in more or less radial tufts of crystals, but showing no resedimentation; d. 3.232,  $n_\alpha$  1.602,  $n_\beta$  1.607,  $n_\gamma$  1.674. A very pure crystal contained  $\text{SiO}_2$  56.49,  $\text{FeO}$  9.67,  $\text{MgO}$  33.83 and  $\text{MnO}$  0.06%. In places the bronzite has passed into bastite, and more frequently into a talc-magnetite mass.

C. A. S.  
The water content of clay substances. O. Koerner, K. Fukaki and H. Salmann *Z. anorg. allgem. Chem.* 225, 69-72 (1935).—At 11.5°,  $\text{H}_2\text{O}$  could be pumped off of Zeolith kaolin so that it had the compn.  $\text{Al}_2\text{O}_3$  2 $\text{SiO}_2$  - 2 $\text{H}_2\text{O}$ . No steps were found in the isothermal diagrams up to 445°; hence 0.5 mol. of  $\text{H}_2\text{O}$  is ad-sorbed by the air-dried material, and only 1.5 mol. is chemically combined. Detns. lasting 7 and 18 months, resp. were made. Decompn. takes place as follows:  $\text{Al}_2\text{O}_3$  2 $\text{SiO}_2$  1.5 $\text{H}_2\text{O}$  aq.  $\rightarrow$   $\text{Al}_2\text{O}_3$  2 $\text{SiO}_2$  1.5 $\text{H}_2\text{O}$   $\rightarrow$   $\text{Al}_2\text{O}_3$  2 $\text{SiO}_2$  0.5 $\text{H}_2\text{O}$   $\rightarrow$   $\text{Al}_2\text{O}_3$  2 $\text{SiO}_2$  aq.  $\rightarrow$   $\text{Al}_2\text{O}_3$  2 $\text{SiO}_2$ .

E. R. Rushton



Morphological types of Swiss titanite Robert L. Parker *Schweiz mineral petrog Mitt* 14, 478-506 (1935) —Crystallographic C A Silberrad

X ray examination of turnerite P Bearth *Schweiz. mineral petrog Mitt* 14, 442-6(1935) —X-ray examination of turnerite from near Sedrun in Val Tavetsch and comparison of the intensities of the various lines with those produced under similar conditions by a mixt. of known compn give the compn (to an accuracy of  $\pm 5\%$ ) as  $\text{ThO}_2$  3,  $\text{V}_2\text{O}_5$  1.4,  $\text{La}_2\text{O}_3$  12,  $\text{Ce}_2\text{O}_3$  30,  $\text{Pr}_2\text{O}_3$  4,  $\text{Nd}_2\text{O}_3$  68,  $\text{Sm}_2\text{O}_3$  2, 4,  $\text{Gd}_2\text{O}_3$  2,  $\text{Dy}_2\text{O}_3$  1,  $\text{Er}_2\text{O}_3$  less than 1, total 12, the remainder being substantially  $\text{P}_2\text{O}_5$  C A S

Rocks and mineral deposits of the Binnental Henri Bader *Schweiz mineral petrog Mitt* 14, 319-441 (1935) —A detailed account, based on previous work (1933 references) and 4 years observations of B. The rocks are described with their proximate constituents and numerous complete analyses, 8 of which (of albite gneisses, biotite schist, talc, phlogopite, asbestos and hornblende) are new. Except the Fe ores the minerals occur in druses (almost confined to the dolomite) or clefts, and are due to the action of hydrothermal solns on the country rock. The various occurrences, including 5 new ones, are described with the paragenesis of the minerals concerned at each, and full accounts compiled of all the minerals (43) found. Of special interest is a crystal of quartz found in a cleft in dolomite, contg inclusions (largest  $10 \times 4 \times 1 \text{ mm}$ ) of liquid  $\text{CO}_2$ , indicating that the crystal was formed under a pressure of 300-550 atm at  $150-370^\circ$  C A Silberrad

Deposits of iron and metal ores of the Rhenish shale mountains and their significance for German economics W Henke *Metall u Erz* 32, 505-11(1935) H S

By what conditions or influences has metal enrichment in middle German copper shale been effected? G Galtzter *Metall u Erz* 32, 533-42(1935) —A study of the geologic forces involved H Stortz

The origin of petroleum Karl Kreyer Graf *Naturwissenschaften* 23, 599-605(1935), cf C A 29, 7879<sup>1</sup> —A review B J C van der Hoeven

Some igneous rocks from the iron producing district of Bulbaio, Province of Vizcaya, north Spain Harold Service *Bull Inst Mining Met* No 374, 23-7(1935), cf C A 29, 7878<sup>1</sup> —Discussion Alden H Emery

Petrographic and tectonic investigation of central granite of Upper Baltscheider Valley W Minder *Schweiz mineral petrog Mitt* 14, 150-75(1934) —After a brief description of the Baltscheider granites the central granite is described in detail with the connected aplite, lamprophyre and inclusions. Numerous chem analyses are given and compared with the results of measurement of thin sections. There is also a variation diagram, and the magmatic differentiation of the different varieties is discussed C A Silberrad

Percy ring-dike complex Randolph W. Chapman *Am J Sci* 30, 401-31(1935) —The ring-dikes and stocks of syenite and granite are described and their origin is discussed Alden H Emery

Basic dike rocks from neighborhood of Brissago (Ticino) Conrad Burri and F de Quervain *Schweiz mineral petrog Mitt* 14, 507-17(1935) —These dike rocks are true gabbrs with proximate compn hornblende with ilmenite 40-60, plagioclase (mostly  $\text{An}_{60-80}$ ), but in one place  $\text{An}_{60-80}$ , and in another  $\text{An}_{80}$  30-45, biotite 12-10.6, titanite 0-2.2, ore (sep ilmenite) 12-2.9%. Thus, as also does a complete analysis, shows very low alkali, the rock falling in Niggli's pyroxene-hornblende-gabbro class C A Silberrad

Kaolin and clay Their geologic origin Albert Vasek *Keram Runtschau* 43, 517-0(1935), cf C A 30, 828<sup>1</sup>

—A review V. maintains that the formation of kaolin or of clay is independent of the weathering and is determined by the nature of the parent rock P S R

A recently discovered kaolin sandstone occurrence in the crystalline limestone of Pargass—Al Island, region of Abo—Turku, SW Finland H Hausen *Acta Acad Aboensis, Math Phys* 8, No 1, 59 pp (1934)(in German) —In the limestone quarry at Skrabblö a kaolin bed was discovered, the deeper part of which appeared to fill a cavity. This kaolin is plastic and is of a predominantly chocolate color. Over the kaolin is a stratum of light rust-colored, brittle sandstone, and over this a dark gray, argillaceous mass in which a no. of different crystal rocks are imbedded. H concludes from detailed study that there occurred (1) formation of deep, enclosed cavities in the limestone during a warm and moist climate; (2) energetic chem weathering of the adjacent crust, Al-rich silicate rocks (diopside, plagioclase, microcline, etc.) and formation of kaolin; (3) covering of the autochthonic kaolin with residual quartz sand from a distance; (4) sliding of the kaolin and sand into the cavity; (5) pre-Quaternary regional erosion; (6) commencement of the Quaternary glaciers and formation of a moraine, further forcing of the kaolin and limestone into the cavity; filling in with moraine clay, glacial removal of various pre-glacial deposits, and (7) at the end of the glacial period a deposition of a sandy moraine. The kaolin-sandstone deposit is considerably younger than the surrounding Archean rocks and older than the overlying glacial formations. Thirty-eight references D Thuesen

Dolomite in Permian limestones of West Texas William A Cunningham *Bull Am Assoc Petroleum Geol* 19, 1678-92(1935) —Analyses for  $\text{CaCO}_3$  and  $\text{MgCO}_3$  in approx 150 samples from 35 oil wells are tabulated. Most of the Blaine limestone in the Permian basin is 75% dolomite Alden H Emery

The travertine deposits near Port St Johns W. Kupferburger *S African Mining Eng J* 46, Pt. 1, 801-5(1935) —See C A 29, 7881<sup>1</sup> Alden H Emery

Chemical classification of metamorphic rocks Paul Niggli *Schweiz mineral petrog Mitt* 14, 464-72 (1935) —Classification of metamorphic rocks is specially difficult because of the difficulty of obtaining a fair average sample. That now proposed is based on chem compn. It consists of 12 groups, closely resembling those of Grubenmann I Alkali-alumino silicate; II Same, with decreasing alkali; III Lume-alkali aluminosilicate; IV Imme-aluminosilicate; V Femic-silicate; VI Femic or Ca rich alkali silicate; VII Aluminosilicate, Al rich; VIII Silica rocks, Si-rich; IX Limestones; X Carbonate; XI Femic-oxide; XII Alumino-oxide rocks. The limits of each group are expressed in N's notation, and exemplified by diagrams, with a statement of the chief rocks falling in each group. C A S

Petrography and geology of the Silretta crystallines (Lower Engadine) Ed Wenk *Schweiz mineral petrog Mitt* 14, 196-278(1934) —Apart from geological details the paper contains descriptions, with proximate constituents, of the various amphibolites, para-, ortho- and mixed gneisses of the area, with a more detailed account of the Monchalp gneiss, showing it to be probably the result of assimilation. A detailed account of mylonitization as it affects the various rocks and their constituent minerals follows, especially as regards orthoclase which is converted into a quartz-sericite via microcline, quartz and albite, and the plagioclases, which are more decomposed the more cataclastic and mylonitized the rocks are in which they occur C A Silberrad

Development of torbanite in South Africa (Duff) 22

## 9—METALLURGY AND METALLOGRAPHY

D J DENOREST, OSCAR E HARDER AND RICHARD RIMBACH

Tabular index of Bureau of Mines Information Circulars on mining and milling methods J R Thoenen *Bur*

*Mines, Information Circ* No 6858, 105 pp (1935).

Alden H Emery

value of various sands, (2) expansion of air, (3) effect of gaseous expansion upon the permeability no, (4) dried permeability value of various sands after correction for expansion of air, (5) dried permeability values obtained on sillimanite bonded with 5% Na silicate, (6) expansion of various sands, (7) expansion of sillimanite, (8) dried compression strength of various sands, (9) dried compression strength of various sands after cooling from elevated temps and (10) max temp reached by the sand in the molds for castings of various thickness Downs Schaaf

Metallurgical researches of the national physical and technical institute for 1934 A Schulze *Z. Metallkunde* 27, 139-40(1935)—A progress report of investigations on the chem and phys properties of metals and alloys, dealing especially with the prepn of high-purity materials, magnetic, thermal and elastic properties, low-temp work, and the development of special app

The elastic limit and the engineer. W. Späth. *Z. Metallkunde* 27, 132-6(1935)—Phenomena which occur when materials are stressed below the endurance limit are analyzed by means of a mech model This is a set of springs, of the material in question, whose displacement due to loading is opposed by a resistance element placed 90° to the direction of load After-effects, damping and the raising of the endurance limit due to loading are interpreted in terms of the model and related to irregular stress distributions along the "structural imperfections" of Smekel Conclusion The mech testing of material can be divided into elastic and inelastic regions, these being sep'd by the endurance limit, and the elastic limit is of little value to engineers until the effects related to it are better understood G Derge

Properties of metals at low temperatures A S Fal'kevich and B A Smurnov. *Khimstrol* 7, 436-42(1935)—Different Fe and Cu alloys, with and without elec welded seams, were treated with boiling liquid O and at from -183° to 50° either once or repeatedly for various periods of time, and then subjected to phys and mech tests The results are presented in graphs and tables and discussed Chas Blanc

Compression requirements for Avional samples K Guler *Z. Metallkunde* 27, 137-38(1935)—Stress-strain curves were obtained for a high-quality Avional contg Si 0.7, Mn 0.7, Mg 0.7 and Cu 4% The compressional elasticity modulus was calcd from these and related to the shape of the specimen The compression expts were made on an app. designed to insure uniform application of load G Derge

Developments and results of investigations of hardening phenomena G Wassermann *Arch. Eisenhüttenw* 9, 241-5(1935)—The discovery of age hardening in Duralumin and the general principles of methods based on it to make metals stronger and harder by natural or artificial means are explained More recent investigations have shown that pptn hardening is preceded by a series of other preparative conditions which are of importance for age hardening Thirty-three references M H

The quenching power of liquid hardening agents K G. Speith and H. Lange *Mitt Kaiser-Wilhelm-Inst Eisenforsch., Dusseldorf* 17, 175-84(1935)—Temp. control in quenching is discussed The cooling velocity is of paramount importance, it depends, at high temps, on the thickness of the vapor film produced around the object to be hardened Lab tests indicate that the quenching power of water can be influenced by addn of "Pektinit," a trade prepn consisting essentially of pectins; an addn of 6-7% produces in water the same effect as that of the usual hardening oils Dried water quenches considerably more slowly than tap water. Cooling curves were det'd in different quenching solns M Hartenheum

New materials for permanent magnets A Kussmann *Z. Ver deut Ing* 79, 1171-3(1935)—Considerable progress in producing permanent magnet steel has resulted from the research of recent years Characteristics of alloy steels formerly used are briefly reviewed Fe-Ni-Cu alloys of the pptn hardening type show magnetic proper-

ties similar to Co steel and are quite workable. The most important group of new permanent magnet steels consists of the Ni-Fe-Al alloys contg. approx. 25% Ni, 10% Al and the remainder Fe (with addns. of Co, Mo, Cr, etc.). The relation between the structure and magnetic behavior of these alloys is briefly discussed. A permanent magnet material is prep'd by sintering ferro magnetic oxides, finely divided Co ferrite ( $\text{CoFe}_2\text{O}_4$ ) and  $\text{Fe}_2\text{O}_3$  contg.  $\text{Fe}_2\text{O}_3$ , and pressing into the desired form An alloy of Pt-Fe with about 78% Pt surpasses all the alloys mentioned in magnetic properties but the cost is too high.

C B Jenni  
Defects in the manufacture of sheet silver III. E. Raub. *Mitt Forschungsanst. Proberamt Edelmetalle* 9, 69-75(1935)—Defects in Ag sheets may be due to impurities which entered the metal during the mfg process (cf *Ibid.* 6, 29(1932); *C. A.* 26, 4018). The present article deals with the influence of pouring temp on the technological properties of Ag alloys and is illustrated by photographs of structures and defects in casting and rolling A definite temp cannot be given as correct pouring temp as this depends on kind of mold (chill mold, sand mold, etc.), shape and capacity of the mold and on the compn of the metal. Only an empirical method will be able to take all factors into account. A few examples show the effects of wrong temps M Hartenheum

Effect of oxygen and nitrogen on the change of ferrite W. Eilender and H. Cornelius *Arch. Eisenhüttenw* 9, 263-4(1935)—Investigations did not reveal a connection between the degree of change in ferrite and the N and O contents. There seems, however, to exist a relation between the  $\alpha$  change and the stresses due to the  $\gamma$ - $\alpha$  transformation Impurities appear to have only a minor effect. The results are illustrated by photographs of the structures M. Hartenheum

Rearrangements in iron between 70° and 700° G. Naeser. *Mitt Kaiser-Wilhelm-Inst. Eisenforsch., Dusseldorf* 17, 185-90(1935)—By detg. the av. sp heat of pure Fe between 70° and 700°, the curve showed 6 distinct, reversible irregularities at 112°, 198°, 292°, 388°, 487° and 591°. These irregularities seem to point to a relation between rearrangement of the mols and the ferro-magnetism of the Fe The distances between the points of irregularity and the heat liberated at these points seem to follow a simple law. More expts are required for full elucidation Thirty-eight references M H

A study of the heats of transformation in iron H. v. Steinwehr and A. Schulze. *Z. Metallkunde* 27, 129-132(1935)—The heats of transformation at the  $A_1$  and  $A_2$  points of vacuum melted electrolytic Fe, contg. 0.03% Mn and a trace of Si, are det'd by a calorimetric procedure already described (*C. A.* 28, 5025). In a series of 5 expts the magnetic transformation was found to begin near 790° and end at 710° giving an av heat of transformation of  $4.8 \pm 0.02$  cal per g. The change from  $\gamma$ - to  $\alpha$ -Fe began at 894° and ended at 870°. For 6 runs the av. value for the heat evolved was  $6.2 \pm 0.03$  cal per g. G Derge

Influence of chemical reactions upon the fatigue of iron F. Roll *Z. anorg allgem. Chem* 224, 322-4(1935)—The effect of oxidation of the metal surface upon its fatigue strength is det'd With Fe this effect varies depending upon what oxide is formed upon the surface H S

Types of cast iron for use at elevated temperatures Lemoine. *Usine* 44, No 44, 27(1935)—Results of recent investigations of heat-resistant cast Fe are summarized Growth and permanent expansion of cast Fe under repeated heating are caused by decompn of the free cementite and the cementite of the pearlite into ferrite and graphite. The outer layers begin to graphitize under heating before the heat has penetrated into the interior, so that cracks develop which also contribute to a growth in vol. Into these cracks, gases of a more or less corroding nature can enter and form Fe compds which increase the wt. and vol Remedies are a cast Fe which remains pearlitic up to 600-50°, or, for temps up to 800-900°, a cast Fe for which the pearlitic structure is not indispensable, by adding 1-2% Cr or diminishing the C content to 2.2-

2.5%. The properties of cast irons of these types are described. M. Hartenheim.

Retardation of the solution of iron in sulfuric acid. V. F. Postnikov and I. P. Kirillov. *Khimizol* 7, 431-4 (1935); cf. Brauer, *J. Chem. Ind. (Moscow)* No. 9, 64(1935).—On the addn of 0.5%  $\text{CS}(\text{NH}_4)_2$  to 20.6%  $\text{H}_2\text{SO}_4$ , the rate of soln. of Fe was reduced to  $1/10$  in 6 hrs. and to  $1/100$  in 72 hrs. Chas. Blane.

Recrystallization of electrolytic iron. G. Wassermann. *Mitt. Kaiser-Wilhelm-Inst. Eisenforsch., Düsseldorf* 17, 203-6(1935).—Rolled electrolytic Fe crystallizes with a fine grain, heating beyond the  $A_1$  point produces, however, a fairly coarse grained structure. By quenching from the  $\gamma$ -state a finer recrystallized grain can be obtained. If electrolytic Fe is cooled from the  $\gamma$ -state so that the transition temp. progresses gradually along the specimen, narrow, long needle crystals are formed, the shape of which is the more perfect the slower the cooling. Adjoining  $\alpha$ -crystals act frequently as nuclei. Armco Fe and C-poor ingot Fe behave differently from electrolytic Fe in recrystn and transformation, the recrystd. structure is always of very fine grain and does not show the narrow needle crystals. The formation of the latter is ascribed to the fact that the no. of nuclei of  $\alpha$ -crystals in the  $\gamma$ - $\alpha$  transformation is at first very small and becomes larger only with increasing undercooling. M. Hartenheim.

X-ray investigation of stress distribution and excess stresses in steel. H. Möller and J. Barbers. *Mitt. Kaiser-Wilhelm-Inst. Eisenforsch., Düsseldorf* 17, 157-60(1935).—The x-ray reflection method is particularly suitable for the study of whether the occurrence of excessive internal stresses is connected with the existence of nonuniform stress distribution and if a relation between these 2 quantities exists. The equipment and theory of the tests are described in detail. A simple linear relation between load and stress exists only under comparatively small loads. Under higher loads, the stress distribution becomes very irregular even below the flow limit. There occur differences in tension between adjoining groups of crystallites, even under theoretically uniform load, which are equal to those produced intentionally in the bending test. This causes in some crystallites a reduction, in adjoining crystallites an increase, in stress. Nine references. M. Hartenheim.

The properties of some low-nickel steels containing manganese. R. H. Greaves. *Engineering* 140, 693-4 (1935).—See C. A. 29, 79114.

Silicon steel in communication equipment. C. H. Crawford and E. J. Thomas. *Elec. Eng.* 54, 1318-53 (1935).—The different grades of Si steel and other magnetic materials are discussed, particular emphasis being given to the selection of the most satisfactory alloy for the applications in communication equipment, which includes transformers for radio frequencies, small rotating app. and relays. W. H. Boynton.

Plasticity and practical behavior of cast light alloys with reference to the bending test. Luigi Locati. *Industria meccanica* 17, 803-7, 802-8(1935).—The plasticity of material helps the stresses of unforeseen origin to remain in limits below the danger point even if they exceed the normal value. The other mech. properties, however, as elongation, must not be overlooked in this connection. The bending test alone will not give sufficient indication of behavior under stress; it can only give a certain quality factor which must be considered together with ultimate strength, elastic limit and max. deviation in bending. Twelve references. M. Hartenheim.

The influence of foreign elements of some industrial aluminum alloys. I. T. Kolenov and B. F. Grashchenko. *Legkie Metal.* 4, No. 4, 40-55(1935).—The influence of Fe, Si, Zn and Cu on the mech. and corrosion-resistant properties of Duralumin, Aludur and Silumin was investigated. H. W. Rathmann.

The influence of alloying elements on aluminum and magnesium alloys. A. S. Lugaskov. *Legkie Metal.* 4, No. 5, 32-4(1935).—L. discusses the influence of Be, Ca, Ce, Si, Mn and Na on the properties of Al and Mg alloys. H. W. Rathmann.

An x-ray study of the alloys of aluminum with barium. K. R. Andrews and L. Alberti. *Z. Metallkunde* 27, 120-8 (1935).—Alloys contg. 0.5% increments of Ba from 0% to the eutectic at 2%, and 5% increments from this point to 50% Ba were studied by back-reflection powder photographs. A const.  $a_0$  value for Al was found throughout this range, indicating no solid-soln. fields. Only one intermetallic phase, corresponding to  $\text{Al}_3\text{Ba}$  and 66% Ba, was observed. Photographs of cleavage sections of  $\text{Al}_3\text{Ba}$  showed that it may be considered as a body-centered tetragonal lattice of Ba atoms with  $a = b = 4.53 \text{ \AA}$ ,  $c = 11.1 \text{ \AA}$ , whose interstices are filled with Al atoms in the positions of the 2 types (00  $u$ ) and (01  $u/2$ ), and  $u$  is detd as 0.28. G. Derge.

The influence of small excesses of magnesium or zinc on the hardening of alloys of aluminum with  $\text{MgZn}$ . R. Schmitt. *Z. Metallkunde* 27, 121-6(1935).—A series of 17 alloys contg. 2.6, 14%  $\text{MgZn}$ , with excesses of 1.15-6.4%  $\text{Mg}$  and 1.7-6.8%  $\text{Zn}$  is annealed at  $475^\circ$  and water quenched, then aged at room temp.,  $80^\circ$ ,  $125^\circ$ ,  $150^\circ$  and  $175^\circ$ . The changes are followed by means of Brinell measurements. After the anneal all samples showed a softening from the as-cast condition, followed by a hardening at room temp. which did not reach a max. after 33 days. The aging data are plotted separately for each alloy. All showed an increase in hardness at all temps., which was asymptotic to the time axis below  $150^\circ$ , but at  $175^\circ$  the hardness decreased after reaching a max. Conclusion. The process is complicated but dependent upon the  $\text{MgZn}$ , primarily, the effect of  $\text{MgZn}$ , being greater from 6-9% than from 0-14%. G. Derge.

Modulus of elasticity of beryllium-copper alloys. Léon Guillet, Jr. *Compt. rend.* 201, 1049-2(1935).—The hardness,  $\Delta$  (Rockwell), modulus of elasticity,  $E$ , and rate,  $H$ , of vibration of the note given out by cylindrical samples  $20 \text{ cm} \times 20 \text{ mm.}$ , were detd. for Be-Cu alloys contg. 2-18.5% Be (with 0.02-0.12  $\text{Fe}$ , 0.1-0.54  $\text{Si}$ , 0-0.21  $\text{Al}$ , 0-0.3  $\text{Mg}$ , 0-0.5  $\text{Mn}$  and 0-0.05  $\text{Zn}$ ).  $E$  increases linearly (70-110) until the amt. of Be is 11%, i.e., until the constitution of the alloy changes (cf. Masry and Dahl, C. A. 24, 3174).  $H$  increases linearly (12,000-20,000) throughout the series.  $H$  also increases with content of Be, but at a decreasing rate. C. A. Silberrad.

Magnetic alloys of iron, nickel and cobalt. G. W. Timen. *Elec. Eng.* 54, 1202-9(1935).—A paper dealing largely with the phys. and elec. properties of the permalloys, permivar and permendur. Curves and tables and 7 references are included. W. H. Boynton.

Iron-cementite-manganese carbide-manganese. Rudolf Vogel and W. Döring. *Arch. Eisenhüttenw.* 9, 217-62(1935).—Former investigations of this system and parts of it were enlarged and completed by establishing the constitutional diagrams on the basis of heats of reaction and structure. Mn and  $\text{Mn}_3\text{C}$  do not form, as indicated previously, an uninterrupted series of solid solns with a max. temp.; a transition equl. occurs at solidification between 1.5 and 2.9% C which leads toward higher C contents, to a min. temp. A new transformation of Mn carbide was observed at  $1070^\circ$ . Four kinds of crystals were found in the system  $\text{Fe-Fe}_3\text{C-Mn}_3\text{C-Mn}$ : Ternary solid solns of  $\alpha(\delta)$ - $\gamma$  e with C and Mn which are restricted to a small range in the Fe corner; ternary solid solns. of  $\gamma$ -Fe with C and Mn with a very extended range; ternary solid solns. of  $\gamma$ -Mn with C and Fe (area  $\alpha + \beta_{\text{Mn}}$ ); and solid solns.  $\text{Fe}_3\text{C}$  and  $\text{Mn}_3\text{C}$  (area  $\text{C} \beta_{\text{Mn}}$ ). The cementite does not form solid solns. with the excess Fe, but the  $\text{Mn}_3\text{C}$  does with excess Mn. The conditions are explained in detail by the diagram. A table shows the location of the characteristic points  $\alpha$ ,  $\beta$ ,  $\gamma$ ,  $\delta$  and  $\epsilon$  for the equilibria of the Fe-Mn-C alloys in solid state. The various structures are illustrated in photomicrographs. M. Hartenheim.

Heat conductivity of pure iron and commercial steels. F. Hollenrath and W. Rungt. *Arch. Eisenhüttenw.* 9, 233-62(1935).—Recent literature on heat cond. is reviewed for practically pure Fe, alloy and plain steels at different temps. in order to derive useful values. This was possible, however, only for plain steels at room temp.

All other information for alloy steels contains so many gaps and contradictions that the influence of the alloying material can be determined only approx. A few curves and numerical values are given for com. steels. Thirty-eight references. M. Hartenheum

**The corrosion problem. Paints and anticorrosive coatings.** Georges Genin. *Industrie chimique* 21, 898-902 (1934), 22, 93-7, 250-5, 402-5, 488-91, 652-4 (1935).—A general discussion of the various aspects of the problem of corrosion in industry, with a review of the means at present available for preventing or combating it, more particularly in the chem. industry. A. P.-C.

**Corrosion in the production of ethylene chlorohydrin.** L. V. Iskra. *J. Chem. Ind. (Moscow)* 12, 947-53 (1935).—Mixts of  $C_2H_5(OH)Cl$ ,  $HCl$ ,  $C_2H_5Cl$ , and  $Cl_2$  such as are used in prep.  $C_2H_5(OH)Cl$  corrode Pb strongly, and the degree of corrosion increases with temp. Acid resisting lacquers and many alloys are badly affected. Acid-resisting cements and Fe contg 13-17% Si are not greatly corroded. A Havg type resin alone is slightly corroded, but when deposited on an Fe surface it resists corrosion. H. M. Leicester

**Substances inhibiting corrosion of iron by acids.** Jacques Bancelin and Yves Cramail. *Compt. rend.* 201, 1033-4 (1935).—While the inhibitory action of rhodamine on the corrosive action of 0.1 N HCl on steel continually increases with concn of rhodamine that of thiourea at first increases, and then decreases with a max. effect for a concn of about 50 mg/l. This concn (c) gives the min. e. m. f. in the cell steel/0.1 N HCl + thiourea (c)/ $Hg_2Cl_2/Hg$ , whereas with rhodamine the e. m. f. decreases continually as concn increases. C. A. Silberrad

**Study of the influence of cold work on the intergranular corrosion of 18-8 rustless steels.** A. Van den Bosch and M. Vialle. *Métaux* 10, 68-75 (1935).—The authors discuss intergranular changes which take place in reheating the annealed 18-8 Cr-rustless steels, the changes on reheating the cold worked material, the pptn. process of the carbides, the rate of heating that produces changes and the temp. range of susceptibility to alteration, the stabilization of the steel, starting with cold-worked metal and its application to welding. Regarding the use of stabilized 18-8 steel, they draw the following conclusions: (1) This steel can be arc welded without alteration. (2) Carbides may ppt. at the grain boundaries when the steel, while not completely stabilized, is used at a temp. below the temp. of stabilization. G. T. Motok

**Rammed mixes for cupola furnaces (Borsch)** 19 Preventing (metal) corrosion and paint (Schleife) 26 Effect of thermal agitation on ar. arrangement in alloys (Bragg, Williams) 2 Pneumatic process for refining ores (Ger. pat. 620,489) 21

**Froth flotation process and apparatus.** Thomas J. Taplin. Brit. 434,352, Aug. 30, 1935. To insure good contact between the air bubbles and the mineral particles, a vibratory movement is imparted to the pulp in addn. to the normal movement produced in the aeration by agitation or by the direct introduction of air, the vibratory movement being produced by imparting vibration to an impermeable surface in contact with the pulp on 1 side only.

**Leaching ores to separate lead and other metals from zinc sulfide.** Maxwell G. Platten. U. S. 2,021,896, Nov. 26. Lead zinc ores, etc., contg. ZnS are leached with a soln. of a chloride of Na, K, Ca or Mg not contg. over about 1% of acid such as HCl, etc., in excess of that neutralized by the acid neutralizing substances in the material being leached, and contg.  $FeCl_3$  as active solvent, thus selectively dissolving the Pb without dissolving the ZnS.

**Treatment of ores, etc., containing iron, nickel and copper.** The International Nickel Co. Inc. Ger. 620,419, Oct. 21, 1935 (Cl. 40a 13.50). See Can. 323,286 (C. A. 26, 3993).

**Ores of lead and zinc.** Ernst Bierbrauer. Fr. 780,684, Sept. 7, 1935. Ores of Pb and Zn are sepd. by selective

flotation; ligninsulfonic acid, its salts or derivs. are used to render the blende passive.

**Purifying zinc ores.** National Processes Ltd. and Thomas B. Oyles. Brit. 433,801, Aug. 22, 1935. Pb and Cd are removed from Zn sulfide ores, concentrates, etc., by a blast-roasting operation in which the ore, etc., is mixed with a granular or nodular material in such quantity as to insure a high porosity in the charge and a Pb content below 1.5% thereof, and in which the draft through the charge bed and the fuel value of the charge are adjusted to give a temp., e. g.,  $1300^\circ$ , above that required to eliminate S without the Pb content of the exit gases exceeding 250 mg per cu. ft.

**Reducing ores of zinc.** American Smelting & Refining Co. Fr. 780,688, Sept. 7, 1935. The ore in intimate contact with a reducing agent such as C and in a finely divided state is passed through a retort heated to and maintained at the temp. of reduction of the ore, so that the ore is reduced in a practically instantaneous manner. The vapors are removed through a coke filter maintained under conditions such that  $CO_2$  is reduced to CO, and the Zn is condensed. App. is described.

**Apparatus (with a series of bowls arranged in step formation) for separating materials such as gold and sand.** Michael Schlank. U. S. 2,023,926, Dec. 3. Various structural, mech. and operative details.

**Treatment of metallurgical by-products.** Wilhelm Witter. Ger. 620,263, Oct. 17, 1935 (Cl. 40a. 15.01). Powd. mixts of metals with oxides or sulfides, obtained as by-products in refining metals and particularly in removing Cu, Zn and Sn from Pb, are subjected to a winnowing process to sep. the metallic and the nonmetallic components. The fractions are then worked up separately in known manner. As a modification, the nonmetallic components may be withdrawn by an air stream from the surface of the metal bath being refined.

**Treating slag.** Edward Perchard Dunn. Ger. 617,510, Aug. 20, 1935 (Cl. 80b. 22.01). App. for filtering, desulfurizing and mixing molten slag is described.

**Granulated slag.** Edgar E. Brohaus. U. S. 2,023,511, Dec. 10. Molten slag is disintegrated by violent agitation and there is added to it simultaneously sufficient water to produce a granulated product but so limited as to insure substantially complete vaporization of the water.

**Pot furnaces for melting metals, etc.** South Metropolitan Gas Co., Dean Chaudler and Alec J. Skinner. Brit. 433,127, Aug. 2, 1935. In a gas-fired furnace for melting metals or heating liquids, where flames and hot gases from burners, after passing upwardly around the pot, escape through a chamber to down flues having outlets at their lower ends, the inner face of the refractory wall is lined with cast-Fe plates which are spaced from the wall by ribs and have vertical ribs on their inner faces to divide the heating space into sep. passages.

**Regenerative furnace for heating metals or fusing ores.** Wilhelm Doderer. Fr. 786,704, Sept. 9, 1935.

**Pusher type furnace for heating metal articles.** Ernst Neubaus. Ger. 620,153, Oct. 15, 1935 (Cl. 18c 10.01).

**Rotating furnace for producing spongy iron.** Fried. Krupp Grusonwerk A.-G. Ger. 614,045, May 31, 1935 (Cl. 18a 18.03). Addn. to 610,520 (C. A. 29, 58027).

**Annealing furnace for iron, etc.** Hans-Arthur Schweichel. Ger. 616,788, Aug. 5, 1935 (Cl. 18c 10.01). Details of a water-cooled conveyor are given.

**Continuous furnace for annealing metal strip in which the strip hangs in a catenary between supporting rollers.** Birmingham Electric Furnaces Ltd., Alfred G. Lobley and Fred Kerfoot. Brit. 433,899, Aug. 22, 1935.

**Means for lifting sheet metal conveyed from an annealing furnace.** Vereinigte Stahlwerke A.-G. Ger. 620,154, Oct. 10, 1935 (Cl. 18c 11.20).

**Continuous pair furnace suitable for heating metal bars.** Ray M. Fenton. U. S. 2,023,318, Dec. 3. Structural, mech. and operative details of a furnace with an endless conveyor extending through a horizontal heating chamber.

**Heat treating furnace suitable for normalizing sheets.**

of steel. Frank A. Fahrenwald. U. S. 2,023,126, Dec. 3. Various structural, mech. and operative details.

Heat-treating materials as in annealing or normalizing steel strip. Albert N. Ous (to General Elec. Co.). U. S. 2,023,283, Dec. 3. Various details of app. and operation.

Apparatus for operating metallurgical Scotch hearths Hugh R. MacMichael (to American Smelting and Refining Co.). U. S. 2,023,484, Dec. 10. Mech. and operative details.

Molten metal displacement device suitable for use with furnace-charging apparatus Paul H. Beitman I. S. 2,022,334, Nov. 26. Structural and mech. features.

Means for charging blast and shaft furnaces Pierre François-Bongarcou Fr. 787,102, Sept. 17, 1935.

Stock support for furnaces such as those for heating metal billets, slabs, etc. John W. Cornston U. S. 2,022,640, Dec. 3.

Linnings for nozzles of foundry ladles Allmanna Svenska Elektriska Aktiebolaget Brit. 434,520, Sept. 3, 1935. The linings, consisting as usual of magnesite, or the oxide of Mg, Cr, Al or other earthy metal mixed in powder form with a binder, e. g., water glass, are heated to considerably below the burning temp. of the material, which then sinters when the hot metal is poured. Substances that lower the m. p. of the lining, e. g., quartz, CaF<sub>2</sub>, alkalis, may be added to the magnesite, etc., so that the bore of the lining increases by wear and compensates for the reduced head of molten metal during pouring.

Pot for holding and expelling molten lead or the like Luther H. Hopkins U. S. 2,022,372, Nov. 26. A construction is described by which city gas or air under pressure may be utilized for expelling molten material from the pot.

Castings of magnesium free aluminum-copper alloys Joseph A. Nock, Jr. (to Aluminum Co. of America) U. S. 2,022,686, Dec. 3. A Mg-free alloy contg. Al together with Cu-12 and Sn 0.005-0.1% is cast and, without aging, the casting is heat-treated by heating to between 400° and the temp. of incipient fusion and then rapidly cooling. The Sn improves the ductility of the product.

Powdering metals Walter Zeiss. Ger. 620,126, Oct. 14, 1935 (Cl. 49/3). See Fr. 780,698 (C. A. 29, 58014).

Fluxes Ernst J. M. Mattsson. Brit. 434,531, Sept. 4, 1935. Fe or steel articles to be coated by dipping in molten Al or an alloy contg. Al as its main constituent, e. g., 1 contg. at least 95% Al, are immersed in a molten flux bath that contains 1 or more fluorides, e. g., Na<sub>2</sub>AlF<sub>6</sub>, and 1 or more alkali fluorides, with or without other salts, e. g., ZnCl<sub>2</sub>, but which does not contain any compd. of Ca. The articles are left in the flux until oxides, etc., are removed and the articles are properly heated and are then transferred to the Al or Al alloy bath. In an example, the flux contains Na<sub>2</sub>AlF<sub>6</sub>, 10, ZnCl<sub>2</sub>, 20, NaCl 30 and KCl 40%.

Container for steel tempering oil Zimmerman & Co. Komm.-Ges. Ger. 617,567, Aug. 21, 1935 (Cl. 18 & 2 10).

Apparatus for forming and quenching automobile leaf springs, etc. John F. Beans (to First National Bank in Massillon, Ohio). U. S. 2,022,632, Nov. 26. Structural and mech. features.

Manufacture of seamless tubes such as those of ferrous metals by extrusion of pierced billet material Adolf Wefing (to Tubus A.-G.). U. S. 2,023,776, Dec. 10. App. and operative details are described.

Brazing together materials such as metal sheets Frank T. Cope (to Electric Furnace Co.). U. S. 2,023,354, Dec. 3. Various details of app. and operation are described, involving use of suction to reduce the pressure between parts to be brazed together.

Brazing joints of curved metal sheets such as automobile panel material. James Velko (to Murray Corp. of America). U. S. 2,022,795, Dec. 3. Various operative details are described, involving the use of a bimetallic strip formed of a layer of retaining material and of spelter material in which a flux is embedded.

Trays suitable for use in heat-treating metal or ceramic

articles. Henry H. Harris. U. S. 2,022,283, Nov. 26. Structural features.

Annealing. Thomas Bolton and Sons Ltd., Wm. E. Atkins and Walter Cartwright Brit. 133,534, Aug. 12, 1935. Current-collectors for traction purposes, e. g., trolley wheels, are made from a known Al bronze contg. a small proportion of Mn and (or) Ni and having a refined structure produced by heat treatment and (or) working. The heat treatment may comprise a normalizing at 400° for 21 hrs. after a quenching from 850°.

The collectors may be produced by casting a blank, forging at 800-900°, quenching and then heating at 400-450°. If the forging cannot be quenched, it may be heated to 850°, quenched and then reheated at 400-500°. In an example, the alloy contains Al 9-11, Mn 0.2-3.0 and, optionally, Ni 0.2-5.0%, the Mn and Ni combined being not more than 5%.

Annealing razor blades Hermann Schmidt. Ger. 617,514, Aug. 20, 1935 (Cl. 18 & 2 28).

Hard metal bodies such as tools Bruno Fethenheuer (to Deutsche Edelstahlwerke A.-G.) U. S. 2,023,413, Dec. 10. Articles such as tools are formed of a material comprising Ti carbide about 60-85%, a carbide of W, Mo or Ta about 10-20%, the remainder being an alloy of Co, Cr and W in the resp. proportions of 40-80, 20-35 and 25% or less. Cl. C. A. 29, 107<sup>9</sup>.

Rotary drum for cleaning and descaling castings, etc., provided with centrifugal devices to inject the cleaning material through apertures in the end walls of the drum. Walter Schemmann and Fritz Kottmann Brit. 434,712, Sept. 6, 1935.

Coated ferrous wire, etc. Frederick M. Crapo (to Indiana Steel & Wire Co.). U. S. 2,023,364, Dec. 3.

A ferrous metal core carries a hot-galvanized Zn coating with an overlying coating of electrodeposited Cd. Cf. C. A. 29, 722<sup>9</sup>.

Coating iron with aluminum. Franz Nilsson, Fr. 780,645, Sept. 6, 1935. Fe or steel to be coated with Al is dipped into a salt bath contg. one or more fluorides, e. g., a double fluoride of Al and Na, and one or more alkali chlorides, with or without other salts except salts of Ca, and then into a fused bath of Al. The salt bath may contain NaAlF<sub>6</sub>, 10, ZnCl<sub>2</sub>, 20, NaCl 30 and KCl 40%.

Iron Mathias Fränkl. Ger. 617,943, Aug. 29, 1935 (Cl. 18 & 16 01). A method is described of refining crude Fe in the molten state by treatment with a blast of air charged with O, and of after-refining by adding solid FeO<sub>2</sub>, in which the heat developed by the oxidation of the P present is used.

Sponge iron Wm D. Brown. U. S. reissue 19,770, Dec. 3. A reissue of original pat. No. 1,979,729 (C. A. 29, 108<sup>9</sup>).

Iron sponge. Mathias Fränkl. Ger. 617,942, Aug. 29, 1935 (Cl. 18 & 18 02). A shaft furnace is described for producing Fe sponge by the acid of reducing gases.

Deoxidation of iron. Soc. d'électrochimie, d'électrometallurgie & des aciéries électriques d'Ugine Fr. 786,872, Sept. 11, 1935. Fe and steel are deoxidized with the cyclic utilization of slags and regeneration thereof, slags are used in which the ratio of MnO content to FeO content is above 4 and preferably above 5. The periodic regeneration is carried out so as to maintain this ratio above 4.

Rustless iron and steel. Wm B. Arness (to Rustless Iron Corp. of America). U. S. 2,021,979, Nov. 26. For the production of a N-contg. rustless iron or steel of fine-grain structure, a bath of molten iron is prep. with an overlying slag contg. oxides of Cr. The Cr oxides are reduced, thus enriching the bath, and N is added to the slag during the reducing operation so that N is absorbed by the nascent Cr and thus carried into the bath.

Steel. Herbert Grubitsch. Ger. 617,765, Aug. 26, 1935 (Cl. 18 & 2 40). See Austrian 142,230 (C. A. 29, 6200<sup>9</sup>).

Alloy steels. Child H. Wills. Fr. 786,597, Sept. 5, 1935. See U. S. 1,992,905 (C. A. 29, 2500<sup>9</sup>).

Forged steel rolls suitable for rolling mills Clifford B. Ferrec. U. S. 2,022,192, Nov. 26. Rolls are formed from

a steel the C content of which approximates and does not substantially exceed the eutectic value and is 0.75-0.95% and contg Cr 1.3-1.8%, by casting, forging and heat-treating in a single elevation of temp to a peak of about 810°, followed by slow reduction of temp, machining, hardening and tempering.

**Composite steel bars.** Max R. Trembour (to Jessop Steel Co.) U. S. 2,023,908, Dec. 10. Various details are given for the manuf. of a welded composite ingot suitable for the manuf. of edged tools or dies and which comprises a body of soft steel with a welded corner piece of harder steel.

**Medium- or high-carbon steel wire rods.** Henry D. L. Lloyd (to Whitecross Co. Ltd.) U. S. 2,023,786, Dec. 10. For producing coiled medium- or high-C steel rods of sorbite structure, the rods are rolled at a temp substantially above that of a molten quenching medium such as molten salt maintained at 450-500° and are immersed in this medium while substantially at rolling temp and are cooled in the medium while the latter is maintained substantially at the quenching temp. App. is described.

**Preparing sheets of metal such as low-carbon steel for deep drawing.** Louis Schulte (to Schulte Grinding and Polishing Machine Co.) U. S. 2,023,904, Dec. 10. The sheet is pickled, cold rolled, fished to provide a concave surface and the latter is engaged with a cushioned rotating grinding surface and ground only sufficiently to remove surface defects and strain-hardened film imparted to the sheet from preceding steps of manuf. App. is described.

**Marking, ornamenting or coloring aluminum and its alloys by photographic processes.** Siemens & Halske A.-G. Ger. 629,664, Oct. 24, 1935 (Cl. 576, 501). Addn. to 607,012 (C. A. 29, 1768<sup>9</sup>), 615,092 (C. A. 30, 75<sup>9</sup>) and 619,450 (C. A. 30, 427<sup>9</sup>). The oxide coating produced on Al and its alloys in the processes of Ger. 607,012, 615,092 and 619,450 is replaced wholly or in part by a coating of different compn, e. g., by a phosphate or fluoride coating.

**Composite metal articles of aluminum metals.** Robert H. Brown (to Aluminum Co. of America.) U. S. 2,023,512, Dec. 10. For producing articles resistant to corrosion, a base of metal such as Al alloyed with Mn 1.25% is provided with a coating of a plurality of aluminum metal layers, each of which (suitably because of alloying with Sn and Bi, etc.) has an electrode potential greater than that of the base metal, and with the layers arranged in the order of increasing electrode potential from the base metal to the surface of the article.

**Separating nickel and cobalt.** Raoul Bévérini and Élie Raymond. Fr. 780,810, Sept. 10, 1935. Ni and Co are sep'd by utilizing the fact that they form complex compds. with ethanolanimes in alk. medium, those of Co being very stable whereas those of Ni are unstable even in the cold, hydrolyzing to form the hydroxide. Under the same conditions Mn does not give a complex and ppts immediately as hydroxide. Fr. 780,808, Sept. 11. Glycylamines are used instead of ethanolanimes to sep. Ni, Co and Mn, the Ni forming an unstable and the Co a stable complex, while the Mn is ppt'd immediately as hydroxide.

**Metallic magnesium.** Fritz Hansgurt (to American Magnesium Metals Corp.) U. S. 2,022,282, Nov. 26. For producing substantially pure Mg, materials such as a crude Mg is heated to liberate Mg vapor, the vapor is removed from the heating zone by a nonoxidizing gas and dust is sep'd from the vapor by effecting suitable variations in its flow to promote gravity sep'n of suspended solids, and the vapor and nonoxidizing gas are passed to a condensing zone. App. is described. Cf. C. A. 29, 4728<sup>9</sup>.

**Refining magnesium and its alloys.** Oesterreichisch-Amerikanische Magnesium A.-G. Brit. 433,859, Aug. 16, 1935. Mg and high percentage Mg alloys are refined by melting with a halide or sulfide of a heavy metal from groups I, II, V, VI, VII and VIII of the periodic system, such metal, base metals excepted, having a sp. gr. above 6 and below 11. Mg halides may be used in addn. The

salts or mixts. of salts must have m. ps. below that of Mg and a sp. gr. above that of molten Mg. In examples, (1) Mg, refined by distn., is melted with a mixt. of equal parts of ZnCl<sub>2</sub>, FeCl<sub>3</sub> and MgCl<sub>2</sub>, and (2) a Mg alloy contg Al 4, Zn 1 and Sb 0.3% is melted with a mixt. of Sb<sub>2</sub>S<sub>3</sub>, 40, FeCl<sub>3</sub>, 20, ZnCl<sub>2</sub>, 10 and MgCl<sub>2</sub>, 30%. Cf. C. A. 29, 443<sup>9</sup>.

**Rolled products from magnesium and its alloys.** John E. Hoy (to Dow Chemical Co.) U. S. 2,023,366, Dec. 3. Mg or a high-percentage Mg alloy is cast to a plastic deformation by extruding and the deformed metal is then rolled. App. is described and a product is obtained which does not tend to crack.

**Recovering copper and silver compounds from alloys.** I. G. Karbennd A.-G. (Hermann Weber, Ernst Hempelmann and Robert Griesbach, inventors). Ger. 620,350, Oct. 19, 1935 (Cl. 40a, 23). Alloys contg 30-70% of Cu, the remainder being wholly or mainly Ag, are treated with Cl without applying heat. A mixt. of AgCl and CuCl is obtained, and is fused by the heat of the reaction. The fused chloride mixt. is run into a solvent for CuCl, e. g., dil. HCl or NaCl soln., and the undissolved AgCl is withdrawn. A neutral substance which increases the soly of CuCl in the solvent used, e. g., NaCl or CaCl<sub>2</sub>, may be added before, during or after the chlorination.

**Alloys.** Thomas W. Iles. Brit. 434,291, Aug. 29, 1935. A draft-excluding strip for the meeting edges of doors, windows, etc., consists of a flat elastic metal strip such as an alloy contg Cu 88.32, Zn 10.30 and Sn 1.16% rolled to 0.18-0.22 mm thickness.

**Alloys.** Pierre G. Adeline. Fr. 786,793, Sept. 9, 1935. Thermo-reaction is used for incorporating metals such as Cr, Si, Ti, W, Mo, Mn, V, Zr, Sr and Th in metals such as Fe, Cu, Zn, Sn, Bi, Sb, Pb, Ni, Na, Al and Mg. An example is given of the prep'n of an alloy contg Al 90, Cr 2 and Ti 2% by using Cr<sub>2</sub>O<sub>3</sub>, 3.7, TiO<sub>2</sub>, 4, fluor spar 0.5 and Al 3.2 kg. An alloy contg Al 80, Cr 10 and Ti 10% may be made first, and molten Al added to this to get an alloy contg a greater amt. of Al.

**Alloys by diffusion.** Fritz Bergmann. Fr. 786,715, Sept. 9, 1935. The diffusion of a metal such as Cr, Ni, W, Mn or Si into the surface of another metal such as Fe takes place in a neutral fused mass of salt. The diffusing metal may be mixed as a powder with the fused salt.

**Light alloy.** Vereinigte Leichtmetallwerke G. m. b. H. Fr. 786,592, Sept. 5, 1935. Alloys of good resistance to traction and of improved elastic limit contain Cu 3-5, Mg 1.3-1.4, Si 0.4-0.7, Mn 1.25-1.25% and Al the rest.

**Hard alloys.** The British Thomson-Houston Co. Ltd. Brit. 434,468, Sept. 2, 1935. In making alloys of the type contg 2 or more hard constituents, e. g., refractory metal carbides, and 1 or more auxiliary metals of lower m. p., each of the hard constituents is separately assoc'd. with 1 or more of the auxiliary metals, e. g., by grinding, and the fractions so formed are afterward mixed and sintered under pressure at high temp. Thus W<sub>2</sub>C 75-97 may be ground with Co 3-25, and Ti carbide 75-92 with Ni 8-25 parts and a final sintered product obtained from the W<sub>2</sub>C mixt. 90-95 and the Ti carbide mixt. 5-10%.

**Hard alloys.** Fagersta Bruks Aktiebolag. Fr. 787,010, Sept. 10, 1935. A mixt. contg Ti carbide 6-25 and inorg. compds. such as oxide or carbonate of one or more auxiliary more fusible metals such as Fe, Co and Ni, the rest being principally W carbide, is used. Pieces are made from this mixt. by pressing, after or not previous decomp'n of the comp'd of the auxiliary metal. The pieces are machined to the desired shape and fritted.

**Alloys suitable for cast dentures.** Baker & Co., Inc. Brit. 434,583, Sept. 2, 1935. See U. S. 1,930,119 (C. A. 28, 919<sup>9</sup>).

**Aluminum alloys.** Horace C. Hall and Reils Royce Ltd. Brit. 434,022, Aug. 29, 1935. The Cu content of those alloys described in Brit. 350,110 (C. A. 26, 1230<sup>9</sup>) that have a Mg content of 0.7-1.75% is increased to 2.6-6.0%. Mn and other elements may also be added as described in Brit. 425,102 (C. A. 29, 5408<sup>9</sup>). The alloys may be subjected to artificial aging at 100-100° for 12-2

hrs., to soln. treatment followed by natural aging in air for 7 days or more, or to soln. treatment and artificial aging.

**Columbium alloys.** Frederick M. Becket (to Electro Metallurgical Co.). Brit 434,400, Aug. 30, 1935. Materials contg. Cb oxides are reduced with Si or Si alloys, e. g., Fe-Si, Cr-Si, Fe-Cr-Si, Cb-Si or alloys with other metals unobjectionable in the final product. The Si in the charge is preferably adjusted to yield an alloy with a Cb-Si ratio of 6-12-1, whereby upward of 75% of the metal may be recovered and the Cb oxide is selectively reduced in preference to the oxides of Ta and Ti present. Fe, Ni, Cr or other metals may be added to the product either by reducing in the presence of a metal bath or making the addn. during or after the reduction. Slags obtained contg. substantial amts of Cb, Ta and Ti may be smelted with enough Si to yield a Cb-Si alloy of a less ratio than 6-1, this being used as reducing agent for Cb oxide-contg. material to form the lower Si alloy. Cf C. A. 30, 425<sup>2</sup>.

**Iron alloys.** Strömsjö Jernverks Aktiebolag Brit. 434,760, Sept. 9, 1935. An alloy for the production of dross comprises C 2.7-3.2, P 0.55-1.0, Cr 0.3-1.0, Mn 0.3-0.6, Si 0.9-1.6 and S 0.07-0.15%, the remainder being Fe.

**Lead alloys.** S. & T. Metal Co. Brit. 433,653, Aug. 19, 1935. See Fr. 772,826 (C. A. 29, 1381<sup>1</sup>).

**Composite rolled forms of magnesium alloys.** Arthur W. Winston (to Dow Chemical Co.) U. S. 2,023,498, Dec. 10. In forming articles such as airplane propeller blades, etc., of integrally joined layers, properly shaped ingots of the metal are arranged in the container of an extrusion press and are extruded through a common die opening at a temp. of about 230-450° so as to form a composite billet, the latter is then heated to about 215-450° and rolled or forged.

**Zinc alloys.** Wm. W. Broughton and Geo. L. Werlev (to The New Jersey Zinc Co.). Brit. 434,047, Aug. 20, 1935. Addn. to 376,988 (C. A. 27, 3191). See U. S. 1,989,368 (C. A. 29, 1769<sup>2</sup>).

**Zinc base alloy dies suitable for casting low-melting metals.** Wm. G. Newton and Howard E. Christie (to

Wm. G. Newton). U. S. 2,023,645, Dec. 10. The face of a die formed of Zn 90-92, Al 4-5, Cu 3-4 and Mg 0.02-0.1% or the like is treated with HNO<sub>3</sub> and then with a soln. of NaOH and Sb oxide, producing a blackish coating which has good hardness.

**Welding rod.** John B. Austin (to Una Welding, Inc.). U. S. 2,022,307, Nov. 26. A low-C steel welding rod contains an effective proportion of Ti, but not more than about 0.2%, an effective proportion of V, but not more than about 0.25% (the total of Ti and V being 0.16-0.29%). A rod of this compn. is suitable for welding rusted steel.

**Welding rod.** Wm. W. Sieg. U. S. 2,022,439, Nov. 26. A welding rod is formed to the extent of over 99% of an alloy of Cu with a lesser quantity of Zn, and with small proportions of Si, Mn, Fe, B and Sn, and with the Si, Mn and Fe in about the proportions of 2:2:1.

**Metallurgical blanks such as welding rods and hollow drill steel.** Burt H. Payne (to Stultz-Sickles Co.). U. S. 2,021,945, Nov. 26. An opening is formed through a drill billet at its center and from end to end, and there is inserted into the opening a Mn-Ni welding steel alloy contg. Mn 11-13.5 and Ni 2.5-3.5%, substantially filling the opening and having a coeff. of expansion greater than that of the billet, the materials are heated to about 925°, the billet is rolled until the diam. of the opening and the core has reached a desired size, and the billet is cooled until the core can be readily removed from it.

**Electrode for arc welding.** Fritz Müller (to N.-V. Machine-en Apparat Fabrieke "Meaf"). U. S. 2,023,818, Dec. 10. A steel core is coated with a mixt. of "technically pure" Mg silicate 70, water glass 30 and deoxidizing and alloying constituents such as ferro-Mn contg. C about 10%, free from mixed elements adapted to transmit impurities "of the oxygen type" to the iron.

**Apparatus and method for coating welding electrodes with flux by extrusion machines.** James G. Jackson. Brit. 433,718, Aug. 20, 1935.

**Apparatus for making metal pipe such as that of ferrous metal by electric welding.** Wm. M. Neckerman (to Republic Steel Corp.). U. S. 2,023,897, Dec. 10. Various structural, mech. and operative details.

## 10—ORGANIC CHEMISTRY

CHAS. A. ROULLIER AND CLARENCE J. WEST

**Productivity of a synthetic methanol installation in relation to various factors.** L. M. Chavnick *Khi-miel* 7, 416-18 (1935).—A math. discussion. Chas. Blare. **Fluorination of halomethyl ethers.** I. Fluorination of trichloromethyl ether. Harold Simmons Booth and Paul E. Burchfield *J. Am. Chem. Soc.* 57, 2070 (1935).—Fluorination of CCl<sub>3</sub>OMe by anhyd. sublimed SbF<sub>5</sub> without a catalyst yielded a mixt. of CFC<sub>3</sub>OMe, b. 55.3°, m. -105.1°, and CFC<sub>2</sub>OMe, b. 30.1°, m. -96.2°. These compds. are only slowly hydrolyzed in H<sub>2</sub>O.

**Geometrical isomers of heptenenitrile.** Charles Hoffmann. *Bull. soc. chim. Belg.* 44, 435-7 (1935); cf. C. A. 27, 266, 5717.—C<sub>6</sub>H<sub>11</sub>Br, prepd. from the alc., was converted into C<sub>6</sub>H<sub>11</sub>CN (I). Bromination of I in the presence of S gave α-bromoheptenenitrile, which on debromination with quinoline gave 2 heptenenitriles. These were sepd. by fractional distn. and converted by treatment with concd. H<sub>2</sub>SO<sub>4</sub> into the corresponding amides, m. 122.8° and 61-1.4°. Dehydration of the amides with P<sub>2</sub>O<sub>5</sub> gave the cis- and trans-α-heptenenitriles (II and III). II b<sub>1</sub>, 60.2-0.4°, has d<sub>4</sub><sup>20</sup> 0.8260, n<sub>D</sub><sup>20</sup> 1.43543, n<sub>D</sub><sup>25</sup> 1.43849, 1.44596, n<sub>D</sub><sup>30</sup> 1.45228, d<sub>4</sub><sup>20</sup> 0.81754, n<sub>D</sub><sup>20</sup> 1.42912, n<sub>D</sub><sup>25</sup> 1.43205, n<sub>D</sub><sup>30</sup> 1.43945, n<sub>D</sub><sup>35</sup> 1.44555. III b<sub>1</sub>, 67.5°, has d<sub>4</sub><sup>20</sup> 0.83088, n<sub>D</sub><sup>20</sup> 1.43978, n<sub>D</sub><sup>25</sup> 1.44292, n<sub>D</sub><sup>30</sup> 1.45067, n<sub>D</sub><sup>35</sup> 1.45719, d<sub>4</sub><sup>20</sup> 0.81875, n<sub>D</sub><sup>20</sup> 1.43369, n<sub>D</sub><sup>25</sup> 1.43688, n<sub>D</sub><sup>30</sup> 1.44377, n<sub>D</sub><sup>35</sup> 1.45082.

**Geometrical isomers of undecenitrile.** R. van Cailie. *Bull. soc. chim. Belg.* 44, 438-40 (1935).—α-Bromo-undecenitrile (cf. C. A. 27, 5716) was heated with an

equimol. quantity of C<sub>6</sub>H<sub>5</sub>N for 40 hrs., first at 110° and then at 140°, giving 45-50% of the undecenitriles, which were converted into the corresponding amides (m. 114-15° and 76-7°) by means of cold concd. H<sub>2</sub>SO<sub>4</sub>. Dehydration of these amides with P<sub>2</sub>O<sub>5</sub> gave the 2 pure nitriles (I and II). I b<sub>1</sub>, 119.7-19.9°, has d<sub>4</sub><sup>20</sup> 0.83255, n<sub>D</sub><sup>20</sup> 1.44536, n<sub>D</sub><sup>25</sup> 1.44816, n<sub>D</sub><sup>30</sup> 1.45515, n<sub>D</sub><sup>35</sup> 1.46000; II b<sub>1</sub>, 127.9-8.1°, has d<sub>4</sub><sup>20</sup> 0.83359, n<sub>D</sub><sup>20</sup> 1.44858, n<sub>D</sub><sup>25</sup> 1.45146, n<sub>D</sub><sup>30</sup> 1.45873, n<sub>D</sub><sup>35</sup> 1.46484.

**Aldehyde acetals.** R. Fornet. *Seifen-Ind.-Ztg.* 62, 847-8, 857-8 (1935).—Synthetic methods are briefly reviewed and the properties of a no. of acetals tabulated.

**Thirty references.** J. W. Ferry. The solvent effect in the addition of hydrogen bromide to allylactic acid. M. S. Kharasch and M. C. McNab. *Chemistry & Industry* 1935, 989-90.—By use of the technic described (C. A. 27, 3444), allylactic acid in C<sub>6</sub>H<sub>6</sub> with small amts. of NHPH<sub>3</sub> or thioacetal gave γ-bromovaleric acid, m. 22°, but with a small amt. of ascaridole it gave δ-bromovaleric acid, m. 40°. Peroxides, not the solvents, control the direction of addn. and it could not be reversed by the use of peroxides or antioxidants in unsatd. compds. in which the double bond is either adjacent to a carboxyl group (crotonic acid) or does not involve a terminal C atom (2-pentene).

**E. R. Rushton.** Addition of hydrogen bromide to olefinic acids. R. P. Linstead and H. N. Rydon. *Chemistry & Industry* 1935, 1009.—The authors obtained δ-bromovaleric acid, m. 40°, from allylactic acid in C<sub>6</sub>H<sub>6</sub> in the presence of

NiHPh, using a current of HBr, whereas Kharasch and McNab (preceding abstr.) used an excess of HBr in a sealed tube and obtained the  $\gamma$ -acid. The facts cannot be explained by any simple peroxide theory which neglects the polar effect of the carbonyl group when near the double bond.

E. R. Rushton

Fractional distillation in vacuo as a method of purification of fatty substances. I. Application to alkyl oleates. L. Kellier *Bull. soc. chim. Belg.* 44, 425-34 (1935). Contrary to the statements in the literature, a single fractional distn of the esters of oleic acid is inadequate if the ester is to be used for precise work, often several successive fractionations are necessary. The heats of combustion of 3 carefully purified alkyl oleates were found to be as follows: Me oleate 9576, Et oleate 9037, and Pr oleate 9702 cal./mole per g. in vacuo and at const. vol. The value for Et oleate is not exact, since the ester was evidently not quite pure. Calen gave the following values for the heats of combustion of oleic and linoleic acids 9170 and 9390 cal. per g., resp.

Louise Kelley

Preparation of  $\epsilon$ -trimethylcaprobetaïne. Francisco Gural *Anales soc. esp. fis. quim.* 33, 752-3 (1935); cf. *Coll. Czechoslov. Chem. Communications* 2, 712 (1930). Heat cyclohexane isooxime ( $\epsilon$ -leucine lactam (Ann 312, 171 (1900)) with 50% aq. HCl, decolorize with C, conc., recrystallize from H<sub>2</sub>O; yield 84% of  $\epsilon$ -leucine-HCl (I), m. 131-2° (cor.). Dissolve 15 g. of I in a little H<sub>2</sub>O, neutralize by solid KOH, add in small increments 25 g. KOH in a little H<sub>2</sub>O, and 51 g. of Me<sub>2</sub>SO, while cooling, then heat under reflux, cool, neutralize with H<sub>2</sub>SO<sub>4</sub>, conc., evap. to dryness on a water bath, ext. several times with cold alc. to remove K<sub>2</sub>SO<sub>4</sub> and KCl, heat 6-8 hrs. under reflux with concd. HCl to hydrolyze Me esters, ppt. by BaCl<sub>2</sub>, filter, evap. to dryness and take up by hot alc. to remove excess BaCl<sub>2</sub>, filter, evap. to dryness, forming the methochloride of  $\epsilon$ -dimethylaminoacetic acid (II), (yield quant.), m. 180° (cor.). Mix 10 g. of II with freshly prep. Ag<sub>2</sub>O, filter, evap. to dryness, evap. repeatedly with abs. alc., recrystallize from abs. alc.; yield, 83% of  $\epsilon$ -trimethylcaprobetaïne, m. 249-50° (cor.) with decompn. E. M. Symmes

The transformation of arginine into citrulline. K. Durr and H. Späth *Z. physiol. Chem.* 237, 121-30 (1935).—Arginine was converted into citrulline by amidation, benzoylation and subsequent hydrolysis. Arginineamide HCl salt (I) was obtained in hygroscopic needles, which sintered at 110°, decompd. at 155° with evolution of gas and showed further decompn. at 270°, by satg. the MeOH soln of arginine Me ester HCl salt with dry NH<sub>3</sub>, pptg. with Et<sub>2</sub>O and recrystg. from H<sub>2</sub>O-EtOH-Et<sub>2</sub>O. It has  $[\alpha]_D^{25}$  13°, gives a strong birefract. reaction, is not attacked by arginase at pH 7, and forms a picrate, decompg. 220°.  $\alpha$ -Benzoylarginineamide HCl salt + H<sub>2</sub>O (II), sinters 115° and decomposes 260°, was obtained by esterification of benzoylarginine and treatment with NH<sub>3</sub>. Esterification of dibenzoylarginine gave the Me ester HCl salt, sinters 167° and m. 171°. Benzoylation of either I or II yielded dibenzoylargininebenzoylamide (III), sinters 236°, m. 245° becoming brown, and evolves gas at 255°. Hydrolysis of III by concd. HCl at room temp., evapn. in vacuo and extn. with EtOH gave dibenzoylcitrullinebenzoylamide, sinters 185° and m. 220°. This on further hydrolysis gave ornithine and proline, identified as picrates. The structure and identity of III were established by prepn. of the same substance from ornithine.  $\alpha$ -Benzoylornithine was condensed with urethan to form  $\alpha$ -benzoylcitrulline, from which the Me ester, decompg. 120°, was obtained by treatment with CH<sub>3</sub>N<sub>3</sub>. NH<sub>3</sub> converted the ester into a benzoylcitrullineamide, decompg. 140°. This was then benzoylated to III. The dihydroperazine of arginine, decompg. 316°, was obtained as a by-product in the prepn. of arginineamide.

A. W. Dox

2,3,6-Trimethylglucose anhydride. Karl Freudenberg and Emil Braun *Ber.* 68B, 1938 (1935).—In reply to the statement of Hess and Neumann (C. A. 29, 6882) that the authors' synthesis of 2,3,6-trimethylglucose anhydride is not reproducible, F and B call attention to their reply (C. A. 27, 3701) to a similar criticism made

before by Hess and Lüttmann (C. A. 27, 3701).

C. A. R.

Dehydration and hydrate formation of lactose. W. Mohr and J. Wellm. *Milchw. Forsch.* 17, 109-17 (1935). Lactose (D. A. B. 6) lost water of crystn. at 87° at abs. humidity from about 3 g./cu. m. downward. At 100° evapn. occurred between 8 and 14 g./cu. m. The anhydride, H<sub>2</sub>O free lactose, obtained by dehydration at 100° in vacuo was highly hygroscopic at room temp. and was converted back immediately to the hydrate. Evapn. of lactose solns. at 70° and 90° yielded in thin layers a H<sub>2</sub>O-free lactose which at 20° and 7 g./cu. m. atm. humidity was not hygroscopic and took up H<sub>2</sub>O over and above the amt. necessary for hydrate formation only in air almost satd. with humidity (17 g./cu. m.), and from that originally in storage in ordinary room atm. (5.2 g./cu. m. humidity) the H<sub>2</sub>O was given up almost completely. Still longer storage in very moist air led to hydrate formation. The H<sub>2</sub>O contained in lactose glass was only absorbed since it was given up under conditions where the H<sub>2</sub>O of hydration does not escape. The  $\alpha$ -lactose hydrate and  $\alpha$ -lactose anhydride 10 min. after dissolving showed  $[\alpha]_D^{25}$  82.2° and 79.6°, while the lactose glass obtained in thin layer from solns. at 70° and 90° showed  $[\alpha]_D^{25}$  60.3°, 58.1° and 56.6°. The Debye-Scherrer x-ray diagrams of the lactose preps. varied as did those of the  $\alpha$ -hydrate and the lactose obtained in thin layers from soln. The  $\alpha$ -lactose anhydride gave a characteristic diagram. The lactose glass showed only a broad, indistinct interference ring and was also solidified amorphously.

H. Macy

Mechanism of addition to double bonds. II. Stereocourse of 2 diene syntheses. Albert Wassermann. *J. Chem. Soc.* 1935, 1511-14, cf. C. A. 29, 5730°.—The 2 possible stereoisomeric forms of cyclopentadiene benzoquinone, the endo- and the exo- forms, are shown in photographs of space models. The ultraviolet absorption was detd. by the method of Smakula and Wassermann (C. A. 26, 2974) in C<sub>6</sub>H<sub>6</sub> and in EtOH. When isolated from C<sub>6</sub>H<sub>6</sub>, EtOH, CCl<sub>4</sub>, C<sub>6</sub>H<sub>6</sub>, and C<sub>6</sub>H<sub>6</sub> at 20°, 40° and 50° and purified by recrystn. from C<sub>6</sub>H<sub>6</sub>, all specimens of this substance have the same m. p., crystal shape and color, and there is a sharp absorption max. at 2220 Å. It is therefore certain that one of the isomerides predominates, and there is no subsequent interconversion. The induction energies corresponding to the different intermediate configurations in the reaction were calcd. and found to be greater for the endo-isomeride. The activation energy of the endo-addn. product is therefore smaller, and since only one is formed, it must be the endo-product. Induction energies corresponding to the different intermediate configurations in the cyclopentadiene maleic anhydride reaction were calcd. and the activation energy was found to be less for the endo addn., indicating that this product is formed more rapidly, which is confirmed by expt. Rate consts. were calcd. The m. p. of cyclopentadienebenzoquinone is 75-76°. At 0°, the soln. in C<sub>6</sub>H<sub>6</sub> is 0.9%.

E. R. Rushton

Fluorinated chlorobenzenes. Harold S. Booth, Howard M. Elsey and Paul E. Burchfield. *J. Am. Chem. Soc.* 57, 2004-5 (1935).—m-C<sub>6</sub>H<sub>4</sub>ClF (I), prepd. in 60% yield from m-C<sub>6</sub>H<sub>4</sub>ClNH<sub>2</sub>, bp<sub>10</sub> 127.6° (cor.), m. less than -78°, has  $d_4^{25}$  1.221 and  $n_D^{25}$  1.4911. The vapor pressure and temp. of I are related by the equation:  $\log P$  (mm.) =  $(-2342.4/T) - 2.4165 \log T + 15.017$ . 2,4,6-C<sub>6</sub>H<sub>2</sub>Cl<sub>2</sub>F (II), prepd. from 2,4,6-C<sub>6</sub>H<sub>2</sub>Cl<sub>2</sub>NH<sub>2</sub>, bp<sub>10</sub> 208.4° (cor.), m. 11.2° with  $d_4^{25}$  1.530,  $n_D^{25}$  1.5429. The vapor pressure and temp. of II are related by the equation:  $\log P$  (mm.) =  $-2452/T - 0.7316 \log T + 9.9389$ . II is thermally unstable. An improved precision technique for the detn. of the vapor pressure of liquids was used in these measurements.

Louise Kelley

Benzotrifluoride and its halogenated derivatives. Harold S. Booth, Howard M. Elsey and Paul E. Burchfield. *J. Am. Chem. Soc.* 57, 2060-6 (1935).—In the prepn. of PhCF<sub>3</sub> (I), the use of an excess of SbF<sub>5</sub> improves the yield; 1 b. 102.3° and m. -29.03°. The following derivs. of I were prepd. m-F (II), prepd. by nitration of I, re-



duction to  $m$ -NH $_2$ C $_6$ H $_4$ CF $_3$  (III), and replacement of NH $_2$  by F, b. 100.9°, m. -51.5°,  $n_D^{20}$  1.3982 (II was not analyzed);  $p$ -F, prep'd. by chlorination of  $p$ -ClC $_6$ H $_4$ Me in the presence of PCl $_5$  and fluorination of the  $p$ -FC $_6$ H $_4$ CCl $_3$  by means of SbF $_5$ , b. 102.8°, m. -41.7°,  $n_D^{20}$  1.3990,  $\alpha$ -Cl, prep'd. from  $o$ -MeC $_6$ H $_4$ NH $_2$  by replacing the NH $_2$  by Cl, and chlorinating and then fluorinating the side chain, b. 152.8°, m. -7.4-7.6°,  $n_D^{20}$  1.4541,  $m$ -Cl, prep'd. by replacing the NH $_2$  group of III by Cl, using the Sandmeyer method, b. 138.4°, m. -55.4°,  $n_D^{20}$  1.4466,  $p$ -Cl, prep'd. by chlorinating  $p$ -ClC $_6$ H $_4$ Me,  $n_D^{20}$  1.4466,  $p$ -ClC $_6$ H $_4$ CCl $_3$  thus formed by means of SbF $_5$ , b. 139.3°, m. -54.0°,  $n_D^{20}$  1.4469,  $3,4$ -di-Cl, prep'd. by successively converting  $p$ -MeC $_6$ H $_4$ NH $_2$  into  $3,4$ -Cl $_2$ C $_6$ H $_3$ Me,  $3,4$ -Cl $_2$ C $_6$ H $_3$ CCl $_3$ , and  $3,4$ -Cl $_2$ C $_6$ H $_3$ CF $_3$ , b. 173.5°, m. -12.3.2°,  $n_D^{20}$  1.4736. Exhaustive chlorination of I apparently converted it into a mixt. of the isomeric tri- and tetra-Cl derivs. L. K.

Evaluation of new methods of production of aniline hydrochloride B. P. Fedorov *Trans. Inst. Chem. Tech. Inst. (U. S. S. R.)* 1, 162-5 (1935).—Patents are critically reviewed. A procedure is described (pat. application No. 112,685 (1932)) for the production of PhNH $_2$ ·HCl with 99.6% yield by the interaction of atomized PhNH $_2$  and dry HCl. Chas. Blanc.

Preparing diphenylthiourea V. I. Kuznetsov and P. M. Aranovich *J. Chem. Ind. (Moscow)* 12, 953-4 (1935).—The yield of (PhNH) $_2$ CS, prep'd. according to the patent of Flemming (C. A. 20, 1631) depends chiefly on the temp. reached during the reaction. Therefore, when only the heat of reaction is used, excess of inert reagents must be avoided, and the app. must be well insulated. Yields of 98% are obtained if the mixt. is heated on the water bath for 1 hr. after it has reached the max. temp. (80°) which it can reach alone. H. M. L.

Synthesis of  $p$ -benzylmercaptobenzenesulfonic acid. Torizo Takahashi *J. Pharm. Soc. Japan* 55, 875-9 (in German 164-5 (1935)).— $p$ -ClC $_6$ H $_4$ NO $_2$  (8 g.) after standing with alc. KOH (200 cc. alc. and 8.5 g. KOH) sat'd. with H $_2$ S, gave  $o$ -nitrophenylmercaptan (I), m. 77°. I (4 g.) in alc. KOH and PhCH $_3$  (3.26 g.) on heating on the water bath for 2 hrs. gave 4-aminophenyl benzyl sulfide (II), PhCH $_2$ SC $_6$ H $_4$ NO $_2$ , m. 123°. II (2 g.) in alc. (90 cc.), HCl (5 cc., ca. 30%), and 15% MeCl (48 g.) on boiling for 0.5 hr. on the water bath gave 4-aminophenyl benzyl sulfide-HCl (III), m. 256°. III (1 g.) and AcOH (3 g.) on boiling for 2 hrs. on the sand bath gave 4-acetamidophenyl benzyl sulfide, m. 133° and 105°. III (0.5 g.) on boiling gently with BrCl (3 cc.) for 3 hrs. gave 4-benzamidophenyl benzyl sulfide, m. 182°. The diazonium comp'd. of III and Na $_2$ AsO $_4$  gave  $p$ -benzylmercaptobenzenesulfonic acid, C $_6$ H $_4$ CH $_2$ SC $_6$ H $_4$ AsO $_4$ H $_2$ , decomp. about 250°, light yellow needles. F. I. N.

Action of dicyanogen on phenols. Georg Hahn and Walter Leopold. *Ber.* 68B, 1974-80 (1935), cf. Machek, C. A. 27, 3922.—In his report of the study of the action of (CN) $_2$  on phenols (the 3 C $_6$ H $_4$ (OH) $_2$  and C $_6$ H $_3$ (OH) $_3$ , and the 2 C $_6$ H $_3$ (OH) $_3$  M. makes the surprising statement that pyrocatechol (I) undergoes nucleus substitution to give the hitherto unknown 3-cyanopyrocatechol (II), all the other phenols and naphthols forming very labile addn. products. On repeating his work, it was found that when purified (CN) $_2$  is passed into aq. I there soon seps an oil, clear at first, which gradually becomes redder and redder. On long standing it decomposes and deposits a small amt. of the crystals of M.'s supposed II, which, after decanting from the water and rubbing repeatedly with ether, he obtained as a reddish powder, m. 198°, in 52% yield. H. and L. find that the conversion of the oil into the cryst. powder can be effected almost instantaneously, with a slight evolution of heat, by means of dil. HCl, giving 70% of a colorless cryst. substance (III), m. 220°. It does not melt without decompn., however. If the resolidified melt is sublimed *in vacuo*, somewhat more than 50% is recovered as carbonyldibenzene (IV) (Einhorn and Lindenberg, *Ann.* 300, 141). Because of its instability, III was not recrystd. from PhNO $_2$ . Neither is M.'s proposed purification method (soln. in dil. NaOH and reprecip. with HCl) suitable; III undergoes cleavage almost instantly

in alkali and darkens because of atm. oxidation, and only when a large amt. is used can a small part be recovered. By pptn. from acetone-ligron, without heating, however, it was obtained in clear prisms which, after repetition of the process, were analytically pure. Their compn. corresponds to C $_6$ H $_4$ O $_2$ ·xCN, instead of C $_6$ H $_3$ O $_2$ ·xCN, as given by M., and mol-wt. detns. point to the formula C $_6$ H $_3$ O $_2$ ·xCN $_2$ . While III can be crystd. unchanged from dil. HCl, boiling alc. HCl decomposes it into I and (CONH) $_2$ . The formation of (CONH) $_2$  shows that the C atoms of the (CN) $_2$  are still attached to each other in III. The further facts that III is sol. in alkalis (i. e., still contains free OH groups), yields IV, forms a diacetate, a dibenzoate and a di- $o$ -Me ether, exclude all other possible structures for III than that of a *dipyrocatechol dimidooxalate*, [C(NH)OC $_6$ H $_3$ (OH)] $_2$  (V). This structure explains, furthermore, why M. obtained I instead of 2,3-(HO) $_2$ C $_6$ H $_3$ CO $_2$ H on alk. sapon. and veratrole with Me $_2$ SO, and only poor yields on benzylation by the Schotten-Baumann method. With CH $_3$ N $_3$  in ether, on the other hand, III in acetone almost quantitatively yields the *di-Me ether (diguacyl dimidooxalate)* (VI), which in 104° without decompn. and can therefore be used for mol-wt. detns. by the Rast method, sapon. with 4 N NaOH gives guaiacol and (CONH) $_2$ . The 1st product of the reaction between aq. I and (CN) $_2$  is assumed to be a comp'd. HOC $_6$ H $_3$ OC(NH)CN (VII) which remains in soln. and reacts with another mol. of I to form the oil, (HOC $_6$ H $_3$ O) $_2$ C(NH) $_2$ CN (VIII). When this oil is converted into III with dil. HCl, the soln. always contains considerable I and NH $_4$ Cl. There must therefore be a partial splitting off of I from the oil and hence the latter must be an addn. product (VIII) of I and VII. H ions (H $_2$ BO $_3$ ) can be used instead of HCl produce a disproportionation of VIII to V, in the course of which a part of the I remains free and the regenerated VII is hydrolyzed to I and (CONH) $_2$ . Definite proof of a further possible change of VII in acid soln. was obtained. If the clear reaction soln. (contg. VII) above the oil VIII is treated with dil. HCl, crystals of IV sep. after a few moments. It is believed they are formed by splitting off of HCN from VII with formation of C $_6$ H $_4$  O C(NH) $_2$  O (IX), which is

then hydrolyzed to IV. This smooth conversion afforded a means for detecting the presence of VII in the reaction mixt. It was found that as long as there was still much I, all the VII combined with it to form VIII, and only toward the end of the reaction was there any considerable amt. of VII. Previous addn. of acid should therefore completely prevent the sepn. of the oil, and, as a matter of fact, by addn. of 10% H $_2$ BO $_3$  (based on the amt. of I used) the reaction VII  $\rightarrow$  III can be effected without any sepn. of VIII; at the same time the reaction VII  $\rightarrow$  IX  $\rightarrow$  IV is favored and the yield of III is decreased to 40-50%. The ratio of the products IV:III depends also on the velocity with which the (CN) $_2$  is passed in, if it is passed in very rapidly the yield of III is increased, while with a very slow rate up to 40% of IV may be formed. This does not yet exhaust the reaction possibilities between (CN) $_2$  and I. If NaH $_2$ PO $_4$  is used to allow the formation of VIII there is formed, often exclusively but generally along with III, a new comp'd. After passing in the (CN) $_2$  for about 2 hrs., there sep. crystals (X) (8.2 g. from 10 g. I), m. 144° (decompn.). At a const. rate of flow of the (CN) $_2$  and a  $pH$  of 3.2-4.0, III is obtained directly without sepn. of oil and with only an insignificant amt. of IV; at  $pH$  4.9 X is formed along with a little III; at  $pH$  4.9-5.3 the ppts. become more and more oily, and at  $pH$  5.3, as also in pure water, VIII is deposited. X has the compn. (C $_6$ H $_3$ O $_2$ ) $_2$ N $_2$ ; it dissolves in dil. NaOH and even in Na $_2$ CO $_3$  without color at first but after a time the soln. is turned brown by the air. Contrary to III, which can be crystd. from dil. HCl, X is decomposed by hot water and by cold dil. HCl into I, NH $_4$ Cl and (CO $_2$ H) $_2$ . It is believed that at the acidity of the NaH $_2$ PO $_4$  soln. ( $pH$  4.9) water adds at the double bond of the NH group to form [C(NH $_2$ )(OH)OC $_6$ H $_3$ (OH)] $_2$  (X). Such addn. does not occur with preformed III, and hence the addn. of H $_2$ O is assumed to be similar to

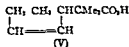
that of I, viz., VII (+H<sub>2</sub>O) → HOC<sub>2</sub>H<sub>4</sub>C(OH)(NH<sub>2</sub>)CN (+I) → HOC<sub>2</sub>H<sub>4</sub>C(OH)(OH)C(NH<sub>2</sub>)OC<sub>2</sub>H<sub>4</sub>OH (+H<sub>2</sub>O) → X. X contains 2 HO groups which can be methylated with CH<sub>3</sub>I, and 4 acetylatable HO groups. In non-aq medium (MeOH was used) I treated a short time with (CN)<sub>2</sub>, gave crystals (XI) of the compn (C<sub>7</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>), sol in dil NaOH, with brown color, only after some length of time, and not methylated by CH<sub>3</sub>I. If, here again, VII is formed primarily, the simplest assumption is that the free HO group in VII plays the same role as the 2nd mol. of I in the formation of VII or the H<sub>2</sub>O at p<sub>H</sub> 4.9 in the formation of X VII → O C<sub>2</sub>H<sub>4</sub> O C(NH<sub>2</sub>)CN → [O C<sub>2</sub>H<sub>4</sub> O C(NH<sub>2</sub>)<sub>2</sub>] (XI). The NH<sub>2</sub> groups are apparently not

acetylatable, for heating with AcCl resumes XI. Hydrolysis with cold dil HCl gives a whole series of cleavage products, depending on the length of action. I, (CO<sub>2</sub>H)<sub>2</sub>, (CONH<sub>2</sub>)<sub>2</sub>, or the so-called quadroxalate, m 130°, in 1 case the o-HOC<sub>2</sub>H<sub>4</sub> ester of (CO<sub>2</sub>H)<sub>2</sub>, was also obtained in small yield. The action of (CN)<sub>2</sub> on phenols therefore always consists in addn. of the phenolic HO at the C N unions, in no case is there CN substitution on the nucleus. An improved form of app. for the prepn of (CN)<sub>2</sub> from KCN and CuSO<sub>4</sub> is described. Dibenzate of III, m 129-30°. Di-Me ether of X, m 129-30°, tetraacetate, m 105°. XI, decomposes 139°. C. A. R.


Preparation of pure benzyl acetate E Shapiro *Masloboino Zhivore Dolo* 11, 321 2(1935) — PhCH<sub>2</sub>OAc, b<sub>p</sub> 93-4°, was obtained from a mixt of PhCH<sub>2</sub>OH, Ac<sub>2</sub>O, and 3% of the catalytic mixt (10 parts of Ac<sub>2</sub>O and 1 part of H<sub>2</sub>PO<sub>4</sub>, (d 1.7) prepd 48 hrs before using) by stirring 24 hrs at 35°. The reaction mixt was dild with H<sub>2</sub>O, AcOH was removed and the product after neutralization with 10% Na<sub>2</sub>CO<sub>3</sub> was dried with anhyd Na<sub>2</sub>SO<sub>4</sub> and redistd. Chas Blanc

Properties and uses of some homologs of α-amylinamaldehyde R Fornet *Seifensieder-Ztg* 62, 697-8 (1935) — Example compns illustrate the use in perfumery of α-phenylcinnamaldehyde and various α-alkylcinnamaldehydes, alkyl representing, resp., Me, Et, Pr, Bu, Am, hexyl, heptyl, octyl, nonyl and decyl. J. W. Perry

New experiments in the camphene series Determination of the structure of camphenelaunonic acid Ossian Aschan *Soc Sci Fennica Commentationes Phys-Math* 8, No 1, 13 pp (1935), d C A 4, 3200, 5, 3577, 3579, 3580, 6, 1144 — Attempts to prep the anhydride of camphenic acid (I) by dry distn at normal pressure resulted in decompn into CO and a viscous product which distd over. This could be sep'd by steam distn into 2 monobasic acids, viz., camphenonic acid (II), the constitution of which is known, and camphenelaunonic acid (III), which was volatile with water vapor. When redistd in *vacuo* III b<sub>p</sub> 129°, d<sub>4</sub> 1.0187, [α]<sub>D</sub> 25.1° (1 dm tube), ketone test neg. The acidity, det'd by titration with NaOH, corresponded to that calcd for III. No salt, non-crystg, soapy. Ba salt, readily sol in water; Ca salt, needles, which ppt on addn of CaCl<sub>2</sub> to an aq soln. of the Na salt, but redissolve in excess of water. Amide, recrystd twice from alc, m 139°, sol in AcOH and alc, slightly sol in benzene and water, insol in cold ligroin. Prepn: Add 0.8 g PCl<sub>5</sub> to 2 g III in a small flask, mix, and let stand for 48 hrs to sep H<sub>2</sub>PO<sub>4</sub>. Cool, decant the acid chloride, drop by drop, into properly cooled 25% NH<sub>4</sub>OH and dry the pptd amide by suction. In view of the formula for I CO can be cleaved in 2 different ways, giving 2 possible formulas for III, viz:

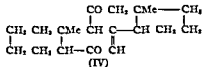
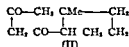
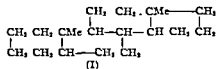


The double bond is evident from the instantaneous discoloration of KMnO<sub>4</sub> and Br solns, resp., when an aq, alk (Na<sub>2</sub>CO<sub>3</sub>) soln of III is added. The cryst., acid oxidation products thereby obtained were in all cases identical with II. The Thiele test, characterizing the group

 as represented by IV, was neg. HCO<sub>2</sub>H

at high temp. and pressure, which in the presence of the same arrangement of 6 C atoms should lead to a hydrolytic decompn of III into COMe<sub>2</sub> and 1-pentanone-3-carboxylic acid (VI), also gave a neg test. A great no. of expts on the oxidation of III with KMnO<sub>4</sub> in neutral, acid or alk solns did not yield VI in a single case. In accordance with these observations it is concluded that the structure of III corresponds to that of V. D. T.

8-Methylhydriindane derivatives, and the cis- and trans-2-methyl-1-carboxycyclopentane-2-acetic acids. Chang-Kong Chuang, Chi-Ming Ma and Yu-Lan Tien *Ber* 68B, 1946-52(1935). — Some 8-methylhydriindane derivs, and similar compds, have been synthesized for the ultimate purpose of synthesizing the parent hydrocarbon (I) of the bile acid and sterol groups. It was assumed that by condensation of 2,4-diketo-8-methylhydriindane (II) with 1-methyl-2-acetylcyclohexene (III) according to Michael, and subsequent ring closure, there might be obtained a compd. (IV) which, on Clemmensen reduction of the CO groups, followed by catalytic hydrogenation of the double bond, would give I. The synthesis of II is described in the present paper; the expts now being carried out on the condensation of II with III are reserved for a later communication. III condenses smoothly with CH<sub>3</sub>(CO<sub>2</sub>Et), in the presence of NaOEt to Et 2,4-diketo-8-methylhydriindane-1-carboxylate (V), which yields II on hydrolysis. II behaves in all respects like dihydrosorcinol, it is a strong acid, easily sol in alkalis and Na<sub>2</sub>CO<sub>3</sub>, and gives a brown color with FeCl<sub>3</sub>. The diketone structure is confirmed by the formation of a dioxime and a characteristic condensation product (VI) with HCHO. Clemmensen reduction gives 8-methylhydriindane (VII). Attempts were made to dehydrogenate VII with Se at 300° to C<sub>10</sub>H<sub>8</sub>, to obtain direct proof of the possibility of expanding a 5- to a 6-membered ring by entrance of the angular Me group into the ring, as is believed to occur in the formation of chrysene from cholesterol. These attempts were unsuccessful, probably because of the relatively low b. p. of VII, so that a sufficiently high temp. could not be attained in the dehydrogenation. II is smoothly oxidized by alk hypobromites to CHBr<sub>2</sub> and cis-2-methyl-1-carboxycyclopentane-2-acetic acid (VIII). Oxidation with hot concd HNO<sub>3</sub> or with alk KMnO<sub>4</sub> gives an oil from which also a small amt. of VIII can be isolated. The cis-configuration is assigned to VIII, by analogy with other acids contg. a CO<sub>2</sub>H group united directly to the ring, on the basis of the instability of VIII toward HCl, with which at 180° it gives the trans-isomer (IX). VIII smoothly forms a cis-anhydride (X) which regenerates VIII on hydrolysis. IX on similar treatment with Ac<sub>2</sub>O likewise forms X instead of a trans-anhydride. X is very stable; long heating at 240° does



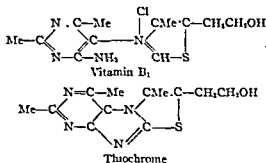
not alter its configuration. The configuration of II has not yet been definitely established, but since it gives VIII by 3 different methods of oxidation it is very probable

that it also has the *cis*-configuration. The same is true of V and of VII. The results reported by Clemo and Dickenson in a paper (C. A. 29, 8834) which appeared after the present one was written agree with those of the authors as regards the synthesis of VII and its attempted dehydrogenation. V (80% yield), thick yellowish oil, b<sub>p</sub> 160–70°, gives a dark red color with FeCl<sub>3</sub>, monosemicarbazone, m. 208–9° (decolor.). II (45% from V with boiling 5 N HCl), b<sub>p</sub> 145–8°, m. 90–2°, also obtained by boiling V with 20% alc KOH dioxime, m. 137–8°; methylenebis-(2,4-dioxo-3-methylhydrazine) (VI), m. 123–4°, sol in dil alkali hydroxides but insol in Na<sub>2</sub>CO<sub>3</sub>. VII, b<sub>p</sub> 80–2° VIII (1.2 g. from 3 g. crude II with NaOBr), m. 165–6°; di-Ag salt, dianilide, m. 202–4°. X, from VIII boiled 40 min with 10 vols Ac<sub>2</sub>O, m. 93–4°. IX, m. 101–2°, mixed m. p. with VIII, 101–25°. Dianilide, m. 156–7°, mixed m. p. with the dianilide of VIII, 160–76°.

**Dumethyldiole** L Petrova and D Al'in Masla-bolno Zhivotce Delo 11, 320 (1935).—A yield of 80–90% of  $\alpha,\beta$ -dimethyldiole, m. 95°, was obtained by following the method of Ger pat 574,849 Chas Blanc

A new synthesis of imidazole derivatives. Rudolf Weidenhagen and Roland Herrmann Ber 68B, 1953–61 (1935).—See C A 29, 7975.

**The antineuritic vitamin IV** A. Windaus, R Tschesche and R Grewe Z physiol Chem 237, 98–104 (1935), cf C A 29, 199°—The sulfate of vitamin B<sub>1</sub> on oxidation with Ba(MnO<sub>4</sub>)<sub>2</sub> yielded a base, C<sub>11</sub>H<sub>13</sub>N<sub>2</sub>, which was pptd. as the picrolonate and purified through the picrate, m. 224° (decolor.) and HCl salt, m. 265° (foaming and sepn. of NH<sub>4</sub>Cl) The base is believed to be a diaminomethylpyrimidine. The 2 NH<sub>2</sub> groups are probably on adjacent carbons. One of these is derived from the thiazole ring of the vitamin. In the oxidation of the vitamin to thiochrome it is believed that the CH of the thiazole ring is changed to CO which in turn condenses with the NH<sub>2</sub> on the pyrimidine ring, forming the 3-ring system of thiochrome.



From the mother liquor of the C<sub>11</sub>H<sub>13</sub>N<sub>2</sub> base, cryst products confg. N but no S, m. 224°, 225° and 180°, resp. were obtained but not in sufficient quantity for identification.

**Glucoside of the flavone of the white flower IV.** Constituents of *Cosmos bipinnatus*, Cav. Tabichuro Nakaoi. J. Pharm. Soc. Japan 55, 967–78 (1935) (in German 173–6); cf. C. A. 29, 7982°.—From the alc. ext. of the flower of *Cosmos bipinnatus*, Cav. N. (cf. C. A. 27, 1883) isolated a glucoside, *cosmosin* (I), C<sub>17</sub>H<sub>17</sub>O<sub>11</sub>, m. 178°. Ac deriv., C<sub>17</sub>H<sub>15</sub>O<sub>10</sub>Ac, m. 207–8°. Hydrolysis of I with 10% H<sub>2</sub>SO<sub>4</sub> for 10–12 hrs gave, according to the equation C<sub>17</sub>H<sub>15</sub>O<sub>10</sub>Ac + H<sub>2</sub>O = C<sub>17</sub>H<sub>17</sub>O<sub>11</sub> (II) + C<sub>6</sub>H<sub>11</sub>O<sub>5</sub> + H<sub>2</sub>O, apigenin (II) and glucose. II m. 347°. Apigenin acetate, C<sub>17</sub>H<sub>15</sub>O<sub>10</sub>Ac, m. 181–2°, and did not depress the m. p. of the acetate prep. from apun. Benzoylation of II gave a compd., C<sub>23</sub>H<sub>19</sub>O<sub>11</sub>, m. 210–12°. Methylation of I with MeI gave a compd., m. 205–6°, which on hydrolysis gave a compd., m. 238–9°, which did not depress the m. p. of acetate. Methylation of I with CH<sub>3</sub>N<sub>3</sub> gave a glucoside, C<sub>17</sub>H<sub>17</sub>O<sub>11</sub>, m. 255°, which on hydrolysis with 5% H<sub>2</sub>SO<sub>4</sub> gave apigenin di-Me ether, C<sub>17</sub>H<sub>15</sub>O<sub>10</sub>(OMe)<sub>2</sub>, m. 207°. The mother liquor (from which I was isolated) gave on hydrolysis with 10% H<sub>2</sub>SO<sub>4</sub>,

quercetin (III), C<sub>15</sub>H<sub>10</sub>O<sub>7</sub>, m. 306°. Oxidation of III gave colorless needles, m. 190–2°, and did not depress the m. p. of pentacetylquercetin (m. 189–91°). The hydrolyzate gave glucosazone and rhamnosazone. Quercetin may exist in the form of rhamnose glucoside. In the course of the isolation of I, N. also isolated isostol, C<sub>15</sub>H<sub>10</sub>O<sub>7</sub>, m. 225°.

**F. I. Nakamura**  
The alkaloids of ergot, ergometrine. Chassarr Moir. Pharm. J 135, 63 (1935); cf. C. A. 29, 5593°, 7019°.—  
Ergometrine. S. Smith and G. M. Timmis. Ibid. 135, 212.—See C. A. 29, 7330°.

**S. Waldbott**  
Alkaloids of Sinomerum and Cocculus. XLIV. Phenolic alkaloid of Cocculus trilobus, D. C. 3. Constitution of normenisarine Heisaburo Kondo and Masao Tomita. J. Pharm Soc Japan 55, 911–13 (1935) (in German 170–1).—Normenisarine, C<sub>12</sub>H<sub>12</sub>(OMe)<sub>2</sub>(OH)(-O-)(-NMe)<sub>2</sub>(-N), m. 223°. On methylation it gave menisarine, C<sub>12</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>(OMe)<sub>2</sub>, m. 164° XLV. Review on the bisco-laurine alkaloids A consideration from the stereochemical and biogenic standpoint Ibid. 914–33.—A review.

**F. I. Nakamura**  
Purification of the alkaloid from Chinese hanfangchi. Shao-Kwang Liu, Chieh Ma and Shih-Yi Li. Pharm. Chem. Research Repts. (China) 1, No. 1, 1–11 (1935); cf. Hsu, C. A. 29, 7570° and J. Chinese Chem. Soc. 3, 365 (1935).—The alkaloid from Chinese hanfangchi can be extd. either by soaking the plant material in 5% AcOH for 2 months and then heating at 40–50° for 2 days, or by extg. 8 times with 95% alc. The free alkaloid is liberated from its salt by Na<sub>2</sub>CO<sub>3</sub>. It is then purified by dissolving in alc., extg. the alc. soln with ether, pptg. as phos-phate, recrystg. the phosphate from alc., recrptg. the free alkaloid by adding NH<sub>4</sub>OH to an aq. soln. of the pure phosphate, and finally recrystg. the alkaloid from acetone or ether. Both hanfangchi and mofangchi (bought from the Nanking market) yield apparently the same alkaloid, m. 215–17° (uncor.).

**C. L. Tseng**  
Organic chemical identification of the alkaloid from Chinese hanfangchi Shao-Kwang Liu and Chen-To Lo. Pharm. Chem. Research Repts. (China) 1, No. 1, 13–28 (1935); cf. preceding abstr.—By applying known methods of study, the alkaloid from hanfangchi, [α]<sub>D</sub><sup>20</sup> + 280 S (in CHCl<sub>3</sub>), was shown to possess in its mol. 1 double bond, 1 carbonyl, 2 MeO and 1 NMe groups.

**C. L. Tseng**  
Purification of the alkaloid from Japanese hanfangchi Shao-Kwang Liu, Chieh Ma and Shih-Yi Li. Pharm. Chem. Research Repts. (China) 1, No. 1, 29–35 (1935); cf. preceding abstr.—The alkaloid from Japanese hanfangchi, which is more difficult to isolate than that from the Chinese plant and is present in very small quantity only, is best isolated and purified by the following procedure. Soak the powd. plant material in 95% alc. for 2–3 weeks (or percolate with alc. until all the alkaloid is extd.), evap. the nearly black soln., filter, ext. the filtrate with petroleum ether to remove oil and fats, treat the mother liquor with Pb acetate, filter again, treat the filtrate with H<sub>2</sub>SO<sub>4</sub> to remove Pb, and then ppt. the free alkaloid by adding Na<sub>2</sub>CO<sub>3</sub> or NH<sub>4</sub>OH to the last filtrate. Shake this ppt. and the mother liquor with ether, evap. the ether, dissolve the residue in dil. HCl and filter again. Add NH<sub>4</sub>OH to the filtrate and shake the mixt. with ether as before. Dry the ether ext. and pass in dry HCl gas, when the HCl salt of the alkaloid is pptd. as a white ppt. This HCl salt is recrystd. several times from acetone and thus obtained pure. The free alkaloid m. 160–3° (uncor.). HCl salt, m. 235–9° (uncor.). This alkaloid is possibly identical with the sinomerine isolated by Japanese chemists from the same plant, but it is evidently different from that obtained from the Chinese hanfangchi bought in Nanking.

**C. L. Tseng**  
Organic chemical identification of the alkaloid from Japanese hanfangchi. Shao-Kwang Liu and Chen-To Lo. Pharm. Chem. Research Repts. (China) 1, No. 1, 37–49 (1935); cf. preceding abstr.—The alkaloid from Japanese hanfangchi has the mol. formula C<sub>17</sub>H<sub>17</sub>NO<sub>4</sub>, [α]<sub>D</sub><sup>20</sup> –66° (in CHCl<sub>3</sub>), and is a mono-acid base. By known methods of study, it is shown to contain 1 double bond, 1 carbonyl,

1 phenolic OH, two MeO and one NMe groups. Its formula can therefore be written as  $C_{11}H_{11}(CO)(OH)(NMe)(OMe)$ . Possible alternative structural formulae are discussed.

C. L. Tseng  
 Quinic acid and derivatives VI Degradation of shikimic acid to acetic acid Hermann O. L. Fischer and Gerda Dangschat *Helv. Chim. Acta* 18, 1204-6 (1935); cf. C. A. 29, 1002<sup>1</sup>.—The necessarily careful oxidation of Me shikimate (I) was carried out by the slow addn of a 10% HIO<sub>4</sub> soln contg 0.02 mol O<sub>2</sub> to a soln of 1.88 g of I (0.01 mol) in 10 cc H<sub>2</sub>O at room temp. After 30 mins the HI formed was removed by shaking with 7 g AcOAg, the soln was neutralized with HCl and filtered through animal black. The filtrate was chilled with ice and treated with a cooled soln. of 50% perpropionic acid (cf. Ans and Frey, C. A. 6, 2737) corresponding to 2.5-3 mols of active O. After standing overnight, the reaction mixt was heated on the steam bath and, on cessation of gas evolution, was evapd *in vacuo*. The partially cryst residue was taken up in 20 cc. of 2 N NaOH, heated for 1 hr. at 50°, and, after precise acidification with H<sub>2</sub>SO<sub>4</sub>, was taken down to dryness *in vacuo*. Repeated extrn of the residue with AcOEt gave 0.6 g (35%) of acetic acid (II),  $C_2H_4O_2$ , m. 184-91° (C. A. 23, 1878), transformed, in 75% yields, to tricarballic acid, m. 157-8°, by catalytic hydrogenation in the presence of Pd II, but with a lower m. p., was prepd in almost the same yields by a corresponding procedure starting with free shikimic acid. Quinic acid similarly yields 30% of citric acid. The dialdehyde resulting from the HIO<sub>4</sub> oxidation of I was pptd from the HI- and Ag-free soln by the addn of 2,4-dinitrophenylhydrazine, as the bis-2,4-dinitrophenylhydrazone,  $C_{11}H_{11}N_4O_8$ , m. 169°, in 75% yields. VII Configuration of shikimic acid *Ibid* 1206-13.—Dihydroshikimic acid (I) (C. A. 24, 464), m. 180°, [α]<sub>D</sub><sup>20</sup> -13.45 (in H<sub>2</sub>O) (tr-Ac deriv, b<sub>1</sub>, 200° (anhydride, m. 171-3°), anilide, m. 145-7°), was heated to 190°, and, after the total evolution of gas, was distd at 0.2 mm. from a bath at 220°. The distillate was taken up in MeOH and, on the addn of AcOEt to the concd soln, yielded dihydroshikimic acid lactone (II),  $C_{11}H_{11}O_5$ , m. 146-7°, [α]<sub>D</sub><sup>20</sup> -145.2°, which was completely inert to (AcO)<sub>2</sub>Pb AcOH (C. A. 29, 1009<sup>1</sup>). Treatment of I with Ac<sub>2</sub>O gave the di-Ac deriv of II,  $C_{11}H_{11}O_5$ , m. 145-6°, [α]<sub>D</sub><sup>20</sup> -128.3° (in ordinary CHCl<sub>3</sub>). The working up of a soln. of 1 g of shikimic acid (III) in 20 cc of dry Me<sub>2</sub>CO contg 1% HCl yielded 1.1 g (90%) of acetoneshikimic acid (C. A. 24, 464), m. 184° (decompn). III also gave a tri-Ac deriv,  $C_{11}H_{11}O_8$ , b<sub>1</sub>, 200-10°, decompn on heating at 240-60°, particularly in the presence of a trace of HCl, to m. AcOCH<sub>2</sub>CO<sub>2</sub>H, m. 130°. The working up of a soln. of 10 g of Me shikimate (IV) in 100 cc of Me<sub>2</sub>CO contg 1% HCl gave Me isopropylideneshikimate (V),  $C_{11}H_{15}O_5$ , b<sub>1</sub>, 150°, volenesulfonate deriv,  $C_{11}H_{15}O_5S$ , m. 66-67°, cleaved with AcOH to the monobenzenesulfonate of IV,  $C_{11}H_{15}O_5S$ , m. 137-8°; Es<sub>2</sub> deriv b<sub>1</sub>, 180°, catalytically reduced to the benzoate, m. 121°, prepd by the acetonization and benzoylation of reduced IV (C. A. 29, 1009<sup>1</sup>) and cleaved to the mono-Es deriv of IV,  $C_{11}H_{15}O_5$ , m. 136°. Treatment of V with MeOH-NH<sub>3</sub> gave good yields of isopropylideneshikimide (VI),  $C_{11}H_{15}ON$ , m. 150°, which could not be further acetonated with Me<sub>2</sub>CO contg HCl. The hydrogenation product of VI could not be isolated but with a mixt of pyridine and Ac<sub>2</sub>O was converted into cryst monooctylisopropylideneshikimide,  $C_{11}H_{15}NO_5$ , m. 125°, and on treatment with BrCl and pyridine gave the monobenzoate of isopropylideneshikimic acid nitrate,  $C_{11}H_{15}NO_6$ , m. 168-69°. From the formation of II, its rotation and that of its di-Ac compd, and the prepn of V a configuration of I is proposed in which the following groups in the 1-, 2-, 3-, 4-, 5- and 6-positions are on 1 side of the plane contg the C atoms CO<sub>2</sub>H, H, H, OH, OH and H and the remaining substituents H, H, OH, H, H and H are on the opposite side. In III the configuration around the OH-carrying C atoms is the same but the CO<sub>2</sub>H group, controlled by the double linkage, will be directly in the plane of the ring in the naturally occurring acid. The posi-

tion of the double bond in I is between C<sub>3</sub> and either C<sub>4</sub> or C<sub>5</sub> but has not been definitely located in the present investigation. The genetic relationship existing between quinic, shikimic and gallic acids is pointed out and the probability of the formation of the latter by loss of H<sub>2</sub>O and dehydrogenation is suggested. C. R. Addinall.

Lignin and related compounds XII Methanol lignin. Fritz Brauns and Harold Hibbert *Can. J. Research* 13B, 28-34 (1935).—Methanol lignin was prepd. by extg spruce wood meal with abs. MeOH, using HCl as catalyst. In 5 small, bomb tube expts, methanol lignin preps having the same MeO content (about 21.6%) were obtained, the product in each case being apparently homogeneous. The methanol lignin was acetylated, partially methylated by treatment with CH<sub>3</sub>N<sub>3</sub> and fully methylated with Me<sub>2</sub>SO<sub>4</sub> and NaOH. From the elementary analyses and the ratio of MeO in the original methanol lignin to that in the diazomethane methylated compd a formula for the smallest building unit of the methanol lignin and for the native lignin can be derived. The latter is represented by the empirical formula  $C_{11}H_{11}O_4$ , or, expanded,  $C_{11}H_{11}O_4(OMe)_2(OH)_2$ . XIII The structure and properties of glycol lignin. K. R. Gray, E. G. King, Fritz Brauns and Harold Hibbert, *Ibid* 35-47.—Lignin was extd from spruce wood meal with (CH<sub>3</sub>OH), contg (a) 0.05% and (b) 0.2% HCl. On purification the two glycol lignins were found to contain 10.9% OMe and 62.8% C, and 16.5% OMe and 64.7% C, resp. The aq glycol mother liquor was found to contain a sol lignin-carbohydrate compd. The glycol lignin could be sep'd into a CHCl<sub>3</sub>-insol and a CHCl<sub>3</sub>-sol fraction, each having the same chem compn and yielding the same methylated products. From the glycol lignin, purified by means of dioxane-ether, acetylated and methylated derivs were prep'd, as were also trityl derivs of glycol lignin and pre-methylated glycol lignin. The introduction of 3 trityl groups indicates the presence of 3 primary alc groups in the original lignin. Glycol lignin can be both partially and completely demethylated by use of HI. The effect of the glycol radical on the MeO value in the Zeisel detn was studied and an equation derived for the correction of the MeO value. The bearing of the results on the formula proposed previously for methanol lignin is discussed. XIV. The action of lead tetraacetate and of mercuric acetate on glycol lignin derivatives. K. R. Gray, Fritz Brauns and Harold Hibbert *Ibid* 48-60.—Fully methylated glycol lignin (OMe, 30.4%) was treated with Pb(OAc)<sub>4</sub> and with Hg(OAc)<sub>2</sub>. In the first case the AcO group was introduced to the extent of 11.5%. With Hg(OAc)<sub>2</sub>, in alc, the sole reaction was the substitution of H by the HgOAc group, there being no addn of EtO or loss of MeO groups. After repeated mercurization the final product contained 42.7% of Hg, i. e., a ratio of introduced Hg atoms to original MeO groups of 1.1. XV. Phenol glycol lignin. Fritz Brauns and Harold Hibbert *Ibid* 55-60.—Phenol condensation products of glycol lignin, of a pre-methylated and a fully methylated glycol lignin have been prep'd. In each case it has been found that, on the basis of the 5 MeO groups present in the original glycol lignin, 3 mols of phenol react with one lignin unit by nuclear condensation, and when the product is worked up immediately no loss of MeO groups occurs. With the fully methylated glycol lignin in 1 expt, some of the MeO groups were removed but the OH groups thus formed could not be remethylated with CH<sub>3</sub>N<sub>3</sub>, thus indicating their probable aliphatic character. Quercetin does not give a phenol condensation product under the same conditions. J. W. Shipley.

Oxidation of 3-epidihydrocholesterol acetate with chromic oxide 3-Epidihydrocholesterolic acid S. Kuwata and T. Toyama *J. Pharm. Soc. Japan* 55, 978-84 (1935) (in German 1934-4).—Oxidation of epidihydrocholesterol acetate (I), m. 96-7°, with CrO<sub>3</sub>, according to Ruzicka, (C. A. 29, 810<sup>1</sup>) gave 3-acetoxycarboxycholesterolic acid,  $C_{27}H_{46}O_6$ , 199.5° (cor.). Lieberman's phytylsterol reaction is pos. The Na salt of I (3 g) on heating with 4% alc KOH (150 cc) on the water bath for 2 hrs gave, after addn of HCl, 3-epidihydroxycho-

cholic acid (III),  $C_{24}H_{40}O_6$ , m. 244° (cor.). Methylation of II gave 3-epihydroxyallocholic methyl ester of II,  $C_{25}H_{42}O_6$ , m. 164.5° (cor.). Methylation of epiachoxyallocholic acid gave 3-epiacetoxyallocholic methyl ester,  $C_{27}H_{44}O_6$ , m. 148° (cor.). Oxidation of II with  $CrO_3$  gave 3-ketoallocholic acid,  $C_{24}H_{38}O_6$ , m. 187° (cor.).

F. I. Nakamura  
Nor- and bisnorlithocholic acids F. Reindel and K. Niederlander. *Ber* 68B, 1969-73 (1935).—For the degradation of the side chain of lithocholic acid (I) by 1 and 2 C atoms, essentially the Wieland, Schlichting and Jacobi method (*C A* 21, 590) was used. In prep. the Me ester of I it was observed that  $CH_3N_2$  gives the known Me ester, m. 125-7°, which seeps with 1.5 mols MeOH, but HCl gas in abs MeOH gives an alc.-free labile ester, m. 90-3°, which, on long standing and recryst. from MeOH or on heating about 0.5 hr above its m. p. rearranges into the higher-melting stable isomer. Both esters give 1, on even mild hydrolysis. *Et* ester, from I with HCl-EtOH, m. 92-3°. *Benzyl* ester, needles with 1  $H_2O$ , m. 145-8°. *3-Ac* deriv. of I, from I boiled with  $Ac_2O$ , m. 189°. *Et* ester, from the *Et* ester of I with  $Ac_2O$ , m. 90-1°, splits off the *Ac* group on attempted sapon. to the free acid. The Me ester (3 g) of I in 30 cc. ether dropped in the course of 1.5-2 hrs into a boiling soln. prep. from 8.5 g MeI and 1.5 g Mg in 15 cc ether gives, after removal of unchanged I with boiling alc. NaOH, about 3 g of *nor*-epicoprostanic-3,24-diol (II), needles from MeOH, m. 141-2°, addn. of water to the mother liquors gives 0.3 g. of leaflets of the same m. p. and mixed m. p. but contg. 1 mol  $H_2O$  of crystn. *Di*-acetate, m. 79-80°, gives with  $CrO_3$  (6 atoms O) in  $AcOH$  50% of 3-acetyl*nor*lithocholic acid, m. 175-6°, hydrolyzed to *nor*lithocholic ( $\alpha$ -3-hydroxy*nor*lithocholic acid) (III), which m. 170-1°, resolidifies and m. again 181-2°. *Me* ester of III, m. 90-100°, *Et* ester, m. 83-5°. With cold  $CrO_3$ - $AcOH$  III gives *dehydranor*lithocholic (3-ketono*nor*lithocholic acid), m. 179-80°. *Me* ester, m. 90°, *semicarbazone*, m. 208-10° (decompn.). *Bis*nor*epicoprostanic*-3,23-diol (IV), prep. like II from the *Me* ester of III, crystals with 0.5  $H_2O$ , m. 196-7°, *di*-acetate, m. 105-6°. *Acetyl**bis*norlithocholic acid, from IV treated with  $Ac_2O$  (it is not necessary to isolate the diacetate) and oxidized with  $CrO_3$ , rosets with 1.5  $H_2O$ , m. 210-12°. *Me* ester, m. 148-50°. C. A. R.

The bile acids XLVII Martin Schenck. *Z. physiol. Chem.* 237, 105-12 (1935), cf. *C A* 29, 68938°. The ketolactamcarboxylic acid  $C_{24}H_{38}NO_6$ , of the bilanic acid series, by treatment with 57%  $HNO_3$  for 6 days was converted into the *aminonitrile*  $C_{24}H_{37}N_2O_6$  (I), decomp. 205-7°. The product was identical with a previous prep. obtained from the oxime lactam of bilanic acid. The yield was increased to 84.8% by shortening the reaction time to 5 days. Refluxing of I with 10% HCl added  $H_2O$  with formation of an *aminoamide*  $C_{24}H_{37}N_2O_6$ , decomp. 226°, identical with a previous prep. The identity of I was further established by a Beckmann rearrangement to the  $\beta$ -acid, m. 203°. A. W. Dox

Synthetic experiments on the constitution of the bile pigment. XIV. Synthesis of glaucobilin, and on urobilin and mesobilirubin. Walter Siedel. *Z. physiol. Chem.* 237, 8-34 (1935), cf. *C A* 29, 4019°. Glaucobilin IX a, so designated because of its derivation from hemin IX by cleavage at the  $\alpha$ -methine bridge of the porphyrin nucleus, has now been synthesized and its structure established in every detail. It is the first bilirubinoide of the unsymmetrical natural series to be prep. artificially by a clear-cut procedure. Moreover, a new proof is advanced for the derivation of bile pigments from the blood pigment. Neoxanthobilirubin acid and isoneoxanthobilirubin acid were esterified with  $CH_3N_2$  and the *Me* esters converted by treatment with dry HCN and HCl into the *formyl* deriv., m. 205.5° and 218-20°, resp. The corresponding free acids obtained by sapon. m. 257° and 273°, resp. Condensation of formylneoxanthobilirubin acid with isoneoxanthobilirubin acid by refluxing the suspension in MeOH with 48% HBr gave the *di*-*Me* ester of glaucobilin IX a, m. 232°, identical with the analytical prep. The

free acid, glaucobilin IX a, m. 316°, was similarly obtained by condensation in the presence of 2 N HCl and treatment of the products with dil.  $NH_4OH$  and  $AcOH$  and extrn. with  $CHCl_3$ , finally recrystg. from MeOH. Condensation in the presence of FeCl<sub>3</sub> yielded ferrobilin IX a, m. 265°, from which the *di*-*Me* ester, m. 263-4°, was obtained by esterification with MeOH-HCl. Two glaucobilins isomeric with the above IX a were prep. for purposes of comparison. Glaucobilin XIII a was obtained as the *di*-*Me* ester, m. 246-7°, by condensation of formylneoxanthobilirubin acid with neoxanthobilirubin acid in MeOH by means of 48% HBr. The corresponding ferrobilin XIII a, m. 275°, was obtained by treatment of mesobilirubin XIII in  $AcOH$  with FeCl<sub>3</sub> and HCl, and esterified to the *di*-*Me* ester, m. 282.5°. The 2nd isomer, glaucobilin III a, was obtained as the *di*-*Me* ester, m. 237.5-8.5°, by condensation of isoneoxanthobilirubin acid with  $CH_3N_2$  and  $Ac_2O$  and esterification with MeOH-HCl. Condensation of formylneoxanthobilirubin acid with isoneoxanthobilirubin acid in MeOH by 48% HBr gave mesobilirubin, while formylneoxanthobilirubin acid with isoneoxanthobilirubin acid yielded mesobilirubin A. W. Dox

Chemistry of plant sterols Jaromír Haňáček and František Fink. *Časopis Českoslov. Lékárnictva* 15, 206-12 (1935).—Of the 0.5% unsaponifiable substances found in *Oleum pruni armeniacae* phytosterol formed 90%. In sample 1 there was found 0.31% of phytosterol, 0.17% of which was free and 0.04% bound as the ester. Recrystd. from ether, sterol forms needle or ruler-like crystals. The I no. of sterol isolated from the above was 61.29. The formula of sterol crystd. from alc was found to be  $C_{27}H_{46}O$   $H_2O$  and that of the sterol crystd. from ether  $C_{27}H_{46}O$ . The Br deriv. of acetylated sterol forms a powder-like substance, m. 86°, with the probable formulas  $C_{27}H_{46}O(OAc)Br$  V. D. Karpenko

Corrosion in the production of ethylene chlorohydrin (Iskra) 9. Dehydroandrosterone (Butenandt, et al.) 11F. Testosterone—transformation of dehydroandrosterone into androstenediol and testosterone (Butenandt, Harnisch) 11F. Reactions of sulfuryl diamide (Wood) 6.

Hydrocarbons I. G. Farbenind. A.-G. (Carl Krauch and Martin Müller-Cunradi, inventors). *Ger.* 617,595, Aug. 26, 1935 (Cl. 12a, 19.01). Unsatd. aliphatic hydrocarbons are obtained by dehydrogenating satd. hydrocarbons at high temps. in the presence of a contact agent free from noble metal. Thus, an active C obtained by heating semi-coke to 800-900° in steam, is heated to 450-500° and  $C_4H_{10}$  vapor led over the mass. The liquid product obtained by cooling is  $C_4H_8$  rich in  $C_4H_6$ . Other examples are given.

Oxidation of hydrocarbons. Frank J. De Rewal (to Atmospheric Nitrogen Corp.). U. S. 2,022,845, Dec. 3. In the vapor-phase oxidation of a hydrocarbon of the naphthalene or anthracene series, with an O-contg. gas, as in the production of anthraquinone from anthracene, the reaction is catalyzed by the presence of N oxides.

Chlorinating ethylene hydrocarbons Meyer Berhner (to Texas Co.). U. S. 2,022,610, Nov. 26. Dichloro derivs. such as those of  $C_2H_4$ ,  $C_2H_2$  or  $C_2H_6$  are produced by the reaction of ethylene hydrocarbons with Cl in the presence of a mixt. of trichloro- and tetrachloro-propanes and -butanes. App. is described.

Olefins and aromatic hydrocarbons from methane and its higher homologs Paul Feiler (to I. G. Farbenind. A.-G.). U. S. 2,022,279, Nov. 26. A gas essentially comprising  $CH_4$  and its higher homologs is brought into contact with heated refractory material contg. 60-95% of SiC with a binder derived from feldspars, cryolites or feldspars, preheated to 700-900° and unsatd. hydrocarbons formed are removed, the refractory material being alternately heated to 900-1300° and brought into contact with the residual gas. App. is described. Cf. *C A* 29, 1430°.

Hydrated olefins. N. V. de Bataafsche Petroleum Maatschappij. *Fr.* 786,687, Sept. 7, 1935. The catalytic hydration of olefins is effected at above 100° under pres-

sure in a vessel the interior surface of which is composed for the greater part of a metal, the normal electrochem. potential of which is between +0.2 and -0.4 on the scale, the zero of which is the normal electrochem. potential of H. In an example Cu or a Cu alloy is used and the catalyst is an aq. soln. of  $H_2SO_4$  contg. a sulfate of Be, Mg, Zn, Cd, Al, Mn, Cr, Co or Ni.

**Alcohols** Bohme Fettechemie-G m b H. (Wilhelm Normann and Hermann Frückner, inventors) Ger. 617,542, Aug. 26, 1935 (Cl. 12a 5 02) Aliphatic alcs with more than 8 C atoms in the chain are prep'd by treating the corresponding acids, free from aging products, with H at high temp. and pressure in the presence of a catalyst. Thus,  $(AcO)_2Cu$  is dissolved in water and added to kieselguhr. Excess of soda soln. is added to ppt. the Cu as carbonate. This is reduced in a stream of H at 190-200°. The product is a catalyst suitable for hydrogenating freshly prep'd acid from coconut oil to give the corresponding alc.

**Alcohols** N. V. de Bataafsche Petroleum Maatschappij Bnt. 433,868, Aug. 20, 1935 Alcs are produced by the hydration of olefins contg. at least 3 C atoms by treatment with an aq. mixt. of a Zn salt and an inorg. acid. Among examples, a mixt. of  $C_4H_8$  and  $C_4H_{10}$ , free from  $iso-C_4H_8$  and contg. 30%  $C_4H_8$ , is treated with an aq. soln. contg.  $ZnSO_4$ , 8 and  $H_2SO_4$ , 4.7% at 225° and 3000 lb. pressure.

**Aliphatic alcohols** I. G. Farbenindustrie A.-G. Bnt. 433,869, Aug. 21, 1935 Alcs of high mol. wt. are produced by condensing aldehydes contg. at least 2 C atoms by means of org. N-contg. catalysts and simultaneously or subsequently hydrogenating the products in presence of a hydrogenating catalyst. In examples, (1) crotonaldehyde is condensed by means of hydrogenated  $\beta$ -naphthoquinoline and  $Ca(OH)_2$  and the product is hydrogenated with a Co catalyst at 150° and 200 atm.; the product contains BuOH and octyl alc. and about 55% of monohydric and polyhydric alcs. of high mol. wt., without the  $Ca(OH)_2$ , only 39% of such alcs. is produced, and (2) a mixt. of AcH, decalhydroquinoline and a Ni catalyst or silica gel is hydrogenated to yield a product contg. EtOH 49, BuOH 21 and higher alcs. 30 parts Cf. C. A. 29, 3355<sup>4</sup>

**High molecular alcohols** Walther Schrauth and Theodor Böttler (to "Umchem" Chemikalien Handels A.-G.) U. S. 2,023,333, Dec. 3 A natural fat such as coconut fat has been added to a hydrogenation catalyst comprising a Cu, Zn, Pb, Mn, Co or Hg salt of palmitic, stearic, linseed oil, colophonic or naphthoic acid or chromous salt of cocoic acid and is subjected to the action of H under about 100-200 atm. pressure at a temp. of about 280-300° until the esters are sepd. into alcs. and acids and the acids are reduced to alc.

**Higher alcohols from waxes** Wilfred A. Sexton and Denis Ward (to Imperial Chemical Industries, Ltd.) U. S. 2,021,926, Nov. 26 For the production of a higher alc., a wax such as spermaceti or beeswax, etc., is heated with a dry mixt. of KOH and NaOH in proportions such as to give a eutectic mixt. of the corresponding soaps, and this mixt. is treated with superheated steam.

**Drying alcohols and esters** Benjamin T. Brooks (to Standard Alcohol Co.) U. S. 2,022,274, Nov. 26 For drying isopropyl alc. and esters or other alcs. and carboxylic acid esters contg. 3 or more C atoms, the material is treated with a concd. aq. soln. of caustic alkali, and the dried compd. is sepd. (suitably by gravity). App. is described.

**Amines** Knoll A.-G. Chemische Fabriken and Wilfrid Klavehn. Ger. 617,536, Aug. 26, 1935 (Cl. 12a 19 03). Unsaid. amines are obtained by condensing 2-methyl-2-hepten-6-one with a primary amine and subjecting the product to the action of a reducing agent. The condensation and reduction may be carried out together. In an example 2-methyl-2-hepten-6-one is dissolved in alc. and treated with aq.  $CH_3NH_2$  soln. Activated Al and alc. are added. The product is 6-methylamino-2-methyl-2-heptene, b. 175-6° (picrate m. 70°). The yield is 90-95%. Other examples describe the prepn.

of the ethylamino and benzylamino compds., b. 66-8° and b. 161-3°, resp.

**Amines** Knoll A.-G. Chemische Fabriken and Wilfrid Klavehn. Ger. 617,579, Aug. 30, 1935 (Cl. 12a 19 03). Addn. to 617,536 (preceding abstr.). Unsaid. amines are obtained by the method of 617,536 by using methylheptenone (2-methyl-1- and -2-hepten-6-one) or mixts. contg. this such as obtained by the hydrolysis of citral or lemon-grass oil. Thus, methylheptenone, b. 168-176°, obtained from citral by hydrolysis with dil. alkali, is dissolved in alc. and treated with aq.  $CH_3NH_2$  soln. and activated Al. The reaction mixt. is then treated with dil. mineral acid and the alc. distd. off. The residue is treated with steam to remove the nonbasic constituents. On adding alkali, a colorless oil seps. and can be driven off by steam. On distn., 6-methylamino-2-methyl-2-heptene, b. 175-6° is obtained.

**Amines** Karl Rosenmund. Ger. 617,647, Aug. 23, 1935 (Cl. 12g 32 10). Secondary and tertiary amines, aralkylamines contg. at least two  $PhCH_2CH_2$ , or their derivs. with one or more OH or alkory groups in the  $Ph$  residue, linked to the N atom, are obtained from  $PhCH_2CH_2NH_2$  or its derivs. Thus,  $p$ -MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> is dissolved in alc. and treated with  $PhCH_2CH_2Cl$ . An 80% yield of  $p$ -methoxyphenethylphenethylamine, m. 188°, is obtained.  $p$ -Methoxyphenylacetaldehyde in alc. or AcOH soln. is treated with H in the presence of Pd to give bis( $p$ -methoxyphenethyl)amine, m. 264°. The prepn. of bis(dimethoxyphenethyl)amine, m. 201°,  $o$ -methoxyphenylisopropylphenethylamine, m. 145°, ( $\beta$ -methoxyphenylphenethyl)phenethylamine, m. 191-193° and bis(dimethoxyphenethyl)methylamine, m. 230°, is described.

**Amines** Röhm & Haas Co. Ger. 617,900, Aug. 30, 1935 (Cl. 12g 5). Vaporized MeOH is treated with  $NH_3$  or primary or secondary amines in the presence of a substance contg. chemically bound P as the catalyst. The reaction preferably takes place at 250-500° with  $(NH_4)_2P_2O_5$  as the catalyst. Examples are given.

**Amines** I. G. Farben A.-G. (Walter Speer, inventor) Ger. 620,510, Oct. 23, 1935 (Cl. 12g 5). Addn. to 618,032 (C. A. 30, 107). Nonaromatic primary or secondary amines are converted into secondary or tertiary amines, resp., by reaction with aldehydes (or ketones) and esters of  $HCOOH$ . The reaction may be effected at 230-250° under pressure. Examples are given in which (1)  $C_6H_5NH_2$ , cyclohexanone (I), and  $HCOOMe$  (II) yield  $C_6H_5NHC_6H_{11}$ , b. 171-4°; (2) piperidine, I and II yield  $N$ -cyclohexylpiperidine, b. 123-3°; (3) cyclohexylamine (III), I and II yield  $(C_6H_{11})_2NH$ , b. 130-2°; (4) piperidine, benzaldehyde and II yield  $N$ -benzylpiperidine, b. 133-4°; (5) III, acetone and II yield  $N$ -isopropyl-III, b. 93-4°.

**Esters of dicarboxylic acids and polyhydroxy compounds.** Carl J. Malm and Charles R. Fordyce (to Eastman Kodak Co.). U. S. 2,023,455, Dec. 10 A nonsaccharide polyhydroxy compd. such as ethylene glycol is caused to react with a dicarboxylic acid anhydride such as phthalic anhydride and at least an equiv. amt. of a tertiary base such as pyridine and the resulting product is pptd. with a dil. inorg. acid such as 5% HCl soln. and purified.

**Esters of cyanohydrins of unsaturated aliphatic aldehydes** I. G. Farben A.-G. (Kurt Meisenburg, inventor) Ger. 617,764, Aug. 24, 1935 (Cl. 12a 22) The above esters are prep'd by the action of an aq. soln. of alkali cyanide on a mixt. of an unsaid. aliphatic aldehyde and an aroyl halide, or substitution products. Thus a mixt. of crotonaldehyde,  $C_4H_8$  and  $BaCl_2$  is cooled to -10° and treated drop by drop with an aq. soln. of NaCN to give the BrOH ester of crotonaldehyde cyanohydrin, b. 128-9°. Other examples describe the prepn. of the 2,5-dichlorobenzoyl acid ester of crotonaldehyde cyanohydrin, b. 158-9°, the BrOH ester of sorbaldehyde cyanohydrin, m. 47°, the BrOH ester of acrolein cyanohydrin, b. 158-8°, and the naphthoic acid ester of crotonaldehyde cyanohydrin, b. 180-182°.

**Tri-alkyl esters of phosphonic acid.** Concorium für elektrochemische Industrie G m b H. Bnt. 433,927,

Aug. 22, 1935. These esters are obtained from  $\text{POCl}_3$  and the corresponding alc. by causing the alc. to react with the  $\text{POCl}_3$  and treating the reaction product with dry  $\text{NH}_3$ , the temp. being kept down to minimize production of alkyl chloride. Examples describe the production of Et and Bu esters.

Polyglycerol esters of aliphatic acids of relatively high molecular weight Benjamin R Harris U. S. 2,023,348, Dec. 3. Numerous examples are given of the production of esters having wetting, emulsifying or antispattering properties, etc., the general mode of manuf. of which is to polymerize glycerol to a desired mol. magnitude, whether it be diglycerol, triglycerol, tetraglycerol or higher polymerized glycerols or mixts thereof, by heating glycerol by itself or in the presence of a catalyst, then freeing the polyglycerol or polyglycerol mixt of unpolymerized glycerol, if any be present, and finally esterifying the polyglycerol material, free of glycerol, with a fatty acid or a fatty acid mixt by causing the two to react with or without the presence of an esterification catalyst, or alternatively with acyl halides or acid anhydrides in or without the presence of a catalyst or condensing agent. As being new compds., general claim is made to esters of a polyglycerol and a relatively high mol "monobasic" aliphatic acid, the ester contg. at least one free hydroxy group.

Vinyl thio ethers and ethylene dithio diethers I G Farbenind A-G (Walter Reppe and Fritz Nicolai, inventors) Ger 617,543, Aug. 26, 1935 (Cl. 12a 23 03). Mercaptans are treated with  $\text{C}_2\text{H}_4$  at high temps. in the presence of a catalyst. The  $\text{C}_2\text{H}_4$  may be dild. by inert gases. In an example, a mixt. of  $\text{PhSH}$ ,  $\text{BuOH}$  and  $\text{KOH}$  is heated to  $160^\circ$  at 15 atm. with a mixt. of  $\text{C}_2\text{H}_4$  and  $\text{N}$  in an autoclave. The product is  $\text{PhSCH}_2\text{CH}_2\text{SCH}_2\text{Ph}$ . The prepn. is described of  $p$ - $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{SCH}_2\text{C}_6\text{H}_4\text{Me}$ ,  $b_1$ , 125–30°, 2-mercaptobenzothiazole vinyl ether,  $b_1$ , 135–40°, ( $\text{PhSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{Ph}$ ),  $m$ , 70°, ( $p$ - $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{SCH}_2\text{C}_6\text{H}_4\text{Me}$ ),  $b_1$ , 81°, ( $\text{EtSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{Et}$ ),  $b_1$ , 91°, ( $\text{EtSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{Et}$ ),  $b_1$ , 90–5°, and 1,8-chlorothioanaphthol vinyl ether,  $b_1$ , 154–5°.

Ether derivatives of morpholine alcohols Henry L. Cox and Thomas I Carruthers (to Carbide and Carbon Chemicals Corp.) U. S. 2,023,872, Dec. 10. Compds. such as  $N$ -( $\beta$ -ethoxyethyl)morpholine and 2,6-dimethyl- $N$ -(2-methoxypropyl)morpholine are formed by heating a 4-morpholine said lower alkyl alc. such as  $N$ -( $\beta$ -ethanol)morpholine or 2,6-dimethyl- $N$ -(2-propanol)morpholine with a dialkyl sulfate such as diethyl or dimethyl sulfate in the presence of an inorg. base such as  $\text{NaOH}$ .

Anthrone derivatives I G Farbenind A-G (Karl Köberle and Otto Schlichting, inventors) Ger 620,345, Oct. 19, 1935 (Cl. 22b 3 14). Leuco compds. of anthrone derivs., the 1- and 9-positions of which form part of a heterocyclic ring, are treated in an alk. medium with an aliphatic, aromatic or heterocyclic aldehyde, e. g.,  $\text{CH}_2\text{O}$ ,  $\text{BzH}$ , or furfural. Suitable starting materials are leuco compds. of 1,9-anthrapyrimidine and its substitution products or of 1,9-anthrapyrimidone or 1,9-pyridinoanthrone. Preferably, the aldehyde is added to an alk. vat of the anthrone deriv. at atm. or raised temp., and the pptd. product is filtered. Treatment of the mixt. with air or another oxidizing agent may be necessary to complete the pptn. The products are believed to be in part substitution products of the starting materials, thus,  $\text{CH}_2\text{O}$  yields Me derivs. and  $\text{BzH}$  yields  $\text{PhCH}_2$  derivs. some of the products are useful as dyes, and others as intermediates for dyes. Examples are given.

Salicylic acid derivatives Gddecke & Co, Chemische Fabrik A-G, Fr. 786,887, Sept. 11, 1935. See Brit 430,130 (C. A. 29, 7587°).

Nitrogenous products of oxazoles and imidazoles. I. G. Farbenind A-G. (Ernst Herdeckerhoff, Werner Zerweck and Heinrich Salkowski, inventors). Ger. 617,544, Aug. 21, 1935 (Cl. 12p. 3). Addn. to 615,526 (C. A. 29, 8003°).

Compds. of the general formula  $\text{X}-\text{arylene}-\text{N}-\text{C}(\text{SO}_2\text{H})_2$ , in which  $\text{X}$  represents  $\text{O}$  or  $\text{NR}$  ( $\text{R}$  being  $\text{H}$ , alkyl, aralkyl or aryl) are treated with  $\text{NH}_3$  or agents

yielding  $\text{NH}_3$ , or with amines of the aliphatic, aromatic, hydroaromatic or heterocyclic series, which have at least one  $\text{H}$  atom bound to the  $\text{N}$  atom. Thus,  $\text{Na}$  2-( $\beta$ -naphthoxazolesulfonate is heated with  $\text{NH}_3$  for 2 hrs. at  $120^\circ$  under pressure to give 2-amino- $\beta$ -naphthoxazole,  $m$ , 199–200°. The prepn. of 2-aminobenzimidazole,  $m$ , 235°, and 2-aminobenzoxazole,  $m$ , 173–4°, is also described.

Acetyl compounds Holzhydrolyse A-G. (Eduard Farber, inventor) Ger 617,989, Aug. 30, 1935 (Cl. 12a 6). Finely powd. carbohydrates are treated with a small excess of  $\text{Ac}_2\text{O}$  in the presence of an acid-condensing agent and the acetyl product, which is insol. in water, is washed or pptd. Thus, wood sugar or dextrin is powdered and treated with  $\text{Ac}_2\text{O}$  in the presence of concd.  $\text{H}_2\text{SO}_4$  or  $\text{ZnCl}_2$ . The products are used as starting materials for artificial masses, paints, etc.

Undecyl compounds Carbide & Chemicals Corp. Fr. 786,734, Sept. 9, 1935. Ethylhexaldehyde is condensed with acetone in the presence of an alk. catalyst to form undecyl ketol of the formula  $\text{C}_{11}\text{H}_{23}\text{CH}(\text{C}_2\text{H}_5)\text{CHOH}-\text{CH}_2\text{COCH}_3$ , which on losing the atoms of water is transformed to an unsatd. undecylene ketone of the formula  $\text{C}_{11}\text{H}_{21}\text{CH}(\text{C}_2\text{H}_5)\text{CH}=\text{CHCOCH}_3$ ,  $b$ , 229°. This is partially hydrogenated to form a satd. undecyl ketone,  $b$ , 93°, and hydrogenated to a secondary undecyl alc. of the formula  $\text{C}_{11}\text{H}_{23}\text{CH}(\text{C}_2\text{H}_5)\text{CH}_2\text{CH}_2\text{CH}(\text{OH})\text{CH}_3$ ,  $b$ , 225°, which can be sulfonated to give a product which has wetting and cleansing properties.

Desulfonating diaminodiphenylamine-2-sulfonic acid compounds Luther M. Lauer (to National Aniline & Chemical Co.) U. S. 2,022,889, Dec. 3. Desulfonation of compds. such as 4,4'-diaminodiphenylamine-2-sulfonic acid, etc., is effected by hydrolysis in the presence of a nonoxidizing inorg. acid, as by heating with aq.  $\text{H}_2\text{SO}_4$ .

Reaction of sodium with aromatic hydrocarbons Norman D. Scott (to E. I. du Pont de Nemours & Co.) U. S. 2,023,703, Dec. 10. An alkali metal such as  $\text{Na}$  is caused to react with naphthalene or a homolog, as in the production of 1,4-disodium naphthalene in a vehicle comprising dimethyl ethylene glycol ether or other polyether derived from an aliphatic polyhydric alc. having all the hydroxyl  $\text{H}$  atoms replaced by alkyl groups and which promotes the reaction but is inert to the reagents and products. Cf. C. A. 30, 490°.

Hydroxybetyl chloride. Clyde O. Henke and Milton A. Prahl (to I. I. du Pont de Nemours & Co.) U. S. 2,023,473, Dec. 10. Di- or tetra-hydroxybetyl chloride is obtained by reaction of  $\text{PCl}_5$  or thionyl chloride upon a hydrogenated rosin. Cf. C. A. 29, 8000°.

Metal carbamates Ernst Kuss and Emil Germann (to I. G. Farbenind A-G.) U. S. 2,023,890, Dec. 10. Carbamate is formed by reaction of  $\text{NH}_3$ , carbamate on  $\text{CaCl}_2$  in  $\text{EtOH}$  substantially in the absence of water. Other carbamates such as those of  $\text{Na}$ ,  $\text{K}$ ,  $\text{Ba}$  or  $\text{Zn}$  may be similarly formed from the chloride, nitrate or oxide of the metals. Several examples are given. Cf. C. A. 29, 2549°.

Dithiocarbamates. Wilhelm Rittmeister (to Deutsche Hydrierwerke A-G.) U. S. 2,022,979, Dec. 3. Dithiocarbamates are produced from  $\text{CS}_2$  and a primary or secondary amine such as piperidine, cyclohexylamine, dibutylamine or the like by reaction in finely divided state and while dild. by an inert carrier gas such as water gas or  $\text{H}_2$ . Several examples with details of procedure are given.

Aldehydes J. D. Riedel-E. de Haën A-G. (Friedrich Boedecker and Hans Volk, inventors). Ger. 617,763, Aug. 24, 1935 (Cl. 12a. 9). Aromatic hydroxy aldehydes are obtained by treating salts of glyoxylic acid with mono-alkyl ethers of pyrocatechol in a neutral or alk. medium, and subjecting the resulting condensation product to the action of a moderate oxidizing agent. Thus, a soln. of glyoxylic acid obtained by the electrolytic reduction of  $(\text{CO}_2\text{H})_2$  is neutralized with  $\text{NaOH}$  and added to a concd. soln. of guaiacol in  $\text{NaOH}$ . The product is oxidized by  $\text{CuSO}_4$  and  $\text{NaOH}$  to give vanillin.

Citrates Giuseppe Bosurgi and Paul Stukart. Ger. 620,152, Oct 16, 1935 (Cl 12o 11). A water-insoluble citrate of Pb or Zn is pptd. from suitable fruit juices or from fermentation mash in which citric acid is formed. The ppt. is then treated with a soln. of a salt derived from an alkali metal, NiH, or Fe and an acid which forms a water-insol. Pb or Zn salt. Carbonates, phosphates, sulfides or sulfates are suitable salts. The soln. of alkali metal, NiH, or Fe citrate so produced is crystal.

Xanthates Ernest D. Wilson (to W-B Chemical Co.). U. S. 2,021,930, Nov. 26. Vapors of CS<sub>2</sub> are brought into contact with a liquid monohydric alc. and a caustic alkali to form a xanthate. App. is described.

Cyclic cyano ketimides and ketones from alkylene dinitriles Schering-Kahlbaum A.-G. (Karl Ziegler, inventor). Ger. 620,904, Oct. 30, 1935 (Cl. 12o 25). Addn to 591,209 (C. A. 28, 2364). The process of Ger. 591,209 is modified by using as the condensing agents compds. of the formula RR'NM, where R is an aryl radical, R' is a nonaryl radical, and M is an alkali metal, preferably Na. The reaction is effected in a concd. or satd. soln. of the condensing agent in an org. solvent and under such conditions that the concn. of dinitrile in the reaction mixt. is always very low. In the examples, (1) CN-(CH<sub>2</sub>)<sub>6</sub>CN and PhMeNNa in etheral soln. yield 1-cyanocyclotetradecanone-2-imide, m. 147-8°, from which cyclooctadecanone, m. 53°, is obtained by boiling with 70% H<sub>2</sub>SO<sub>4</sub>; (2) CN-(CH<sub>2</sub>)<sub>6</sub>CN and PhC<sub>6</sub>H<sub>5</sub>NNa yield a cyano ketimide from which cyclohexadecanone is obtained as in (1).

Refining crude alkyl phenols Reuben Schuler (to Stanco Inc.). U. S. 2,022,256, Nov. 26. For removing alkyl phenyl ethers from crude alkyl phenols, the alkyl group of which contains 4 or more C atoms, such as *sec*-hexyl cresols, the crude material is dissolved in liquid NH<sub>3</sub>, the soln. is sepd. from the undissolved residue of the alkyl phenyl ethers, and the dissolved product is recovered from the liquid NH<sub>3</sub>.

Alkylated phenols Röhm & Haas Co. Ger. 616,786, Aug. 5, 1935 (Cl. 12g 16). Alkylated phenols of the general formula RR'OH, in which R' is a mono- or polycyclic aromatic or substituted aromatic nucleus and R is an alkyl group derived from disubutylene are obtained by treating PhOH or its homologs or substitution products with the equiv. amt. of di- or triisobutylene in the presence of a small amt. of concd. H<sub>2</sub>SO<sub>4</sub> at 25-35°. H<sub>2</sub>SO<sub>4</sub> of 96% strength is used, 0.075 to 0.30 mol. being used to each mol. of PhOH. Examples describe the prepn. of disubutylphenol, and disubutyl-*p*-cresol, m. 46-47°. The substances are used as intermediates for the prepn. of dyes, drugs, tanning and wetting agents.

Asymmetrical ureas Josef Hilger and Carl Taube (to General Aniline Works). U. S. 2,022,935, Dec. 3. Compds. of the general formula RNHCONXY, where R stands for the radical of an aminoazo compd. free from a free carboxylic acid or a free sulfonic acid group, but in which the nuclei may be otherwise substituted, for example by alkyl, hydroxyalkyl, halogen, the hydroxy group, an alkoxy group, the nitro group or an esterified carboxylic acid group, and X stands for H or alkyl and Y stands for radicals of the benzene or naphthalene series, are obtainable by causing an isocyanate of an aminoazo-compd. free from a free carboxylic acid or a free sulfonic acid group and ammonia or a primary or secondary amine of the aliphatic, aromatic or aliphatic-aromatic series, to act upon each other in a solvent. The process is favorably carried out by working with about mol. quantities of the two reacting components, and as a general rule can be performed at temps. of 30-100°, and due to the evolution of heat produced in the exothermic reaction, in some cases the process can be performed without heating. The products are suitable for use as dye intermediates and various examples are given.

Nitriles of resin acids Otto Nicodemus and Otto Wulff (to I. G. Farbenind. A.-G.). U. S. 2,023,337, Dec. 3. Gaseous NiH<sub>2</sub> is caused to react upon a resin acid or a resin acid ester such as colophony (suitably at a temp.

of about 350°) in the presence of a catalyst promoting dehydrations, such as silica gel. Nitriles are formed which form highly viscous balsams.

Synthesis of aliphatic acids Alfred T. Larson (to E. I. du Pont de Nemours & Co.). U. S. 2,022,344, Nov. 26. Acids such as propionic, butyric and valeric acids are produced by the reaction in the liquid phase, of an olefinic hydrocarbon such as C<sub>3</sub>H<sub>4</sub>, C<sub>4</sub>H<sub>6</sub> or C<sub>5</sub>H<sub>8</sub> with CO and water (suitably under a pressure of 25-900 atm. and at temps. of 150-385°). Various examples with details of procedure are given. The reaction may be catalyzed by the use of CuSO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub>, cupric phosphate and H<sub>2</sub>PO<sub>4</sub>, Cu<sub>2</sub>Cl<sub>2</sub> and HCl, an ammoniacal Cu soln., MgCl<sub>2</sub> and HCl and activated charcoal, CaCl<sub>2</sub>, Cd phosphate, silicotungstic acid, ZnCl<sub>2</sub>, CaI<sub>2</sub>, NaBr, KCl, etc. Cl. C. A. 30, 491.

Formic acid Gilbert B. Carpenter (to E. I. du Pont de Nemours & Co.). U. S. 2,023,003, Dec. 3. A gaseous mixt. of CO and steam (suitably at a temp. of about 325°) is passed over a catalyst contg. primary Ca phosphate or other suitable acid salt of the acidic oxides of P, As, W, Mo, U, Cr, V, B, Si or Zr. Pressures of 25-900 atm. may be used. Cl. C. A. 29, 4379.

Polyhydroxy monocarboxylic acids Oskar Spengler and Adolf Pfannenstiel. Ger. 620,248, Oct. 17, 1935 (Cl. 12o 11). Monosaccharoses, or higher sugars having reducing properties, are treated in aq. alk. soln. with air at about 45-90°, the air being fed to the soln. in such a state of subdivision that the soln. becomes turbid. Polyhydroxy carboxylic acids contg. a lower no. of C atoms in the mol. than the starting materials are obtained. An example is given of the prepn. of *d*-arabonic acid from glucose. Cl. C. A. 30, 111.

Aromatic arsenic acids Karl Streitwoll, Alfred Fehrlé and Hubert Oesterlin (to Winthrop Chemical Co.). U. S. 2,023,751, Dec. 10. See Ger. 543,114 (C. A. 26, 3263).

Aminopyrenesulfonic acids I. G. Farbenindustrie A. G. Brit. 434,123, Aug. 26, 1935. The acids are obtained by (1) treating 3-aminopyrene (I) with a sulfonating agent at 20-50°, (2) converting I into its acid sulfate and heating the product, preferably *in vacuo*, to a high temp. and (3) nitrating 8-pyrenesulfonic acid (II) and reducing the product. In examples, (1) I is sulfonated with H<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>O to give a mixt. of 3-amino-8- and -10-sulfonic acids, (2) I is converted to the sulfate, which, on heating *in vacuo*, gives the 3-amino-4-sulfonic acid and (3) II, obtained by treating pyrene in CCl<sub>4</sub> with CH<sub>3</sub>SO<sub>3</sub>H, is nitrated and then reduced with a Ni catalyst and H<sub>2</sub> under pressure. In 434,160, Aug. 27, 1935, divided on and addn. to 434,123, a 3-aminopyrenesulfonic acid is obtained by treating I with CH<sub>3</sub>SO<sub>3</sub>H, whereby it is converted into the chlorosulfonate, and heating in a solvent, e. g., *o*-dichlorobenzene, which causes the evolution of HCl. Cl. C. A. 29, 6164.

Replacing the sulfonic acid group by hydrogen. Chemische Fabrik von Heyden A.-G. (Bayer-Krmer and Karl Willy Rittler, inventors). Ger. 617,720, Aug. 24, 1935 (Cl. 12g 14 02). The HSO<sub>3</sub> group in salts of aromatic, hydroxy sulfonic acids is replaced by H by heating the salts with water under pressure in the presence of an equiv. amt. of a water-sol. alk. earth salt. Thus, Na phenolsulfonate is heated to 200° with CaCl<sub>2</sub> in water and a small amt. of HCl in an autoclave at 14-15 atm. On removal of the resulting CaSO<sub>4</sub>, a 90% yield of PhOH can be steam-distd. off. Other examples are given.

Acetaldehyde. Chemische Forschungs G. m. b. H. (Hans Deutsch and Willy O. Herrmann, inventors). Ger. 617,762, Aug. 30, 1935 (Cl. 12o 7 02). Acetaldehyde is made distn.-stable in mono- or trimeric form by treatment with acid-binding substances with the exception of org. salts, in such quantity that a negligible amt. of alkol is formed. Preferably agents which bind only the inorg. or *inorg*-substituted org. acids are used. In an example, C<sub>6</sub>H<sub>5</sub>I is led over AcOH and H<sub>2</sub>SO<sub>4</sub> to give ethylidene diacetate. This is split into AcOH and AcH by water and C<sub>6</sub>H<sub>5</sub>SO<sub>3</sub>H. The AcH is an unstable mixt. of AcH and



paraldehyde. If shaken with  $\text{AcONa}$ , the  $\text{AcH}$  is rendered sufficiently stable for sepn. from the paraldehyde by fractional distn. Other examples are given.

Acetaldehyde from acetylene I. G. Farbenind A-G (Walter Rosinsky, inventor) Ger. 620,402, Oct. 21, 1935 (Cl. 120 7 02) This corresponds to Brit 313,864 (C. A. 24, 1125), but gives addnl examples

Dichloroethylene Compagnie de produits chimiques et électrometallurgiques Alus, Iroges et Camargue Fr 786,803, Sept. 10, 1935  $\text{CCl}_2\text{CH}_2$  is made by heating metals such as Fe, Zn and Cd with  $\text{CCl}_2\text{CH}_2\text{Cl}$  in the presence of water

Ethylene oxide Edgar C. Britton, Gerald H. Coleman and Iverson Mate (to Dow Chemical Co.) U. S. 2,022,182, Nov. 26 In the prepn of  $\text{C}_2\text{H}_4\text{O}$ , a base such as  $\text{NaOH}$  is gradually added to  $\beta$ -chloroethyl acetate while agitating the reaction mixt and maintaining it at a temp. of between about  $40^\circ$  and about  $150^\circ$

Ethylene oxide N. A. de Bitaafche Petroleum Mart-schappij Brit 434,011, Aug. 23, 1935  $\text{C}_2\text{H}_4$  is oxidized with the use of a catalyst prepd by the thermal decomposn of an org. Ag compd., particularly the oxalate. One or more heavy metals, e. g., Cu, Au, I, Fe, Mn, Co, Ni, Ce, Th or Zn may be present as activators. The reaction may be effected at  $350\text{--}400^\circ$ . The gas mixt. should contain 8–9%  $\text{C}_2\text{H}_4$ , diluents, e. g., steam or N, may be present

Mono- and dimethylamine I. G. Farbenind A-G (Paul Herold and Karl Schmalz, inventors) Ger. 620,298, Oct. 17, 1935 (Cl. 124 4) Mixts. contg.  $\text{MeNH}_2$ ,  $\text{Me}_2\text{NH}$  and water are subjected to fractional distn. so as to sep. a mixt. of the amines from the water. The amines are then sepd. from one another by further fractionation. The

process may be applied to mixts. obtained by the reaction of  $\text{MeOH}$  or  $\text{Me}_2\text{O}$  with  $\text{NH}_3$ . An example is given.

3,3-Dimethyl-1-bromobutane. Frank C. Whitmore and Walter R. Trent (to Mallinckrodt Chemical Works). U. S. 2,022,485, Nov. 26 This compd., a colorless oily liquid  $b_m$  134–7° and of pleasant odor, is produced by reaction of  $\text{PBr}_3$  on 3,3-dimethyl-1-butanol.

Musk xylene Walter V. Wirth (to E. I. du Pont de Nemours & Co.) U. S. 2,023,865, Dec. 10. Nitration of *tert*-butyl-*m*-xylene, in prep. musk xylene, is effected in one step by the use of a com. grade of mixed acid.

*tert*-Butyl-*m*-xylene Walter V. Wirth (to E. I. du Pont de Nemours & Co.) U. S. 2,023,566, Dec. 10. *tert*-Bu chloride is caused to react with *m*-xylene in the presence of  $\text{AlCl}_3$  in less than 2% of the amt. of the *tert*-Bu chloride, while keeping the temp. below  $50^\circ$  during the entire reaction.

Pentaerythritol Lonza I. kktirizitätswerke und Chemische Fabriken A-G Fr 786,000, Sept. 14, 1935 See Swiss 176,018 (C. A. 30, 1129)

Hydrogenated benzoisotriazoles Chuno Gygyszer és Vegyszeri Termék Gyára R. T. (Kereszty & Wolf). Ir 786,884, Sept. 11, 1935 See Ger. 613,123 (C. A. 29, 50019)

Acenaphthylene I. G. Farbenind A-G (Carl Wulff, Otto Nicodemus and Max Truppenhauer, inventors). Ger. 617,004, Aug. 26, 1935 (Cl. 120 19 01).

Acenaphthene vapor is heated to high temps. in the presence of a dehydrogenating catalyst consisting of difficultly reducible oxides of metals of the 2nd to 7th groups of the periodic table. Examples of such catalysts are  $\text{ZnMoO}_4$  and  $\text{MgO}$ ,  $\text{ZnO}$  and  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$  and C. Cf. C. A. 29, 51294.

## 11— BIOLOGICAL CHEMISTRY

PAUL E. HOWE

### A—GENERAL

ARTHUR W. DOX

Biochemistry of zinc Oswaldo de Almeida Costa *Rev. quim. e farmacia* 1, 3–11 (1935)—A review with 4 references. E. S. G. B.

Cozymase. H. v. Euler, H. Albers and F. Schlenk *Z. physiol. Chem.* 237, 1–11 (1935), cf. C. A. 29, 58634.—The purification procedure has now been perfected to the point where a uniform product with cozymase activity of 400,000 can be obtained in 25% yield. Analyses correspond to the formula  $\text{C}_{11}\text{H}_{16}\text{O}_4\text{N}_4\text{P}_2$ . The substance is believed to be pure cozymase. It contains 19.5% adenine. On the assumption that the substance is a dinucleotide, 5 of the 8 N atoms are in adenine, 2 belong to a basic component which yields nicotinic amide on hydrolysis and the 8th may account for the  $\text{NH}_3$  evolved. The picric acid salt has N content and cozymase activity in close agreement with the calcd. values. A. W. Dox

Enzymic histochemistry. XVI. The digestion of keratin by the larvae of the clothes moth (*Tineola bisellella* Humm). K. Landerstrom-Lang and F. Duspiva *Z. physiol. Chem.* 237, 131–58 (1935); cf. C. A. 29, 47014.—In the intestinal cells as well as in the succus entericus, a very active proteinase is present which shows optimal cleavage of casein at  $\text{pH}$  9.3. The proteinase does not attack native wool either at its optimum  $\text{pH}$  or at the  $\text{pH}$  of the intestinal juice (9.6–10.2). The alk. reaction alone is not sufficient for hydrolysis of keratin. The intestinal juice of larvae which have eaten wool gives an intense nitroprusside reaction, which, however, does not occur if they have not eaten or have been fed a nutrient poor in S (cotton-casein), but reappears when cystine is added to the feed. The nitroprusside reaction is neg. if the intestinal juice of wool-fed larvae is exposed to air a few min; hence the SH-substance is autooxidizable. In cleavage exps. with wool at  $\text{pH}$  10 and addn. of reducing agents ( $\text{Li-H}$  and  $\text{HSClHCO}_2\text{H}$ ) the wool dissolves with liberation of  $\text{NH}_3$  and  $\text{CO}_2\text{H}$  groups in equal proportions, indicating the cleavage of peptide linkages. In the mid-

intestinal lumen of living larvae there is a reduction which can be demonstrated by feeding oxidation-reduction indicators. The reduction state is independent of the presence of major quantities of SH-compds. and persists in larvae which have been fed cotton-casein. The existence of a reducing agent is thus demonstrated. The proteinase of *Tineola* is somewhat sensitive to SH in the hydrolysis of casein. Trypsin-kinase is inert in the reducing medium required for the breakdown of keratin. Larvae of *Tineola* thus possess a keratinase, in the sense of a system which consists of a proteinase active in a reducing medium at alk. optimum and a reducing agent which breaks down the molecular structure of keratin and so renders the protein accessible to enzyme action. A. W. Dox

The free diffusion of nucleic acids and mononucleotides as a means of determining their molecular magnitude. With especial consideration of the molecular magnitude of the pentosepolynucleotide from pancreas and a comment on the molecular magnitude of cozymase. K. Myrback and E. Jorpes *Z. physiol. Chem.* 237, 159–64 (1935).—Two important factors must be considered in detg. the mol. wt. of nucleic acids by the diffusion method. The elec. charge of the substance affects the apparent mol. wt., so that adenylic and guanylic acids give about half, and yeast nucleic acid gives about one-third, of the true value, while nonelectrolytes such as sucrose show the correct value. Salts exert an inhibitory influence on the diffusion of the electrolytes, increasing with increase in mol. wt. Pancreatic nucleic acid diffuses more slowly than yeast nucleic acid, because of the greater size of the mol. For a polynucleotide structure, as the analysis indicates, the mol. wt. is calcd. to be 1649. In 1%  $\text{NaCl}$  soln., they give values near the theoretical for guanylic, adenylic and yeast nucleic acids, the value for pancreas nucleic acid is about 3000. Whether this is because of a union of 2 pentanucleotide complexes or the result of an increased salt effect on the larger mol. is difficult to decide. At any rate the pentosenucleic acid in the pancreas cannot be a mixt. of yeast nucleic acid with guanylic acid, but must be considered a particular type among the polynucleotides.

Expts with mononucleotides, especially those with yeast adenylic acid, have a certain bearing on the question of the mol magnitude of cozymase. The value of approx. 500 was not materially altered by diffusion in 5% NaCl. Possibly this is because cozymase is a monobasic and less strongly dissociated acid than adenylic acid. The expts. indicate a mol wt of cozymase not far from 500.

A. W. Dox

Glycerophosphatase activity in the tissues of animals deprived of vitamin A. Luc Emerique *Bull. soc. chim. biol.* 17, 1372-7 (1935), cf. C. A. 29, 4795.—Lack of vitamin A caused a slight decrease in the glycerophosphatase content of the kidneys (rats) and a considerable increase in that of the lungs. There was no change with the kidneys. Individual variations were large. L. E. G.

Energy of growth. XIV Action of toxic concentrations of zinc and manganese salts on energy yields of germinating seeds. Germaine Boy *Bull. soc. chim. biol.* 17, 1414-26 (1935).—When  $ZnSO_4$  or  $MnSO_4$  was added to water cultures of rice in such concn that germination and growth were retarded the "growth energy" yield, as detd by the heat of combustion of the seedlings, was considerably decreased. L. E. Gilson

Dehydrogenase of the S and R forms of Aertycke's bacillus. Eugène Soru *Compt. rend. soc. biol.* 120, 232-5 (1935).—Methylene blue was used as the H acceptor. With glucose, asparagine, Na lactate and Na succinate as H donors the dehydrogenase of the S form was much more active. With glycerol the difference was slight and with glycine there was no apparent difference in the activity of the 2 forms. L. E. Gilson

Compounds of proteins with o-nitrophenols. Adolph Bolliger *Australian J. Exptl. Biol. Med. Sci.* 13, 189-91 (1935).—Edeston, casein and globin form compds. which are approx. const. in compn by "saturating" with picric acid, picrolonic acid or 2,4-dinitronaphthol. C. G. K.

Photochemical oxidation of hemoglobin. F. G. Lennox *Australian J. Exptl. Biol. Med. Sci.* 13, 193-6 (1935).—The presence of KI accelerated the conversion of hemoglobin to methemoglobin when irradiated with ultraviolet light. C. G. King

Studies on invertase. I A new modified method for the isolation and purification of invertase. Takekazu Kōzaki *Japan J. Gastroenterol.* 7, 125-34 (1935).—Brewers' yeast with a high invertase value was autolyzed 3 months in the presence of  $(NH_4)_2HPO_4$ . After decantation it was filtered with "Glashter". Alc. pptn. was employed twice to remove N compds. The yeast gum was next removed by adsorption on raw kaolin at a pH of 3.2 followed by elution with dil.  $NH_4OH$  after 30 min. The gum remains fixed to the kaolin and the detail of importance is the prompt removal of the enzyme from the untreated kaolin. Only 20% of the enzyme is lost after this prepn. is dialyzed through a fish bladder. The time value of this prepn. was nearly equal to that of Willstätter. The prepn. is the "abnormal" type of  $\beta$ -D-fructosidase. II Thermal analysis of the invertase action. I General treatise and the determination of the reaction heat. *Ibid.* 135-47.—The inversion heat of sucrose was detd. in a special calorimeter and found to be 4.1 cal per g mol. The inversion of sucrose follows the equation of  $dx/dt = k\sqrt{a_1 + x}$  in the earlier stage and  $b_1(a - x)/(a_1 - cx)$  afterward. III Thermal analysis of the invertase action. 2 The enzyme amount and the reaction velocity. *Ibid.* 148-53.—In these equations  $k_1$  and  $b_1$  bear a direct relation to the amt. of enzyme, but the time of the first reaction is not influenced by the concn of invertase. IV. Thermal analysis of the invertase action. 3 The sucrose concentration and the reaction velocity. *Ibid.* 154-61.— $k_1$  and  $b_1$  are little influenced by the sucrose concn, while  $a_1$  is inversely proportional except in concns of 2-7.5%. The time of the first reaction shortens with decrease in initial sucrose concn. V. Thermal analysis of the invertase action. 4 The hydrogen ion concentration and the reaction velocity. *Ibid.* 162-6.— $k_1$  and  $b_1$  are const. at pH 2.9-3.1 and then decrease at 6.2-7. The first reaction is longer at pH 6.2-7.0. VI

Thermal analysis of the invertase action. 5 The temperature and the reaction velocity. *Ibid.* 167-72.— $k_1$  and  $b_1$  have an optimum at 37° and heat inactivation starts at 35°. VII. A theoretical consideration of the invertase action. *Ibid.* 173-8. C. M. McCay

Are serum proteins affected by the time the blood stands before removing the serum from the coagulate? Jens Bing *Acta Med. Scand.* 86, 367-9 (1935).—In the detn. of serum proteins by the Henriques-Klausen method (C. A. 27, 329) the results are not affected by the time blood is allowed to stand before removing the serum. S. M.

Proteolytic activity of saliva. I Autoproteolysis of saliva. Paul Fantl and Josef Weissmann *Biochem. Z.* 281, 37-41 (1935).—Human saliva contains proteinases, derived apparently almost entirely from the cellular elements and not from the oral flora, which for the same individual show a more or less definite activity. The protein of saliva undergoes hydrolysis under the influence of these salivary proteinases without any marked alteration in the pH. II. Reaction of human saliva with proteins. *Ibid.* 42-8.—Human saliva cannot hydrolyze fibrin or egg albumin which has been denatured by boiling or pepsin, at room temp. Even the hydrolysis of fibrin, prepd. according to Fuchs (C. A. 20, 3465, 21, 427, 2001; 25, 5182), obtained with mixed saliva is shown not to be due to the salivary proteinases but to the presence in the fibrin of a fibrinolytic enzyme. S. Morgulis

Pectin enzymes. III Complete direct hydrolysis of pectic acid to d-galacturonic acid by pectolase. Felix Ehrlich, Renate Guttman and Ruth Haensel *Biochem. Z.* 281, 93-102 (1935), cf. C. A. 26, 5588.—Pectic acid is hydrolyzed almost quantitatively to d-galacturonic acid with pectolactonic acid as an intermediate product of the reaction. S. Morgulis

Optical studies on the hair pigment. H. Zwicky and F. Almsay *Biochem. Z.* 281, 103-10 (1935).—The light extinction of alk. exts. (in 5% KOH) from white, red and black hair as well as from melanomas of the horse was investigated. The black pigment, the pigment from red hair and the melanin from melanomas, could not be distinguished from each other spectroscopically. The alk. solns. of the pigments obeyed the Lambert-Beer law quite consistently and did not alter in their optical properties on long standing. S. Morgulis

Effect of cholesterol and insulin on yeast fermentation. Siegwart Hermann and Richard Neiger *Biochem. Z.* 281, 121-7 (1935).—Cholesterol sol. inhibits the fermentative activity of living yeast, but increases that of yeast autolyzates. This is probably not simply a colloidal phenomenon, since it is not reproducible either by paraffin or matrix sol. The stimulation of yeast fermentation by some insulin preps. can be much inhibited by cholesterol sol. The insulin has no effect on the fermentation by yeast autolyzates. The identity of the stimulating factor of the insulin preps. with cozymase is questioned. S. M.

Coupling of the chemical reactions in alcoholic fermentation. I. Synthesis and cleavage of adenosinetriphosphoric acid and its relation to the processes of cleavage of sugar. C. Lutwak-Mann and T. Mann *Biochem. Z.* 281, 140-56 (1935).—In muscle, through the transfer of P from adenosinetriphosphoric acid (ATP) to glycogen, the glycogenolytic process is set in motion, and the resulting adenylic acid is again phosphorylated by the intermediate products of glycogenolysis (phosphopyruvic acid) and by the phosphocreatine, thus being prevented from deamination. Subsequently, phosphocreatine is rephosphorylated from ATP and creatine. An investigation of the role of adenylic acid and of adenosinetriphosphoric acid in alc. fermentation leads to the extension of this idea of their part in the process of sugar fermentation by yeast. Analyses of Lebedev's yeast juice show that this contains only very little adenylic acid and ATP, just as autolyzed or dialyzed muscle juice. The Lebedev juice as well as fresh yeast can accumulate ATP from added adenylic acid, when glucose ferments in the presence of phosphate. The P for the phosphorylation of the adenylic acid arises from 2 sources of fermentation intermediate products. Hexosediphosphoric acid causes a phosphorylation of 80%

of added adenylic acid and this reaction is practically insensitive to F poisoning. Phosphoglyceric and phosphopyruvic acids likewise act as P donors for adenylic acid, with the formation of ATP and of pyruvic acid, and this reaction is completely inhibited by F. The ATP added to Lebedev's juice in the presence of glucose leads to its phosphorylation, thus  $\text{ATP} + \text{glucose} \rightarrow \text{hexosediphosphoric acid} + \text{adenylic acid}$ , whereas in the presence of hexosediphosphoric acid there is no dephosphorylation of the ATP, which is thus responsible for the synthesis of the first fermentation product, the fructose diphosphate. Furthermore the ATP is the essential agency for the transformation of phosphoglyceric or phosphopyruvic acid to pyruvic acid. Phosphoglyceric acid inhibits the cleavage of ATP in the Lebedev juice while free adenylic acid stimulates the transformation of the phosphoglyceric acid.

**II. Formulation of a theory** J. K. Parnas, C. Lutwak-Mann and T. Mann *Ibid* 168-74—(1) The induced reaction of fermentation  $\text{Glucose} + \text{H}_2\text{PO}_4 \rightarrow \text{hexose ester}$  is dependent upon the ATP and adenylic acid present in the maceration yeast juice  $\text{ATP} + \text{glucose} \rightarrow \text{hexosediphosphoric acid}$ , and  $\text{hexosediphosphoric acid} + \text{adenylic acid} \rightarrow \text{ATP}$ , which is unaffected by F. But, as with muscle, phosphoglyceric acid + adenylic acid  $\rightarrow \text{ATP} + \text{pyruvic acid}$ , or aldehyde +  $\text{CO}_2$ , which reaction can be inhibited by F. In yeast fermentation, as in muscle, 2 enzyme systems are found which promote the splitting of P from hexosediphosphoric acid and phosphoglyceric acid by an acceptor or the phosphorylation of sugar by a donor. (2) The fermentation induction is supposed to involve 3 distinct reactions the summation of which is expressed by  $\text{ATP} + 2 \text{ glucose} + \text{H}_2\text{PO}_4 \rightarrow 2 \text{ phosphoglyceric acids} + 2 \text{ glycerophosphoric acid} + \text{adenylic acid}$ . Two subsidiary reactions are assumed to take place. (a)  $2 \text{ phosphoglyceric acid} + \text{adenylic acid} \rightarrow 2 \text{ pyruvic acid} + \text{ATP} + 2 \text{H}_2\text{O}$ ; (b)  $\text{pyruvic acid} \rightarrow 2 \text{AcH} + 2 \text{CO}_2$ . The entire induction is, therefore, a summation of these various reactions:  $2 \text{ glucose} + 2 \text{H}_2\text{PO}_4 = 2 \text{ glycerophosphoric acid} + \text{ATP} + 2 \text{AcH} + 2 \text{CO}_2 + 2 \text{H}_2\text{O}$ , in other words, the P-transferring agent ATP is again resynthesized. The reaction is also identical with Neuberg's second form of fermentation, since the yeast phosphatase hydrolyzes the glycerophosphoric acid to glycerol. (3) The fermentation of glucose in the presence of F and of an excess  $\text{H}_2\text{PO}_4$ , since there is no splitting of P from phosphoglyceric acid, is summed up in the reaction:  $n \text{ glucose} + 2n \text{H}_2\text{PO}_4 = n \text{ phosphoglyceric acid} + n \text{ glycerophosphoric acid} + 2n \text{H}_2\text{O}$ , with the retention of the original ATP and no accumulation of hexosediphosphoric acid. (4) The fermentation in the presence of an excess of hexosediphosphoric acid is attributed to the increased velocity of the ATP  $\rightarrow$  adenylic acid reaction. (5) In the stationary condition of fermentation a reaction between hexosediphosphoric acid and AcH takes place. The hexose ester is formed by phosphorylation of glucose by ATP, which in turn is resynthesized by the phosphopyruvic intermediate product with the formation of AcH. In this way the summation of the reactions becomes  $\text{glucose} \rightarrow 2 \text{ alcohols} + 2 \text{CO}_2$ , while the 2 catalysts (ATP and AcH) are again reconstituted. (6) In the presence of an excess of  $\text{H}_2\text{PO}_4$ , it is shown that the summation of the reactions involved becomes:  $2 \text{ glucose} + 2 \text{H}_2\text{PO}_4 = 2 \text{EtOH} + 2 \text{CO}_2 + 2 \text{H}_2\text{O} + \text{hexosediphosphoric acid}$ ; in other words, the fermentation proceeds according to Harden-Young (cf. C. A. 4, 1870; 5, 110, 2822) with an accumulation of the hexose ester. (7) In the alc. fermentation, as in muscle glycolysis, the coenzyme of the internal P transfer system is ATP  $\rightleftharpoons$  adenylic acid. This in conjunction with coenzyme, the coenzyme of oxidation-reductions, and  $\text{Mg}^{++}$  constitutes the coenzyme of fermentation. S. M.

Further studies on the optical activity of hemoglobin and some hemoglobin derivatives with special reference to the sulfur content. István Simonovits and Georg Balassa. *Biochem. Z.* 281, 186-97 (1935); cf. C. A. 25, 3369—Horse hemoglobin is found in 2 types with 0.43 and 0.49% S; beef hemoglobin contains 0.51% S. One % horse CO-hemoglobin with 0.43% S has  $[\alpha]_{578}^{20} = +11.6^\circ$  and with 0.49% S,  $+12.3^\circ$ . The values for 1% horse oxy-

hemoglobin are about  $+14.4^\circ$  and  $+12.4^\circ$ , resp. With repeated crystal the specific rotation decreases. Once crystal. 1% beef CO-hemoglobin has a sp. rotation of  $+7.9^\circ$ . Horse hemoglobin in 0.5% aq. soln. reduced with  $\text{Na}_2\text{S}_2\text{O}_4$  has an av. sp. rotation of about  $+28.30$ . The sp. rotation of horse CO-hemoglobin in 0.1%  $\text{NaHCO}_3$  is independent of the concn of the pigment. S. M.

**Polysaccharide proteins. VI The state of glycogen in muscle.** 2. Edmund M. Mystkowski, Arnold Stiller and Aleksandra Zysman *Biochem. Z.* 281, 231-7 (1935); cf. C. A. 29, 8020—The press juice of mammalian muscle contains in addition to the characteristic protein fractions also small amts of glycogen. In frog muscles as much as 17% of the total glycogen can be found in the press juice. It is argued that this indicates the presence of a glycogen-myosin and possibly also of a glycogen-globulin symplex in the muscle tissue. These symplexes are difficult to split, and 4-10% of the glycogen seems to be irreversibly bound, since it cannot be washed out with  $\text{H}_2\text{O}$ . They are not easily attacked by salivary amylase even at the opt. pH, although the glycogen hydrolyzed from the protein by KOH is easily so digested. S. Morgulis

**Dilatometric studies in the enzymic hydrolysis of polysaccharides. III Hydrolysis of starch, amylose and amylopectin by takadiastase.** H. B. Sreerangachar. *Proc. Indian Acad. Sci.* 2B, 333-41 (1935), cf. C. A. 29, 2980—Lutner's sol starch, potato starch and amylopectin were used with a two-bulb dilatometer. The amylopectin was prep'd from potato starch by the methods of Nanji and Ling and of Eckert and Marzin (C. A. 26, 3135). The depression in the dilatometer in cu. mm. was linearly proportional to the release of maltose and to the fall in rotation. The same depression per millimol of maltose was found for all the starch and amylopectin preps, with an av. value of 4.8. J. J. Willaman

**Bile acids (Schenck) 10 Synthetic expts on the constitution of the bile pigment (Siedel) 10**

**Soluble lactalbumin.** Geo. E. Flanigan and Geo. C. Supplee (to Borden Co.) U. S. 2,023,014, Dec. 3. In the prep'n. of sol. lactalbumin, whey is brought to a pH of about 4.35-4.85, heated to above  $70^\circ$ , pptd. lactalbumin is sep'd. and agitated with a soln. such as one of an alkali metal or  $\text{NH}_4$  bicarbonate, carbonate or hydroxide, the pH of the resulting fluid is adjusted to about 6.4-6.99 and it is evap'd to dryness substantially instantaneously.

## B—METHODS AND APPARATUS

STANLEY R. BENEDICT

**Determination of nitrogen and carbon in small amounts of plankton (in sea water).** Theodor v. Brand. *Biol. Bull.* 69, 221-32 (1935)—Fifty cc. of sea water is treated with 1 cc. 2% KOH in a centrifuge tube and centrifuged at 3400 r. p. m. for 10 min. The liquid is decanted, the ppt. dissolved in 3%  $\text{H}_2\text{SO}_4$  and transferred to a 25-cc. centrifuge tube, treated dropwise with 2% KOH until just turbid. The mixt. is again centrifuged 10 min. The supernatant liquid is transferred to another tube, more KOH added until the mixt. is again just turbid and again centrifuged. The first of the latter tubes contains the plankton with inorg. hydroxides; the second is used as a blank for adsorbed C or N. N analyses are made by the method of Krogh and Keys (C. A. 28, 6656). In the range 0.5-5%, the accuracy is about 10%. A modification of the method of Krogh and Rehberg (C. A. 24, 5667) is used for C. Amts. of the order of 10 to 100  $\gamma$  C can be det'd. with an accuracy of  $\pm 3\gamma$ . L. W. Elder

**The estimation of minute quantities of atabrin in the blood.** R. N. Chopra and A. C. Roy. *Indian Med. Gaz.* 70, 504-5 (1935)—Oxalated blood contn. atabrin is soaked into strips of filter paper, dried 1 hr. at  $50^\circ$ , ext'd. with  $\text{Et}_2\text{O}$  for 2 hrs. and the ext. evap'd. The residue is dissolved in 4 cc. 0.1 N HCl by heating and shaking, the mixt. cooled and filtered and a 3-cc. aliquot taken for colorimetric comparison with standards prep'd. from a 0.01 mg./cc. soln. of atabrin hydrochloride. The unknown and standards are treated with 0.1 cc. 20% NaOH and 1

cc. amyl alc. and thoroughly mixed. The yellow color of the amyl alc. layer is matched with that of the standards to det. the atherin content. Data are presented showing recoveries of 0.005-0.025 mg. to within 0.003 mg.

L. W. Elder

Embsden's cholesterol assay method. Sadaharu Miyamori. *Nagoya J. Med. Sci.* 8, 135-7 (1935) (in German).—A modification of the method of Embsden and Lawaczek (C. A. 17, 2448). Two g. of tissue or 2 cc. of blood is saponified in 20 cc. of 25% KOH for 2 hrs. under reflux on a water bath. The mixt. is extd. 6 times by shaking 5 min. with 50-cc. portions of  $\text{Et}_2\text{O}$ . Each fraction is washed with 10 cc.  $\text{H}_2\text{O}$  by brief shaking. The  $\text{Et}_2\text{O}$  is distd. from the combined fractions, the residue dried at 60-80°, taken up in  $\text{CHCl}_3$  and distd. to 10 cc. A 5-cc. aliquot is treated with 2 cc.  $\text{Ac}_2\text{O}$  and 0.1 cc. concd.  $\text{H}_2\text{SO}_4$  and after 15 min. in the dark is compared colorimetrically with a standard made up from 10 cc. of 0.1% cholesterol soln. 4 cc.  $\text{Ac}_2\text{O}$  and 0.2 cc.  $\text{H}_2\text{SO}_4$  under similar conditions. Results are slightly higher than by the digitonin method.

L. W. Elder

Anticoagulant properties of pyrogenation products of citric acid. Auguste Lumière and Suzanne Sonnerly. *Compt. rend. soc. biol.* 120, 213-14 (1935).—The min. quantities required to prevent coagulation of 100 g. rabbit blood were, in g., tri-Na citrate 0.3-0.4, di-Na citrate 0.4-0.5, mono-Na citrate 1.0, di-Na itaconate 1.0, tri-Na aconitate 1.0, di-Na citraconate 2.25, di-Na mesaconate 2.5, K or Li citrate 0.3, Pb citrate 0.5-1.0 and Co or Mg citrate 1.25.

L. E. Gilson

Histochemical determination of potassium during the development of the oocytes of the hen. V. D. Marza and L. T. Chocua. *Compt. rend. soc. biol.* 120, 345-7 (1935), cf. C. A. 29, 1117.—The K contents of different parts of the cells were detd. by the method previously described.

L. E. Gilson

Chemistry of the panoptic staining reaction applied to the detection of adrenaline in tissues. T. Pawlikowski and T. Siebodziński. *Compt. rend. soc. biol.* 120, 465-6 (1935).—Adrenaline is detected by staining first by the Heule  $\text{CrO}_3$  method and then by the Pappenheim "panoptic" method.

L. E. Gilson

Color reaction exhibited by adrenaline in mammalian erythrocytes. Thadée Pawlikowski. *Compt. rend. soc. biol.* 120, 467-9 (1935).—Adrenaline was detected in red corpuscles by a modification of the Heule staining method. Presumably it is adsorbed from the plasma. Dog erythrocytes contained more than those of man, cat, guinea pig, sheep or rat.

L. E. Gilson

Investigation of the reliability of the blood alcohol determination of Widmark. Herbert Elbel. *Dent. Z. ges. gericht. Med.* 25, 124-9 (1935); cf. Kanitz, C. A. 29, 6263.—Vein puncture is the best method of drawing blood for alc. detn. The factor 1.2 can be used to calc. whole blood values from serum values.

Frances Krasnow

Respiration apparatus for small animals. Sándor Belak and Andor Illényi. *Biochem. Z.* 281, 27-9, *Magyar Orvosi Arch.* 36, 207-9 (1935).—The app. consists of a closed glass jar suspended in a thermostat. The animal is placed in a small wire cage about 8 cm. above the bottom where there is a layer of  $\text{Ba(OH)}_2$  soln. As the  $\text{O}_2$  is being used up new  $\text{Ba(OH)}_2$  is admitted from a buret until the manometer registers normal pressure. The vol. of  $\text{O}_2$  used up at standard pressure and temp. is calcd. from these data. The  $\text{CO}_2$  is detd. by analysis of an aliquot of the total  $\text{Ba(OH)}_2$  soln. To facilitate the absorption of  $\text{CO}_2$  the entire app. is agitated by a constant back and forth horizontal motion.

S. Morgulis

The determination of the sum of glycine and serine. S. Rapoport. *Biochem. Z.* 281, 30-6 (1935).—Glycine is decarboxylated with  $\text{NaNO}_2$  to glycolic acid, which is then oxidized with  $\text{KMnO}_4$  to  $\text{H}_2\text{C}_2\text{O}_4$  and detd. as such. Only the dicarboxylic acids and serine interfere in this detn. but the former can be quantitatively removed. Attempts have been made to det. the serine separately but the final results of this will be published later. The sum of glycine and serine (calcd. as glycine) for fibron is 40.23, for elastin 31.82, gelatin 26.45, casein 8.18, sericin 7.91 and edestin

1 4.42%. Preliminary detns. on sericin, with the colorimetric procedure, shows a serine content of 8.71% and of glycine of 1.69%.

S. Morgulis

2 Fermentation of carbohydrates, a means for the observation of cellular physiological processes. Günther Malyoth and Eckart Sommerfeld. *Biochem. Z.* 281, 49-79 (1935).—A modified Krogh macrorespirometer that detn.  $\text{CO}_2$  with an accuracy of  $\pm 2\%$  is used. Fermentation expts. with similar quantities of glucose and yeast give likewise consistent results with a max. error of 4%, of which 2% is due to the instrument. The  $\text{CO}_2$  tension developing during fermentation depends primarily upon the vol. of  $\text{H}_2\text{O}$  and the amt. of sugar used. Addn. of buffers contg.  $\text{KH}_2\text{PO}_4$  or of  $\text{K}_2\text{H}_2\text{P}_2\text{O}_7$  alone stimulates the fermentation, especially with small amts. of yeast (<40 mg.) or with washed yeast. The "fermentation deficit" is due to the utilization of sugar in synthetic processes of the yeast as well as in by-reactions of the fermentative process itself; also to the respiration of the cells, whereby the  $\text{O}_2$  consumed tends to mask the  $\text{CO}_2$  developed. These factors disappear when the fermentation is carried out with cell free yeast juice and the glucose is fermented 100%, i. e., without any deficit. The stimulation of the fermentation by  $\text{KH}_2\text{PO}_4$  is greater with the cell-free yeast juice than with the living yeast.

S. Morgulis

3 Benzene determination with the Pulfrich step-photometer. Ladislav v. Szécsényi-Nagy. *Biochem. Z.* 281, 178-80 (1935).—Acidify the material (blood, urine, org. matter) with  $\text{H}_2\text{SO}_4$ , dil. with 200 cc.  $\text{H}_2\text{O}$  and distil in a special app. at 90° for 20 min. Increase the flame and distil at boiling temp. Then add another 50 cc.  $\text{H}_2\text{O}$  and distil at boiling temp. for 10 min. to drive off traces of benzene, and wash the cooler with 5 cc. saturating mixt. The distillate is collected in a cooled mixt. of 20 cc. fuming  $\text{HNO}_3$  and 10 cc.  $\text{H}_2\text{SO}_4$ . Dil. with about an equal vol.  $\text{H}_2\text{O}$  and make alk. with  $\text{NaOH}$ . Ext. the dinitro compd. with ether, evap. and dissolve the residue in 20 cc. acetone. To 10 cc. of the acetone soln. add 2 cc. 33%  $\text{NaOH}$ , stir vigorously 5 min. and after 30 min. exam. the violet-colored soln. in the step-photometer. The max. absorption of this soln. is obtained with filter S 57. Up to a concn. of 3.2 mg. % the color obeys the Lambert-Beer law. The chief loss in this detn. is during the distn. and the app. should be carefully tested.

S. Morgulis

4 Recent methods of clinical value in biochemical analysis. G. A. Harrison. *Pharm. J.* 135, 183-5 (1935).—Recent tests in urinary exarans. and blood analyses are reviewed, 26 references (cf. Godfried, C. A. 29, 2565<sup>9</sup>, 6266<sup>9</sup>, 6293<sup>9</sup>; Bloem, C. A. 27, 391<sup>2</sup>; Harris and Ray, C. A. 29, 3005<sup>9</sup>; Kay, C. A. 23, 1054, 24, 182; Roberts, C. A. 24, 3290<sup>9</sup>).

S. Waldbott

5 Device for the rapid detn. of certain gases or vapors in the atm. (or in viscera) (Kohn-Abrest) 7. App. for drying and grinding materials such as blood (U. S. pat. 2,023,247) 1.

## C-BACTERIOLOGY

LAWRENCE N. JAMES

6 An unnoted hemolytic streptococcus associated with milk products. James M. Sherman and Helen Upton Wing. *J. Dairy Sci.* 18, 657-60 (1935).—*Streptococcus hemiothermophilus* (n. sp.) is suggested as the name for an organism believed to represent a new species. It differs from pathogenic streptococci in its higher max. temp. of growth, lower min. temp. of growth, higher thermal death point, a more acid limiting  $\text{pH}$  of growth, its ability to hydrolyze Na hippurate and its inability to ferment sucrose.

Philip D. Adams

7 The action of water and physicochemical factors on the tubercle bacillus. I. Veissfeller and K. M. Dvolaitzskaya-Barucheva. *Beitr. Klin. Tuberk.* 80, 151-9 (1935).—Tubercle bacilli die rapidly in water, the no. of living bacilli being reduced 1 to 100 after 10 to 20 days. Various strains differ in their resistance in this respect so that old lab. strains may even reveal multiplication in water. This "water treatment" may result in variation forms, actinomycetes forms, diphtheroides or micrococci like

forms. These forms occur with slow growth and in dissoen. expts. Const. results in dissoen. forms can be obtained in water. A  $pH$  of 8.0 and trivalent metals give the best results. Tubercle bacilli suspended in  $N/204$   $FeCl_3$  are rapidly ( $1/2$  to 3 hrs.) transformed into non-acid-fast forms which are stable and cannot be transformed back to tubercle bacilli.

H. J. Corper

Some methods of investigation of indole in bacteria cultures. Fgidio Tosatti *Biochim. terap. sper.* 22, 286-97(1935), cf *C A* 29, 6621<sup>1</sup>—The method of Jolles (*C A* 28, 4445<sup>1</sup>) in which *o*-nitrobenzaldehyde is used is more sp. and sensitive than the methods of Salkowski (cf *C A* 14, 1550), Legal, Eleig and Siere, but is less sp. and sensitive than Ehrlich's method (cf. *C A* 5, 2254). Jolles' method is preferable because color of the products of condensation is most stable. A E Meyer

Sulfur bacteria from a warm sulfur spring in Santa Rosalia, Mexico Oskar Baudisch *Svensk Kem. Tids.* 47, 191-204(1935)(in German)—A microorganism from a sulfur spring is cultured and the compn of its natural and artificial medium studied. A R Rose

A study of the flora of human, gaseous gangrene. A new anaerobic organism A Sordelli and S Soriano *Folia biol.* 1934, 197, *Anales asoc. quim. Argentina* 23, 10B—The organism described differs from *Clostridium sporogenes*, *Cl. centrosporogenes*, *Cl. tyrosinogenes* and *Cl. oedemaloides*. It has not been positively identified as *Cl. parapsorogenes*. For comparative study of the various species, and for literature study, it should be considered as a new species. E. M. Symmes

## D—BOTANY

THOMAS G. PHILLIPS

The reaction of the culture medium and the activity of ordinary and preformed mycelia of an *aspergillus* Virgilio Boleato *Ind. sacchar. ital* 28, 454-9(1935).—Citric acid is formed in a medium with a reaction of  $pH$  3.4 or less, and gluconic acid is produced when the  $pH$  is above 3.4, while the mycelia are under development. The sp. acid forming activity of a transplanted mycelium is influenced by the reaction of the original medium, and more markedly in the case of citric acid production. This is explained by a stabilizing influence on the enzymes involved. Great variations in the  $pH$  of the media result in weakening the mold. L. Cusachs

Does the radon content in the air in soil influence seed development? Walter Kosmath and Volkmar Hartmar. *Protoplasma* 24, 8-13(1935).—These expts. show that the germination of *Vicia faba equina* in quartz sand was not influenced by a radon content in the soil air of 1000  $\times 10^{-12}$  curies/cc. F. L. Dunlap

Growth substances of microorganisms in embryonic tissues and in exudation sap. Jonas Dagys *Protoplasma* 24, 14-91(1935).—Resting seeds of *Zea mays* and *Triticum vulgare* contain in the embryo and scutellum a large amt. of a growth substance which aids the cell increase of yeast and the increase in dry substance of *Aspergillus niger*. In the wheat endosperm this growth substance is present in considerable amts. but only in traces in maize endosperm. In the swelling of seeds, there is first of all a transport of the growth substance toward the embryo, resulting in the case of wheat seeds of an emptying of the endosperm of growth substance. During germination there is an active new formation of growth substance in the embryo and scutellum. In wheat seeds which were 21 years old and which were no longer capable of germination, no decrease in growth substance could be noted. In the buds of *Betula verrucosa* the growth substances were found in greatest amts. in late summer; but in going over into the winter resting period the amt. of growth substance in the buds gradually decreased. The growth substance minimum was reached in February. In forced buds there is again a manifold increase of this growth substance. The greatest content in growth substance was found in young and rapidly growing birch leaves. In the summer leaves this sinks to about a half. In the falling autumn leaves there is a considerable increase in growth substance. The

active birch cambium contains the growth substance in rich amts., the secondary cortex very little. The leaves are to be looked on as the place of origin of this growth hormone. The growth hormone is sol. in 57% alc. and in 90%  $CH_3OH$ , insol in  $Et_2O$ , decomposed by long boiling with  $H_2O_2$ , adsorbed by animal charcoal but not by fuller's earth, heat-stable at  $135^\circ$ , and stable to acids and alkalis. It is thus closely related to bios. The spring exudation sap from the birch contains no growth hormone (bios) which acts on yeast, but it does act on *Aspergillus* and is closely related to the chemically produced growth substance of Group B of Nielsen and Hartelius (*C A* 27, 1019). In a similar fashion an active and probably identical growth substance can be made by autoclaving a fructose-malic acid soln., when the soln. is of the same concn as birch sap. The ash of birch sap contains large amts. of a co-growth substance. Birch sap growth substance (growth substance B) differs from bios in its complete resistance to oxidation by  $H_2O_2$  and small adsorption by animal charcoal. On account of its destructibility by heating with  $H_2O_2$ , bios does not belong to the growth substance group B. Exudation sap from *Cucurbita pepo* contains neither bios nor growth substance B. The ash from the exudation sap of *Cucurbita* contains no co-growth substance. One hundred and forty-seven references. F. L. Dunlap

The sensitivity loss of Nitella in alkaline solution. Karl Umrat. *Protoplasma* 24, 101-7(1935). F. L. D

Tissue physiology of roots. Analysis and methods of vital staining of plant cells with neutral red. Siegfried Strüger. *Protoplasma* 24, 108-27(1935).—Neutral red in acidified distd.  $H_2O$  colors first of all only the cell membrane of root hairs of *Trisanea bogotensis*. Neutral red in weakly alk. cond.  $H_2O$  stains immediately the cell juice in the well-known fashion. If the soln. is more acid than  $pH$  6.4, there is a pure membrane coloring of the root hairs. At  $pH$  6.4 only certain membrane portions of root hairs are stained. Above 6.4 and toward the neutral point and beyond this, there follows a storage of the color in the vacuoles. Membrane staining does not occur. The H-ion concn of the color soln. and that of the medium in which the hair is washed and investigated are of equal significance in the color distribution in vital staining of root hairs. If the staining is carried on in acid soln. the cell membrane is first stained and if the roots are then placed in a colorless soln. which is weakly alk., there is a rapid transference of the color adsorbed by the membrane to the vacuoles. This transference from the hair ends and base is more rapid than from the middle portions. Young hairs show a more rapid carrying out of this process than the older ones. This action is reversible. Hairs colored in alk. soln. show at first only a diffuse and drop-like storage in the cell juice. If the root is then placed in acid soln. (uncolored buffered soln. or distd.  $H_2O$ ) the color begins to leave the cell juice and go to the membrane. The colored drops in the cell juice disappear in a short time almost completely. These expts. on the reversibility of the color distribution can be repeated a number of times with the same root without affecting its vitality. Also the epidermal cells show in their dependence on the H-ion concn a strongly regular distribution of the dyestuff. The epidermal cells of the meristem zone store the dyestuff only in the plasma and cell juice when in an approx. neutral medium. In the extension zone the color is stored in the radial and inner membranes. In the permanent zone there is a pure membrane coloring of the outer walls, the plasma and cell juice are not affected. The membranes of the hair base which border immediately on the root tissue show a fundamentally different action. If color is stored in the vacuoles of the root hairs, it begins to wander from them into the basal membranes. From there it can be further taken up by the adjacent root cells. With the aid of vital staining in accurately defined media it is possible to show the inhomogeneity of various cell regions. Bethe's reaction theory is not applicable to the whole cell. It can be applied only to certain cell parts. F. L. Dunlap

Absolute viscosity measurements by means of the Brownian molecular movement. VIII. Cell juice viscosity and its dependability on temperature and light. J. Pekarek,

*Protoplasma* 24, 128-73(1935); cf. C. A. 28, 2971<sup>1</sup>—The expts. were conducted on the cell juices of *Vaucheria sessilis*, *Forma repens*, *Leptobryum pariforme* and *Rheum palmatum*. The viscosities all decreased in light with increasing temps up to 33°. Beyond this critical temp the viscosities all increased to a max., in the case of *L. pariforme* at 48° and for *V. sessilis* and *R. palmatum* at 39°. By further increase in temp these viscosities decrease again. With all the cell juices at 33°, the viscosity is approx. 1.9 times that of H<sub>2</sub>O at the same temp. Above 33° the ratio of the viscosity of water to cell juice increases to a max., either at 39° (*Vaucheria*, *Rheum*) or at 48° (*Leptobryum*). The viscosity of the cell juice is in the case of *Vaucheria* approximately 2.5 times, with *Rheum* 2.7 times and with *Leptobryum* 3.3 times greater than the viscosity of H<sub>2</sub>O at the same temp. The relative viscosity of the cell juices, referred to H<sub>2</sub>O of the same temp., does not change up to 33°, increases between 33° and 39°, respectively 48° and at still higher temps it has a const. value. The viscosity of the cell juice of the chlorophyll-free petiole epidermal cells of *R. palmatum* within the temp. range of 18-48° is unaffected by either light or darkness, its viscosity is independent of any light factor. The viscosities of the chlorophyll-contg. cell juices of *V. sessilis* and *L. pariforme* differ in a characteristic fashion when subjected to light and darkness, at different temps. In the dark, the cell juice viscosity of protonema cells of *L. pariforme* up to 24°, and the thallus threads of *V. sessilis* up to 27°, show a decrease of approx. 20%. After these critical temps it increases and in the case of *Vaucheria* at 33° it is 5%, and in the case of *Leptobryum* between 33° and 39° it is 7-10% greater than the value which characterizes the cells in the light. Above 33° (*Vaucheria*) and 39° (*Leptobryum*) the cell juice viscosity decreases with increase in temp., the relative viscosity is furthermore const., in the case of *Vaucheria* approximately and with *Leptobryum* approx. 2.2-2.7 greater than that of H<sub>2</sub>O at the same temp. Above 39° (*Vaucheria*) and 48° (*Leptobryum*) the viscosities of the cells in the dark are about 20% less than in the light. In all cases, both in the light and in the dark, changes in viscosity brought about by temp. changes are reversible. Likewise changes in viscosity in cell juices by light and darkness are reversible. The cause of these changes in viscosity induced by temp. is to be sought in the degree of dispersion of the cell-juice colloids. The changes in viscosity in the chlorophyll-contg. cells of *V. sessilis* and *L. pariforme* through light and darkness are in all probability related to CO<sub>2</sub> assimilation.

F. L. Dunlap  
The problem of bios. Haehn. *Wochschr. Brau.* 52, 95-6(1935)—General discussion. S. Józsa  
Initiation of roots by growth substance paste. F. Laubach and O. Fischrich. *Ber. deut. botan. Ges.* 53, 528-39(1935)—Expts. are described in connection with root initiation in *Coleus*, *Vicia faba* var. *equina* and *Solanum lycopersicum* by  $\beta$ -indolylacetic acid.

Lawrence P. Miller  
Measuring the oxygen consumption of yeast by a new principle. Georg Neumann. *Biochem. Z.* 281, 181-3 (1935).—A suspension of yeast in an oxyhemoglobin soln. of definite vol. is studied spectrophotometrically. The amt. of the oxyhemoglobin being known, the O consumption in a given time is easily calcd., since each g. corresponds to 1.34 cc. O. The physically absorbed O is detd. from the available data (pressure, temp. and absorption coeff.). Hemoglobin solns. of 0.005-2.0% concn. were employed and the cu. mm. O used up by 1 mg. yeast per hr. was detd.

S. Morhul  
The metabolism of the colorless alga, *Prototheca zopfii* Krüger. H. Albert Barker. *J. Cellular Comp. Physiol.* 7, 73-93(1935)—*Prototheca zopfii* develops only in the presence of complex organic materials (as in yeast autolyzate). It utilizes NH<sub>3</sub> as well as more complex nitrogenous substances. It utilizes fatty acids, monosaccharides and alcohol but apparently no keto, hydroxy or dibasic acids. Glucose aerobically yields cell material and CO<sub>2</sub>; anaerobically it is quantitatively fermented to L-lactic acid.

T. H. Rider

1 The chemistry of grass crops. Artturi I. Virtanen. *Chemistry & Industry* 1935, 1015-20—A review of V's work on N fixation in legumes, changes in carotene and vitamin C content in plants during growth, the use of acids to preserve ensilage and the effect of feed on the vitamin content of milk. A method for diagnosing pregnancy is described. L. R. Rushton

2 Effect of frost on wheat at progressive stages of maturity. I. Physical characteristics of the kernels. R. Newton and A. G. McCalla. *Can. J. Research* 10, 414-29(1934)—Susceptibility of wheat to superficial injury by frost continued generally over nearly the whole maturation period. Marquis wheat is apparently more susceptible than Garnet, Reward and Red Bobs, to degradation by frost during the maturation period. Grade is more sensitive to frost than are chemical compn. and baking quality. Classification of the kernels of Marquis, in 1930, into vitreous, starchy, immature, green, bran frosted and heavily frosted, confirmed the earlier conclusion (cf. C. A. 26, 2219) that only the sound class (vitreous and starchy) has enough relation to grade to be useful as a grading factor. The germination of Garnet was improved by moderate freezing.

J. W. Shipley  
The course of stone cell formation in pear fruits. Wm. W. Smith. *Plant Physiol.* 10, 587-611(1935); cf. Crist and Batjer, C. A. 26, 1980—No decrease occurred in the abs. amts. of lignocellulose of either Kieffer or Bartlett pears sampled periodically throughout the growing season. But, owing to the large increase in materials sol. in EtOH, the amts. of lignocellulose calcd. on a percentage basis decreased rapidly. The alleged conversion with advancing maturity of part of the lignocellulose into reducing substances is consequently questioned. The Shaffer-Hartmann method (C. A. 15, 1327) gave, with the ext. exam., values greater than the amts. of Cu reduced (cf. Phillips, C. A. 26, 5123). Storage studies at 33°F. with Kieffer fruits were also made. These showed that the percentage of hemicelluloses decreased more than that of reducing sugars, lignins or pectins. Thirty-seven references. Walter Thomas

Osmotic pressure and water content of prairie plants. Lawrence A. Stoddard. *Plant Physiol.* 10, 661-80(1935)—Colloidal imbibition is capable of exerting much greater force than is osmotic pressure and is probably the more important factor acting to secure water from a dry soil (cf. Shull, *Bot. Gaz.* 62, 1, 1916). Soil moisture and humidity were found to be the major factors affecting the osmotic pressure of plant tissues. Walter Thomas

Boron-deficiency disease of beets. J. E. Koutla and G. H. Coons. *Facts about Sugar* 30, 373-6(1935)—The symptoms of heart and dry rot, previously reported only in Europe, have been found in beets grown in certain areas in Michigan and Ohio, and are described and illustrated. The symptoms disappear upon the addn. to sand cultures, of small quantities of boric acid or borax. P. W. Z.

Research and the rubber industry [botanical problems] (Eaton) 30. Glucoside of the flavone of the white flower (Nakaoki) 10.

## E—NUTRITION

PHILIP B. HAWK

Studies of the vitamin B complex. I. Effect of fats and of the individual esters upon the vitamin B requirement of rats. W. D. Salmon and J. G. Goodman. *Ala. Agr. Expt. Sta., 45th Annual Rept.* 21-2(1934); cf. C. A. 28, 7309<sup>1</sup>—When the rat's diet contained glycerol or Et esters equiv. to 23% of the fatty acid, the acids ranked in the order of decreasing effectiveness in their vitamin B-sparing action as follows: caprylic, caproic, heptylic, lauric, myristic, nonylic, undecylic and oleic. Valeric, propionic and acetic acids had very low efficiencies; palmitic and stearic acids had no demonstrable sparing effect; this was probably due in part to their high m. ps. and poor absorption. Butyric glyceride was toxic to rats even when the diet contained adequate vitamin B. Spastic cases of vitamin B deficiency were cured by the administration of caprylic, caproic, heptylic or lauric acids without

any vitamin B. Bakers' yeast after autoclaving at 17-20 lb. pressure for 8 hrs. still contained traces of vitamin B. Such a product should not be given *ad libitum* to rats. The onset of vitamin B deficiency is not hastened by the substitution of 23% of lactic acid as the glyceride for sucrose in the diet. II Quantity of glycogen in the vitamin B-deficient rat and its ability to deplete this glycogen during starvation G A Schrader. *Ibid.* 21-2, cf. C A 28, 7306.—There is no large accumulation of glycogen in vitamin B-deficient rats as compared with rats receiving adequate amounts. The rate of depletion of glycogen in both sets of rats was approx. the same. III Ability of the vitamin B-deficient rat to utilize D-lactic acid *Ibid.* 22.—A much poorer utilization of lactic acid occurred than for D-glucose. There was no indication of a breakdown in the lactic acid metabolism of the vitamin B-deficient rats. The use of 0.3-0.5 g. daily of lactic acid in the diet did not hasten the onset of vitamin B symptoms. IV Apparent ability of the vitamin B-deficient rat to transform carbohydrate into fat *Ibid.* 22-3.—Respiratory quotients averaging 1.26 were found for all vitamin B-deficient rats on the high-carbohydrate diet, irrespective of whether they received no vitamin B, or vitamin B with either a limited or an unlimited energy intake. At no time was a R Q of 1.00 secured from the rats on the high-fat diets, in fact, after the 1st week the R Q averaged only 0.8, indicating that the energy was mainly secured from fat. Conclusion.—Rats on a high-carbohydrate diet can convert carbohydrate into fat. C. R. Fellers

Cane molasses as a feed for dairy cows L. A. Henke. *Hawaii Agr. Expt. Sta., Bull.* 73, 1-18(1934).—A ration containing 25% cane molasses was compared with a similar ration without molasses. The percentages of crude protein and digestible nutrients in both rations were the same. The wt. gains, milk and butter-fat production as well as the fat content of the milk were almost identical in the 2 rations. C. R. Fellers

Sunflower silage for milk production. F. W. Atkeson. *Idaho Agr. Expt. Sta., Bull.* 215, 2-8(1935).—Sunflower silage contains approx. 75.4% water, 2.3% ash, 2.4% crude protein, 7.6% crude fiber, 11% N-free, and 1.2% fat. The sunflower silage was equal to corn silage in wt. gain, milk and butter-fat production when fed to dairy cows. C. R. Fellers

Utilization of energy-producing nutrient and protein as affected by individual nutrient deficiencies III. Effects of the plane of protein intake. E. B. Forbes, R. W. Swift, Alex. Black and O. J. Kahlenberg. *J. Nutrition* 10, 461-79(1935); cf. C. A. 28, 7383.—The effects of 4 planes of protein intake, 10, 15, 20 and 25%, were studied by means of 2, ten-weeks' growth, metabolism and body analysis expts. on 48 rats each. The effects of the increasing protein content of the eucaloric diets were: increase in gain in body wt. at decreased cost in terms of dry matter of food; increase in efficiency of digestion and retention of protein and of energy-producing nutrient; increase in urinary N at an increasing rate, and increase in protein of the body at a decreasing rate; increase in energy of the urine coincident with decrease in the energy of the feces; the metabolizable energy therefore remaining practically constant; diminished efficiency in the utilization of food N; no regular change in amt. of fat gained, but usually a decrease in fat gained in proportion to protein gained. Increases in the protein of eucaloric diets made no change in the basal heat production per unit of computed surface area, but diminished the total heat production of the animals. C. R. Fellers

Pathological skin changes in the tail of the albino rat on a diet deficient in vitamin G (B<sub>2</sub>). Susan Gower Smith and Douglas H. Sprunt. *J. Nutrition* 10, 481-92(1935).—Rats fed the Bourquin and Sherman vitamin G-deficient diet for 90 days or longer showed varying degrees of atrophy of the sebaceous glands and thinning of the epithelium in cross sections of the tail. The fat normally present around the sebaceous glands was replaced by connective tissue. There was no cellular infiltration. The above diet supplemented with autoclaved yeast produced rats with normal sebaceous glands and epithelium. These

1 pathol. changes were corrected by the addn. of autoclaved yeast to the diet. C. R. Fellers

Human iodine balance. Versa V. Cole and Geo. M. Curtiss. *J. Nutrition* 10, 493-506(1935).—Iodine balance was studied in 3-day periods for 15 days on 3 subjects, 9 on a fourth and 6 on a fifth. The combined urine and stool I may account for less than 50% of the I intake. There was a fairly uniform output of I for an individual on a monotonous diet. There is an indication of increased loss in fecal I in hyperthyroidism. The fecal I was higher than usually reported by previous investigators. C. R. Fellers

Utilization of inulin for growth by the young white rat. Adelaide Bendaña and Howard B. Lewis. *J. Nutrition* 10, 507-15(1935).—When young white rats were maintained on a limited caloric diet, the addn. to the diet of inulin (from chicory), sucrose or fructose as a supplement produced a definite growth response. The response from inulin was inferior to that obtained from sucrose or fructose. As a sole source of energy in the white rat inulin was not effectively utilized. There was marked fermentation of the polysaccharide in the intestine with distention which resulted in death in a few days. C. R. F.

Effects of cereal diets on the composition of the body fat of the rat. Harold S. Olcott, Wm. E. Anderson and Lafayette B. Mendel. *J. Nutrition* 10, 517-23(1935).—Where corn, barley, wheat or oats furnished 82% of the total energy value of individual rations, and corn and oat oil supplied 8 and 10%, resp., of the total cal., the body fat produced by the corn- and oats-fed rats differed slightly in degree of unsatn. as measured by I no., e g., 83 and 79, resp. The oil of wheat and barley constituting 1.7 and 2.0% of the resp. grains supplied only 3 and 4% of the total energy value of the resp. wheat and barley diets. Body fat yielded by animals fed wheat and barley rations was less unsatd., e g., 70 and 71, than fat produced on either corn or oats diets and was similar in character to the fat from rats fed fat-free diets. The unsatd. acid content of the body fats from cereal-fed rats ranged from 68.5% for fat from those fed the barley diet to 75% for fat of rats fed on the corn ration. C. R. Fellers

Influence of diet on the glucose tolerance of the dog. Esther M. Greisheimer and F. W. Hoffbauer. *J. Nutrition* 10, 525-34(1935); cf. C. A. 29, 2114.—The effects of different dietary regimes on the carbohydrate tolerance of each of 8 dogs were studied. Dogs require approx. 3 g. of glucose per kg. of body wt., though 1 g. per kg. is sufficient for man. High-protein diet gives a decreased glucose tolerance. This decrease is even more marked after a high-fat diet. The higher the protein level, the more marked the decreased tolerance became. However, beyond 24.1% no further effect was noticeable. With both 55 and 80.1% of fat in the diet, definite decreases in tolerance were noted. Great variation among the animals was found. The differences in glucose tolerance are not due to differences in the rate of absorption. C. R. Fellers

Adequacy of simplified rations for the complete life cycle of the chick. Albert G. Hogan, Robert V. Boucher and Harry L. Kempster. *J. Nutrition* 10, 535-47(1935); cf. C. A. 28, 8097.—Four successive generations of chicks were reared on simplified rations. While growth was rapid, the fertility of the males was low. The females were normal until they attained maturity, and their egg records compared favorably with those of normally fed birds. After periods of extensive egg production, fowl mortality was high. Since earlier investigations indicate low fertility is common to all lab.-raised birds, it seems entirely possible that the present synthetic diet is complete. One of the promising rations was No. 2159 and consisted of casein 35, cornstarch 30.5, Cellophane 3, lard 11, Osborne and Mendel salt mixt. 4, wheat germ oil 4, acid hydrolyzed yeast 4, liver ext. 6, tikiiki 2 and CaCO<sub>3</sub> 0.5. In addn. to the above each chick received daily 0.2 mg. carotene and 240 International units of irradiated ergosterol. C. R. Fellers

Effect of one % cod-liver oil on the rat, with particular reference to the thyroid gland. Clay B. Freudenberg and Fred W. Clausen. *J. Nutrition* 10, 549-55(1935).—

The addn. to a good stock diet of 1% cod liver oil produced no differences in body growth, wt., nose-ans length, tail length, incidence of infections, wt. of head, suprarenal glands, hypophysis, thymus or ovaries. The thyroid gland was significantly smaller in the rats fed cod-liver oil. C. R. Fellers

Relation of rate of growth to diet. III. Comparison of stock rations used in the breeding colony at the Connecticut Agricultural Experiment Station. Lafayette B. Mendel and Rebecca B. Hubbell. *J. Nutrition* 10, 557-63 (1935), cf. C. A. 26, 4086—A gradual increase in growth and reproductive ability has followed each improvement in the stock ration of the albino rat colony. C. R. Fellers

Comparative rachitogenic property of oats and corn. Lawrence L. Lachat and Leroy S. Palmer. *J. Nutrition* 10, 565-77 (1935)—HCl exts. of oats, when purified and freed from excessive amts. of NaCl, may exhibit rachitogenic properties when fed to rats in a mildly rachitogenic ration, but not when the ration is severely rachitogenic. Rolled oats and yellow corn are rachitogenic to both rats and chicks, especially to the latter, and rolled oats appears to be more rachitogenic when the rations are otherwise only mildly rickets-producing or the susceptibility of the animals is low. Divergent results obtained by others in the study of the relative rachitogenic properties of oats and other cereals probably may be explained by lack of control of the severity of the rickets produced by the ration with which the cereal rations have been compared. C. R. Fellers

The cereal grains and their use in poultry nutrition. I. Hatchability studies. J. B. Smith. *Sci. Agr.* 16, 73-6 (1935)—In hatchability studies pens of hens were raised from the time of hatching on a single source of vegetable protein such as wheat, oats, barley and corn, but supplemented by animal sources of protein. Whereas both wheat and barley gave good hatchability records, better results were obtained when the cereals were mixed. There was usually a heavy mortality in the very young chicks from the cereal-fed hens. A crushed grain mix of wheat, oats and barley with milk gave excellent results in both hatchability and egg production over a 2-yr. period. Buckwheat was used satisfactorily in combination with oats and barley, when milk was used as an animal protein supplement. II. Influence on live weight gains and distribution of fat in fattening stock. W. A. Maw. *Ibid.* 77-8—The different cereals cause deposition of fats in the fattening cockerel in entirely different manners, e. g., cornmeal causes a high percentage of body fat to be deposited in the flesh and much less fat in the abdominal cavity and in the skin of the bird, whereas the cereals, barley, oats and wheat, show the reverse in varying degrees. The flesh from the corn-fed birds was judged to have the highest flavor and as being moist, whereas the wheat flesh decidedly lacked flavor and was dry. The barley and oat flesh was intermediate to the corn and wheat flesh. C. R. F.

Relation between the vitamin C contents of various plant tissues and the presence or absence of chlorophyll. Lucie Randoit, A. Giroud and C. P. Leblond. *Compt. rend. soc. biol.* 120, 297-300 (1935), cf. C. A. 29, 3702<sup>1</sup>.—Feeding expts. with guinea pigs showed that the green parts of leeks, beets, carrots, leafy turnips and lettuce were satisfactory sources of vitamin C while the nongreen parts contained very little. L. E. Gilson

Hemostatic action of ascorbic acid. P. Lambin and W. van Hecke. *Compt. rend. soc. biol.* 120, 536-8 (1935)—In rabbits weighing 1900-2400 g. the intravenous injection of 12-20 mg. ascorbic acid produced a 17-22% decrease in bleeding time (detd. less than 1 hr. after the injection). L. E. Gilson

Vitamins in dermatology. Werner Jadassohn. *Z. Vitaminforsch.* 4, 324-8 (1935)—A review. J. S. H. The distribution of vitamin C in lower organisms. G. Bourne and R. Allen. *Australian J. Exptl. Biol. Med. Sci.* 13, 165-74 (1935), cf. C. A. 29, 7413<sup>1</sup>—The AcO<sub>2</sub>-AgNO<sub>3</sub> staining technique was used to demonstrate the presence of vitamin C in *Chlamydomonas* (Protista), *Oscillatoria* (Cyanophyceae), *Scenedesmus* and *Spirogyra*

(Chlorophyceae), diatoms, lichens, yeasts, mold mycelia, *Es. coli*, and the fungi *Fusarium*, *Ophiobolus*, *Coleotrichum*, *Trichoderma*, *Hellinotriporum*, and *Rhizoctonia*. The connective tissue, liver and epithelial cells of the tadpole also showed typical granules. The uniformity in types and distribution of granules in the cells of the higher and lower plants and animals led B. and A. to conclude that the reducing granules are probably composed of the same substance, reduced vitamin C, in all of the tissues studied. C. G. King

Occurrence of ascorbic acid in the lens and vitreous humor. V. Demole and H. K. Muller. *Biochem. Z.* 281, 80-5 (1935)—Three cc. of vitreous humor produces in feeding expts. an antiscorbutic effect equal to that of 0.5 mg. ascorbic acid, which corresponds more or less to the estn. by the indophenol titration of the humor. The lens likewise produces an antiscorbutic effect which is equiv. to about 50% of the indophenol titration value and is attributable to ascorbic acid. In animals with scurvy the ascorbic acid content of the vitreous humor is decreased, but in young less than in adult animals. S. Morgulis

Growth in length of young rats on sufficient and insufficient vitamin A administration. Maj. Malmberg. *Biochem. Z.* 281, 215-18 (1935)—On a vitamin A free diet the growth of the tail practically ceases when the increase in body wt. stops. On the addn. of 20  $\gamma$  carotene to this diet the tails become elongated. Carotene also antagonizes the effect of tyrosine, which causes a loss in body wt. and checks the growth of the tail, in that the wt. loss is moderated and the tail increases in length. S. Morgulis

Vitamins. M. van Laer. *Ann. zymologie* (2), 2, 101-7 (1935)—An address discussing very briefly the importance of vitamins in the foodstuffs industry, problems in relation thereto and progress accomplished in 20 yrs. in the study of vitamins. A. Papineau-Couture

Fractionation studies on provitamin D. Elizabeth M. Koch and F. C. Koch. *Science* 82, 394-5 (1935).—Com. cholesterol and purified cholesterol which has been heated at 185-95° for 1 hr. in the presence of traces of O<sub>2</sub> were much superior to vosterol or purified unheated cholesterol in preventing leg weakness in Leghorn chicks. Ten rat units of either prepn. per 100 g. diet were equiv. to 0.25% or 10 rat units of cod liver oil. The provitamin D of heated cholesterol evidently seps readily in the crystal. from alc., since the filtrate residue has no potency when given in comparable doses to rats or chicks. The chicks receiving 100 rat units of vosterol per 100 g. of diet were in no better condition than those receiving the basal ration only. Felix Saunders

The antineuritic vitamin (Windaus, et al.) 10.

## Γ—PHYSIOLOGY

HOMER W. SMITH

The effect of endocrine glands on the composition of the skeletal muscle. IV. The effect of the tests on the composition of the skeletal muscle. S. Osada. *Folia Endocrinol. Japon.* 11, 21-2 (1935); cf. C. A. 29, 7417<sup>2</sup>. The creatinine content of the skeletal muscle of male rabbits remained unchanged after oral administration of testis powder (I) or after castration (II). Residual N, NH<sub>4</sub>, urea, creatine and amino acid concn. increased after I but remained unchanged after II. Glycogen and lactadogen decreased and lactic acid increased after I. II produced the opposite effect. V. The effect of the ovary on the composition of the skeletal muscle. *Ibid.* 22-3—The creatinine content of the skeletal muscle of female rabbits remained unchanged after oral administration of interstitial tissue powder (I) or of corpus luteum powder (II) or after oophorectomy (III). Creatine and lactic acid increased and glycogen and lactadogen decreased after I, II and III produced the opposite effect. Amino acid and NH<sub>4</sub> increased after I, but remained unchanged after II or III. Residual N and urea increased after I, decreased after II and remained unchanged after III. Ruth Berggren



**Dehydroandrosterone.** Adolf Butenandt, Hans Dannenbaum, Günter Hamisch and Helmut Kudsus. *Z. physiol. Chem.* 237, 57-74 (1935); cf. *C. A.* 29, 4997.—Dehydroandrosterone, m. 148°,  $[\alpha]_D^{25}$  10.9°, occurs in male urine in practically the same amt. as androsterone. It is the source of the chloro ketone previously isolated from urine by a procedure involving the use of HCl. In addition, to the benzoate previously described, the following new derivs. have since been prep'd.: acetate m. 168.9°,  $[\alpha]_D^{25}$  3.9°, oxime m. 191°, semicarbazone decomp. 202-4°. Its activity is about 1/3 that of androsterone in both the comb test and in the vesicular gland test. Although without activity in the Allen-Dowsy test on castrated female mice, it brings on estrus in the infantile females. The acetate has about the same activity as the mother substance, but the benzoate is only slightly active, probably because of its extreme insol. Isolation of dehydroandrosterone from urine is best performed by digitonin pptn and purification through the semicarbazone and acetate, from 100 l. of urine some 20 mg. was thus obtained in pure form, probably less than a 50% recovery. Treatment with HCl, or with SOCl<sub>2</sub>, gave the chloro ketone but only in small yield along with other products not identified. Dehydroandrosterone differs from androsterone only in the presence of a 5-6 double bond and in the spatial arrangement of the OH at C<sub>3</sub>. Catalytic hydrogenation converts it into isandrosterone (3-hydroxy- $\Delta^4$ -cholestanone-17). In steric configuration it corresponds to cholesterol from which it is obtained by replacement of the side chain by O. The transformation is effected by converting cholesterol into the acetate-dibromide, oxidizing off the side chain with CrO<sub>3</sub>, removal of the 2 Br atoms and sapon. of the Ac group. The product is therefore 3-hydroxy- $\Delta^4$ -cholestanone-17. The yield was 885 mg. from 21 g. of cholesteryl acetate, or 2.8%. Sitosterol or stigmasterol may be used in place of cholesterol. As intermediate products cholesterol yielded the  $\Delta^3$ -3-hydroxycholesterol, while stigmasterol gave the interesting  $\Delta^3$ -3-hydroxyvinylcholesterol, a substance from which the corpus luteum hormone progesterone has already been prep'd. Neither acid in 4-mg. dosage showed physiol. activity in the comb test. The conversion of dehydroandrosterone into androsterone consists in satg. the double bond and epimerizing the OH. The reaction is performed by treatment with SOCl<sub>2</sub>, hydrolyzing the chloro ketone, and treatment of the dihydrochloro ketone with KOAc, whereby androsterone acetate is obtained. It is probable that the Walden inversion occurs in the last step of the process. A. W. Dux.

**Androstenedione, a highly active male hormone.** The genesis of gonad hormones. Adolf Butenandt and Helmut Kudsus. *Z. physiol. Chem.* 237, 75-88 (1935).—Cholesterol is probably the mother substance from which all the known sex hormones are derived. A simple replacement of the side chain by O yields dehydroandrosterone which is intermediate between the male hormones of the androsterone type and the female hormones of the estrone type. As the probable precursor of both types dehydroandrosterone is of especial interest, and particularly its dehydrogenation product, androstenedione. The dehydrogenation of dehydroandrosterone was effected by adding 2 Br at the double bond, oxidizing the product with CrO<sub>3</sub> to the dibromo diketone, and removal of the Br by means of Zn dust. The intermediate products were not isolated. The end product was androstenedione, with the keto groups at C<sub>3</sub> and C- and double bond at C<sub>4</sub>. It m. 169°, has  $[\alpha]_D^{25}$  18.5°, absorption max. at 235 m $\mu$  (characteristic of  $\alpha$ , $\beta$ -unsatd. ketones), and forms a oxime m. 143°. Androstenedione is structurally related to all 3 types of known sex hormones, and was therefore tested for all 3 types of activity. In the capon comb test it showed approx. the same activity as androsterone, but in the vesicular gland test on rats the activity was considerably greater than that of androsterone. The corpus luteum test was entirely neg. The Allen-Dowsy test for estrus was neg. in castrated females, but the Curtis-Dowsy test on infantile females was pos. in 200-400  $\gamma$  dosage. It is not improbable that the pos. result with infantile females

is due to a biol. splitting off of the angular Me at C<sub>10</sub>, thereby forming estrone itself. A. W. Dux.

**Testosterone.** Transformation of dehydroandrosterone into androstenediol and testosterone, a way to the preparation of testosterone from cholesterol. Adolf Butenandt and Günter Hamisch. *Z. physiol. Chem.* 237, 89-97. *Ber.* 68B, 1859-62 (1935). cf. *C. A.* 29, 5004.—Reduction of dehydroandrosterone in EtOH with Na and sublimation of the product in a high vacuum gave the corresponding alc.,  $\Delta^4$ -androstene-3,17-diol, m. 177-8°,  $[\alpha]_D^{25}$  -53.5° (diacetate m. 159.5°,  $[\alpha]_D^{25}$  -56.6°). This showed unexpectedly low activity in the comb test, a 300  $\gamma$  dose giving no definite response. The diacetate was partially sapon. by treatment of the dil. soln. in MeOH with KOH in the cold, yielding the 17-monooacetate, m. 148°, it has  $[\alpha]_D^{25}$  -62.4°. Addn. of Br to protect the double bond, followed by oxidation with CrO<sub>3</sub> in AcOH and removal of the Br by Zn dust and AcOH, gave  $\Delta^4$ -androstene-17-diol-3-one acetate, m. 138°, it has  $[\alpha]_D^{25}$  87.5°. Sapon. of this acetate then yielded the corresponding androstosterone, m. 151°, it has  $[\alpha]_D^{25}$  104°, identical in all respects with the testosterone obtained by David, et al. (*C. A.* 29, 5163 $\gamma$ ) from bull testes. The oxime m. 215°. Testosterone exceeds all known male sex hormones in physiol. activity, both in the comb test and in seminal vesicle test. It has 6 times the activity of androsterone and twice that of androstenediol in the comb test. In the vesicle test it is 5 times as active as androstenedione and 10 times as active as androsterone. This synthesis establishes the structure of the testicular hormone in every detail. A. W. Dux.

**Glycogen formation from lower fatty acids with even carbon number.** III. The fate of acetoacetic acid in the animal organism. Richard Stöhr. *Z. physiol. Chem.* 237, 165-70 (1935), cf. *C. A.* 27, 5343.—The influence of AcCH<sub>3</sub>CO<sub>2</sub>H on the respiration of surviving organ sections (liver, kidney, diaphragm of the rat) was detd. in the Warburg app. In concns. of 1, 2 and 4 mg. per cc. of Ringer phosphate AcCH<sub>3</sub>CO<sub>2</sub>H inhibits the respiration of liver and kidney tissue and increases that of the diaphragm. Feeding expts. with 0.2 g. AcCH<sub>3</sub>CO<sub>2</sub>Na and with 0.2-0.3 g. AcCH<sub>3</sub>CO<sub>2</sub>Et in the presence of glucose did not show an increase in glycogen content in the liver of fasting rats. A. W. Dux.

**Influence of thyroid hormone on vitamin C metabolism.** Janos Mosonyi. *Z. physiol. Chem.* 237, 173-7 (1935).—Subcutaneous administration of thyroid hormone (thyroid and thyroid ext.) to guinea pigs resulted in a decrease in the ascorbic acid content of the liver and especially of the suprarenals. This decrease did not occur in rats. In guinea pigs it was prevented by simultaneous administration of vitamin A. Exstirpation of the thyroid in rats increased the ascorbic acid content, apparently by increased synthesis in the suprarenals. A. W. Dux.

**The occurrence of L-glyceroisophingosin in beef lung.** Caspar Tropp. *Z. physiol. Chem.* 237, 178-80 (1935).—One g. of L-glyceroisophingosin was isolated from 5 kg. of beef lungs, a yield of 0.06% on the dry basis. Similar quantities of heart, kidneys, lymph glands, testes and white and red bone marrow, worked up by the same procedure, yielded none of this substance. A. W. Dux.

**Constant value of the ratio of the skeletal dimension to the active mass in the rat.** Andree Roche and Isabelle Garcia. *Compt. rend. soc. sci. 120*, 160-2 (1935).—As young rats weighing 5 g. at the start grew in wt. to 194 g. the P content of the total fresh carcass, 0.37% in the 5-g. rats, increased regularly to 0.75%, the N content, 1.54% at the start, increased to 2.89%. The P/N ratio remained within the range 0.19-0.25 throughout.

**Change in the glycerol content of blood during glycolysis in vitro.** Michel Polonovsky, H. Warenbourg and P. Lamour. *Compt. rend. soc. sci.* 120, 191-2 (1935).—Fresh hog blood and also a suspension of washed dog erythrocytes were incubated at 37° after addn. of glucose. After several hrs. there was a small increase in glycerol (detd. by the method of Zeisel and Fanto). The residual chromic index increased proportionately. L. E. G.

Changes in blood composition during short-wave treat.

ment A Compère *Compt. rend. soc. biol.* 120, 237-40 (1935).—Dogs were treated with a 6-mm wave length. The max rectal temp reached was 41.9°. The most important effect was a large decrease in the alkali reserve and  $p_{\text{H}}$  (decreased 0.07-0.33 unit) of the blood. Non-protein N increased 20-44% and total blood protein increased slightly. Total fixed bases did not change. P and Cl showed irregular fluctuations. L E Gilson

Disappearance of [injected] prolactin from the blood of nephrectomized female rabbits A Lipschütz, A. Fehreman and T. Vivaldi *Compt. rend. soc. biol.* 120, 333-6 (1935), cf C A 29, 3012<sup>1</sup>.—In the normal female 80% of the injected prolactin disappears from the blood in 10 hrs, part being excreted in the urine. In the nephrectomized female about 30% disappears in 10 hrs. L E Gilson

Effect of pregnancy on the survival period of adrenalectomized animals A Gradinesco, N Santa and F. J. Lucanonescu *Compt. rend. soc. biol.* 120, 356-8 (1935).—The survival period was the same or slightly shorter than that of nonpregnant cats and dogs. Apparently the fetal adrenals offer no protection to the mother. Twenty references. L E Gilson

[Absence of] influence of the thyroid on the receptivity of rabbits toward herpes virus P. Haber *Compt. rend. soc. biol.* 120, 420-1 (1935). L E Gilson

Effects of hypophysectomy on pregnancy and lactation in dogs B A Houssay *Rev. soc. argentina biol.* 11, 196-201; *Compt. rend. soc. biol.* 120, 496-7 (1935).—Total hypophysectomy produces abortion without any lactic secretion. If performed a few days after normal parturition it causes a rapid decrease in milk flow. The anterior lobe is necessary for the production of lactic secretion after parturition or abortion, and for its maintenance after it has begun. Extirpation of the posterior lobe does not affect pregnancy, parturition or lactation. Injury to the tuber cinereum does not suppress lactation. L E Gilson

Respiratory quotients of normal and neoplastic tissues cultivated in vitro A. H. Roffo and Luis M. Correa. *Rev. soc. argentina biol.* 11, 202-9 (1935).—The app and method are described. The R Q of growing chick heart was 0.80-0.97, av. of 10 expts 0.86. That of growing rat sarcoma and carcinoma was 1.01-2.02, av. for 15 cultures 1.31. L E Gilson

Production of milk secretion in female and male dogs by anterior hypophysis extract B A Houssay. *Rev. soc. argentina biol.* 11, 240-9; *Compt. rend. soc. biol.* 120, 502-3 (1935).—Repeated injections of the ext. (made by the method of Evans and Simpson, C. A. 26, 3020) produced lactation in adult females which had never been pregnant but not in very young females. It also produced lactation in females which had been castrated, thyroidectomized, hypophysectomized and thyroidectomized, or had the splanchnic nerves cut and the lumbar sympathetic chains resected. The activity of the ext. was destroyed by boiling 15 min. The ext. did not produce lactation in male dogs directly, but if the mammae were first hypertrophied by prolonged administration of folliculin and repeated injections of the ext. then made, lactation was produced in whole, castrated and castrated hypophysectomized males. L E Gilson

Production of lactation in male guinea pigs by anterior hypophysis extract J. L. Sardi. *Rev. soc. argentina biol.* 11, 250-7; *Compt. rend. soc. biol.* 120, 503-4 (1935).—The ext., or the ext. and folliculin together, did not cause lactation, but treatment for 10-17 days with folliculin to produce hyperplasia of the mammae followed by repeated injections of the ext. produced abundant lactation. L E Gilson

Morphine and ether hyperglucemia in hypophysectomized dogs Mme Elena Di Benedetto de Sabelli and Enrique J. Di Benedetto. *Rev. soc. argentina biol.* 11, 405-9 (1935); *Compt. rend. soc. biol.* 120, 738-9 (1935).—The hyperglucemia produced by ether anesthesia is much greater in normal than in hypophysectomized dogs. Morphine produced a slightly greater hyperglucemia in the latter group. L E Gilson

The corpus luteum hormone. Enrico Fels. *Rev. soc. argentina biol.* 11, 417-27; *Compt. rend. soc. biol.* 120, 730-1 (1935).—The hormone, recently renamed progesterone, is a mixt of luteosterones C and D. Previous conclusions concerning the activity of these (cf C. A. 28, 7238<sup>4</sup>) were confirmed. Progesterone differs from estrone in that it has no effect on the male genital tract and does not modify the changes in the anterior hypophysis which follow castration in the female rat. L E Gilson

The influence of tyrosine on the thyroid function I. Abelin. *Naturwissenschaften* 23, 528-9 (1935).—The hormones of thyroid and adrenals are both derived from tyrosine. Duodotyrosine is active in disturbances of either of these glands. Tyrosine itself proved to have therapeutic effects in the severe secondary effects of exptl hyperthyreosis. Evidently tyrosine as well as I participates in the regulation of the thyroid action. B J C van der Hoeven

Distribution of C vitamin in cerebral basal ganglia particularly in the globus pallidus and the substantia nigra. F. Plaut and K. Stern. *Naturwissenschaften* 23, 557-8 (1935).—Vitamin C (ascorbic acid) was detd. in different parts of the brain. Typical values obtained (av) were 11.7 mg. % for globus pallidus, 15 mg. % for substantia nigra, max. 10 in front cerebrum, 17.7 in cornu ammonis. These results are significant in connection with the high Fe content of the globus which is taken as an explanation for the CO and HCN sensitivity of these regions. Ascorbic acid is likewise a reactive substance probably involved in the cell respiration but present in relatively lower concn. in the globus. B J C van der Hoeven

Influences of amino acids and choline upon the pigment-excreting function of the liver. Tatsuo Matsumura and Akio Kashimura. *Japan J. Gastroenterol.* 7, 115-19 (1935).—A series of amino acids and amines failed to stimulate the excretion of azofuchsin-G from the livers of rabbits. C M. McCay

Uric acid elimination in man. Hilding Berglund and A. Rune Frisk. *Acta Med. Scand.* 86, 232-68 (1935).—The ratio urinary uric acid/blood uric acid, or elimination index (E. I.) bears a high correlation to the Rehberg test. A linear relation exists in the E. I. in both normal persons and in those with severe kidney damage following intravenous injection of moderate amts. of Li urate, which is, however, broken over variable periods because of periodic alterations in glomerular circulation and function. The elimination can be greatly increased and the periodic "breaks" in the linear relationship avoided by the administration of salyrgan, neocinephren or ephylline. But under all circumstances the elimination of uric acid is inferior to that of creatinine, the mechanism unaltered by drugs being only 1/5 to 1/4 as efficient. No correlation apparently exists between diuresis and uric acid elimination. S Morgulis

Relationship between hydrochloric acid and total chlorides in gastric juice and the possibility of standards for gastric secretion. Hilding Berglund, Richard Johnson and H. Chien Chang. *Acta Med. Scand.* 86, 269-91 (1935).—The fundamental phenomenon in gastric secretion is the secretion of chlorides and, when the mechanism for HCl production may be lost, chloride secretion persists. The gastric acidity is very variable and no standards can be established without stimulation (with histamine) producing max response. S Morgulis

Influence of some external factors on the insensible perspiration A van Harreveld, B. W. Grutternik and A. K. M. Noyons. *Biochem. Z.* 281, 1-26 (1935).—Expts show that even as large a change as +35% in relative humidity does not affect the insensible perspiration, provided that the person has attained equilibrium with his environment. Sweating during an expt may give results which would indicate that more water is given off in a moist than in a dry atm or when the relative humidity is changed suddenly. Within a temp range of 20° to 28.5° the insensible perspiration of a person wearing a swimming suit varies but insignificantly. Neither does the wt of the

clothing have any effect so long as the subject is in equilibrium with his environment. The insensible perspiration even of the same subject shows considerable variability, and the relation between this and the metabolism may deviate by as much as 20%, so that the latter cannot be deduced from the insensible perspiration. S. Morgulis

**The carbohydrate metabolism of guinea pigs.** Friedrich Gottschalk. *Biochem. Z.* 281, 128-39 (1935).—In late fall the blood sugar of guinea pigs after a brief fast (16-26 hrs.) rises 250% or more, and this hyperglycemia is not affected by eating. During the winter this hyperglycemia is not found in the same animals. Feeding 2 g. glucose per kg. by mouth produces an alimentary hyperglycemia which attains a max. in 30-60 min. The glycemia curve then falls, occasionally even below the original level, but by the end of 7 hrs. shows invariably a renewed large rise, even beyond the previous max. values. Administration of 0.2 cc. adrenaline (1:1000) per kg. causes a marked hyperglycemia, up to 300% above the initial level. Injection of 2 units per kg. of insulin causes in the great majority of the animals severe hypoglycemic convulsions without materially lowering the glucemic level. In fact, in some guinea pigs there was no lowering of the blood sugar and others have shown considerable hyperglycemia at the height of the insulin shock. The high reducing values of the blood were not due to nonsugar reducing substances as was detected by the fermentation with yeast. S. Morgulis

**Coupling of chemical processes in the muscle.** X. Ostern and T. Baranowska. *Biochem. Z.* 281, 157-67 (1935). cf. *C. A.* 29, 8091.—The heart muscle of the rabbit differs definitely from the skeletal muscle by the presence of free adenylic acid and of very little phosphocreatine; the total P and the adenosinepolyphosphoric acid are only about 50% of that of skeletal muscle. In heart muscle the adenylic acid is stable, its hydrolysis proceeding very slowly even in exts., and apparently it does not need to be phosphorylated at once, as in the skeletal muscle, to prevent its deamination. The stability of the adenylic acid in the heart makes it unnecessary to have a high phosphocreatine content which acts as a P donor in the rephosphorylation of the adenylic acid. In exts. from heart muscle the formation of adenosinetriphosphoric acid from adenylic acid proceeds the same as in muscle exts., the phosphoglyceric or phosphopyruvic acid being the P donor. In inactive exts. there is also a synthesis of phosphocreatine besides the synthesis of adenosinetriphosphoric acid, while in dialyzed exts. there is no synthesis of phosphocreatine and a much smaller synthesis of adenosinetriphosphoric acid. The splitting of the phosphoglyceric acid occurs only in the presence of adenylic acid and the adenosine does not function as coenzyme. In the absence of the adenylic acid the phosphoglyceric acid only changes to phosphopyruvic acid. It was also found that the Mg ions play an important role in the synthesis of adenosinetriphosphoric acid, and function therefore in the muscle as activators of the P transfer. The Mg ions seem also to play an important part in the synthesis of phosphocreatine from creatine and adenosinetriphosphoric acid. S. Morgulis

**Some biochemical aspects of fats.** II. Wm. E. Anderson and Harold H. Williams. *Oil and Soap* 12, 232-4 (1935). cf. *C. A.* 29, 5088.—Recent studies in animal fat metabolism are discussed. E. Scherubel

**Artificial and natural regulation of body function.** Ivan de Burgh Daly. *Pharm. J.* 135, 593-4; *Chemist and Druggist* 123, 674-5 (1935).—An address on the interrelation of endocrine functions in the body. S. W.

## G—PATHOLOGY

H. GIDEON WELLS

**Adsorbable fraction of serum calcium in acute and chronic parathyroid insufficiency.** Franz Mathieu. *Compt. rend. soc. biol.* 120, 94-102 (1935). cf. *C. A.* 29, 6644.—The serum was shaken 2 hrs. with much BaSO<sub>4</sub>.

1 From normal dog serum (contg. inorg. P 3.8 and Ca 10.2 mg. %) 58% of the Ca was adsorbed, while 75% of the Ca was ad-orbed from the serum of dogs with acute (inorg. P 5.7 and Ca 6.1) and latent (inorg. P 6.4 and Ca 6.4) tetany due to removal of the parathyroids.

L. E. Gilson  
**Anaphylactic sensitization and desensitization of guinea pigs with mixtures of serum and lanolin [and olive oil].** C. Gernex. *Compt. rend. soc. biol.* 120, 186-8 (1935); cf. *C. A.* 29, 5054.—Sensitization was readily produced by subcutaneous injections of the mixt. but desensitization by the mixt. was slow and difficult compared to results obtained with pure serum. L. E. Gilson

**Kopczewski's lactogelification reaction as an indicator of neoplasms.** Laura Capper. *Compt. rend. soc. biol.* 120, 235-6 (1935).—With normal persons the reaction is always neg. In bloods from 110 cancer cases 85.4% of pos. and 14.6% of neg. reactions were obtained. The reaction was always pos. at the beginning and in the early stages but in the advanced stages, with cachexia, it was often neg. In other diseases (tuberculosis, gallstones, various chronic inflammations) pos. reactions were obtained with about 8%.

L. E. Gilson  
**Existence in the liver of substances capable of inhibiting benzenepyrone cancer in albino mice.** J. Maisin, H. Vassiliadis and A. Godenir. *Compt. rend. soc. biol.* 120, 259-61 (1935). cf. *C. A.* 29, 5912.—The acetone-insol. fraction of the Et<sub>2</sub>O ext. contains a thermostable substance which has a retarding or inhibiting effect on the cancer when given by mouth. In whole liver the inhibiting substance is masked by the cancer-activating substances present. L. E. Gilson

**Antigenic power of staphylococcus toxin and anatoxin.** Resistance to heat. G. Ramon and R. Richou. *Compt. rend. soc. biol.* 120, 290-2 (1935).—The toxin loses its antigenic property slowly at 30-55° and rapidly at 55-60°. The anatoxin (toxin + HClO<sub>4</sub>) is not changed below 55° but loses its antigenic power slowly at 55-60° and rapidly at 60-70°. L. E. Gilson

**Effect of the serum of mice with tar cancer on the fluorescence of uranin solution.** Augustin Boutanc and Jean Bouchard. *Compt. rend. soc. biol.* 120, 293 (1935); cf. *C. A.* 29, 4419, 6307.—In tar cancer the serum does not decrease the fluorescence as it does in other forms of cancer. L. E. Gilson

**Blood indican in nephritis.** I. Gavril and Mihailescu. *Compt. rend. soc. biol.* 120, 358-60 (1935).—Normal human blood contains 0.22-0.92 mg. indican per l., as determined by the Jolles-Rosenberg method. In many pathological cases, with nonprotein N retention, the blood indican was within the normal range; in about 50% it was definitely increased. No relation between indican content and other variables of the blood was found. L. E. Gilson

**Conditions for flocculation and gelification of pathological serums.** E. Benhamou and R. Gille. *Compt. rend. soc. biol.* 120, 428-9 (1935). "Flocculability index" of serums in flocculation and gelification reactions. *Ibid.* 430-2.—Discussion. L. E. Gilson

**Hypocalcemia in pancreatostomized and hypophysectomized pancreatostomized dogs.** R. Gerschmann and A. D. Mareni. *Riv. sci. argent. biol.* 11, 391-404; *Compt. rend. soc. biol.* 120, 737-9 (1935).—Hypophysectomy does not affect blood Ca. Removal of the pancreas causes a decrease in blood Ca caused by the degenerative changes which take place in the parathyroids. The decrease is more rapid if the hypophysis is also removed. Insulin may partially restore the blood Ca, parathyroid hormone brings it back to normal. L. E. Gilson

**Sources of nickel eczema. Report of two examples and review of the pertinent literature.** Paul D. Foster and Franklin I. Ball. *Arch. Dermatol. Syphilol.* 31, 461-9 (1935).—The literature on nickel eczema produced by plated objects and alloys is brought up to date and 2 cases are described in detail. Hypersensitivity to Ni appears to be acquired rather than inherent in all cases known. O. Hartley

**Arsenic as an etiologic agent in certain types of epithelioma.** Hamilton Montgomery. *Arch. Dermatol. Sy-*

*philol* 32, 218-33(1935)—A review with 25 references

O. Hartley

The relation between bacterial agglutinins and precipitins. Guglielmo Ascione *Arch sci biol.* (Italy) 21, 90-5(1935)—Precipitins and agglutinins are antibodies of different nature since with antityphoid, anticholeric and antimelittic serums one can demonstrate agglutination after the pptg power has been exhausted with the corresponding precipitin

P. F. Metildi

The lipolytic power of the liver of normal and tuberculous rabbits on the glycerides of lower and higher fatty acids and on the acetone soluble fraction of the lipides of tubercle bacilli. Roberto Rordorf. *Arch sci biol* (Italy) 21, 97-101(1935)—During the course of the disease in rabbit livers there is a diminution of lipase and tributryase activity. Neither the normal liver nor one from a diseased animal shows a hydrolytic action on acetone sol. lipides of tubercle bacilli

P. F. Metildi

Urinary urobilinogen value of routine estimation. P. H. Levinson *J. Tenn. State Med. Assoc.* 28, No. 1, 17-19(1935)—Routine testing for urobilinogen is valuable in liver dysfunction, biliary obstruction, cholangitis, pyelophlebitis and excessive hemolysis

J. C. M.

Lack of mineral salts, esp. phosphoric acid in soils [causing osteomalacia] (Rubino) 15

## H—PHARMACOLOGY

A. N. RICHARDS

Caffeic acid in prunes and its behavior as a laxative principle. E. Mraz, J. Fessler and C. Smith. *Science* 82, 304(1935)—Caffeic (I) and chlorogenic (II) acids cause an increase in tonus and amplitude of contraction of isolated rabbit jejunum or duodenum (C. A. 28, 1403) similar to that effected by various prune exts. Attempts to isolate these acids from the alk. hydrolyzed aq. ext. of prunes by the method of Plucker and Keilholz (C. A. 28, 5898) gave no indication of the presence of II but yielded 0.03% of crystalline I. The source of I in prunes is uncertain but I and quinic acid may be combined in complex caffeannins. Feeding tests with live rabbits, dogs and human beings failed to show any significant laxative effect but I caused a slight change in tonus and amplitude in tests with isolated rabbit duodenum. I is not the substance responsible for the laxative action caused by the ingestion of prunes

C. R. Addinall

The effects of certain acid treatments for coccidiosis on the hydrogen ion content of the fowl intestine. W. R. Kerr and R. H. Common. *Vet. J.* 91, 309-11(1935)—The reaction of various organs of birds treated with buttermilk or HCl varied very little from the values secured with the controls. The  $pH$  of the first portion of the small intestine was somewhat increased by buttermilk treatment

K. C. Beeson

The effect of pituitrin on the composition of the skeletal muscle. S. Osada. *Folia Endocrinol. Japon.* 11, 23(1935)—After one injection of pituitrin (amt. not stated) the lactic content of the skeletal muscle increased, glycogen and lactacidogen decreased, and residual N,  $NH_4$ , urea, creatinine, creatine and amino acid remained unchanged

Ruth Berggren

Therapeutic uses of colloidal sulfur. Goff P. Lilly and Benjamin S. Gershwin. *Am. Med.* 41, 546-9(1935)

Rachel Brown

Acute potassium permanganate poisoning. Report of a case. Edmund W. Klinefelter. *Am. Med.* 41, 570-2(1935)

Rachel Brown

Better general anesthesia in animals. I. Oxygen-ether controlled anesthesia by the closed method in the dog with morphine as preanesthesia hypnosis. N. C. Covington. *J. Am. Vet. Med. Assoc.* 40, 662-72(1935)

Rachel Brown

Treatment of staphylococcus infections with staphylococcus toxoid. M. A. Gohar. *J. Trop. Med. Hyg.* 38, 249-60(1935)—Formalinized anaerobic broth culture filtrates of staphylococcus were found effective in subcutaneous injections for the treatment of staphylococcus skin infections

Rachel Brown

1 Treatment of amebic colitis with iodoform. Aldo Castellani. *J. Trop. Med. Hyg.* 38, 208-9(1935).—Satisfactory results were obtained in the treatment with iodoform of a series of 9 cases of amebic colitis

Rachel Brown

Effect of atropine on the augmented salivary secretion in the cat. F. C. MacIntosh and H. E. Rawlinson. *Quart. J. Exptl. Physiol.* 25, 199-205(1935)—After small doses of atropine the secretory response of the cat's submaxillary gland to sympathetic stimulation is increased by previous stimulation of the chorda tympani, although the latter nerve is itself unable to cause any secretion. Stimulation of the chorda tympani must therefore cause a true increase in the irritability of the secretory cells to sympathetic stimulation

Rachel Brown

The effect of sympathetic stimulation and of autonomic drugs on the paralytic submaxillary gland of the cat. A. J. Fleming and F. C. MacIntosh. *Quart. J. Exptl. Physiol.* 25, 207-12(1935)—In cats after section and degeneration of the chorda tympani, the submaxillary gland shows a greatly increased secretory response to sympathetic stimulation and to adrenaline. This increased response is due to a true heightened irritability of the secretory cells for sympathetic stimulation. Pilocarpine and acetylcholine act more strongly on the normal than on the paralytic gland

Rachel Brown

4 The effect of adrenaline and of increased work on the carbohydrate metabolism of the mammalian heart. J. Yule Bogue, C. Lovatt Evans, F. Grande and F. Y. Hsu. *Quart. J. Exptl. Physiol.* 25, 213-28(1935)—Increase in the energy expenditure of the dog's heart by the addn. of adrenaline to the circulating blood or by raising the mech. work done increases the utilization of both sugar and lactate. Cardiac muscle uses up lactate more readily than skeletal muscle. It is suggested that the lactate is burned by the tissue to yield energy, whereas the sugar is used to replace glycogen usage. When the blood sugar and lactate have fallen to a low level the heart glycogen is drawn upon, especially if the energy expenditure has been high

Rachel Brown

The selective action of histamine and the effect of prolonged vagal stimulation on the cells of gastric glands in the dog. D. J. Bowie and Arthur M. Vineberg. *Quart. J. Exptl. Physiol.* 25, 247-57(1935)—After repeated subcutaneous administrations of histamine during several hrs., there was no diminution in the amt. of pepsinogen granules in the peptic cells. The gastric juice produced by histamine was copious and of high acidity but low in peptic power. A few hrs. after the administrations of histamine were begun pepsin became practically absent from the secretion. A max. discharge of pepsinogen granules from the peptic cells occurred after prolonged rhythmic stimulation of the vagi in the neck by means of a strong induction current. The increase of pepsin in the gastric juice following vagal stimulation coincided with the discharging of the pepsinogen granules from the peptic cells. Vagal stimulation applied immediately after a period of administration of histamine soon activated the previously inactivated peptic cells. Histamine does not stimulate the peptic cells to discharge their pepsinogen granules

Rachel Brown

8 Novarsenobenzene poisoning. Wilhelm Keri. *Wien klin. Wochschr.* 48, 647-9(1935)—In 2 of 47 patients the use of novarsenobenzene was harmful. Clinical descriptions are given.

D. B. Dill

The influence of diuretics on the alkali reserve of the blood. Georg Gottsegen. *Wien klin. Wochschr.* 48, 1116-18(1935)—If diuretics, ephylline and Na dehydrocholate increase the alk. reserve from 1 to 12 vols. %

D. B. Dill

9 Extract of viper skin. L. Piton. *Bull. med. Jan.* 27, (1935); *J. pharm. Belg.* 14, 263, *Anal. farm. belguim.*, Suppl. 6, 77.—In cancer the ext. has all the effects previously described in C. A. 29, 7493.

L. E. Gilson

Retarding action of formaldehyde, acetaldehyde and acetone on the enzymic hydrolysis of sucrose. A. Chaudin. *Bull. soc. chim. bel.* 17, 1340-50(1935), cf. C. A. 29, 4789.—In concns. of 2-15% the compd.

retard the hydrolysis although the rate curves show an apparent increase in the enzyme concn., owing to the compds. acting as nonsolvent diluents, as in the case of glycerol and the lower alcs. L. E. Gilson

Physiological and pharmacodynamical experiments on the [isolated] human uterine tube. C. Daniel, I. I. Nitescu, A. Somaru and I. D. Georgescu. *Compt. rend. soc. biol.* 120, 54-6 (1935).—At  $pH$  about 7.4 adrenaline produced a transient increase in tone, Doryl or very small doses of nicotine a prolonged increase in tone and no. of contractions, and eupaverine, papaverine or large doses of nicotine, paralysis. Folliculin in small doses had no effect, very large doses caused a decrease in tone and cessation of contractions. Ext. of corpus luteum caused a decrease in tone. Prolan alone had no apparent action on tone or contractions but sensitized the organ to the action of estrin or folliculin. Increasing the  $pH$  of the bath to 7.6 or higher produced an increase in tone and amplitude of contractions, compared to the values for  $pH$  7.4, while decreasing the  $pH$  to 7.1 had the opposite effect. L. E. Gilson

Some effects of piperidinomethylbenzodioxan (F933) on the circulatory system. C. Heymans and J. J. Bouckaert. *Compt. rend. soc. biol.* 120, 79-82 (1935). L. E. Gilson

Effects of adrenaline and of excitation of the cervical sympathetic nerve on the intracranial circulation of the isolated [dog] head. J. J. Bouckaert and F. Jourdan. *Compt. rend. soc. biol.* 120, 81-7 (1935). Existence of cerebral vasoconstrictor nerves and the action of adrenaline on the blood vessels of the brain. *Ibid.* 88-90. Cerebral vascular effect of adrenaline. *Ibid.* 255-7.—In the isolated dog head adrenaline causes a constriction of the blood vessels of the brain, but in anesthetized dogs with the vagus cut and the carotid sinus denervated it causes a slight dilation of the cerebral vessels. Apparently there are 2 simultaneous and antagonistic effects. L. E. Gilson

Action of organic peroxides in the treatment of experimental cancer in mice. W. Koch and J. Maass. *Compt. rend. soc. biol.* 120, 106-8 (1935).—Injections of a 1 in 20,000 aq. soln. of diformaldehyde diperoxide had an ameliorating effect in benzopyrene cancer in mice. L. E. Gilson

Action of commercial preparations of histidine. A. Schwartz, L. Israel and A. Jacob. *Compt. rend. soc. biol.* 120, 121-6 (1935).—Com. histidine and histidine-HCl contain traces of histamine and so produce a wheal when applied to scarified skin. Five  $\gamma$  of histamine is sufficient to produce a noticeable effect. Highly purified histidine has no effect on the skin. L. E. Gilson

Hemolysis by saponin and solanine and cholesterologenesis in vivo. V. de Laverge and P. Kivell. *Compt. rend. soc. biol.* 120, 149-50 (1935); cf. C. A. 29, 4829.—In guinea pigs and rabbits injections of acid solns of solanine and saponin produced hemolysis, hypocholesterolemia and death. Neutral or alk. solns of saponin produced hemolysis and hypercholesterolemia. L. E. Gilson

Effects of injections of spleen extract on carbohydrate metabolism. Michel Polonovski, H. Warenbourg and J. Driessens. *Compt. rend. soc. biol.* 120, 181-2 (1935).—In normal persons the injection of ext. equiv. to 100 g. fresh spleen had practically no effect on the residual chromic index of the blood. In diabetic and cancer patients where the residual chromic index was above normal the injections caused a drop to nearly the normal level. In no case was there any significant change in glycemia. L. E. Gilson

Effect of radium on the glucolytic activity of neoplastic tissues. C. Grandclaude, M. Polonovski, H. Warenbourg and J. Driessens. *Compt. rend. soc. biol.* 120, 183-4 (1935).—In 16 out of 25 cancer cases treated several days with Ra the residual chromic index decreased, and in 6 it increased slightly. The decrease is ascribed to a slowing of glycolysis in the irradiated tissues. L. E. Gilson

Lesions of the liver and kidneys in experimental intoxication [of rabbits] with apiole. A. Patour, G. Patour,

Bedrines and Payen. *Compt. rend. soc. biol.* 120, 192-4 (1935). L. E. Gilson

Effects of intravenous injections of compounds of iron and ascorbic acid on human and experimental animal cancers. F. Arlong, A. Morel and A. Joverand. *Compt. rend. soc. biol.* 120, 201-4 (1935).—See C. A. 29, 7471<sup>1</sup>. Accelerating action on experimental cancer of intravenous injections of ascorbic acid without iron and associated with copper. *Ibid.* 205-6.—In rabbits with grafted cancer

of the testicle repeated daily injections of 0.1 g. ascorbic acid alone or 0.01 g. of the cupric salt of dehydroascorbic acid accelerated the growth of the cancer and increased the no. of visceral metastases formed. L. E. Gilson

Action of adrenaline and pelletierine on the heart of the oyster. A. Julien. *Compt. rend. soc. biol.* 120, 211-13 (1935), cf. C. A. 29, 3923<sup>1</sup>. L. E. Gilson

Ascorbic acid of the adrenals in experimental diphtheria toxin poisoning. G. Mouriquand, P. Sédallian and A. Coeur. *Compt. rend. soc. biol.* 120, 216-17 (1935); cf. Cardoso, C. A. 29, 6644<sup>1</sup>.—In acute intoxication of guinea pigs by diphtheria toxin the ascorbic acid of the adrenals (as detd. by the Giroud-Leblonde reaction) does not begin to decrease rapidly until 18 hrs. after injection of the toxin. L. E. Gilson

Respiration of brain tissue. L. Crasnanu and N. Gavrilescu. *Compt. rend. soc. biol.* 120, 226-8 (1935).—

Novarsenbenzene decreased the O consumption of brain tissue *in vitro* but increased the O consumption of muscle, blood, liver and yeast. L. E. Gilson

Action of diacetylmorphine on the liver. G. Baltacanu and C. Vasiliu. *Compt. rend. soc. biol.* 120, 229-32 (1935), cf. C. A. 28, 6843<sup>1</sup>.—In dogs heroine greatly decreases the flow of bile. The compn. of the bile is not affected. L. E. Gilson

Effect of cortin on adrenaline secretion. Edgard Zunz and Jean La Barre. *Compt. rend. soc. biol.* 120, 218-50 (1935).—The hyperglycemia produced by ext. of adrenal cortex is partly, but not entirely, due to adrenaline discharge. L. E. Gilson

Action of caffeine, papaverine and barium chloride on the cerebral blood vessels. J. J. Bouckaert and F. Jourdan. *Compt. rend. soc. biol.* 120, 257-9 (1935).—In the perfused isolated dog head caffeine benzoate and papaverine salts caused dilation of the blood vessels of the brain and BaCl<sub>2</sub> caused constriction. L. E. Gilson

Effect of cobra venom on the pain threshold of man and guinea pig. D. I. Machit. *Compt. rend. soc. biol.* 120, 284-5 (1935).—The sensitivity to pain produced by elec. stimulation of the skin is decreased. L. E. Gilson

Action of conessine on certain protozoa *in vitro*. R. K. Goyal. *Compt. rend. soc. biol.* 120, 206-7 (1935).—A 0.01% soln. of conessine quickly kills parameria. A 0.1% soln. kills *Spirochaeta duttoni*, *Trypanosoma evansi* and *Leishmania histolytica* in 2 hrs. or less. L. E. Gilson

Influence of experimental hypertrophy of the adrenal cortex and of adrenal cortex extract on the resistance to certain intoxications. G. Benetato, I. Gama and C. Oprisu. *Compt. rend. soc. biol.* 120, 353-5 (1935).—One lot of rats was given daily injections of a suspension of total hypophysis until the adrenals were about 60% above normal wt. Another lot was given several injections of cortical ext. No increase in resistance to morphine or antityphoparatyphoid vaccine was found in either case. L. E. Gilson

Absence of the "all or none" effect in the frog heart poisoned with potassium chloride. A. Chaybany. *Compt. rend. soc. biol.* 120, 407-9 (1935). L. E. Gilson

Treatment of acute mercurial poisoning with sodium formaldehydesulfoxylate. Juan M. Muñoz. *Re. soc. argentina biol.* 11, 224-9, *Compt. rend. soc. biol.* 120, 300-1 (1935).—Na formaldehydesulfoxylate (I) reduces Hg salts to Hg metal *in vitro*. Rats and guinea pigs given HgCl<sub>2</sub> by mouth and then I, also by mouth, in less than 15 min. usually survived. Intravenous injections were of little or no value in rats but were effective in guinea pigs. The lethal dose of I for rats and guinea pigs, when given alone by mouth, is between 5 and 10 g./kg. L. E. Gilson

Magnesium in experimental uremia. I. Austo Amantea,

*Arch. farmacol. sper.* 60, 353-64 (1935).—MgCl<sub>2</sub> introduced into rabbits with exptl. uremia produces an immediate azotemia which diminishes to its original value after several hrs. and a lowering of body temp. The physiol. mechanism is discussed. Helen Lee Gruel

Drugs which affect cellular oxidation. Bruno De Biaso. *Arch. farmacol. sper.* 60, 389-94 (1935).—In rabbit and guinea-pig expts. alkaloids (papaverine and perparine) with peripheral depressive effect did not inhibit or attenuate the excitatory-metabolic action of dinitrophenol. Helen Lee Gruel

Japanese camphor and its synergic association with boric acid in the treatment of skin diseases. Giuseppe Ganino. *Boll. chim. farm.* 74, 671-4 (1935). Helen Lee Gruel

Glycocoll treatment in myopathic progressive muscular atrophy and creatine exchange. Luigi Perria. *Boll. soc. ital. biol. sper.* 10, 751-2 (1935).—In 6 persons glycocoll treatment produced no beneficial results and no change of creatine bodies toward normal values. H. L. G.

Glucemic, chloremic and proteinemic curves after insulin injection. G. Dell'Acqua. *Boll. soc. ital. biol. sper.* 10, 761-5 (1935).—The subcutaneous injection of 20 units of insulin into normal persons caused a lowering of the glucemic and chloremic curves in the first hr. followed by a rise in the second hr. with subsequent inversion of the glucemic-chloremic ratio in the third hr. When similar injections of insulin were made into diabetics the inversion of the glucemic-chloremic ratio occurred earlier and was more marked. Intravenous injections of 12 units caused still greater differences. Insulin injections into normal persons usually produced an initial diminution in proteinemia followed by a rise and subsequent fall. The curve after insulin injection into diabetics was similar but much more irregular. Helen Lee Gruel

Action of compressed air on animals. XVII. Combustion of ethyl alcohol injected into rats. Alberto Agazzotti. *Boll. soc. ital. biol. sper.* 10, 782-4 (1935).—Combustion of EtOH in rats was slightly higher at atm. pressure than at 6 atm. XVIII. Combustion of ethyl alcohol injected in increasing doses. *Ibid.* 784-6.—The amt. of alc. consumed per 100 g. body wt. increased progressively at normal and at 6 atm. pressure. XV. Oxygen and carbon dioxide in expired air of rabbits subjected to compressed air. G. Buccardi, M. Leonardi and E. Ferrarini. *Ibid.* 787-8.—Increase of pressure increased the no. of respirations but the R. Q. remained practically unchanged. Helen Lee Gruel

Action of thyroxine on the catheptic and amylase power of the liver in guinea pigs. G. Scoz and L. De Caro. *Boll. soc. ital. biol. sper.* 10, 829-8 (1935).—Guinea pigs given 3 injections of 0.3-0.4 mg. thyroxine and killed 2-42 days after the last injection showed an increase of cathepsin and amylase in the liver during the period of diminution in body wt. (4-6 days after injection). Subsequently the enzyme titer fell within normal limits. H. L. G.

Effect of thyroxine on protein sulfur of the liver in guinea pigs. G. Scoz, P. L. Micheli and T. Gualtierotti. *Boll. soc. ital. biol. sper.* 10, 829-30 (1935).—In general there was a decrease in protein S during the first 4-6 days after the last of 3 injections of 0.3-0.4 mg. thyroxine. This was followed by a const. increase. Helen Lee Gruel

Effect of thyroxine on extractable sulfur and vitamin C content of guinea pig liver. G. Scoz and T. Gualtierotti. *Boll. soc. ital. biol. sper.* 10, 830-2 (1935).—The period of diminution in wt. is accompanied by an increase of extractable S and a diminution in vitamin C. During the second stage after thyroxine injection the vitamin C titer increases and the extractable S values return to normal. Helen Lee Gruel

Effect of thyroxine on the phosphatase power of the liver in guinea pigs. G. Scoz and G. Cantoni. *Boll. soc. ital. biol. sper.* 10, 833-4 (1935).—The period of wt. diminution after thyroxine injection is accompanied by a 10% reduction in liver phosphatase. Subsequently this gives way to a marked increase in phosphatase. Helen Lee Gruel

Hematologic-biochemical changes in the blood from neoparsphenamine. I. R. Bakhroneev and L. N. Pavlova. *Arch. Dermatol. Syphilis* 170, 543-9 (1934).—Changes in blood leucocyte count, Ca and K are given for 3 healthy cows injected with neoparsphenamine. O. Hartley

The influence of pharmaceuticals on experimental urticaria sensitization in animals. F. Marquardt. *Arch. Dermatol. Syphilis* 171, 430-9 (1935).—In guinea pigs, the development of hypersensitivity of the skin to urticin (II) can be prevented only by large doses of adrenaline (II) injected simultaneously. II has no effect when given prior to I, after reactions from I have set in, its use, or that of pilocarpine, in small doses, causes exacerbation of symptoms. Atropine, morphine-scopolamine, phenobarbital and thyroglucandol were without effect. O. Hartley

Experimental investigations of arsenphenamine sensitization of the skin. H. Haxthausen. *Arch. Dermatol. Syphilis* 171, 553-9 (1935).—Diazotized arsenphenamine alone or coupled with horse serum produced hypersensitivity to As in all persons tested (4 in each instance). When human serum was substituted for horse serum, sensitization occurred in only 1 person out of 8 tested. O. Hartley

Psoriasis and the suprarenal cortex. II. The influence of cortical extracts and vitamin C on psoriatic conditions. Theodor Grünberg. *Arch. Dermatol. Syphilis* 173, 1-28 (1935), cf. C. A. 28, 1769.—Fifty-eight cases of psoriasis were injected with cortical ext. (Cortidyn, Promonta-Hamburg). Of these, the 12 severest cases showed striking improvement. Two badly crippled arthritic psoriatics left the clinic on foot. No patients reacted unfavorably; the most highly pigmented skins healed fastest. The theory is advanced that there is hypofunctioning of the suprarenal cortex in psoriasis. Expts. with vitamin C failed to give conclusive results. O. Hartley

Disturbances in liver metabolism in arsenobenzene poisoning. Albert Wiedmann. *Arch. Dermatol. Syphilis* 173, 173-80 (1935).—Arsenobenzene causes disturbances in both carbohydrate and protein economy of the liver. A glycogen impoverishment of the latter can be avoided by administration of sugar, according to animal investigations. Seventeen references. O. Hartley

Oral administration of potassium bismuth tartrate in treatment of syphilis. John A. Kolmer. *Arch. Dermatol. Syphilis* 31, 9-14 (1935).—Adults tolerate doses of 0.19-0.4 g. K Bi tartrate 3 times a day, orally, much better than HgCl<sub>2</sub>, Hgl, or Hgl, and it is distinctly curative. Its toxicity for rats and rabbits is extremely low, and its curative value for syphilis in these animals much higher than that of Hg compounds. O. Hartley

Excretion of mercury after oral administration of mercury with chalk, yellow mercurous iodide and corrosive mercuric chloride. Torald Sollmann, Nora E. Schreiber, Harold N. Cole, Henry DeWoll and John V. Amlder. *Arch. Dermatol. Syphilis* 31, 15-25 (1935).—Urinary excretion of Hg when taken orally in doses of 0.2 g. Hg with chalk daily or of 0.016 g. Hgl 4 times daily was found to be essentially the same as that obtained with 4 g. 50% Hg ointment daily by inunction. O. Hartley

Use of maize oil (unsaturated acids) in the treatment of eczema. Preliminary report. Theodore Cornbleet and Earle R. Pace. *Arch. Dermatol. Syphilis* 31, 224-6 (1935).—Eighty-seven patients with chronic eczema were cured or much improved by oral administration of maize oil in 4 1/2 yrs' experience. The theory that eczema is related to the unsatd. fat acid level of the blood is being investigated. O. Hartley

Colloidal sulfur in dermatology. Hiram E. Muller. *Arch. Dermatol. Syphilis* 31, 516-25 (1935).—A review with 24 references. O. Hartley

Fatality from exacerbation of latent tuberculosis due to thioisomol in a case of yaws. C. M. Hasselmann. *Arch. Dermatol. Syphilis* 31, 695-91 (1935). O. Hartley

Reactions due to phenolphthalein. A study of their pathogenesis. E. Wm. Abramowitz. *Arch. Dermatol. Syphilis* 31, 771-92 (1935).—A review covering 43

references. Eruptions are probably due to allergy rather than to cumulative toxic effects or overdose. O. H.

Acne and furunculosis. Preliminary report of treatment with physiologic solution of sodium chloride locally or by intravenous injection. Herman Goodman. *Arch. Dermatol. Syphilol.* 31, 829-30(1935).—Several hundred patients have been treated successfully. Dosage varies from 100 to 200 cc. per injection. Therapeutic action may be due to lowering of the blood sugar. Growth of streptococci and staphylococci is inhibited in 2-3% NaCl solution. O. Hartley

Treatment of vitiligo with gold sodium thiosulfate given intravenously and subcutaneously. Jacob L. Grund. *Arch. Dermatol. Syphilol.* 31, 867-9(1935).—A case of patch depigmentation in a negro was repigmented by Au Na thiosulfate. The normal tissues remained unchanged. O. Hartley

Excretion of mercury after clinical intramuscular and intravenous injections. Torald Sollmann, Nora E. Schreiber and H. N. Cole. *Arch. Dermatol. Syphilol.* 32, 1-48(1935).—Detailed exptl. data are given on Hg excretion after injection of 12 different mercurials. Urinary excretion is an index of diffusible Hg; fecal excretion is negligible except with flumerin. Antisyphilitic efficiency is conditioned on concn of ionized Hg, that fixed in the tissues plays no therapeutic role. In all effective treatment excretion is progressively cumulative. From 31 to 77% of the Hg of diffusible org. mercurials is fixed indefinitely in the tissues, from 9% to 99% of the Hg of colloidal solns. The retained Hg has no therapeutic value and may be harmful. O. Hartley

Mercury injections. Torald Sollmann, H. N. Cole and N. E. Schreiber. *Arch. Dermatol. Syphilol.* 32, 242-57(1935).—Metallic Hg in 50% ointments was found the most effective. Hg oleate was absorbed as well, but colloidal Hg was not. Absorption was measured by detn. of urinary excretion. It was found to be distinctly greater in whites than in negroes. O. Hartley

Modified composition of iodobismutol. Results on local irritation. P. J. Hamzik, C. W. Barnett and A. P. Richardson. *Arch. Dermatol. Syphilol.* 32, 284-7(1935).—Observations on 1174 patients indicate a better tolerance for iodobismutol injections when saligenin is added as a local anesthetic. Propylene glycol is substituted for ethylene glycol as solvent to avoid the cumulative toxicity attributed to the latter. O. Hartley

Role of iodine in the therapy of syphilis. A discussion of its relationship to lipoids. E. T. Burke. *Arch. Dermatol. Syphilol.* 32, 404-12(1935).—Although I has no spirocheticidal value, it should always accompany As or Bi therapy in order to iodinate the unsatd. lipoids which prevent lymphocytic enzymes from having spirocheticidal effect. O. Hartley

Treatment of arsenical hepatitis with sodium dehydrocholate. Experimental and clinical studies in cases of arsenamine poisoning. Bernard Appel and I. R. Jankelson. *Arch. Dermatol. Syphilol.* 32, 422-45(1935).—Toxic hepatitis during syphilis treatment is largely due to liver damage by As. The latter appears in the feces after intravenous injection of arsenamine (I) as a result of excretion from the liver via the gall bladder. Addn. of Na thiosulfate (II) increases the proportion of As excreted by the liver, Na dehydrocholate (III) is still more effective. In expts. on rabbits III increased the ratio of the wt. of the liver to the percentage of I retained more than II. Clinically III promoted recovery from hepatitis and gave increased tolerance for I. O. Hartley

Dimorphophenol in treatment of ichthyosis. Matthew Molitch and Richard F. Cousins. *Arch. Dermatol. Syphilol.* 32, 486-7(1935).—Oral administration for 4 weeks of doses increased progressively to the max. recommended for obese adults failed to produce sweating or loss of wt. in a 108-lb. 16-yr. old boy with ichthyosis. Basal metabolism increased 18% but no effects were produced on temp., blood count, blood sugar, blood urea or urine vol. O. Hartley

Pre-iodine and post-iodine days. A review of 37,228 cases of goiter at the Mayo Clinic. Charles H. Mayo and

1 Charles W. Mayo. *West. J. Surgery Obstet. Gynecol.* 43, 477-82(1935).—In the 15,973 cases of exophthalmic goiter treated since 1892 the mortality in pre-iodine days was nearly 4 times the rate averaged since I therapy was begun. Much of this improvement is due to increased operability induced by preoperative I therapy. O. Hartley

Chemotherapy of bacterial infections. Gerhard Domagk. *Angew. Chem.* 48, 657-67(1935); cf. C. A. 29, 4831<sup>1</sup>.—A very comprehensive treatise including the following subjects: streptococci, staphylococci, pneumococci, tuberculous bacilli, leprosy, coli, typhus, paratyphus, anthrax, erysipelas, Malta fever, Bang's disease, gas gangrene and tropical bacterial infections. Fifty-two references. Karl Kammermeyer

The therapeutic value of inhalation for patients suffering from inflammation of the upper air passages due to inhalation of vapors of dichloroethyl sulfide (mustard gas).

3 H. Osswald. *Gesellschaft u. Luftschutz* 5, 235-7(1935).—Expts. were made with rabbits. The treatment consisted in exposing the poisoned animals to smokes composed of salts of Ce, Ca and Na. Although the treatment did not affect the percentage of recoveries it is claimed that the severity of the effects was considerably mitigated.

A. L. Kibler. Experimental investigation of indelible-pencil poisoning. J. Guerlich. *Deut. Z. ges. gericht. Med.* 25, 156-63(1935).—Methyl violet, the poison of the indelible pencil, created a local necrotic center from which it passed into the circulation. It is a strong protoplasmic poison, destroys erythrocytes, causes methemoglobin formation and results in anemia. The growing organism appeared able to decolorize and detoxify comparatively large amounts without fatal poisoning. Histological examn. presented great similarity to aniline or nitrobenzene poisoning.

4 Frances Krasnow. Atebrin and plasmochin in the treatment and control of malaria. D. L. Seckinger. *Am. J. Trop. Med.* 15, 631-49(1935).—A review. G. H. W. Lucas

5 Allergic shock from local and general anesthetics. G. L. Waldbott. *Anesthesia and Analgesia* 14, 199-204(1935).—Sensitization to local and general anesthetics may produce allergic shock and death when given as an overdose to an extremely sensitive patient. Autopsy findings in sudden death from anesthesia are identical with those of allergic shock from pollen and serum injections. G. H. W. Lucas

Cyclopropane anesthesia. Harold R. Griffith. *Anesthesia and Analgesia* 14, 253-6(1935).—A review of 1108 cases in which cyclopropane has been used successfully. G. H. W. Lucas

Clinical use of vinethene. Frank W. Marvin. *Anesthesia and Analgesia* 14, 257-62(1935).—A review. G. H. W. Lucas

7 Cyclopropane anesthesia in thoracic surgery. E. A. Rovenstone. *Anesthesia and Analgesia* 14, 270-5(1935).—Statistical data recorded in 160 thoracic operations show that certain characteristics of cyclopropane endow it with many advantages in thoracic surgery. Its main disadvantages are its explosibility, which necessitates a special closed technic for its administration, and its high cost. G. H. W. Lucas

8 Leucocytosis following inhalation anesthesia. Ivan B. Taylor and Ralph M. Waters. *Anesthesia and Analgesia* 14, 276-81(1935).—A leucocytosis, reaching a max. in 4 to 8 hrs. after operations and continuing for 2 to 3 days, occurs after the use of such inhalation anesthesia as ether, N<sub>2</sub>O, ethylene and cyclopropane. The return to normal requires from 3 to 5 days. G. H. W. Lucas

9 Experimental convulsions induced by administration of thujone. A pharmacologic study of the influence of the autonomic nervous system on these convulsions. H. M. Keith and G. W. Stavsky. *Arch. Neurol. Psychiatry* 34, 1022-40(1935).—A study of the effects of other drugs on the convulsions produced by thujone showed that sympathetic stimulants such as adrenaline, pitresin and nicotine, as well as histamine, when added to the thujone markedly increased the severity of the convulsions and lowered the min. convulsant dose of the drug.

tolerance to As is increased, permitting the use of larger doses. A. E. Meyer

The therapeutic action of arsenobenzene associated with sodium dehydrocholate. L. Jacchia and G. Truffi. *Arch. Dermatol. Syphilis* 170, 550-71(1934)—Add clinical evidence is presented to show the favorable influence of Na dehydrocholate in As therapy. O. H.

The enterochromaffin cells of the guinea pig and the rabbit after treatment with histamine, pilocarpine and acetylcholine. Vittorio Lrpamer. *Biochim terap sper* 22, 390-4(1935)—The enterochromaffin cells are not stimulated by either of the drugs tested. Conclusion The chromaffin tissue examd does not secrete a substance taking active part in the digestion, since the drugs used stimulate the secretion of digestive juices. A. F. M.

Criminal poisoning with seeds of *Datura* and *Mandragora*. J. Bouquet. *Bull sci pharmacol* 42, 450-9(1935)—Description of two cases. A. F. M.

Physiological investigation of the optical isomers of diethylaminomethylbenzodioxan. D. Boyet and A. Simon. *Bull sci pharmacol* 42, 466-73(1935)—The *l*-compd is twice as toxic to fishes as the *d*-compd. The hypertensive action of adrenaline in cats is inverted by the *l*-compd at  $\frac{1}{2}$ , to  $\frac{1}{3}$  of the quantity necessary for the *d*-compd. The myotic action on the mouse iris is 4 to 8 times stronger in the former compd than in the latter. A. E. M.

Intravenous use of mercury oxyacynide against tenias. F. Prado Moreira. *Publicações med* (Brazil) 6, No. 9 (1935). *Rev sud-americana endocrinol imunol. quimioterap* 18, 634—Two cc of a 1% soln was given with good result. A. E. Meyer

The action of indole and skatole on the isolated intestine. A. M. Michelazzi and G. Bellucci. *Rass terap patol clin* No. 9 (1934). *Rev sud-americana endocrinol imunol. quimioterap* 18, 637—Both substances applied on the smooth muscle of the intestine cause an inhibition of the tonus and the contractions. A. E. Meyer

Treatment of psoriasis with organ extracts. Th. Grüneberg. *Rev méd Germano-Ibero-Americana* 7, Nos. 5 and 6(1934). *Rev sud-americana endocrinol imunol. quimioterap* 18, 650-1—Adrenal cortex and liver exts. give favorable results. A. E. Meyer

Ephedrine in treatment of shock affecting the hepatobiliary system. J. Lauer. *Rev med chir des mal du foie* (July, 1934); *Rev sud-americana endocrinol imunol. quimioterap* 18, 653-4—Ephedrine combines the action of adrenaline and caffeine. It is recommended as a cardiotonic and for treatment of symptoms caused by liver dysfunction as asthma, urticaria, etc. A. E. Meyer

Treatment of diabetes with testicle extract. G. Odio de Granada. *Rev med cir Habana* 40, No. 3(1935). *Rev sud-americana endocrinol imunol. quimioterap* 18, 655—The ext. was prepd. from testes taken from animals after previous ligation of the vas deferens. Treatment by injections caused cure in cases of mild diabetes. A. F. Meyer

Alkali therapy in leprosy and other diseases. Nicolas V. Greco. *Semana med.* (Buenos Aires) 1935, II, 597-604. *Rev asoc med Argentina* 49, 1381-94. A. F. Meyer

Treatment of arterial hypertension with octyl alcohol. Carlos Rossi Belgrano. *Semana med* (Buenos Aires) 1935, II, 1073-80—Intravenous injections of 10-20 cc of a soln. 1:10,000 of octyl alc. were given. The effect is a slight hydemia with decrease of urea and Cl in the blood. The influence on the blood pressure was not very significant; subjective symptoms were relieved, but occasionally fever was observed. A. E. Meyer

Hematological changes caused by intoxication with barbiturates. R. Carratalá. *Soc. med. legal toxicol* July 11(1934); *Rev. sud-americana endocrinol imunol. quimioterap* 18, 675-6—The red cell count is reduced. An increased white count with relative mononucleosis gives unfavorable prognosis. Leucocytosis with accentuated polynucleosis is a favorable sign. Eosinophilia appears a few days after the onset of symptoms. A. F. M.

Absorption and oxidation of alcohol in alcoholics. Carl G. Bernhard and Leonard Goldberg. *Acta Med. Scand.* 86, 152 215(1935)—The blood alc. curve has been studied in several chronic alcoholics and in nondrinking persons. The alc. was administered by mouth in doses of 0.41-0.79 g per kg. The detns were made by Widmark's micromethod. In the post-absorptive phase the blood alc. curve in both alcoholics and in temperate subjects follow a straight line. The av. velocity of oxidation of the alc. in the male alcoholics was  $0.0028 \pm 0.00017\%$  per min and in the female  $0.0028 \pm 0.0001$ . In the abstemious male and female subjects this value was  $0.0024 \pm 0.0001$  and  $0.0026 \pm 0.0001$ , resp. The distribution coeff *r* was  $0.72 \pm 0.02$ ,  $0.64 \pm 0.10$ , and  $0.70 \pm 0.01$ ,  $0.59 \pm 0.01$ , resp. The alc. oxidation per kg and hr. was  $115 \pm 4.4$  mg.,  $108 \pm 13$  mg., and  $93 \pm 3.1$  mg.,  $80 \pm 4.1$  mg., resp. The max. absorption occurs in  $32 \pm 4.5$  min, 30 min, and  $46 \pm 2.3$  min,  $44 \pm 6.8$  min, resp. There is, therefore, no difference from these stand-points between the chronic alcoholic and the abstemious persons. The max. absorption, however, seems to come somewhat earlier in the former group. It is pointed out that single blood alc. curves give no basis for diagnosing chronic alcoholism. S. Morgulis

Results from sanocrysin therapy in nonsurgical joint diseases. Knud Secher and E. Gudiksen. *Acta Med. Scand* 86, 370-95(1935)—Sanocrysin is regarded as an efficient remedy against a series of joint diseases. S. Morgulis

Amidopyrine and agranulocytosis. Knud Brochner-Mortensen. *Acta Med. Scand* 86, 396-406(1935)—Leucocytosis followed by pronounced leucopenia was observed in a polyarthrititis patient treated with amidopyrine. S. Morgulis

Effect of adrenaline on liver cell suspensions. E. Geiger. *Biochem Z* 281, 86-92(1935)—Sedimentation of the proteins pptd by  $CCl_4CO_2H$  in liver cell suspensions is increased through the addn of adrenaline, which apparently so alters its phys. state as to diminish the colloid protective action. S. Morgulis

The effect on gaseous metabolism of poisons paralyzing the vegetative nervous system. F. Peter. *Biochem Z* 281, 111-20(1935)—Ergotamine causes a rise, up to 42%, of the metabolism of the adult rat at the crit. temp. 28°. This effect may last 5-6 hrs. Atropine causes a lowering of the metabolism and Na phenylethylbarbiturate has no effect. The effect of various combinations was also studied. S. Morgulis

Composition of the residual nitrogen in experimental uranium poisoning. Martin Jacoby. *Biochem. Z* 281, 198-9(1935)—In normal rabbits the urea N constitutes 35.7 to 51.9% of the total residual N of the blood. In animals poisoned with  $UO_2(NO_3)_2$ , this fraction is always and very markedly increased (69 to 96%). S. Morgulis

Effect of salyrgan, theophylline and caffeine on diuresis, glomerular filtration and proteinuria. Hilding Berglund and Björn Sundh. *Acta Med. Scand.* 86, 216-32(1935).—Salyrgan may be used in primary renal disease, and no harmful results were noted even in extremely contracted kidneys. Its diuretic action is due to diminished tubular reabsorption. Administration of NaCl accelerates the action of salyrgan, and this may account for the poor results obtained in patients who have been on a salt-free diet a long time. With a given damage to the glomerular structure, the glomerular blood flow is the chief factor detg. the protein loss. Theophylline causes a certain degree of kidney irritation, but there is no evidence that salyrgan has a deleterious effect. S. Morgulis

Behavior of ethynal in the animal and human organism. Shigeru Tsunoo. *J. Biochem* (Japan) 21, 409-16 (1935)—Ethynal (furylacrylyl- $\beta$ -1-hydroxyphenylurea) administered to dogs undergoes hydrolysis to furanacrylic acid and  $\beta$ -hydroxyphenylurea. The former is conjugated with glycine, the latter with glucuronic acid, and both are excreted as these conjugated products. In the rabbit the glucuronic compd. was found but neither the furanacrylic acid nor any of its derivs was found in the urine. In the human organism ethynal is likewise hydrolyzed but no conjugation with glucuronic acid occurs while the furanacrylic acid is changed to pyromucic acid which is eliminated conjugated with glycine. S. Morgulis



Vinyl ether (vinesthene) anesthesia in dogs Wesley Bourne and Bernard B. Raginsky. *Brit. J. Anesthesia* 12, No. 2, 62-9(1935), cf. following abstr.—Liver function was not altered in normal or partially starved dogs. Death in dogs following exposure to vinyl ether resulted from respiratory failure. In general, vinyl ether is a poor anesthetic for dogs. James C. Munch

Vinyl ether obstetrical anesthesia for general practice. Wesley Bourne. *Can. Med. Assoc. J.* 33, 629-32(1935), cf. C. A. 29, 34014 and preceding abstr.—Vinyl ether in concn. suitable to maintain anesthesia for obstetrical procedures does not cause liver damage. Pharmacol. studies show that the musculature of uterus and intestines remains unaffected. It appears safe for both mother and child. As it is extremely volatile it should be given in a closed system with  $O_2$ . Also in *J. Am. Med. Assoc.* 105, 2047-51(1935). G. H. W. Lucas

Poisoning from phenobarbital (luminal). E. P. Scarlett and D. S. Macnab. *Can. Med. Assoc. J.* 33, 635-41(1935)—Rept. of a case of phenobarbital poisoning when the drug was administered under hospital control, together with a review of phenobarbital poisoning. G. H. W. Lucas

Biological testing of trypanamide. L. Launo and M. Friess. *Bull. Soc. Path. Expt.* [5], 28, 289-98(1935); *Trop. Diseases Bull.* 32, 696-7(1935)—Intravenously injected into 5 male rabbits, 3 survived 750 mg/kg in 10% soln for 10 days. Many brands were tolerated in doses of 1250 mg/kg, the LD<sub>50</sub> being 2500 mg/kg. More regular results were obtained by using a 20-day limit instead of a 10-day limit. In tests upon 20 g mice, 80% survived 60 mg/mouse for 10 days, 50% survived 90 mg, and 38% survived 100 mg. Somewhat smaller percentages survived a period of 30 days. James C. Munch

Further observations on the vessels and nerves of the rabbit ear, with special reference to the effects of denervation. R. T. Grant, P. D. Camp, Ashton Graybiel and Paul Rothschild. *Clin. Sci.* 2, No. 1, 1-33(1935)—The recovery of vascular tone was due to an increased responsiveness of the denervated vessels to stimuli, including an adrenaline-like substance circulating in the blood stream. The source of this stimulus is unknown, but is not the adrenal gland nor the pituitary gland. The concn. of this adrenaline-like substance is increased by nervous or muscular activity, and is reduced by rest. It may play a part in the maintenance of normal tone and temp. increase may produce pallor and hyperthermia. James C. Munch

The manner in which necrosis arises in the fowl's comb under ergot poisoning. Sir Thomas Lewis. *Clin. Sci.* 2, No. 1, 43-56(1935)—Intramuscular injections of 10 mg of ergotamine as ethane sulfonate were made daily to white leghorn hens. After a single dose recovery was complete in 2 to 3 days. After daily injections the effects range from cyanosis to stasis to necrosis. Ergot vasoconstriction does not lead directly to tissue necrosis but to damage of the vessel walls, which is followed by dilatation, stasis or thrombosis, thus producing dry necrosis. Maintained arterial constriction produces serious nutritional changes in the endothelium, loss of plasma, capillary stasis, vascular clotting and necrosis. Mech. occlusion or clamping produces all of these changes. James C. Munch

Dinitrophenol poisoning. N. L. Beebe. *Colorado Med. J.* 32, No. 1, 30-31(1935)—A woman, aged 32, showed high fever and rash after taking one 1/16 grain capsule daily for 3 days, then 2 capsules daily for 3 days more. James C. Munch

A case of quinine hemoglobinuria. R. N. Chopra, B. Sen and S. N. Bhattacharya. *Indian Med. Gaz.* 70, 453-4(1935)—A Hindu girl, aged 11, excreted black urine within 4 hrs. after the administration of quinine for the treatment of malaria. The administration of 6 grains of quinine orally produced black urine for 3 days. The administration of 0.1 g of atabrin 3 times and also of 0.0099 g of plasmochin did not produce black urine, but 3 daily administrations of 3 g. of quinine subsequently

produced black urine once more. Blood examn. showed increased fragility of corpuscles, decrease of serum albumin, with increase in Ca, cholesterol, euglobulin and pseudoglobulin. Hemoglobinuria depends upon factors other than quinine. J. C. Munch

Sodium thiocyanate as a prophylaxis and in the treatment of bacillary dysentery with special emphasis upon the shiga type. L. D. Massey. *J. Ark. Med. Soc.* 31, No. 12, 216(1935)—In the treatment of 73 cases, 1 to 3 intravenous injections of 20 mg of NaSCN per kg were given, with a mortality of 13.7%. Oral administration of 20 mg/kg daily for 3 days was effective in prophylaxis. James C. Munch

Recent cardiovascular therapy. J. Curtis Lyter. *J. Missouri State Med. Assoc.* 32, No. 4, 138-41(1935)—Lacornel or padutin, combined with supervised exercise, was useful in the treatment of 21 cases of angina pectoris. James C. Munch

What may we expect of dinitrophenol? O. S. Jones. *J. Missouri State Med. Assoc.* 32, No. 5, 106-7(1935)—Clinical studies have shown that 10% of patients receiving dinitrophenol show urticaria or some untoward effect. A period of 4 weeks is necessary to establish a lack of sensitivity. Ice packs and inhalation of  $O_2$  decreased toxicity of dinitrophenol for animals. James C. Munch

The use of dilaudid in general practice. Clifford M. Bassett. *J. Oklahoma State Med. Assoc.* 28, No. 5, 178-81(1935)—Dilaudid was 5 times as potent as morphine in relieving coughs, but did not produce nausea, constipation, vertigo or anuria. James C. Munch

Agranulocytosis with report of a case. B. H. Teasdale. *Tri-State Med. J.* 7, No. 12, 1514-16(1935)—Agranulocytosis has been observed after treatment with atabrin and plasmochin for Vincent's angina. For antidotes, liver ext. and thiosulfonate were employed. J. C. M.

Chemical injury of cornea in the newborn with report of experiments. Sidney Trattner. *Virginia Med. Monthly* 62, No. 3, 163-5(1935)—Topical application of 10%  $AgNO_3$  produced negligible corneal irritation, but phenol produced definite injury. Recovery was complete after 2 months. James C. Munch

Chronic lead poisoning in early childhood. H. H. Donnelly, C. A. Schutz and A. Nimetz. *Virginia Med. Monthly* 62, No. 2, 83-9(1935)—Chronic lead poisoning increases the density of the ends of rapidly growing bones. Three cases of chronic lead poisoning in children under 5 yrs. of age were diagnosed by x-ray studies of this factor. James C. Munch

The acute narcotic action of aliphatic and aromatic hydrocarbons. I. The action of a single inspiration of different concentrations of benzene, benzene, toluene and xylene on rabbits and cats. Wilhelm L. Englehardt and Wilhelm Estler. *Arch. Hyg. Bakt.* 114, 249-60(1935)—Cats are more sensitive to hydrocarbons than rabbits except that the rabbits are almost as sensitive toward xylene. In low concn. the homologs of  $C_6H_6$  bring about light narcosis earlier than  $C_6H_6$  and are also more toxic in higher concns. (100 mg/l.)  $C_6H_6$  seems to be more toxic than xylene. Benzene in concn. of 200 mg/l. is only slightly active. II. The action of repeated inspiration of different concentrations of benzene, benzene, toluene and xylene on the white mouse. W. Estler. *Ibid.* 261-71—No characteristic action of benzene could be shown within the limits of the concns. used. In general the homologs of  $C_6H_6$  were more toxic than the  $C_6H_6$ . Results are summarized in tables. Felix Saunders

Strychnine poisoning or tetanus? Hans Bludau. *Arch. Hyg. Bakt.* 114, 287-303(1935)—A report of an unusual case of tetanus which simulated strychnine poisoning. Felix Saunders

Goutier prophylaxis with iodized salt. Roy D. McClure. *Science* 82, 370-1(1935)—The general use of iodized salt in Michigan began in 1924. The first effect was an increase in the no. of thyroid operations and the death rate from gout which reached its peak in the 2nd year. The increase was in the nodular gout or adenoma group and except for this group there was no increase in hyperthyroid-

ism. The no. of operations for toxic diffuse and toxic nodular goiter rapidly and steadily decreased after the apex of the 2nd year increase had been reached. The incidence of endemic goiter or enlarged thyroid has been reduced to almost nil since iodized salt has been widely used and cases showing ill effects from the use of iodized salt are no longer seen.

**Felix Saunders**  
The nature of the barbiturate-picrotoxin antagonism Charles R. Linegar, James M. Dille and Theodore Koppanyi. *Science* 82, 376-7 (1935).—The findings of Maloney and Tatum (*C* 26, 3301) have been confirmed. The antidotal effect of different doses of picrotoxin was manifested in 4 different ways. (a) Occasional rise in blood pressure, (b) prevention of the steep fall in blood pressure and hastening the recovery from the fall produced by intravenous barbiturate injection, (c) stimulation of respiration when stoppage was produced by barbiturates, (d) maintenance of respiration after barbiturate injection even after cardiac stoppage. The action of small doses of adrenaline and ephedrine was especially marked after picrotoxin in the barbiturized animals.

**Felix Saunders**  
The toxicity of local anesthetics György Gömöry *Magyar Orvosi Arch* 36, 223-36 (1935).—Procaine, tucocaine and nupercaine in small doses do not produce fatal collapse in guinea pigs, excessive doses do

**Henry Tauber**  
The sensitivity of animals to histamine István Karády *Magyar Orvosi Arch* 36, 244-50 (1935).—Three  $\times$  1 mg of histamine, injected subcutaneously daily for 1 or 2 weeks, caused the animals to become more resistant to histamine. This may be due to an increased histaminase production.

**Henry Tauber**  
The action of narcotics on the brain of white rats György Mayer *Magyar Orvosi Arch* 36, 251-6 (1935).—Ether, chloroform and EtCl diminished the respiration of the cortex cells (Warburg method). Evipan (barbiturate), however, had no effect on these cells but it affected the white substance, especially the thalamus. When fatal narcosis was produced the respiration of cells of the medulla oblongata was diminished by ether and chloroform more than by evipan.

**Henry Tauber**  
The chemistry of the blood in ammonia poisoning Gyula I. Fazekas *Magyar Orvosi Arch* 36, 285-95 (1935).—In rabbit blood in exptl.  $\text{NH}_3$  poisoning Ca, alkali reserve and  $p_{\text{H}}$  are decreased; inorg. P and glucose are increased.

**Henry Tauber**  
The effect of small doses of copper upon erythropoiesis János Somogyi *Magyar Orvosi Arch* 36, 317-26 (1935).—A single intravenous injection of  $\text{CuSO}_4$  (0.3-0.6 mg of Cu per kg.) into rabbits increases erythropoiesis. The number of red cells rises 16-39.2% and the hemoglobin 6.3-24.4% above the normal. The increase is maintained for 18 days. In hemorrhagic anemia copper hastens regeneration of blood. Numerous references are given.

**Henry Tauber**  
Pharmacological study of an alkaloid isolated from Chinese fengchianghi. Li-Pin King and Yuan-Kao Shih. *Bull. Natl. Acad. Peking* 6, No. 3, 13-50 (1935); cf. *C* A. 30, 726<sup>1</sup>, 727<sup>1</sup>.—From Chinese fengchianghi, of the family Menispermaceae, bought from the Shanghai market, an alkaloid (*fengchirin*) is isolated by leaching with slightly acidified 70% alc., then concg. under vacuum, treating with strong alkali and extg. with ether. The yield of this alkaloid, after several purifications through the hydrochloride, is 6 g. from 1 kg. of raw material. Pure fengchirin forms colorless radiating crystals, is bitter and turns yellowish in contact with air. It is insol. in water, but sol. in abs. alc. and easily sol. in ether and acetone; it m. 218-218.5° and has  $[\alpha]_D^{25} +268.7^\circ$ . Fengchirin-HCl is similar to other alkaloids of Menispermaceae in physiol. action, and the strongest of all of them. The minimum lethal dose of this compd. for rats is 0.5 mg. per g. body wt. when injected under the skin; that for rabbits, 18 mg. per g. body wt. when injected intravenously. A large intravenous dose causes an immediate lowering of blood pressure. Small doses excite respiration centers and increase rate and depth of respiration. Medium doses cause excitation and deepening of respiration at first,

and then paralysis and weakening of respiration. Lethal doses cause at first a momentary weakening of respiration, then a deepening, and finally cessation of respiration. Small doses have little effect on the heart, but large doses are decidedly harmful. Fever caused by typhoid germs can be relieved by fengchirin-HCl.

**C. L. Tseng**  
Cosmetics and skin irritation. I. Effect of synthetics on the skin Frank Atkins *Pharm J* 134, 669 (1935).—Aside from the factor of idiosyncrasies, Albeck's irritation index (*Aromatics*, Sept., 1931) is a useful general guide. This index (the degree of diln. producing an effect of definite, tolerable intensity) varies from 4 for esters to 24 for aliphatic and 37 for aromatic aldehydes. The index should not be above 6 (alcs = 5) and should never exceed 10 (terpenes). With benzylidene acetone (not examd by Albeck) an index of about 12 (ketones) is expected; but it is one of the strongest irritants, as a perfume of the sweet-pea type it can be used only on fabrics. The mellowness of the rose and similar perfumes is connected with a low index on the basis of their chem. compn. Possibly the aldehyde problem for creams can be solved by conversion into acetals.

**S. Waldbott**  
II Influence of the medium and other factors *Ibid* 135, 346.—Alky. and rancidity in creams may cause irritation of the skin. To avoid alky. it is recommended to det.  $p_{\text{H}}$  after manu. and again after storage. To avoid rancidity, hydrocarbons, hydrogenated oils and nonoxidizable waxes should form the basic materials. Colors are also factors in skin irritation.

**E. D. Walter**  
Survival and increase of adrenaline in tissue cultures of adrenal glands from chick embryos Margaret R. Lewis and E. M. K. Geiling *Am J Physiol.* 113, 529-33 (1935). cf. *C* A. 23, 869.—The presence of adrenaline was demonstrated by blood-pressure changes following injection of exts. into cats under phenobarbital anesthesia, by dilatation of the pupil in frogs, and by the FeCl<sub>3</sub> reaction. Exts. of adrenals of chick embryos under 7 days old had little effect on blood pressure. Exts. of adrenals of chick embryos grown 3 days in tissue cultures showed a definite increase in the amt. of adrenaline. In embryos at different stages (5-14 days) progressive increases of adrenaline occurred.

**E. D. Walter**  
Reversible loss of the all or none response in cold-blooded hearts treated with excess potassium. Geo. H. Zwister and T. E. Boyd. *Am. J. Physiol.* 113, 560-7 (1935).—The ventricle of the turtle or bullfrog heart, soaked in modified Ringer soln. contg. 0.2-0.4% KCl, shows perfectly graded mech. responses to graded stimuli. The ventricles take up K from the soln. Normal response to stimulation is restored when the preps. are soaked for a time in normal Ringer soln.

**E. D. Walter**  
The influence of peptones and certain extracts of the small intestine upon the secretion of succus entericus. E. S. Nasset and H. B. Pierce. *Am. J. Physiol.* 113, 568-77 (1935); cf. *C* A. 29, 6297<sup>1</sup>.—A potent secretagog was extd., free of vasodpressor substances, from Witte's peptone with 70% EtOH. It is digested by erepsin or trypsin, and is thermostable. When given intravenously to dogs with denervated jejunal transplants, it may cause a 3-5 fold increase in the vol. of juice secreted and a 2-8 fold increase in enzyme production. Five other peptones showed little or no secretagog activity. Small intestines from dogs and swine, extd. with 70% EtOH acidulated with HCl, yield an ext. which stimulates intestinal secretion when given by vein. It is thermostable and resists ereptic and tryptic digestion. The vasodpressor and the secretion-promoting substances of these exts. are not identical because the latter is destroyed by boiling in alkali, while the former are scarcely affected.

**E. D. Walter**  
The effect of ergotamine upon glucosuria and hyperglucemia produced by stimulation of the superior cervical sympathetic ganglion David A. Cleveland *Am. J. Physiol.* 113, 592-4 (1935).—Ergotamine inhibits the action of the sympathetic nervous system. Smaller doses usually prevent and larger doses are more certain to prevent glucosuria and hyperglucemia following stimulation of the superior cervical ganglion in cats which have a sufficiently high glycogen reserve.

The clearance, extraction percentage and estimated filtration of sodium ferrocyanide in the mammalian kidney. Companson with inulin, creatinine and urea. Donald D. Van Slyke, Alma Miller and B. T. Miller. *Am. J. Physiol.* 113, 611-28(1935), cf. *C. A.* 29, 4810<sup>1</sup>.—In the dog  $\text{Na}_2\text{Fe}(\text{CN})_6$ , inulin and creatinine show the same excretory behavior in the following respects: Their plasma clearances are approx equal. Their percentage extn from the plasma are approx equal, averaging approx 20. Their clearances and extn percentages are independent of plasma concn. The av urea clearance is 0.57 of the clearances of the above 3 substances, and is independent of plasma concn. These observations appear to support the filtration reabsorption theory of renal excretion. The distribution of ferrocyanide, inulin, creatinine and urea in the blood and its effect on the significance of their extraction percentages. *Ibid.* 629-41.— $\text{Na}_2\text{Fe}(\text{CN})_6$  and inulin injected intravenously into a dog circulate in the plasma without entering the erythrocytes in measurable amts. Creatinine absorbed or injected into the circulation gradually enters the blood cells. Urea diffuses so quickly that when added to blood it reaches equil between cells and plasma before they can be sep'd for analysis. The effects of these diffusibility differences on the relative concn. decreases of these substances in plasma and whole blood during perfusion of the kidneys have been calcd. E. D. Walter

## I—ZOOLOGY

R. A. CORTNER

Adrenaline in the venom of *Bufo arenarum*. Venancio Deulofeu. *Z. physiol. Chem.* 237, 171-2(1935).—Adrenaline was isolated in cryst. form from the venom of the Argentine toad, *Bufo arenarum*, and identified by m. p., optical rotation and the usual color reactions.

A. W. DOW  
Chloride and total osmotic pressure in the blood of marine teleosts. Allan L. Grafflin. *Biol. Bull.* 69, 245-58(1935).—Plasma chloride in pollack, cod, sculpin, flounder, mackerel and conger ranges from 150.6 to 172.6 millimols. per l. There is no correlation between chloride

concn. and f.-p. depression for the whole blood ( $\Delta$ ). Both chloride and  $\Delta$  are higher in fish bled after delay in landing or rough handling. L. W. Elder

Osmotic pressure and molecular weight of hemerythrin of *Sipunculus nudus*. Andr  e Roche and Jean Roche. *Bull. soc. chim. biol.* 17, 1494-1508(1935).—See *C. A.* 29, 2006<sup>1</sup>. L. E. Gilson

Acetylcholine in invertebrate tissues. Z. M. Bacq. *Compt. rend. soc. biol.* 120, 243-5(1935).—No acetylcholine was found in ascidians, coelenterates or porifera (entire animals) or in the blood of any other marine invertebrate. Certain crustacean tissues contained traces, and tissues of various mollusks, marine worms and echinoderms contained 0.4-1.4  $\gamma$  per g. The cerebral ganglions of *Octopus vulgaris* contained 77  $\gamma$  per g. The nerves of mollusks, worms and echinoderms are probably cholinergic. Identification of acetylcholine extracted from the ganglions of *Octopus vulgaris*. *Ibid.* 246-7.—Crystals of the chloroaurate were obtained. L. E. Gilson

Choline esterase in invertebrates. Z. M. Bacq. *Compt. rend. soc. biol.* 120, 247-8(1935), cf. *C. A.* 29, 6017<sup>1</sup>.—Choline esterase is present in the blood and tissues of marine mollusks, worms, holothurids and ascidians. It is not present in crustaceans, insects, coelenterates and sponges exclud. so far. Its absence in crabs explains their insensitivity to acetylcholine and also indicates that their motor nerves cannot be cholinergic. L. E. Gilson

Attempted treatment of amebiasis in snakes. J. Rodhain and M. T. van Hoof. *Compt. rend. soc. biol.* 120, 523-5(1935).—Rivanol and emetine-HCl were ineffective in snakes (*Tropidonotus natrix*) infected with *Entamoeba invadens*. L. E. Gilson

Presence of vitamin A and carotenoids in the frog. Charles Rand. *Biochem. Z.* 281, 200-5(1935).—Considerable quantities of carotenoids are present in various organs (liver, skin, kidney, lung, ovary, ova, oviducts, testes and fat bodies) of summer or winter frogs. The carotene is almost always accompanied by variable amts. of xanthophyll. The highest percentage of carotene was found in the liver and fat bodies. Vitamin A was found regularly in the liver of both summer and winter frogs. S. M.

## 12—FOODS

F. C. BLANCK AND H. A. LEPPER

Problems of chemistry in the new Germany. XII. Biology in food conservation. W. Schwartz. *Angew. Chem.* 48, 629-32(1935), cf. *C. A.* 29, 4896<sup>1</sup>.—A general discussion with tables on the methods of food conservation, the application of the most important processes of conservation and the ripening processes and decomposition by microorganisms in refrigeration. Nine references.

Karl Kammermeyer  
Sifting and mixing machinery for foodstuffs. H. Thomas. *Food S.* 93-7(1935). A. Papineau Couture

Presence and identification of silicic acid in various foodstuffs. R. Stroecker, R. Vauhel and K. Breitwieser. *Z. Unters. Lebensm.* 70, 345-53(1935).—The most important details of the step-photometric method for the detn. of  $\text{SiO}_2$  are given. Thirteen samples of milk were examd., and the  $\text{SiO}_2$  content was found in every one to be less than 1 mg. per l. Milk concentrates contained relatively more  $\text{SiO}_2$ , which must have had its source in the method of prep'n. Under certain conditions it is possible from a  $\text{SiO}_2$  detn. to adduce that milk has been watered. The  $\text{SiO}_2$  has been detd. in different types of meat, in internal organs and in the most important kinds of sausages, and these results have been compared with data found in the literature. The detn. of  $\text{SiO}_2$  can be used for the detn. of added  $\text{H}_2\text{O}$  to minced meat, but not for sausages, for spices bring about a significant increase in the  $\text{SiO}_2$ . A whole series of different types of cacao beans has been studied for their  $\text{SiO}_2$  content. The cacao shell contains 2-10 times the  $\text{SiO}_2$  found in the kernel. The  $\text{SiO}_2$  found in the fat free bean (dry basis) averages somewhere in the neighborhood of 1 mg. per g. The preservation of eggs

in water glass does not cause an increased  $\text{SiO}_2$  of the inner portions of the egg, but only of shell. As compared with this, hmed eggs produced an increased alkali of the egg white. Twenty-three references. F. L. Dunlap

The nature of food poisoning. S. Lancelfield. *Food S.* 125-6(1935). A. Papineau Couture

Changes in stored corn meal. Charles O. Willets and Frank J. Kosokis. *Ind. Eng. Chem.* 27, 1491-6(1935).—Corn meal can be stored for a long period of time without change in the crude fat content. (a) When the moisture content is 14% or higher, the storage temp. must be maintained at 18° or lower; (b) with a moisture content less than 8%, the storage temp. may be as high as 37°. The "degrees of acidity" of corn meal do not indicate changes in crude fat and therefore cannot be used as an index to crude fat losses. F. L. Dunlap

Varietal differences of wheats with reference to the ash content in grain and flour. M. I. Knyaginichev. *Bull. Applied Botany, Genetics, Plant Breeding* (U. S. S. R.) Ser. III, No. 5, 169-201(1935).—A max. ash content is found in grain grown under conditions of sufficient moisture and strong insolation. The ash content decreases with a shift toward the more arid regions. The winter varieties of wheat contain 13-14% less ash than the soft and hard spring wheat varieties. The flour of winter wheat contains 25% less ash than that of soft spring wheat. The ash content of flour does not necessarily correlate with its cellulose content. In general, however, a variety with a high cellulose content has a high ash content. The index for the milling qualities may be judged from the cellulose content. J. S. Joffe

**Fermentable carbohydrates of wheat flour and the fermentation of dough.** R. Genfroy. *Bull. soc. chim. Ind.* 17, 1351-71 (1915).—The flours examd. contained 0.5% or less of sucrose. This is less than reported by other workers. Less than 1%  $\alpha$ -glucose, and 0.1-0.15% glucose were also present. Maltose and probably more glucose were formed in the dough by the enzymes present in the flour. The amylase content is greater in the latter grade flours. Flour contains a fermentation factor "7" or something very similar. Fourteen references. L. E. G.

**The microbial content of soft wheat flour.** D. Frank Holtman. *J. Bact.* 30, 379-61 (1935).—Overbleaching of flour may permit microbial or atm. oxidation causing it to darken in color. John T. Myers.

**Biochemistry of bread making. The carbohydrate sequence.** C. H. Bailey and R. C. Sherwood. *Ind. Eng. Chem.* 27, 1426-30 (1935).—Bread making is a dynamic process in which various organisms and tissues, and numerous enzymes are involved. Flour from normal wheat contributes starch, sucrose, a trace of glucose, and the saccharifying enzyme  $\beta$ -amylase. Flour milled from germinated wheat, or active malt exs., contributes  $\alpha$ -amylase, the starch-hydrolyzing enzyme. In dough or flour suspensions to which small amts. of wheat or barley malt have been added, the rate of saccharification is greatly increased. The effect on sugar production is substantial and prompt. A small proportion of wheat malt flour (1%) approx. doubled the rate of sugar (maltose) production, when added to either a low or high diastatic flour. The optimum condition in a dough is approached when fermentation rate and saccharogenesis are so adjusted as to maintain a const. level of sugar in the dough from the time it is mixed until it goes to the oven for baking. This condition tends to impart stability and fermentation tolerance which in turn implies that, at the close of the fermentation period, the fermentation shall be vigorous and that there shall be an adequate residue of sugar to result in a brown crust and a suitable sweetness of flavor in the baked loaf. F. L. Dunlap.

**Bakers' yeast.** Englebert Rosenbaum. *Z. Untersuch. Lebensm.* 70, 366-78 (1935).—A general discussion. Some tabular data are given as to moisture losses at ordinary temps. and in cold storage, difference in fermentation time with the same yeast but with varying amts.; difference in fermentation time with the same yeast but the yeast contg. varying amts. of moisture, influence of cold on the stability and the fermentation time of bakers' yeast; difference in the fermentation time with the same yeast but with different good flours. F. L. Dunlap.

**Foreign bakers' yeast.** Englebert Rosenbaum. *Z. Untersuch. Lebensm.* 70, 378-83 (1935).—An examn. of 26 samples of yeast from 13 European countries. Analytical data are given for moisture, N, protein and  $P_2O_5$  (in the dry substance), acidity and stability at 35°. A table is also presented of the properties of these yeasts, such as color, odor, consistency and fermentation time. F. L. Dunlap.

**The determination of chlorides in milk.** A. Mavrot and H. Lestra. *Bull. soc. pharmacol.* 42, 523-6 (1935).—(1) The method of Deméché becomes more accurate when the  $HPO_4$  is transformed into  $H_2PO_4$  by boiling with  $AgNO_3$  and  $HNO_3$  before titration. (2) Laudat's method is modified as follows: Mix 10 cc. milk first with 5 cc. 0.1 N  $AgNO_3$  soln. and then add 20 cc. satd.  $KMnO_4$  soln. Heat to boiling; add 40 cc.  $HNO_3$  and heat until a clear liquid is obtained. (3) To 60 cc. of a mixt. of 3 parts  $FeOH$  and 1 part  $Me_2CO$  add, drop by drop, 10 cc. of milk and fill to 100 cc. with the same mixt. Filter, mix 75 cc. with 5 cc.  $HNO_3$  and titrate with  $AgNO_3$  in the usual way. The last method is the quickest and most accurate. A. E. Meyer.

**The substitution of methylene blue thiocyanate for methylene blue chloride in the reduction test of milk.** H. R. Thornton, R. B. Sandin and C. S. Miller. *Can. J. Research* 13B, 258-63 (1935); cf. *C. A.* 29, 8159.—Methylene blue thiocyanate has been found to be superior to methylene blue chloride in the reduction test for bacteriol. quality of milk, because the thiocyanate is

easily prepd. in a state of purity approaching 100%. It reduces over the same oxidation-reduction potential as the chloride and has approx. the same reduction time and the same poisoning and lethal effects. J. W. S.

**Production of artificial cream and sour cream.** V. Bochkov and V. D'yachenko. *Maslobolno Zhivoe Delo* 11, 359-70 (1935). Production of artificial milk products at margarine works. V. Bochkov. *Ibid.* 451.—Cream with 25-30% fat content was obtained by centrifugal emulsification of a mixt. of 75 parts of skim milk and 25 parts of mixed compd. fat and sunflower oil and then converted to sour cream and cheese. Chas. Blanc.

**Differentiation of butter from pasteurized and non-pasteurized cream by aid of the peroxidase reaction.** L. Waters and A. Zürn. *Z. Untersuch. Lebensm.* 70, 353-6 (1935).—Place a piece of butter the size of a hazel nut in a test tube, add 2 cc. of a satd. soln. of  $MgSO_4$  and 10 drops of a 4% alc. benzidine soln. Shake to make a homogeneous mixt. Add 5 drops of 3%  $H_2O_2$  and, after shaking, add 5 cc. of  $I_2O$  and again shake until the butter fat has completely dissolved in the  $I_2O$ . Let stand and observe the color of the aq. layer. If a blue color develops at once or within a half hr., the butter has been made from cream which has been subjected to a temp. not over 80°. Occasionally this color may be greenish instead of blue, but this is not significant for butter made from pasteurized cream always gives a pure white color in the aq. layer. A neg. color result in a test cannot unconditionally be due to butter from pasteurized cream, for it has been found that older country butters fail to respond. Fresh country butters, however, respond to this test. Very rancid and strong tallowy butters fail to respond to the test. A neg. color reaction, therefore, cannot be taken as abs. assurance of pasteurization, for the peroxidase can be destroyed not only by heat but also by storage. F. L. Dunlap.

**The flavor of butter.** H. M. Langton. *Food* 5, 95-100 (1935).—A brief review of present knowledge of Ac, its presence in butter and methods of detg. it, of defects in flavor of butter and of the chemistry of rancidity in butter. A. Papineau-Couture.

**Control of production of margarine works.** A. Siyazhkina. *Maslobolno Zhivoe Delo* 11, 431 (1935); cf. Sandomirskii, *C. A.* 29, 81617.—Methods of control of various operation steps of processing are discussed. Chas. Blanc.

**Moisture contents in margarine in relation to the churning temperature.** V. Bulin-Sokolov and A. Kopteva. *Maslobolno Zhivoe Delo* 11, 378-9 (1935).—To obtain margarine from cottonseed-oil fat mixt. and cottonseed oil with a standard moisture content, the churning should be carried out at 2° below the m. p. of the fat base. Chas. Blanc.

**Effect of the degree of aging of cheeses on their fat content.** Ch. Brioues and Ldg. Jouris. *Ann. Jais.* 28, 535-7 (1935).—Analyses of a lot of Camembert cheese immediately after manuf., and after 3 weeks and 2 months, gave the following results: total solids by drying in vacuum at atm. temp. 43.43, 40.88, 65.87; total solids by drying in air at 100° 43.11, 19.40, 64.61; total solids by drying at 105° 43.10, 49.20, 64.31; fat 20.31, 22.80, 32.55; fat on dry basis 47.19, 47.20, 50.61; total N 2.90, 3.38, 4.21; insol N (Trillat method) 2.65, 2.26, 2.63; sol. N 0.25, 1.11, 1.65; ammonia N —, 0.55, 0.85; sol. N as percentage of total N 8.64, 32.92, 33.00%. The loss of N during drying at 105° of the fresh cheese and also at the end of 3 weeks and 2 months was 0.05, 0.13 and 0.22%, resp., though the amt. of nitrogenous matter lost on drying cannot be calcd. owing to the complexity of the products formed, the results clearly indicate that they are not the sole factor that accounts for the increase in fat on a dry basis, but that there are very probably other losses caused by the volatilization of volatile acids or other nonnitrogenous compds. A. Papineau-Couture.

**Changes in meat proteins, determined by their digestibility with pancreatin.** I. A. Smorodintzev and I. N. Laskovskaya. *Z. Untersuch. Lebensm.* 70, 355-65 (1935).—1 or the complete activation of 10 mg. pancreatin,

a mixt. of 1 cc enterokinase soln with an equal vol of glycerol is necessary. A neutral enterokinase soln. with glycerol 1:1 can be preserved in the cold without change for some time ( $1\frac{1}{2}$  month). Various animals furnish activator preps of the same strength. Dry pancreatin preps made according to the methods of Willstätter and Waldschmidt-Leitz (C. A. 18, 91) retain their activity for a long period ( $1\frac{1}{2}$  year). The optimal conditions for meat digestion by means of pancreatin were obtained with 1 g meat, heated to  $37^\circ$  for 1 hr. The degree of digestibility of meat one hr after slaughter by pancreatin is of const magnitude, corresponding to an increase in acidity of 1.3 cc 0.2 N KOH. The max. digestibility of meat is found in the sixth hr after slaughter and is independent of the temp at which the meat has been held. The meat digestibility after the sixth hr following slaughter is less than it was during the first hr. The mean increase in digestibility is const. for different animals. An increase in the storage temp. of meat from  $1-3^\circ$  up to  $37^\circ$  accelerates protein changes, but in no way alters the character of the curve.

F. L. Dunlap

Carbon dioxide in handling fresh fish. Maurice E. Stansby and Francis P. Griffiths. *Ind. Eng. Chem.* 27, 1452-8(1935); cf. C. A. 30, 1761. Whole haddock, stored in  $\text{CO}_2$  from the time they were caught until they are unfit for food, keep approx. twice as long as those stored in air. Haddock which are just passing out of rigor mortis are benefited if stored in the presence of  $\text{CO}_2$ , a pronounced difference existing after 4-day storage in ice in favor of the gas-stored fish over those stored in air. Haddock which are in rigor mortis are not greatly benefited by  $\text{CO}_2$  storage, as long as rigor exists. Fillets maintained in a  $\text{CO}_2$  atm will be of better quality than those kept in air, especially after prolonged storage. If the best sanitary conditions are used during filleting, the use of  $\text{CO}_2$  will be of the greatest benefit. The use of hypochlorite solns will not offset careless handling of the fillets.

F. L. Dunlap

Some chemical changes exhibited in sterile and in contaminated haddock muscle stored at different temperatures. J. T. R. Nickerson and B. E. Proctor. *J. Bact.* 30, 383-94(1935), cf. C. A. 29, 1171. The pH of sterile samples decreased while that of contaminated muscle increased. There was an increase in amino acid followed by a decrease in sterile muscle, while contaminated muscle showed an increase particularly toward the latter part of the storage period, probably because of bacterial action. This will eventually cause spoilage; hence low-temp. storage is essential.

John T. Myers

Chemical composition of pig stomach. I. A. Smorodintzev and V. V. Palmin. *Z. Untersuch. Lebensmittel* 70, 385-6(1935). Pig stomach is a N-free foodstuff. Its fat content is small, but shows great variation. The ash content of the stomach is less than that of other organs. A tabular result is given of the analysis of 15 samples of pig stomachs, the data being the av. value, max and min, and variations from the av. values for the following detns: moisture, total N, protein N, residual N, fat, extractives and ash. Another table gives the moisture, N substance, fat, ash and calorie value of the pig stomach as compared with other organs of the pig, such as heart, lungs, liver, kidneys and spleen.

F. L. Dunlap

Quantitative estimation of vitamins A and D in food substances, cooked and fresh. Katharine H. Coward and Barbara Morgan. *Brit. Med. J.* 1935, II, 1041-4. Inconsistencies in the methods of detg the vitamin content of foods are discussed. The use of the international standard is recommended. The vitamin A content of several foods has been detd in comparison with a cod-liver oil which in turn was compared in 9 different labs, with standard carotene. This standard oil was likewise employed in detg the vitamin D content of certain foods. Boiling vegetables did not decrease their vitamin A content. Boiled carrots, cabbage and runner beans were shown to be valuable sources of A, their potencies being  $1\frac{1}{2}$ ,  $1\frac{1}{2}$ , and  $1\frac{1}{2}$ , resp., of an av. sample of summer butter which contains 60 units/g. The following vitamin D contents of several foods in international units were re-

ported: calf liver 0, milk 0-0.1/cc.; butter 0.4-4.0/g.; egg yolk 1.5-5.0/g.; cod-liver oil 60-300/g.; halibut-liver oil 2000-4000/g.; olive oil 0. J. B. B.

The vitamin C content of Indian fruits. J. P. Spruyt and W. F. Donath. *Geneeskund. Tydschr. Nederland. Indie* 75, 1944-50(1935).—The 2,6-dichlorophenolindophenol titration method was used for the detn of ascorbic acid in some native fruits. Pending the definite proof by animal tests, the results given are preliminary values. The results are tabulated.

J. C. Jurejans

Vitamin C (ascorbic acid) content of the paprika preparation "Vitapric." Eugene Becker. *Z. Vitaminforsch.* 4, 255-9(1935).—Vitapric, a bright-red, marmalade-like product manufd from the tomato paprika, contains 26-27% total solids and 0.45-0.46% ascorbic acid (approx. 20 times the concn of that acid in lemons). In feeding expts with guinea pigs for periods in excess of 60 days, a daily dose of Vitapric contg 1 mg ascorbic acid produced normal growth and freedom from scurvy. Animals receiving twice this daily dose had a greater resistance to other diseases.

Joseph S. Hepburn

[Effect of boiling on the ascorbic acid content of cabbage.] B. Ahmad. *Nature* 135, 797(1935).—By boiling cabbage before extg with 20%  $\text{CCl}_3\text{CO}_2\text{H}$ , the ascorbic acid content is increased from 13 mg to 40 mg per 100 g of vegetable. This is perhaps due to hydrolysis of an ester of ascorbic acid.

W. T. H.

Relation of maturity and handling of Bartlett pears in the Pacific Northwest to quality of the canned product. B. D. Ezell and H. C. Diehl. U. S. Dept. Agr., *Tech. Bull.* 450, 1-24(1934); cf. C. A. 29, 7388. Results of 3 years' investigation are detailed. As the season advances there is a decrease in the alc-insol solids and in the acids and reducing sugars and an increase in alc-sol, materials, sucrose and total sugars. There is also a similar increase in the alc-sol materials in ripened fruit, though variations in total solids obscure this relation. In the early-picked fruit the insol solids make up 23-30% of the total solids, dropping in storage and ripening to 22-23%. In the later-picked fruit insol solids amount to only 18-20% of the total solids, dropping to 15-18% on ripening. Sucrose increases both in total amt. and in percentage of total sugars. Acid changes are but little affected by ripening changes. A progressive decrease in astringency takes place during ripening. Time of picking has little influence on the color of the sirup. Ripening temps. of 21-1-23 $^\circ$  at relative humidities of 78-85% give best results. Soil types have no appreciable effect upon the quality of the canned product.

C. R. F.

Extraction of pectin from apple thinnings. H. W. Gerritz. *Ind. Eng. Chem.* 27, 1458-9(1935).—Apple thinnings were found to be rich in pectin-yielding material. Best results were obtained by slicing turgid apple thinnings into 0.5% HCl and allowing them to stand for 2-3 days at room temp. before pressing. Similar results were obtained from the pomace after freezing the apples, thawing and pressing to remove the juice. A quantity of acid soln. equiv. to  $1\frac{1}{2}$  times the wt. of the unpressed apples gave a convenient vol. of rich pectin soln. A second extn with hot  $\text{H}_2\text{O}$  gave an addl. yield of pectin. The pectin obtained either as the original soln., as a concd. soln., or as dried powd. pectin produced an excellent gel. Solns. of 0.5%  $\text{H}_2\text{SO}_4$  or tartaric acid were about 75% as efficient as HCl in extg the pectin. The nature of HCl, which makes it an efficient agent for sugar hydrolysis, is probably responsible for its efficiency in hydrolyzing the insol. pectin. The HCl pectin soln. obtained at room temp. did not need decolorizing with charcoal.

F. L. Dunlap

Consistency changes in starch pastes. Tapioca, corn, wheat, potato and sweet potato. G. V. Cascar and E. E. Moore. *Ind. Eng. Chem.* 27, 1447-51(1935).—A new and improved consistometer and technic for the study of the pasting phenomena of starches are described. Charts give the consistency record of pastes of tapioca flour, heavy- and thin-boiling corn, potato and wheat starches. The usual concn. is 20%. For tapioca, concns. of 10, 20 and 30% are given. The temp. range extends from the

zone where starch and  $H_2O$  constitute a milk, to boiling and back to room temp. The record covers, therefore, the whole paste history of a starch from cooking to cooling. The degree of degeneration of a starch is sensitively revealed in the form of curves, where paste temp is plotted against the net power in w required to maintain a const. speed of agitation. Each starch thus assumes a characteristic form, and a classification into types exhibiting similar characteristics may readily be made. The scientific and practical interest of the method is described.

F. L. Dunlap

Effect of soybeans, soybean-oil meal and tankage on the quality of pork. C. M. Vestal and C. L. Shrewsbury Ind. (Purdue) Agr. Expt. Sta., *Bull.* 400, 47 pp. (1935). — When supplementing a full feed of corn, minerals added to the corn-soybean rations reduced the feed requirement and increased the rate of gain in growing pigs. Roasted soybeans and cooked soybeans had high nutritive values, and surpassed tankage, soybean-oil meal and raw soybeans in wt. gain production and reduction in feed requirements. Soft, low-melting fats were produced only when soybean products were fed in amounts exceeding 14% of the ration. Soybeans should be fed as a supplement and not as a substitute for corn.

C. R. Fellers

Following the removal of bitter substances of the soybean with the aid of the hemolytic reaction. M. Krayčnović. Z. *Untersuch. Lebensm.* 70, 391-4 (1935), cf. C. A. 29, 1172. — This method depends on following the disappearance of saponin, present in the bean, by means of the hemolytic reaction. The lower the temp., the longer the time needed for the complete deodorizing of unhulled beans. On the other hand, the coeff. of digestion of the protein of the deodorized bean is more favorable on treatment at lower temps. The last trace of saponin disappears in unhulled beans after heating them with water at  $100^\circ$  for 25 min.; the previously dried and hulled beans under the same conditions of treatment show a neg. hemolytic reaction after 21 min. In deodorizing the crude soybean meal in boiling water, the pos. hemolytic reaction disappears after 3 min. because of greater contact area. Bread made from crude soybean meal shows only a partial destruction of the saponin during the baking process. For a complete quant. removal of the residual saponin, it was necessary to heat the bread with boiling  $H_2O$  for 15 min. The addn. of 0.05% HCl to the unhulled beans heated to  $100^\circ$  with  $H_2O$  hastened the hydrolytic decom. of the saponin, reducing the time for the disappearance of the hemolytic reaction to 21 min. Through the action of moist water vapor at  $100^\circ$  on unhulled beans, the saponin is destroyed only after 40 min. This increase in time, as compared with other results, is due to the minimal absorption of  $H_2O$ , but it has the advantage that subsequent drying of the beans is more rapid and more easily carried out. When the bean is heated with said.  $H_2O$  vapor at  $115^\circ$  under pressure, a neg. hemolytic reaction is obtained in 2 min. The deodorizing of the bean at higher temps. under pressure has the advantage of a minimal time for the disappearance of the saponin. While the coeff. of digestibility of the protein of the raw bean is the highest (94-92%), the lower the temp. at which deodorization takes place, the more favorable is this coeff. The digestion of the bean with weakly acidulated  $H_2O$  protects the vitamins from destruction. Deodorizing with boiling  $H_2O$  produces a lighter-colored product than when the vapor is used. In the latter case the color of the bean becomes still darker, particularly when dried at higher temp. so it is necessary to dry beans deodorized with steam at lower temp. (80-85°). The dark color of the bean is, for the most part, caused by pectin substances. A method is described for the identification of saponin in the soybean.

F. L. Dunlap

Action of dilute acids on aluminum. Charles F. Poe, R. M. Warnock and A. P. Wyss. *Ind. Eng. Chem.* 27, 1505-7 (1935). — Data are returned in mg. loss per 100 sq. cm. of sheet Al. The strengths of acids used were N, 0.1 N and 0.01 N. Expts. were carried out at  $25^\circ$  and at boiling temps. Of the inorg. acids, the halogen acids seemed most active. Of the org. acids, acetic and its Cl

deriv., formic and lactic are rather active. A no. of acids showed a rather great activity for the first few weeks, but thereafter decreased. Among these are glycolic, *III*, malonic, oxalic,  $H_2SO_4$ , etc. The metal may have become coated with a fine film of the Al salt and further soln. thus made difficult. Most of the acids found in fruits and vegetables are more or less active. It cannot be said at present how much of the soly. of Al would be altered by the other food constituents. Nothing stated in the paper should be construed as claiming that Al is not a proper material for use in the manuf. of cooking utensils. The physiol. actions of Al salts were not considered. Nineteen references.

F. L. Dunlap

The mineral content and feeding value of natural pastures in the Union of South Africa. III. P. J. Du Toit, A. I. Malan, J. G. Louw, C. R. Holzappel and G. Roets. *Onderstepoort J. Vet. Sci.* 5, 201-14 (1935); cf. C. A. 28, 5593. — Data are given on the P, Ca, Mg, K, Na, Cl, crude protein and crude fiber contents of numerous samples of natural pastures. Practically all samples were low in P for the greater part of the year. Crude protein varied more or less directly as the P content of the pasture and was extraordinarily low during certain seasons of the year, particularly in herbage of advanced stage of growth. In general, the Na content of the samples was so low that it suggested an insufficiency of Na for producing animals on pasturage. IV. The influence of season and frequency of cutting on the yield, persistency and chemical composition of grass species. P. J. Du Toit, J. G. Louw and A. I. Malan. *Ibid.* 215-70. — Grasses cut at monthly intervals were highest in percentage P and crude protein and lowest in crude fiber. With decreased frequency of cutting the crude fiber content increased to a max. at maturity, while the P and crude protein dropped markedly to minima in the old mature herbage cut prior to the commencement of new growth in the following season. The percentages of Ca and Mg were not appreciably affected by the frequency of cutting. Pure species grown on the same soil and exposed to the same climatic conditions showed appreciable differences in mineral and protein content when harvested after definite intervals, e. g., 1 month. Fluctuation in the chem. compn. of species cut at monthly intervals during the growing season is attributed mainly to the stage of growth attained by the species and to the rainfall insofar as it influences the stage of growth. During dry seasons the P content of herbage cut at monthly intervals tended to be low; at the same time the percentage of Ca was high. The crude protein content did not seem to be adversely affected by drought. The percentages of mineral constituents and crude protein were low and that of crude fiber was high during seasons of plentiful rains. The highest yields of dry matter, crude protein and P were obtained from pasture species by cutting at bi-monthly intervals during the growing season. The difference in nutritive value between herbage cut at monthly and bi-monthly intervals was insignificant during seasons of dry weather conditions. Numerous analytical data are tabulated.

K. D. Jacob

Colorimetric method for detg. nitrate N in grass (Ashton) 7. Thermal properties of fats (Ravich) 27. Colorimetric detn. of phosphoric acid in grass (Greenhill, Pollard) 7. Chemistry of grass crops (Virtanen) 11D. Effect of frost on wheat at progressive stages of maturity (Newton, McCalla) 11D. App. for drying and grinding materials such as fruit pulps (U. S. pat. 2,023,247) 1. Grease- and moisture-proof paper for wrapping foods (U. S. pat. 2,021,947) 13. Moistureproof material such as regenerated cellulose for wrapping foods (U. S. pat. 2,022,490) 13.

Food product. Soc. des crédits internationaux S. A. Fr. 787,018, Sept. 16, 1935. Flakes of oats, barley, rice or maize are mixed with milk powder and salt, sugar, fruit juice or other flavoring, and the mixt. is compressed. Lecithin, vitamins, fatty material or other food may also be added.

Food preservation Per D. Peterson and Göran A. Möller. Fr. 786,962, Sept. 14, 1935. Meat, fish, eggs and vegetables are disinfected by means of  $H_2O_2$ , formalin or other disinfectant and then preserved by an atm. of  $CO_2$ .

Vitamin concentrates Lloyd A. Hall (one-half to Carroll L. Griffith) U. S. 2,022,464, Nov. 26. A vitamin D emulsified concentrate suitable for addition to various foods is prepared by mixing with an oil base vitamin D prep (such as one from irradiated ergosterol or a strong cod liver oil extract) an emulsifying agent such as gum acacia and gum tragacanth or the like and adjusting the pH to 5.5-6.0 by adding water containing a mild alkali such as  $NaHCO_3$  or a weak acid such as citric acid or  $HOAc$ .

Bread, etc. Standard Brands, Inc. Brit. 433,413, Aug. 14, 1935. The crust color of baked goods is improved by bringing the surface of the dough into contact with  $NH_3$  or an  $NH_3$  compound that rapidly decomposes under heat treatment, e.g.,  $NH_4OH$ ,  $NH_4$  tartrate,  $NH_4$  carbonates,  $NH_4$  malonate. The process is used preferably when a sugar, other than cane sugar, is incorporated in the batch.

Stable dry leavening material Hans Wögerbauer. U. S. 2,023,500, Dec. 10. A culture of acid-producing bacteria such as lactic acid bacteria, with which yeast may be associated if desired, is mixed with a food flour in the presence of only such an amt. of water that the flour is moistened without becoming sticky, and the mixt. is allowed to undergo a long period dough fermentation (sufficiently until it becomes sufficiently dry to permit milling without artificial drying). Various details and modifications are described.

Use of hydrogen peroxide and other per compounds such as sodium percarbonate or perborate as dough leavening agents. E. I. du Pont de Nemours & Co., Joseph S. Reichert and Wm. J. Sparks. Brit. 433,471, Aug. 15, 1935. See U. S. 1,953,567 (C. A. 28, 3897).

Process and apparatus for the continuous or intermittent concentration of milk to obtain evaporated or condensed milk Alfred R. Jahn. Brit. 433,678, Aug. 19, 1935.

Removing milk or beer scale. Henkel & Cie. G. m. b. H. (Helmuth Firsau, inventor) Ger. 617,583, Aug. 22, 1935 (Cl. 53e 2). Scale and other deposits occurring in food-making app. are removed by treating the app. with enzymes, such as pancreatin soln., which have no corrosive action on metals.

Rotary-drum apparatus with internal scraper, etc., for desiccating materials such as milk, casein, etc. Julius F. Dietrich U. S. 2,023,468, Dec. 10. Various structural, mech. and operative details.

Whey. Josef Sirek U. S. 2,023,350, Dec. 3. A whey prep. suitable for use with various dietetic compns. such as those containing vitamins and enzymes is obtained by freeing whey from milk proteins and their products of decomposition and from all milk ferments and adding a colloidal soln. of edestin-calcium derived from hemp seed and about 0.05% of  $MgSO_4$ . Various details of prep. are described.

Whey Kraft-Phenix Cheese Corp. Brit. 434,058, Aug. 26, 1935. Whey is condensed, to give a product having a high vitamin G content by evap. at a  $p_H$  not higher than 6 and not below about 5.5. In an example,  $H_2PO_4$  is added to give the desired  $p_H$  prior to or subsequent to the introduction of the whey into a vacuum pan, where it is evap. until it will set on cooling. While still hot, the free  $H_2PO_4$  is preferably neutralized by adding  $CaO$ . After setting, the product is subdivided and dried in a drying tunnel at a temp. sufficiently low to prevent caramelization of the sugar.

Margarine Marvin C. Reynolds U. S. reissue 19,767, Nov. 26. A reissue of original pat. No. 1,917,273 (C. A. 27, 4600).

Margarine Marvin C. Reynolds. U. S. 2,022,924, Dec. 3. A normally liquid vegetable oil such as cottonseed oil is hydrogenated to a point at which it is solid at

room temp., the oil is melted at a temp. above its clearing point, crystd., heated to a temp. below its clearing point to melt the relatively lower-melting constituents, and the oil is churned with an aq. liquid such as cultured milk to form a relatively liquid emulsion, the emulsion is crystd. to solidify it and the solid emulsion is processed to form a final plastic margarine product.

Jacketed tubular processing apparatus suitable for congealing ice cream or lard, etc. Bruce DeH Miller (to Vogt Processes, Inc.) U. S. 2,023,607, Dec. 10. Structural details.

Cheese Pomosin Werke G. m. b. H. Ger. 617,858, Aug. 27, 1935 (Cl. 53e 6 01). See Brit. 420,563 (C. A. 29, 3418).

Cheese treatment Ernest Schneider (to Kraft-Phenix Cheese Corp.). U. S. 2,021,899, Nov. 26. An emulsifier such as Na phosphate or whole-milk powder is added to cheese and the material is worked to a smooth consistency at a temp. not substantially above about 93° and is subjected to heat and to the action of a vacuum without heating to swell the casein, the operations being so conducted and timed as not to impair the flavor. App. is described.

Preserving eggs Göran A. Möller. Fr. 786,963, Sept. 14, 1935. The whites and yolks of eggs are preserved, after removing the shells, in hermetically closed containers in an atm. of  $CO_2$ .

Preserving fruit. Wilhelm J. H. Hinrichs. Brit. 434,137, Aug. 27, 1935. See Fr. 779,631 (C. A. 29, 6324).

Drying fruits. Charles C. Moore (to Vacuodri Fruit Corp.) U. S. 2,023,536, Dec. 10. A "dehydrated" fruit having a considerable fructose sugar content is brought to a substantially dry condition by subjecting it for a predet. time to a pressure-heating medium such as steam at a temp. considerably in excess of that at which fructose sugar inverts, to heat the fruit to a temp. approx. the point of inversion, withdrawing the heating medium before the fruit has absorbed sufficient heat to cause inversion, and immediately reducing the pressure of the surrounding medium to vaporize suddenly a portion of the moisture in the fruit, and maintaining a reduced pressure until the moisture is substantially removed.

Pectin Suzanne G. Rabaté (nee Grillon), Adrien Macé and Jean Clément. Brit. 434,037, Aug. 26, 1935. See Fr. 769,210 (C. A. 29, 5187).

Pectous preparation suitable for use in jelly making Herbert T. Leo, Frederick A. Beck and Clarence C. Taylor U. S. 2,022,470, Nov. 26. A dry powd. mixt. is prepd. with particles of greater than colloidal size comprising fibrous crushed fruit or vegetable cellulose tissue containing substantially the original content of water-insol. pectose substantially free from water-sol. substances and bitter components of the original fruit or vegetable, together with a filler and such as diatomaceous earth to standardize the material to a desired jelly strength. Numerous details of prep. are given. U. S. 2,022,471 relates to a similarly standardized prep. of water-sol. pectin.

Flavoring material Albert K. Epstein and Benjamin R. Harris U. S. 2,023,877, Dec. 10. A flavoring suitable for use with margarine, etc., in very small proportion to impart a butter-like flavor comprises butyryl and another diketone such as 2,3-pentadione or 2,3-hexadione.

Soluble coffee Wilbert A. Heyman U. S. 2,022,467, Nov. 26. A closed porous container such as a cheese-cloth bag is filled with a partly sol. coffee in the form of granular particles of coffee ext. and granular particles of roasted coffee, from both of which powd. material has been sepd. Numerous details of prep. are given.

Decaffeinating coffee Jean MacLang (to Coffex A. G.) U. S. 2,023,333, Dec. 3. Selective extraction of caffeine is effected with a dichloroethane. Various details of operation are described.

## 13—CHEMICAL INDUSTRY AND MISCELLANEOUS INDUSTRIAL PRODUCTS

(PLASTICS, RESINOIDS, INSULATORS, ADHESIVES, ETC.)

HARLAN S. MINER

Systematizing chemical technology. H. Hoppmann. *Chem. Fabrik* 1935, 468-77.—Chem. technology is discussed from the material or consumptive, the operating or process, and the app. or construction sides, and various methods and equipment employed are classified. Ten tables.

Systematizing chemical technic. H. Hoppmann. *Chem. Fabrik* 1935, 467.

Industrial gas masks. F. Rutledge Davis. *India Rubber World* 93, No. 3, 27-9 (1935).—An illustrated description of present developments, with special reference to the part played by rubber.

Plastics in chemical industry. Lester W. Tarr. *Ind. Eng. Chem.* 27, 1281-6 (1935).—A description of Ilavog, a PhOH-CH<sub>2</sub>O resin reinforced by asbestos fiber and available in large molded shapes (0 × 10 ft.). Some advantages are chem. resistance, lightness combined with strength and toughness, resistance to thermal shock and to temps. up to 150°, machinability and elimination of electrolytic corrosion.

The significance of plastic masses, ceramic materials and glass for the development of domestic raw materials. W. Spielvogel. *Glastech. Ber.* 13, 381-90 (1935).

A new method for determining phthalic acid in alkyl resins and other phthalic acid esters. C. P. A. Kappelmeier. *Farben-Zig.* 40, 1141-2 (1935).—This new simple method isolates and weighs the phthalic acid (I) as a previously unknown K phthalate contg. one mol. of alc. of crystn., viz., CaH<sub>2</sub>O<sub>4</sub>, EtOH (II). The sample is weighed out into a 150-cc. Erlenmeyer flask, dissolved in a few cc. of benzene and treated with 2-3 times as much 0.5 N alc. KOH as is consumed by the sapon. of the sample. After attaching a reflux air condenser, the flask is warmed first to a temp. not exceeding 40° and then gradually heated to refluxing. Sapon. is complete after 1-1½ hrs. If the fat acids are to be detd. simultaneously, an amt. of ether sufficient to give an alc.-ether ratio of 3:1 is added, the cryst. II filtered off and washed with a 3:1 alc.-ether mixt., the fat acids passing into the filtrate. If the fat acids are not to be detd., the possibility of errors due to K<sub>2</sub>CO<sub>3</sub> formation can be forestalled by neutralizing the sapon mixt. with linseed oil fat acids. Next an amt. of ether sufficient to give an alc.-ether ratio of 1:1 is added. The resulting mixt. is cooled and quickly filtered through a porcelain or glass filter. The filter residue, II, is washed with a 1:1 alc.-ether mixt.; sucking air through II is avoided as II is hygroscopic. II is dried to const. wt. by warming for a few min. in a drying oven and then allowing to stand for 2-3 hrs. over H<sub>2</sub>SO<sub>4</sub> in a vacuum desiccator. The formation of II can be used as a qual. test for I. A confirmatory test consists of fusing a small portion of the substance thought to be II with resorcinol and observing the formation of fluorescein. Cf. following abstr.

The reliability of the resorcinol test for detecting phthalic acid. F. Krämer. *Farben-Zig.* 40, 1142-3 (1935).—On heating resorcinol with oxidizing agents, e. g., concd. H<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>O<sub>2</sub>, chromic acid salts, etc., and dissolving the reaction mixt. in caustic soln., a green fluorescence is observed. For this reason, the test for phthalic acid using resorcinol and concd. H<sub>2</sub>SO<sub>4</sub> is apt to give misleading results when applied to resins.

Starch and dextrin adhesives. Fred Holt, Jr. *Paper Ind.* 17, 482-5 (1935).—A brief outline of the history and chemistry of starch and dextrin, of the manuf. and analysis of starch and of its use in the paper industry.

Pastes, vegetable glues, adhesives from dextrin, casein, and gums, gelatin and animal glue. IV. Adhesives from casein. Carl Becher, Jr. *Gelatine, Leim,*

*Klebstoffe* 3, 163-5 (1935); cf. C. A. 29, 8172'.—Recipes and a brief discussion of characteristics are given for 11 different casein glues consisting of combinations of casein with NaOH, Na<sub>2</sub>CO<sub>3</sub>, borax, NH<sub>4</sub>OH, "Preventol C," rosin soap, turpentine, and lude glue. The formulas differ considerably from those combinations of casein-lime-sodium salt that are commonly used in the U. S. V. Adhesives from gums. *Ibid.* 165-8.—A brief discussion of the physical properties of gum arabic and recipes for 9 adhesives based on gum arabic are given.

The prevention of ice accretion (on aircraft). B. Lockspeiser. *Aircraft Eng.* 7, 278-81 (1935).—The requirements for a suitable liquid are low-setting point, miscibility in all proportions with H<sub>2</sub>O, large effect in depressing the f. p. of H<sub>2</sub>O, low vapor pressure below 0°, and high flash point. Both ethylene glycol itself and a 10% EtOH mixt. with ethylene glycol have been used with success in flight. In test flights under ice-forming conditions, a Hart and a Gordon airplane used 1.5 and 1.7 pints ethylene glycol per hr., resp.

Waxes—animal, mineral, vegetable, synthetic. Ibert Mellan. *Chem. Industries* 37, 639-45 (1935).—A review.

Chloropicrin in modern fumigation. Conrad C. Johnson. *Soap (Sanitary Products Sect.)* 11, No. 11, 105, 107, 109 (1935).—CCl<sub>3</sub>NO<sub>2</sub> is used in house, rodent or mill fumigation, it has a high toxicity to insects and high penetrative powers. It is sprinkled or sprayed in the space to be fumigated by an operator wearing a gas mask and requires little time and trouble in application. Its strong odor serves as an efficient warning during and after fumigation.

Household insecticides. R. C. Roark. *Soap (Sanitary Products Sect.)* 11, No. 11, 101, 103, 117 (1935).—HCN, (CH<sub>3</sub>Cl), ethylene oxide + CO<sub>2</sub>, p-Cl<sub>2</sub>CH<sub>3</sub>, P, NaF, certain fluosilicates, pyrethrum, derris and cube are the principal household insecticides. The properties of the last three are discussed. The trend seems to be toward the development of org. compds. that are highly toxic to insects but relatively nontoxic to man and warm-blooded animals. Phenothiazine is an example and R. suggests it should prove effective against some household pests.

Effect of lead on pyrethrum extracts. Louis S. Bake. *Soap (Sanitary Products Sect.)* 11, No. 11, 111, 113 (1935).—Oil exts. of pyrethrum stored in darkness in glass bottles with small pieces of Pb or solder were nearly decolorized in 14 days with the formation of an amorphous, light-yellow ppt. contg. 26% Pb. Similar exts. stored with Zn or Cu became greenish. Larger ppts. formed with Zn, Al, Sn, Ni and Fe appeared to be without effect. B. concludes that oil exts. of pyrethrum should not be stored in containers having either Pb or solder present, even in caps, but exts. are apparently safe from decomposition when stored in the presence of Sn, Al, Ni or Fe.

Effects of sunlight on fly sprays. David G. Hoyer. *Soap (Sanitary Products Sect.)* 11, No. 12, 105, 107, 109, 111 (1935).—Pyrethrum exts. of the same strength in 3 samples of specially treated odorless petroleum distillates and in one sample of ordinary petroleum distillate were exposed in tight flint-glass bottles to sunlight and daylight for 4 mos. and were tested at intervals biologically with house flies by the Peet-Grady method (C. A. 22, 4731) and chemically from pyrethrins I and II by the Seil method (C. A. 28, 4531<sup>1/2</sup>). The sprays prepd. with an odorless base gained slightly in toxicity during the first 4 days. The other spray lost toxicity from the start and after the 4th day all sprays lost rapidly, with about a 65% loss for each of the 4 sprays at the end of 2 mos. and an addnl. loss of about 25% during the next 2 mos. II. suggests that the light caused the formation of an un-



stable toxic substance in the odorless base sprays which accounted for the increase in toxicity at the start. These sprays took on a hazy appearance within 3 hrs, this haziness increasing and being accompanied by a loss of the typical amber color. Finally a white ppt. settled out. The spray prepd with the ordinary base remained clear but had lost some of its amber color by the end of 4 mos. The chemical analyses were not consistent for the different sprays. The "apparent" pyrethrin content of one rose rapidly during the entire period while that of the one prepd. with the ordinary distillate fell slightly at first and then remained const., though during this time there was a loss in the "apparent" pyrethrin I which was compensated for by an equal gain in the "apparent" pyrethrin II. The pyrethrin contents of the other 2 sprays followed the toxicity curve well but H. believes that the development of an acidic substance in the sprays interfered with the analysis and gave a false pyrethrin content. Fifteen references

Henry H. Richardson

Lubricating comps.—elec. insulations (Brit. pat. 434,626) 22. Rubber comps. [in manuf. of leather substitute] (Brit. pat. 433,396) 30. Urethyl comps. [having wetting and cleansing properties] (Fr. pat. 759,734) 10. [Red brown resin from] lubricating oils (Brit. pat. 434,295) 22. Pitch [for castings] (Brit. pat. 433,826) 21. Polyglycerol esters of aliphatic acids of relatively high mol. wt. [having wetting and emulsifying properties] (U. S. pat. 2,023,288) 10. Coating parchmentized paper for use as imitation leather (U. S. pat. 2,023,711) 23. Alkylated phenols [as wetting agents] (Ger. pat. 616,786) 10.

Gas masks Hugo Stoltzberg (trading as Chemische Fabrik Hugo Stoltzberg). Brit. 433,848, Aug. 21, 1935.

Plastic masses I. G. Farbenind. A.-G. (Wilhelm Schreier and Georg Niemann, inventors). Ger. 617,733, Aug. 24, 1935 (Cl. 336.5) High-mol. org. plastic masses, especially rubber, are colored by azo dyes insol. in water, obtained by coupling 1 or 2 mols. of diazotized *o*-aniline or its derivs. with 1 mol. of diacetoxyl-*o*-toluidine, or its derivs. Examples are given.

Plastic compositions John P. Henharen Brit. 433,994, Aug. 23, 1935. See Fr. 781,902 (C. A. 30, 1889).

Removing volatile solvents from solutions of plastic compositions Adolf Kämpfer. Brit. 434,640, Sept. 5, 1935. In the removal of solvent from layers of plastic comps. applied to the single sheets in the prepn. of laminated glass having nonsplintering intermediate layers, the sheets being dried in the sep. compartments of a container, the formation of skins on the surface of the layers is prevented by replacing the solvent-charged atm. above the layers, just prior to its satn. point, with fresh air. This substitution may be repeated until the solvent is removed, or the absorptive power of the atm. may be increased by step-by-step increase of the temp. of the atm., each compartment being heated by tubular steam heaters.

Lining pipe fittings with bituminous material Pipe Linings (Australasia) Ltd. and Einar V. Langevad. Brit. 434,221, Aug. 28, 1935. The fitting is heated, cold dies or molds forming a core are introduced into the fitting and the material, hot and liquid, is introduced between the core and the fitting. Diatomaceous earth to 10-13% may be added to the bitumen, which may be treated by the "air blown" process and respond to the penetration test at 77°F. with 35-40 units and to the ball-and-ring test between 180° and 190°F.

Bituminous coverings Deutsche Röhrenwerke A.-G. Ger. 615,175, June 28, 1935 (Cl. 293.24). Addn. to 501,539 (C. A. 28, 2927). The method of 501,539 for making masses for coating metal tubes by mixing molten dipolymerized soft rubber vulcanizates with molten mineral oil bitumen is modified by adding the molten rubber to the molten bitumen. Filling material, such as asbestos fiber, and coloring matter may be added. The mass is used for coating the insides and outsides of metal tubes.

Synthetic resin Geo. W. Seymour (to Celanese Corp.

of America). U. S. 2,022,389, Nov. 26. A synthetic resin which may be used with cellulose acetate, etc., is prepd. by treating  $\text{CH}_2\text{O}$  with the sulfonamide of a phenol ether such as an anisole sulfonamide. Various examples are given.

Synthetic resin. Charles A. Thomas (to Monsanto Petroleum Chemicals, Inc.). U. S. 2,023,493, Dec. 10. A resinous reaction product which is suitable for use as a coating is formed from a polyhydric alc. such as ethylene glycol or glycerol and a preformed unsatd. hydrocarbon resin obtained by the polymerization of a cracked petroleum distillate.

Synthetic resins Marie B. Rousset and Auguste V. Keller. Brit. 434,112, Aug. 21, 1935. Solid, opaque or transparent condensation products are made by causing urea, thiourea or their derivs. to react with an aldehyde, e. g.,  $\text{CH}_2\text{O}$ , in the presence of a preferably monohydric phenolic substance of low m. p., which acts as an autocatalyst, the mol. proportions of the reacting substances being so balanced that a gradual increase of H-ion concn. is developed from the neutral soln. In an example, 33 parts  $\text{CH}_2\text{O}$ , calcd. on the 35% solid content of com. 40%  $\text{CH}_2\text{O}$  soln., is nearly neutralized to a pH of 7-8 by addn. of alkali, e. g., NaOH, 12.5 parts urea and 2 thiourea, mixed with 0.05 *p*-cresol, are added and the neutral soln. is condensed by heating at low temp. under reflux until the reaction subsides, after cooling and filtering, the mixt. is evapd. at elevated temp. under partial vacuum to remove the  $\text{H}_2\text{O}$  and the viscous product is poured into molds and dried at 70-100°. Decolorizing agents, e. g., charcoal, softeners, sucrose, glycerol, and fillers, e. g.,  $\text{SiO}_2$ , metal dust, wood flour and paper pulp, may be added.

Synthetic resins. Dynamit-A.-G. vorm. Alfred Nobel & Co. Ger. 617,855, Aug. 27, 1935 (Cl. 396.22). The hardening of  $\text{PhOH-CH}_2\text{O}$  condensation products by org. bases is accelerated by using high-boiling aliphatic bases or base mixts. with a straight chain, with a pH value of over 10 and with both primary and secondary amino groups in the mol. Thus, a  $\text{PhOH}$  resin is mixed with wood meal and diethylenetriamine to form a homogeneous mass, which is pressed to form and rapidly hardens.

Synthetic resins Kurt Albert G. m. b. H. chem. Fab. Ger. 620,201, Oct. 18, 1935 (Cl. 120.26.02). A resin prepd. in known manner from a polyhydric alc., a polybasic acid and a resin acid at 220-250° (resin A) is added to a mixt. comprising a polyhydric alc., a polybasic acid and a higher aliphatic acid or its glyceride (mixt. B), and the whole is heated to 170-200°. Alternatively, resin A is mixed with a resin previously prepd. at 170-200° from mixt. B, and the whole is heated to 170-200°. Undiscolored products useful as lacquer components are obtained. Examples are given.

Synthetic resins Firma Louis Blumer. Ger. 620,202, Oct. 18, 1935 (Cl. 120.26.02). Resins prepd. from polyhydric alcs. and polybasic acids, with or without monobasic acids or their derivs., are refined by dissolving them at atm. or raised temp. in a lower aliphatic alc., e. g., MeOH or EtOH, and either cooling the solns. or adding a precipitant thereto. Resins free from unchanged starting materials and reaction products of low mol. wt. are pptd. The refined resins have a lower acid no. than the unrefined resins and are more easily hardened. Examples are given.

"CO-amide" Carleton Ellis (to Unyte Corp.). U. S. 2,022,233, Nov. 26. A urea-formaldehyde condensation product is incorporated with phthalimide and caused to react to form a resinous product which is suitable for coatings, etc. Numerous examples are given.

Molded resin ware. Poy H. Cunningham (to Bryant Elec. Co.). U. S. 2,022,587, Nov. 25. Various operative details are described for use of a highly polished mold for molding articles with a smooth nonabsorptive outer surface and a more porous texture beneath the surface, some of the surface material being removed for application of liquid coloring material to color certain areas in a desired pattern, as in the production of decorated ware of urea resin.

Coating surfaces with synthetic resins N. V. Philips' Gloeilampenfabrieken. Brit. 434,872, Sept. 10, 1935.

A layer of moldable material containing a hardening synthetic resin is applied by heat and pressure to a compressible base by first applying a high pressure for such a short time as to liquefy the material and then applying a considerably lower pressure while heating is maintained until the material has hardened. A wooden plate is covered with a thin layer of a mixt. comprising a  $\text{PhOH}\cdot\text{CH}_2\text{O}$  condensation product 50 and wood dust 50%; a layer of paper impregnated with resin may be inserted between the wood and the resin powder, the plate is then pressed 3 min. at 150–160° under 70 kg. per sq. cm. pressure and afterward 7–10 min. under 10 kg. per sq. cm.

**Surface coloring of synthetic resin articles** Gustavus J. Esselen (to Specialty Guild, Inc.). U. S. 2,022,710, Dec. 3. The surface to be colored is softened with a water-sol. softening agent such as a phenol in aq. soln. and is then dyed with an aq. soln. of a water-sol. dye. Several examples are given.

**Crank press suitable for use with artificial resins, insulating materials, etc.** Karl Götz U. S. 2,023,319, Dec. 3. Mech. features.

**Heat insulation** Carl G. Munters and John G. Tandberg (to Carl G. Munters). U. S. 2,023,204, Dec. 3. Heat insulation comprises a low sp. gr. and highly vesiculated mass of polystyrol substantially composed of closed cells.

**Thermal insulation** Martin C. Huggett (to Research, Inc.). U. S. 2,023,422, Dec. 10. A film of nonmetallic mineral material having not more than a slight luster but containing a binder, such as a compn. containing Ag sulfide and casein, is secured to a supporting base sheet and then subjected to friction to give it a highly polished heat-reflective surface.

**Heat-insulating material from mineral wool** Edward A. Tooley (to Johns-Manville Corp.). U. S. 2,022,750, Dec. 3. After molten mineral material has been blown into fibers, the fibers, while still suspended and heated, are mixed with a normally solid, inherently water-repellent material such as Zn stearate and talc in a finely divided form. App. is described. Cf. A. 30, 191.

**Insulated wire** Samuel Ruben (to Vega Mfg. Corp.). U. S. 2,022,827, Dec. 3. A wire carries a surface insulation including an oxide such as finely divided Cr oxide with a resinous binder and a B compd. such as boric acid, and an overlying textile winding is impregnated with a similar mixt.

**Compositions for coating cables** Siemens & Halske A.-G. (Ernst Fischer, inventor). Ger. 620,232, Oct. 17, 1935 (Cl. 21c. 7.50). Comps. for protecting submarine cables, insulated with gutta-percha or like material, from marine fauna comprise a powder light metal, particularly Al, mixed with insulating or semiconducting materials.

**Nonconducting coverings for heat and sound** Dynamit A.-G. vorm. Alfred Nobel & Co. Brit. 433,251, Aug. 12, 1935. Addn. to 429,316 (C. A. 29, 6977\*). In the prepn. of porous heat- and sound-insulating substance from polymerization products of a vinyl compd. by the addn. of a volatile solvent and subsequent heating, or by the addn. of decomposable substances evolving gases on heating, as described in 429,316, the operation is effected under vacuum. In an example, a vacuum of 3–0.2 mm. Hg was applied in the prepn. of porous polystyrol, a product of sp. gr. 0.1–0.02 being obtained.

**Adhesives** I. G. Farbenindustrie A.-G. Brit. 434,260, Aug. 28, 1935. Materials, e. g., wood, metal, glass, concrete, leather or rubber, are stuck together by applying to the surfaces to be united a high-mol. polymer of a purely aliphatic mono-olefin having a branched C chain and bringing the surfaces together.

**Adhesive base from cornstarch** Gordon G. Pierson (to Perkins Glue Co.). U. S. 2,023,973, Dec. 10. Substantially dry powd. corn starch is used with an admixed oxidizing agent such as  $\text{BaO}_2$ , 0.4–1.5% adapted to release O and capable of liquefying the starch, and with urea, which serves as a liquefier and stabilizer and retards evapn. of water from the adhesive after its application to an object to be glued. The adhesive is prepd. for use by mixing it with water and NaOH and heating the mixt. Several examples are given.

**Wetting and other agents** Edmund Waldmann and August Chwala. Brit. 431,358, Aug. 27, 1935. Sulfonic acids of aliphatic amino ethers, which are carbonizing agents in free form and wetting, washing and emulsifying agents in the form of their salts with alkalis or organic bases, are prepd. by acylating a sulfonic acid of an aliphatic amino-ether, or a salt thereof, with a fatty acid containing at least 10 C atoms or a resin acid, or a chloride thereof. The original acids may be prepd. by treating with  $\text{NH}_3$  or a primary amine the halogenated ether sulfonic acids obtained by the reaction of dihalo aliphatic ethers with alkali or  $\text{NH}_3$  sulfite. In an example,  $\beta,\beta'$ -dichlorodiethyl ether is treated with alk.  $\text{Na}_2\text{SO}_3$  and the product is treated with  $\text{MeNH}_2$  and acylated with lauric acid chloride. The latter may be replaced by abietic acid chloride and  $\text{EtNH}_2$  or  $\text{PhNH}_2$  may be used for the  $\text{MeNH}_2$ .

**Wetting and other agents** Imperial Chemical Industries Ltd. Fr. 786,925, Sept. 6, 1935. Wetting, washing and emulsifying agents are made by converting to their sulfuric esters the aliphatic hydroxy sulfides obtained by causing alkyl ( $\text{C}_{12}$ – $\text{C}_{20}$ ) mercaptans to react with halohydrins. The esters are obtained by causing the hydroxy sulfides to react with the addn. product resulting from the action of  $\text{SO}_2$  on a tertiary amine or with substances capable of forming an addn. product of this nature (e. g., a pyrosulfate and pyridine). Thus, 2-hydroxyethyl ethyl sulfide (from cetyl mercaptan and ethylene chlorohydrin) or  $\beta,\gamma$ -dihydroxypropyl ethyl sulfide (from cetyl mercaptan and glycerol  $\alpha$ -mono-chlorohydrin) is caused to react with Na pyrosulfate and pyridine.

**Wetting and other agents** Soc. pour l'ind. chim. à Bile. Fr. 786,911, Sept. 14, 1935. Tertiary amines which contain an aliphatic or hydroaromatic radical formed of at least 6 C atoms are treated with oxidizing agents capable of transforming the amines to their oxides. Examples are given of oxides formed from 1-dodecyl-4-dimethylaminobenzene, 4-dimethylamino-1-laurophenone (by condensing  $\text{PhNMe}_2$  with lauric acid chloride in the presence of  $\text{ZnCl}_2$ ,  $b.p.$  220–60°), lauryldimethyl (and ethyl) amine, cetyldimethyl (and ethyl) amine, stearyldiethylethylenediamine, and  $N$ -laurylpiperidine (from lauryl chloride and piperidine),  $b.p.$  170–82°, and octadecenyldimethylamine.

**Molded objects** The Ferro Engineering Co. Fr. 786,570, Sept. 5, 1935. Objects are molded from an intimate mixt. of an aggregate such as silica sand, a sol. glass and not more than 1% of a common metal sulfate, e. g.,  $\text{FeSO}_4$ . The molded mass is placed on a metal plate with the interposition of a film of a sol. chloride other than NaCl, e. g.,  $\text{CaCl}_2$ , and the plate is put into a furnace to bake the mass.

**Molded articles such as electric-switch cover plates** James T. Walsh and Rene P. Pperoux (to Cellulose Corp.). U. S. 2,022,364, Nov. 26. A decorative facing of synthetic thermoplastic cellulose-deriv. material such as cellulose nitrate or acetate compn. is fused to a backing of a synthetic cellulose-deriv. material of nonflammable character such as one containing cellulose nitrate or acetate. Several examples are given.

**Molding articles such as containers from fibrous pulp** Ernest Huff and Andrew Keiding (to American Lacle Paper Co.). U. S. 2,023,200, Dec. 3. Various operative details suitable for making cups, etc. App. is described.

**Molding powder containing cellulose acetate and synthetic resin** Wm. H. Moss (to Celanese Corp. of America). U. S. 2,022,383, Nov. 26. Cellulose acetate is used with at least its own wt. of synthetic resin consisting essentially of a diphenylol propane formaldehyde resin, these 2 materials forming most of the plasticizable compn., which, however, also may be used with other ingredients such as china clay or triacetin.

**Bitumen-fiber mixture suitable for molding, trowelling, etc.** Harry C. Fisher (to Richardson Co.). U. S. 2,023,675, Dec. 10. Fibers such as those of cotton, wood or flax are mixed with and coated by bitumen and are then dispersed in water while heated.

**Buttons, etc., from powdered casein** Geo. Morrell (to Geo. Morrell Corp.). U. S. 2,022,895, Dec. 3.

Various details of app. and of a pressure, extrusion and heating process are described.

Casein composition suitable for sizing, etc. Carl Iddings (to Muralo Co.). U. S. 2,023,353, Dec. 3 A stable compn retaining its viscosity undiminished over long periods of time is prep'd by heating casein to about 75-99° with  $\text{NH}_4\text{F}$  or an alkali metal fluoride, finely divided silica and water.

Putty Douglas H. B. Cowman Brit 434,447, Sept. 2, 1935 A putty, particularly for glazing metal windows, comprises a filler, a drying oil,  $\text{H}_2\text{O}$  and a peroxide, with or without driers. It may be produced shortly before use by mixing a component comprising the filler, drying oil and  $\text{H}_2\text{O}$  and a component comprising a peroxide, both components preferably being pastes. In an example, the components comprise white, linseed oil,  $\text{H}_2\text{O}$  and  $\text{CCl}_4$ , and  $\text{BaO}$ ,  $\text{CaO}$ ,  $(\text{AcO})_2\text{Pb}$ , vaseline and  $\text{PbNO}_3$ , resp.

Leather substitute Le cuir contreplaqué Fr. 787,080, Sept. 16, 1935 Rubber latex is mixed with wood fibers, a soln of  $\text{Al}_2(\text{SO}_4)_3$  is added and finally hardening or suppling agents. The paste may be made into bands, plates or cylinders using high pressure.

Coated fabrics Mitchell G. Thomson and Imperial Chemical Industries Ltd Brit 434,359, Aug. 30, 1935 In the manuf. of coated fabrics, e. g., leather-cloth or tarpaulin, comprising a cotton, hessian, paper or other base coated with a compn contg. dissolved nitrocellulose and a softening agent therefor, the latter is a brominated tritriol phosphate corresponding substantially to a di-bromotriol phosphate in which both Br atoms are substituted in the nucleus. Other softening agents, e. g., castor oil, may also be used. The softener may be obtained by treating tritriol phosphate with Br at 55-60° in the presence of powd. Cu.

Condensation products I. G. Jarbenind A-G (Michael Jahrstorfer and Hans G. Hummel, inventors) Ger. 616,429, July 30, 1935 (Cl. 12a 26 02). Addn to 608,591 (C. A. 29, 5202a) The method of 608,591 for obtaining products by condensing polyamides of carboxylic acids with  $\text{CH}_2\text{O}$  is modified by replacing the polyamides or thioamides of polyamino-N-carboxylic acids. Thus, hydrazine carboxylic acid diamide (from hydrazine sulfate and  $\text{KCN}$ ) is condensed with aq.  $\text{CH}_2\text{O}$  of  $\text{pH}$  value 7.0 to give a syrupy soln hardening to a glossy transparent product. Other examples are given.

Condensation products from natural resins Josef Linapff (to I. G. Jarbenind A-G) U. S. 2,022,456, Nov. 26 A natural resin such as colophony or an ester of a resin such as the glycerol ester of colophony is treated with a phenol of an alkylated phenol or an alkyl ether or ester of a phenol, in the presence of  $\text{BF}_3$  or a complex compd of  $\text{BF}_3$  with an org. acid. Numerous examples are given and products are obtained with a much lower  $\text{I}$  value and higher softening point than the starting materials. Cf. C. A. 28, 919<sup>1</sup>, 29, 6781<sup>1</sup>.

Halogenated diphenyl oxide Edgar C. Britton, Wesley C. Stoesser and Gerald G. Goergen (to Dow Chemical Co.). U. S. 2,022,634, Nov. 26 Diphenyl oxide or a lower halogenated diphenyl oxide is treated with  $\text{Cl}$  or  $\text{Br}$  at an elevated temp. (suitably above about 145° and 75°, resp.) sufficient to maintain the reaction mixt. in a mobile fluid condition to form a halogenated diphenyl oxide mixt. having a halogen content corresponding to more than 4 and less than 10 atoms of combined halogen per mol. of halogenated diphenyl oxide. Products thus produced are of a resin like character. Numerous examples are given.

Emulsifying agents containing fatty acid esters of polyglycerols Benjamin R. Harris. U. S. 2,022,766, Dec. 3 A mixt. of polyglycerols is produced from glycerol, and the polyglycerols (usually in excess) are caused to react with higher fatty acids such as those of coconut oil, neat's foot oil, corn oil or lard or with the triglycerides of the oils and fats. Products are obtained which may be used in the prep'n of cosmetic creams, shaving preps, detergents, insecticides, etc. Various examples are given.

Emulsifying and dispersing agents Imperial Chemical Industries Ltd, Henry A. Puggott and Geo. S. J. White

Brit. 434,424, Aug. 27, 1935 The agents are prep'd by treating sod oil, degrass, molleon or like oil recovered in the manuf. of chamois leather with ethylene oxide (I), preferably in the presence of caustic alkali or a surface-active solid, e. g., infusorial earth, silica gel. The products give emulsions with animal, vegetable and mineral oils and are useful in the textile industry as leveling agents in dyeing and printing and for improving the cleansing action of sulfonic esters of the higher alcs. Among examples, (1) sod oil is treated with I to prep. products which are used to emulsify a lubricating oil with  $\text{H}_2\text{O}$ , and (2) wool is dyed with indigo in a vat contg. the product of (1).

Emulsion for filling water-cooling systems Karl Kreleler (to Shell Development Co.). U. S. 2,023,367, Dec. 3 Water is used with a small proportion of an emulsifiable mineral oil together with an alkali soap of a sulfonated naphthemic acid or mineral oil sulfonic acid.

Sulfonated fatty substances Deutsche Hydrierwerke A-G Brit. 434,452, Sept. 2, 1935 See Fr. 778,014 (C. A. 29, 3430<sup>1</sup>) When mixts. are sulfonated, the mean  $\text{I}$  value must be at least 100. The products are wetting, washing, dispersing and emulsifying agents.

Washing agent for skin and hair. Ehrhardt Franz Fr. 786,533, Sept. 5, 1935 Washing agents comprise or contain products of the formula  $\text{RXSO}_3\text{Y}$  or  $\text{RXP}(\text{O})_2\text{Y}$ , (where Y is an alkali metal or an org. or an inorg. radical forming a salt, R is an org. radical, preferably aliphatic, of 10 or more C atoms, X is O, S, imino group in which the H atom is substituted by alkyl, aralkyl or aryl, a methylene, alkylene or arylene group, substituted or not, which is joined to the R either directly or by a carbonamide or sulfamide group). Examples are given.

Polishes. Walter R. Weeks Jr. 787,035, Sept. 16, 1935 Emulsions used for polishing lacquered or varnished surfaces are improved by regulating the consistency of the emulsions to a value of the order of 60-120 secs., measured by the Gardner viscometer, and incorporating a buffer soln. to stabilize the  $\text{pH}$  value at 5.2-8.

Treating clay or shales to remove impurities Marshall Lasky U. S. 2,023,426, Dec. 10 Clay or shales contg. volatilizable impurities and a high proportion of hydrated, plastic clay substance are heated in finely divided condition in contact with an oxidizing atm., at a temp. sufficient to oxidize and volatilize impurities and the material is then heated sufficiently partially to dehydrate the clay substance and to form a product that is still of plastic character. App. is described, with superposed hearths.

Alkali-resistant composition for use in manufacture of hard tiles Edmund Claxton and Martin K. Bare (to Armstrong Cork Co.) U. S. 2,022,707, Dec. 3 A considerable mass contg. cumar, m. 100-140°, contains as a toughening modifier an oxidized siccativ oil gel such as one derived from linseed oil and menhaden oil in sufficient proportion to overcome the brittleness of the cumar but in such minor proportion that the hardness of the cumar is substantially unimpaired.

Colloidal dispersions of metals in oils John C. Bird (to Standard Oil Development Co.) U. S. 2,021,893, Nov. 26 A metal such as Ag, Bi, Hg or Cu is colloiddally dispersed in an oil such as white oil, kerosene or gasoline and as stabilizing agents there are employed org. products of disocn or reduction (suitably produced by heating) of an oil-sol. sulfonic acid salt of a metal displaceable by H and a heavy metal sulfonate of an oil-sol. sulfonic acid, such as a Ag sulfonate.

Decalcomania paper. Lewis Davis (to McLaurin-Jones Co.) U. S. 2,023,813, Dec. 10. A decalcomania paper is formed with a suitable backing sheet such as paper with a water-resistant coating to take decalcomania printing and an intervening layer of reousous water-resistant material such as gum sandarac and rosin which is substantially insol. in a solvent such as gasoline, etc., which readily dissolves the other coating, contg. "cumar". Cf. C. A. 29, 5056<sup>1</sup>.

Adhesive carbon paper Harold W. A. Dixon and Robson S. Moore (to Columbia Ribbon and Carbon Mfg. Co.) U. S. 2,022,276, Nov. 26 A base membrane such as paper carries a layer of sol. adhesive material such as

gum arabic or dextrin or glue and a superposed layer of transfer ink compn.

**Colloid reliefs.** I. G. Farberind A.-G. Ger. 614,358, June 7, 1935 (Cl. 57d. 2 02). Addn. to 605,995. The method of 605,995 (C. A. 29, 1907\*) for forming the above by thermoplastic masses is modified by using insol. masses which at ordinary temps. have a low viscosity. Polyvinyl chloride contg. 65% Cl dissolved in MeOH is given as an example of such a mass.

**Grease- and moisture-proof paper suitable for wrapping foods,** etc. John E. Schopp U. S. 2,021,917, Nov. 20. Paper or the like is treated with an aq. dispersion of approx. 23% of rubber latex, casein, modified starch, a filler such as clay, a hygroscopic plasticizing material such as glycerol or glucose and about 4% of shellac, forming when dried a flexible nontacky coating.

**Moistureproof material such as regenerated cellulose suitable for wrapping foods** Wm. H. Charch (to Du Pont Celophane Co.) U. S. 2,022,490, Nov. 26. A smooth nonfibrous sheet of material is provided with a thin moistureproof surface coating which does not become loosened upon prolonged contact with moist substances and which is deposited from a varnish contg. a wax, and a film-forming drying oil or synthetic resin compn. having drying characteristics (the article being flexible without losing its moistureproofness at a temp. of 0°). Numerous examples are given.

**Coated sheet material suitable for films and foils** Arthur L. Petersen (to Celluloid Corp.) U. S. 2,022,360, Nov. 26. A base is coated with a lacquer such as one contg. nitrocellulose and with an overlying layer of gelatin soln., another gelatin soln. layer, and with dichromate hardening soln. Several examples are given.

**Draw sheets or tympan sheets for rotary printing presses** Vulcan Proofing Co. Brit. 433,910, Aug. 22, 1935. A tympan for covering the impression blanket consists of fabric layers united by a resilient layer of natural or artificial rubber or of synthetic resin, with suitable fillers if desired, 1 of the layers having an oil-repellent coating composed of a reaction product obtained from the interaction of a halo-substituted olefin and an alkali polysulfide, e. g., ethylene dichloride and Na polysulfide. A suitable compn. for the uniting layer consists of rubber, 30, ZnO 25, Sb sulfide 5.25, S 3 lb., adol-a-naphthylamine 6, CaO 6, diphenylguanidine 5.4 and stearic acid 0.5 oz.

**Tanning gelatinous printing layers** Valentin Dietz (to Erwin B. Elliott and Maurice C. Boyd) U. S. 2,023,660, Dec. 10. Parts of a gelatinous layer such as one on celluloid or glass are tanned in agreement with the image to be printed by the layer to render such parts incapable of swelling in varying degree by the swelling medium employed, the distensible parts of the layer are caused to swell and are rendered ink-repellent in a bath contg. water, glycerol, a bile salt and an amine such as *p*-phenylenediamine, the unmoistened printing surface of the layer is inked with an ink contg. ox. gall and the inked surface is used for printing.

**Noncorrosive solution suitable for use in radiators,** etc. Frederick A. Weihe, Jr. (to McAleer Mfg. Co.) U. S. 2,023,755, Dec. 10. An aq. alc., glycerol or ethylene glycol soln. is used contg. a small proportion of the reaction product of triethanol and blown castor oil. Blown soybean oil, diamylamine, etc., also may be used.

**Cores for pencils.** American Lead Pencil Co. Brit. 413,995, Aug. 23, 1935. See U. S. 1,985,894 (C. A. 29, 1184\*).

**Transparent partition material.** Jules E. Malivert (to L'accessoire de precision) U. S. 2,023,332, Dec. 3. At least one rigid transparent support such as a glass sheet carries a gelatinous layer of a moisture absorbing

transparent material such as a gelatin or cellulose compn. on at least one side of the support which is protected by a grid material of large mesh.

**Sizing asbestos pulp.** The Raybestos Co. Brit. 433,974, Aug. 23, 1935. Asbestos fibers to be felted to form sheet material are sized by adding to a dispersion thereof in a beating-engine a "glutinous single-phase soln. of a sizing material in colloidal form which is adsorbable by the asbestos" to an extent such that the adsorbed material is substantially insol. in an aq. medium of pH 8-10, the mixt. being beaten until it runs evenly and freely. The sizing material may comprise rosin, causticized starch, glue-CH<sub>2</sub>O or casein. In an example, 2500 lb. and 500 lb. of asbestos of av. lengths of fiber of about 1/4 in. and 1/16 in., resp., are mixed in a beating-engine and causticized starch added slowly.

**Coating wood with metals.** Grumpelt Propellerbau G. m. b. H. Brit. 434,636, Sept. 5, 1935. Wood surfaces are metallized by spraying on a coating of a readily fusible metal, e. g., Al, and then spraying on a layer of a metal, e. g., bronze or steel, having a higher m. p. than that of the 1st coating.

**Composition for waterproofing materials such as fabrics of garments** John B. Cleveland U. S. 2,022,405, Nov. 26. About 2.75 oz. of a mixt. contg. paraffin 4, paracoumarin resin 2, white beeswax 1 and Al palmitate 4 parts is dissolved in each gal. of a solvent mixt. comprising xylol 3 and CCl<sub>4</sub> 1 part by vol.

**Artificial straws,** etc. British Celanese Ltd. Brit. 433,911, Aug. 22, 1935. Addn. to 328,312 (C. A. 24, 5497) and 354,273 (C. A. 26, 4482). A heavy artificial filamentary product formed from a spinning soln. contg. a cellulose deriv. and a plasticizer therefor is subjected to a molding operation under heat to produce permanent irregularities of shape, e. g., crinkles, twists, patterns, frizzed effects. The heavy filamentary product may be formed by coalescing a no. of fine filaments with a total wt. of about 75 denier. The plasticizer preferably acts as a solvent for the cellulose deriv. at elevated temps. Triphenyl or tritolyl phosphate, *p*-toluenesulfonamide, triethylene glycol, diethylene glycol, triacetin, diacetin, di-lb. tartrate, and mixts. thereof, are suitable plasticizers.

**Viscous composition** Nelio Nardelli Jr. 786,553, Sept. 5, 1935. An elastic gum is melted and 5-20% of colophony or other resinous or bituminous substance is added. The product may be used with various addns. on roads or for waterproofing paper and cloth or as a substitute for glue.

**Puncture-sealing composition** Matsuharu Hagino Brit. 431,630, Sept. 5, 1935. The compn. consists of a mineral oil and powder, plaster or gypsum, suitable proportions being 10 of oil to 1 of powder. A granular material buoyant in oil, e. g., cork, may be added.

**Fly-repelling livestock spray** Gideon J. Malherbe (to Shell Development Co.) U. S. 2,023,140, Dec. 3. A pine oil and a mineral oil of approx. the same b. p. range are used together.

**Fireproofing compositions** Rudolph J. White and Wilberforce R. Dauncey. Brit. 433,645, Aug. 19, 1935. The compns. consist of equal parts of K<sub>2</sub>CO<sub>3</sub> and NaHCO<sub>3</sub> dissolved in a suitable liquid, e. g., H<sub>2</sub>O or a paint.

**Fire-extinguishing charge** Francis R. Bichowsky (to General Motors Corp.) U. S. 2,021,981, Nov. 26. A F deriv. of a hydrocarbon such as CCl<sub>4</sub> is used with a chemical decompd. by heat to form extinguishing gases, such as NaHCO<sub>3</sub>. An extinguisher for use with such mixts. is described.

**Automatic and manual carbon dioxide fire-extinguishing system** Scott E. Allen and Orzo N. Wiswell (to C-O-Two Fire Equipment Co.) U. S. 2,023,569, Dec. 10. Various structural, mech. and operative details.

## 14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

**Softening of industrial (process) water by means of trisodium phosphate** Jeannet Tiba 13, 261, 263, 337,

330, 341, 410, 421 (1935) —Expts. are described on the processing of process water for the tanning, textile and other

industries. **Conclusions.** Practically complete softening (hardness of 1-1.5°) can be obtained, even in the cold, by the use of the theoretical quantity of  $\text{Na}_2\text{PO}_4$ . To attain this result it is necessary first to remove  $\text{CO}_2$  and bicarbonates, which in practice can be accomplished by preliminary treatment with lime, or with lime and soda, in a suitable app. The treatment must be adjusted to reduce the hardness to a min. while at the same time producing a softened water having an alk. such that, after  $\text{Na}_2\text{PO}_4$  treatment, the alk. to phenolphthalein is appreciably higher than half the total alk. to methyl orange. The  $\text{Na}_2\text{PO}_4$  treatment is carried out in a relatively small softener, as 30 mins is sufficient to obtain equl and the voluminous  $\text{Ca}_3(\text{PO}_4)_2$  ppt settles rapidly. For a water having a hardness of 6° after preliminary softening, the theoretical  $\text{Na}_2\text{PO}_4$  consumption is about 150 g. per cu. m., and this is readily attained in practice. When the alk. is too high for certain purposes, it can readily be corrected by addn. of small quantities of  $\text{H}_2\text{SO}_4$ . A Papineau-Couture

Arc spectral analyses of some Spanish medicinal waters. III. S. Piña de Rubies and M. Amat Bagues. *Anales soc. esp. fis. quim.* 33, 765-6 (1935); cf. C. A. 26, 1049. —The presence of Ag, Mo and Be is shown in some medicinal waters. E. M. Symmes

Developments during the past three years in the treatment of boiler feed water and water used in the plant. *Inst. Papierfabr.* 33(Tech. Teil) 297-300 (1935).—An address. Louis E. Wise

Removal of oil from condensate for use in boilers. A. C. Stutton. *Power* 79, 582-4 (1935).—Clean  $\text{H}_2\text{O}$  was obtained from a condensate contg. 30 p. p. m. of emulsified oil by coagulation with  $\text{Fe}_2(\text{SO}_4)_3$  at pH 5.5-8.6 and filtration. Boilers are treated to maintain 20-30 grains per gal. of alk., 30 p. p. m. of phosphate and 1000-17,000 p. p. m. of sulfate. Alden H. Emery

Volatility of salts on boiling of their solutions. P. V. Zolotarev. *Trans. Inst. Chem. Tech. Ivanovo* (U. S. S. R.) 1, 54-5 (1935).—Preliminary expts. indicate that the contamination of distd. feed waters with salts is caused by the atomization of overheated boiler scale on contact with water. Chas. B. Banc

The adsorption of phenol by activated charcoals. B. N. Rutovskii and N. I. Antropova. *J. Chem. Ind. (Moscow)* 12, 940-7 (1935).—A. Removal of 99.5% of  $\text{PhOH}$  from waste waters can be reached by passing the soln. over peat charcoal at 40° at a rate of 0.6 m. per hr. For recovery, the  $\text{C}_6\text{H}_5$  from previous expts. is distd. directly onto the C in amts. equal to 30% of the amt. of  $\text{H}_2\text{O}$  used. The C is regenerated by passing steam over it at 170-80° for 1-1.5 hrs. H. M. Leicester

The nature and amount of the colloids present in sewage. VII. The effect of bubbles of gas and agitation on sewage liquors. E. Williams. *J. Soc. Chem. Ind.* 54, 355-61T (1935). cf. C. A. 29, 8192.—Samples of domestic sewage in 1-l. cylinders were treated with streams of (1) air, (2) O, (3) H and (4) N, which passed through glass wool, soda-lime and  $\text{H}_2\text{SO}_4$  and entered the cylinders at the bottom through thin sintered glass filters; the rate of flow was maintained const. at 25 l. per hr. Exptl. results indicate that: (1) Coagulation of the dispersed matter in sewage can be produced by bubbling gas, all 4 gases producing the same amt. of coagulation at 25° over a 6-hr. period. (2) Over a period of 24 hrs. at 25°, O and air are superior to  $\text{H}_2$  and  $\text{N}_2$ , after 24 hrs. the org. C and total N values fall below those of the ultrafiltrate of the control sample. (3) Coagulation can be produced by mech. agitation out of contact with air. (4) The temp.

affects the amt. of coagulation produced, increased temp. increasing the coagulating effect. Complete exptl. data are included. G. H. Young

Treatment of effluents of paper manuf. (Brit. pat. 434,225) 23. Ca hypochlorite pellets for treating water (U. S. pat. 2,023,459) 18. App. for supplying chemicals to water mains (U. S. pat. 2,023,552) 1.

Apparatus (with a column and spiral passageway) for clarifying water by aeration and filtration. Kiyoji Tsuda (to G. K. Tsudashiki Pump Seisakusho). U. S. 2,022,329, Nov. 26. Various structural and operative details.

Apparatus for removing dissolved gases from water or other liquid by heating. Géza Székla. Austrian 143,057, Oct. 10, 1935 (Cl. 85c).

Sand filters. Norman G. Elhot. Fr. 786,601, Sept. 6, 1935. Means for removing and washing the top layer of sand.

Conduit and associated self cleaning screen suitable for water. Joseph E. Bower. U. S. 2,022,336, Nov. 26. Structural, mech. and operative details.

Water-softening apparatus. Dennis Thomas and Thomas & Clement Ltd. Brit. 433,543, Aug. 14, 1935. The app. comprises means interposed between a hopper, contg. water-softening agent, and a mixing tank, adapted to be moved to a charging position with respect to the hopper when the tank is filling and to be moved to a position to discharge its contents into the tank after the latter has been emptied and has begun to refill.

Apparatus for softening and filtering water. John A. Montgomery and Robert O. Friend. U. S. 2,021,919, Nov. 26. Various details are described of an app. comprising a tank contg. a graduated gravel bed on which other water-treating material such as a zeolite bed is placed and through which water may be passed upwardly.

Continuous apparatus for softening boiler feed water by addition of reagents. Chem. Fab. Budenheim A-G. Ger. 620,500, Oct. 22, 1935 (Cl. 85b, 2 01).

Apparatus for degassing feed water. Sergius v. Le Juge. U. S. 2,023,630, Dec. 10. Various details are described of an app. suitable for use on board ship.

Base-exchange substances. Wm. H. Sullivan, Horace Ingleson and Basil A. Adams. Brit. 434,663, Sept. 6, 1935. Base-exchange substances are obtained from clays by treating with concd.  $\text{HCl}$  or with  $\text{H}_2\text{SO}_4$  of equiv. concn., removing adherent acid, if desired, drying and grading, heating in an oxidizing atm., e. g., to 550-600°, and treating the product with a Na silicate-Na aluminate soln. The last step may be carried out by boiling the mixt. and may be followed by treatment with a  $\text{NaCl}$  soln.

Treatment of sewage. Georg Herrmann. Ger. 620,320, Oct. 18, 1935 (Cl. 85c, 6 05). Means is described for directing a stream of fresh water or purified sewage through scum layers in sludge digestion tanks to facilitate the biol. degradation of the scum.

Apparatus for purifying sewage. Office français de travaux d'assainissement. Fr. 787,112, Sept. 17, 1935.

Sewage clarification and disposal. John Schaaf. U. S. 2,022,901, Dec. 3. A granulated filtering medium is continuously deposited upon the dry portion of a screen which is subjected to atm. pressure only and is partially submerged to intercept a sewage stream (various details of app. and operation being described).

Sewage-sludge digesting tank and associated apparatus. Henry L. Thackwell (to Dorr Co.). U. S. 2,023,295, Dec. 3. Structural, mech. and operative details.

## 15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

M. S. ANDERSON AND E. D. JACOB

A study of the uniformity of soil types. Franklin L. Davis. Ala. Agr. Expt. Sta., 45th Ann. Rept. 1934, 18-19.—Lab. and greenhouse studies with 22 soils of 6 different soil types showed greater differences within the type than between types in many cases. The phys. and

chem. properties of soils within a type were very variable. C. R. Fellers

The soils of Scotland. III. The central valley and southern uplands. W. G. Ogg. *Empire J. Exptl. Agr.* 3, 295-312 (1935); cf. C. A. 29, 6341<sup>1</sup>.—Typical soil pro-

files are described and examples are given from a wide variety of parent material. Numerous data are given on  $pH$  values, loss on ignition, exchangeable Ca, Mg and H and compn. of the clay fractions. K. D. Jacob

**Investigation on the soil formation near the apex of the alluvial fan of Azusa River in the central highland of Japan.** Toyotaro Sakai. *J. Sci. Soil Manure* (Japan) 9, 211-51(1935), cf. C. A. 29, 1917.—The morphological feature of the profile and the chem. compn. indicate that the soil is of the weakly acid, sub-alluvial brown soil type according to S's designation. Y. Kamoshita

**The properties of the soil at different degrees of unsaturation in relation to the effects of calcium.** L. P. Magaram. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1934, No. 12, 49-61.—A clay chernozem soil was treated with 0.06 N HCl until it became unsatd. Portions were treated with  $CaCO_3$ , the quantities were varied to obtain degrees of unsatd., and their moisture properties were examd. It was found that the soil fully satd. with Ca possesses the highest hygroscopicity. A slight shift toward alk. is conducive to mineralization of the humus. The various methods for detg. the exchange and hydrolytic acidity give a definite idea about the lime requirement. One and the same method is not applicable to all soils. J. S. Joffe

**An attempt to intensify the cultivation of virgin loess-like podzol by means of chemization.** V. I. Pgorov. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1935, No. 1, 18-24.—Lupines are an important green manure crop for intensifying the cultivation of podzols. The podzol horizon is poor in nutrients and the incorporation of org. matter with the addn. of N, P and K fertilizers makes it possible to plow deeper and increase the feeding grounds of plants. J. S. Joffe

**The theories of the soil absorbing complex from the agronomic viewpoint as a current problem in the chemization of socialist agriculture in the subtropics.** V. G. Tarasovskaya. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1934, No. 12, 36-48.—A discussion of the properties and behavior of the soil absorbing complex in the subtropics, of methods of fertilization and of schemes, based on the laws of the colloidal behavior of soils, which are to be adopted for the most effective utilization of the soils in the subtropics. J. S. Joffe

**Agrochemical study of the absorbing soil complex.** F. N. Germanov. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1934, No. 12, 22-35.—Data of K. K. Gedroitz and his associates are presented on the importance of the exchange cations, the relation between the various cations within the complex and their effect on plants, soil microorganisms and concn. of ions in the soil soln. The disruption of the complex leads in certain soils to demobilization of  $P_2O_5$  by chem. and microbial agencies, the activity of the latter being greatly influenced by the conditions of the weather. It is considered that a study of the relationships existing among the exchange cations, as demonstrated in the 17 tables of data, will result in a productivity classification of soils. J. S. Joffe

**The peptization of soils and the separation of the organic fraction of the soil-absorbing complex.** S. N. Alechin. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1934, No. 12, 65-9.—Mixts. of NaOH and oxalic acid in various concns. and dilns. were used in extg. the org. fraction of the soil complex. The more alk. the mixts. the more org. matter was extd. Dets. were made on the swelling of the soil, depth of color of ext., surface tension of ext., and viscosity. The surface tension was affected but little and the viscosity changed slightly, indicating low hydrophilic properties. J. S. Joffe

**The problem of studying the energy of cation adsorption.** N. I. Gorkunov. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1934, No. 12, 61-4.—Expts. conducted on differential absorption and retention energy of Ca, Mg and H by the soil-absorbing complex show that the energy of adsorption of Ca is higher than that of Mg which is contrary to the work of Wiegner. It is suggested that with the permutites used by Wiegner the formation of

Mg hydroxides took place, whereas, in soils it might not have formed the hydroxides. J. S. Joffe

**The crystalline structure of soil colloids and new data on the exchange of cations and anions in soils.** I. D. Sedletzki. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1934, No. 12, 69-77.—A crit. review of the subject and a discussion of the theories involved. J. S. Joffe

**The influence of exchangeable sodium in soils on the growth of plants and the physical properties of the soil.** I. I. Ratner. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1935, No. 3, 35-45.—A deep chernozem soil was treated with NaCl of different concns. making up soils with different percentage satns. of Na. It was found that even a 5% satn. with Na affects unfavorably the percolation rates of water, capillary rise of water and dispersion. Injury to plants does not become apparent until 50% satn. At 60-70% satn. the plants died. Soils high in org. matter exhibit the unfavorable properties in a higher degree than soils poor in org. matter. The injury of high-Na soil is partly due to the lack of Ca for plant nutrition. In carbonate rich soils the limit of Na injury is lower than in soils free from carbonates of Ca or Mg. J. S. Joffe

**Annual report of the assistant director of agriculture, Bengal, in charge of the agricultural chemist's section, for the year ending 31st March, 1934.** M. Carbery. *Ann. Rept. Dept. Agr. Bengal* 1933-34, II, 81-128.—Compn. of paddy plants. Data are given on the N, P, K,  $SiO_2$  and  $SiO_2$ -free ash contents of the leaves and stems and of the whole plants at different stages of growth and under different fertilizer treatments. There were extremely wide and inconsistent variations in the  $SiO_2$  content of the plants and these variations showed no correlation with either the stage of growth or the fertilizer treatment. Soils. The N NaOH method of dispersing soils for mech. analysis was found to be inapplicable to the salt soils of Bengal which contain a considerable amt. of humified org. matter, and also to salt soils contg. but little org. matter. Chem. analyses of soils from Setabganj, Dinajpur, are given. K. D. Jacob

**The nonprotein nature of a fraction of soil organic nitrogen.** A. W. J. Dyck and R. R. McKibbin. *Can. J. Research* 13B, 261-8(1935).—Not all the N in org. soils is determinable by the Kjeldahl method. In every sample tested the Dumas method gave a considerably higher percentage of N. The differences in N content, as shown by the 2 methods, vary from 6.4 to 29.6%, hence it is believed that an appreciable fraction of the soil org. N may be of nonprotein nature. J. W. Shipley

**Influence of light in the nitrogen cycle in the soil.** N. R. Dhar. *Science & Culture* 1, 213-6(1935).—A review. E. H.

**The decomposition of lime nitrogen and some derivatives of cyanamide in soils.** Shutaro Teshima. *J. Sci. Soil Manure* (Japan) 9, 269-80(1935).—The formation of dicyanodiamide is retarded by the addn. of Japanese acid clay and by the volcanic Komaba soil, because the cyanamide is absorbed and hydrolyzed to urea by the soils. Dicyanodiamide formation in the soil from  $CaCN_2$  is affected not only by the reaction of the soil but also by the nature of soil colloid, which causes the cyanamide to be hydrolyzed. The stabilities of dicyanodiamide and guanidine phosphate in soil depend upon the reaction of the soil; acid reaction is favorable to the latter but not to the former. In both paddy field and dry farm guanidine phosphate, guanidine nitrate and guanidine carbonate decomposed slowly at low temp. but rather rapidly at higher temp.; these salts were decomposed more easily than was dicyanodiamide. Y. Kamoshita

**The chemical compositions of some algae and weeds developing in the paddy field and their decomposition in the soil.** Matsunosuro Shioiri and Shingo Mitui. *J. Sci. Soil Manure* (Japan) 9, 201-8(1935).—In pot expts., slim-forming algae (*Euglena* sp. and *Pleurococcus* sp.) and *Lemma paucicostata* Hegelin, cultured in water-logged soil fertilized with  $(NH_4)_2SO_4$ , converted 6.09-15.28 mg. of total N of the pot into org. form. Analyses of algae and weeds (*Hydrodictyon* sp., *Spirogyra* sp.,

*Iuglena* sp., *Lemna paucicostata* Hegelm, *Hydrilla verticillata* Casp var. Rorburghii, *Najas graminea* Dehliile gave total N 2.20-6.62%, C:N ratio 20-14-6 and lignin 2.91-6.15%. Water-sol N increased with total N. In the paddy field algae and weeds of narrow C:N ratio accumulated NH<sub>4</sub> more vigorously than those of wide ratio in the earlier stage of decomposition, later no difference in NH<sub>4</sub> accumulation was observed between the two types. In dry farming the types of narrow C:N ratio formed more nitrate than did those of wide ratio through the whole period of decomposition. Y. Kamoshita

Reduction of nitrate to nitrite by the green plants. A. L. Sommer. Ala Agr. Expt. Sta. 45th Ann. Rept. 1934, 10-17, of C. A. 29, 5576<sup>9</sup>—Where precautions were taken to prevent the growth of microorganisms little or no reduction occurred in the expressed juices of the roots and tops of the tomato plant. Juices from buckwheat, cotton and wheat showed similar results. No evidence of reduction of nitrate to nitrite for soils with or without the presence of algae in the dark was obtained with the Allison app. On exposure to light the presence of nitrite was detected, the amt. increasing rapidly with the time of exposure. The presence of algae seemed to be favorable to nitrite formation. Where phosphate was not added to the soil and the light intensity was not sufficiently low, more nitrite was sometimes found in the filtered than in the unfiltered soils. The addition of phosphate to the soil greatly increased the rate of formation of the nitrite in the presence of the algae but produced no change when the algae were absent. C. R. Fellers

The lack of mineral salts, especially phosphoric acid, in our soils. Miquel C. Rubino. Rev. mensual asoc. rural Uruguay 1935, No. 6, 35-7—The widespread occurrence of osteomalacia in cattle in some parts of Uruguay is due to deficiency of phosphoric acid in the soil, the phosphoric acid content of which may be as low as 0.10 and 0.3 part per thousand. This condition is aggravated by the annual exportation of approx. 3000 toneladas of phosphoric acid in the bodies of cattle. Remedial measures are suggested. Colin W. Whittaker

The salt soluble phosphoric acid in volcanic ash soils. M. Miyoshi. J. Sci. Soil. Manure (Japan) 9, 232-60 (1935).—Normal soils of KNO<sub>3</sub>, K<sub>2</sub>SO<sub>4</sub>, KOAc, K<sub>2</sub>CO<sub>3</sub> and K nitrate were employed to ext. the P<sub>2</sub>O<sub>5</sub> from volcanic ash soils. The amt. of P<sub>2</sub>O<sub>5</sub> extd. varied with the pH, the kind of salt and the nature of soils. Y. K.

The fineness of grinding raw phosphates. N. D. Smirnov. Khimicheskaya Sotsialist. Zemledeliya (Moscow) 1935, No. 3, 45-52—Coarsely ground rock phosphate—from 80 to 60 and lower mesh—can be used on strongly acid soils, high in moisture. Where P<sub>2</sub>O<sub>5</sub> has to be quickly mobilized, as for spring grains, when applied in the row, when small quantities are available, or on soils satd with bases, finely ground rock is to be preferred. There is no advantage in grinding the phosphate to colloidal size (0.001 mm.). J. S. Joffe

Drying superphosphate in relation to its physical and chemical properties. P. A. Baranov and P. A. Ruzanov. J. Chem. Ind. (Moscow) 12, 1054-7 (1935).—Superphosphate, dried at about 400° with good stirring for 3 min., has excellent properties. A satisfactory product is obtained even if the drying lasts only 0.5 min.

The influence of the composition of the absorbed cations on the development of barley and clover. O. K. Kedrov-Zikhman and O. E. Kedrova-Zikhman. Khimicheskaya Sotsialist. Zemledeliya (Moscow) 1934, No. 12, 9-21—A podzol soil was treated with the carbonates of Ca, Mg, K, Na and Mn in quantities to satisfy the hydrolytic acidity, as detd. by the Calky method. Some samples were prepd with a combination of the cations, and an excess of Ca was added to a no. of combinations. A complete fertilizer was added, the soils were placed in Mitterlich pots, and planted with barley and clover. The soils receiving lime up to 50% of the unsatn. gave a high yield of barley grain. An increase in lime beyond this point decreased the grain yield. Satisfying 80% of the unsatn. with Mg increased the yield of grain and straw

1 Above this point the increase in Mg rapidly decreased the yield. The addn. of mixts of Ca and Mg to satn. the soil from 50 to 100% had no injurious effects and the yields were just as good as those with Ca alone at 50% satn. Thus the injurious effects of Mg are diminished upon the addn. of Ca. Normal yields of barley were obtained with Ca and Mg sol. in a 5 to 1 water sol. ext. at ratios of 13.4 Ca to 1 Mg down to 1Ca:2.3Mg. With clover a 20% satn. of the hydrolytic acidity with Ca and 50% with Mg gave the highest yield in pot. expts. With mustard, winter wheat, buckwheat and beans the results were analogous. The tolerance to high concns. of Mg as compared with Ca varies somewhat with the individual crops. Addns. of Na<sub>2</sub>CO<sub>3</sub> up to 30% and of K<sub>2</sub>CO<sub>3</sub> up to 10% satn. showed favorable results. An increase of K markedly reduced the grain yield. MnCO<sub>3</sub> also gave increased yields, but the limits of favorable effects were a good deal lower—with respect to percentage satn.—than with the other cations. J. S. Joffe

The utilization of calc. tufa-travertine. O. K. Kedrov-Zikhman and V. I. Vinogradov. Khimicheskaya Sotsialist. Zemledeliya (Moscow) 1935, No. 1, 41-53—Travertine as a source of lime was compared with ground limestone and chemically pure CaCO<sub>3</sub> on podzol soils with clover, spring wheat, vetch and oats, and beans. This material compared favorably with other lime materials even without grinding. When ground, the travertine was superior to ground limestone. A no. of analysis of travertine and of the soils used is given. J. S. Joffe

Pasture studies. VII. The effect of fertilization on the nutritive value of pasture grass. E. W. Crampton and D. A. Finlayson. Empire J. Exptl. Agr. 3, 331-45 (1935), of C. A. 29, 1143<sup>1</sup>.—The nutritive value of immature timothy for rabbits was greater than that of immature reed canary-grass. This superiority could not be explained on the basis of quant. differences in crude protein, Ca, P or total nutrients, for in these respects the grasses were very similar, and were equally palatable to the animals. There were strong indications of a marked difference in the nutritive value of herbage from pastures treated with mineral fertilizers (potash 100 and 16% superphosphate 500 lb./acre) as compared with herbage from similar unfertilized pastures, the chem. analyses showed no significant differences. The nutritive value of the herbage in either series was not significantly increased by the addn. of cystine. However, the difference in the nutritive values of the diets contg. fertilized and unfertilized herbage disappeared when a mixt. of cystine and casein was added to the diet of rabbits receiving the unfertilized grass. It is believed that the effect of fertilizers in increasing the nutritive value of mixed pasture grasses is due to their effect in altering the nature of the protein-complex in particular species as well as to their effect in changing the botanical compn. K. D. J.

The significance of sodium in fertilizers for increased yields of sugar beets on a number of soils. D. V. Druzhinin. Khimicheskaya Sotsialist. Zemledeliya (Moscow) 1935, No. 1, 53-62—Sylvinit is superior to the concd. K salts as a source of K<sub>2</sub>O. Its superiority decreases whenever NaNO<sub>3</sub> is substituted for NH<sub>4</sub> sulfate which indicates the effect of the Na of the sylvinit. The NaNO<sub>3</sub> is especially efficient for beets on degraded and deep chernozem, being superior to (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and Ca(NO<sub>3</sub>)<sub>2</sub>. On gray forest loams the nitrate and sulfate are almost alike, sulfate being a little better. This is ascribed to the differential content of Na in the various soils. Similar favorable results with Na-contg. fertilizers are noted on table beets. It is pointed out that in connection with nitrogenous fertilizers contg. no Na the addn. of NaCl is feasible. J. S. Joffe

Chlorine as a factor of crop yields. V. V. Butkevich. Khimicheskaya Sotsialist. Zemledeliya (Moscow) 1935, No. 2, 14 27—Exptl. evidence is presented showing that Cl is energetically absorbed by plants even though they need only small quantities of it. This fact is ascribed to the mobility and diffusion capacity of the Cl which was detd. by membrane and plant expts. It is pointed out that in view of the soly. of its compds. the Cl ion mobilizes

cations. The nitrate ion is still more efficient for it is utilized by the plant and is therefore conducive to a high cation absorption. The more efficient utilization of *Ammophos* upon the addn. of NaCl is explained by the mobility of the Cl ion which stimulates  $\text{NH}_4^+$  absorption. An excess of the chlorides of Na or K decreases the Mg content of the plant and reduces the chlorophyll content. J. S. J.

The causes of the injurious effects of overliming N. V. Katalumov *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1935, No. 2, 42-8.—Adjusting the Ca Mg ratio on 3 types of soils to which lime was added did not correct the injurious effects of overliming on flax. Addns of mixts of Mn, B, Cu, Zn, I and F, of Mn and B, Mn and Balone to the overlimed soils to which a base fertilizer of N, P and K was added brought out the following results: with the mixt of microelements, B + Mn, and B alone the flax grew normally notwithstanding the overliming. The same results were obtained with mustard. J. S. Joffe

The alleged fixation of nitrogen by germinating peas Elizabeth M. Smyth and P. W. Wilson *J. Biol.* 30, 330 (1935)—Vita (cf. C. A. 28, 5831) has maintained that legumes may fix N nonsymbiotically. This may be due to inaccuracies in the Kjeldahl method. The problem should be studied by using gasometric methods of N detn. John T. Myers

Farm manure (sources, use, composition and preservation). José Manáns *Rev. mensual asoc. rural Uruguay* 1935, No. 3, 21-8. Colin W. Whittaker

Coloring mineral fertilizers P. A. Baranov *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1935, No. 1, 77-84.—I or  $\text{NH}_4\text{NO}_3$  metanil yellow or anil yellow could be used to color it for identification purposes. For  $(\text{NH}_4)_2\text{SO}_4$ , Congo red or anil blue, for ferrous sulfate rose color, for  $\text{NH}_4\text{Cl}$  orange B, for a mixt. of  $\text{NH}_4\text{NO}_3$  and *Ammophos* methyl violet, malachite green or orange, depending on whether the mixt. is 1:1, 2:1 or 1:2. J. S. J.

The preparation of concentrated fertilizers by treating phosphorite with nitric acid III. A laboratory study of the treatment of Izyum phosphorite with nitric acid and ammonia or ammonium carbonate G. I. Gershteln and T. I. Khakharina *J. Chem. Ind. (Moscow)* 12, 1057-61 (1935), cf. C. A. 28, 3167.—When the phosphorite is extd. with the theoretical amt. of 25%  $\text{HNO}_3$ , 98% of the  $\text{P}_2\text{O}_5$  is removed. When  $(\text{NH}_4)_2\text{SO}_4$  is added to the  $\text{HNO}_3$ , removal drops to 95%. If 5% less than the theoretical amt. of  $\text{NH}_3$  is added to the soln. obtained by the action of  $\text{HNO}_3$ , the product of evapn. of the filtrate is very hygroscopic. It is better to add  $(\text{NH}_4)_2\text{CO}_3$  and filter the  $\text{CaCO}_3$  off at 45-50°. There is then almost no  $\text{Ca}(\text{NO}_3)_2$  in the evapn. residue, and it is not hygroscopic. Filtration of the solid in the original extn. goes faster if the phosphorite has first been heated to 700° for 0.5 hr., but the  $\text{P}_2\text{O}_5$  removal is only 90-95%. Filtration should go at a higher temp. and the layer on the filter should be thin. The presence of  $\text{NH}_4\text{NO}_3$  in the soln. slows filtration. H. M. Leicester

Plant analyses as a method of determining the fertilizer requirements on a production basis A. Yu. Levitzkii and A. A. Lesyukova *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1935, No. 2, 48-58.—From analyses made at the various stages of growth of oats and wheat, certain ratios of N to P and K<sub>2</sub>O to N were established for the best growth at these stages, and by detg. these ratios from production field units at the first stage of growth it is possible to add the proper fertilizer. These analyses may even afford an opportunity to remedy the conditions on the field examd. by supplementing with a top dressing. A crit. analysis of the various availability methods, as well as of the field method, is given. J. S. Joffe

Determining the placement of fertilizers with the aid of *Aspergillus niger* and *oryzae*. E. V. Bobko, O. G. Nadina and N. V. Yashnova. *Khimizatsiya Sotsialist. Zemledeliya* (Moscow) 1935, No. 2, 35-41.—Expts. are presented showing that *A. niger* and *oryzae* cultures could be used as detectors of fertilizer localizations and thereby utilize them to det. the proper placement of fertilizers. J. S. Joffe

Study of some methods for the determination of phosphoric acid by precipitation as ammonium phosphomolybdate. Application to the analysis of fertilizers. H. Terlet and A. Brian. *Ann. fals.* 28, 546-55 (1935); cf. C. A. 29, 2284.—Scheffer's method, which is based on that of von Lorenz, consists in pptg.  $\text{P}_2\text{O}_5$  as  $\text{NH}_4$  phosphomolybdate by addn. to the hot soln. of a large excess of  $\text{NH}_4$  molybdate soln. all at one time, washing the ppt. with  $\text{Na}_2\text{SO}_4$  soln., dissolving the ppt. in excess of 0.5 N NaOH in presence of  $\text{CH}_3\text{O}$  and titrating the excess of alkali with phenolphthalein as indicator. T. and B. confirmed that, in order to obtain complete pptn. in presence of citric acid, a large excess of precipitant is required, but if it is added all at once there is danger of entraining molybdic acid, thereby giving high results by either gravimetric or volumetric detn. of the ppt. According to Scheffer the ppt. has the following compn.:  $\text{PO}(\text{MoO}_4)_2(\text{ONH}_4)_2 \cdot 2\text{H}_2\text{O}$ , and soln. takes place according to  $4\text{PO}(\text{MoO}_4)_2(\text{ONH}_4)_2 \cdot 2\text{H}_2\text{O} + 112\text{NaOH} + 18\text{CH}_3\text{O} = 4\text{Na}_2\text{MoO}_4 + 8\text{NaNO}_3 + 4\text{Na}_2\text{HPO}_4 + 3(\text{CH}_3)_3\text{N} + 8\text{H}_2\text{O}$ , so that  $1\text{P}_2\text{O}_5 = 56\text{NaOH}$ . This latter ratio was confirmed, and the end point in presence of  $\text{CH}_3\text{O}$  is quite sharp, but no nitric N could be detected in the ppt., which is taken as an indication of entrainment of molybdic acid by the ppt. This entrainment can be prevented by adding the precipitant, drop by drop, and using only a slight excess when no citric acid is present.

When the phosphomolybdate ppt. is washed with a neutral  $\text{KNO}_3$  soln., as suggested by Blair (*Analysis of Iron and Steel*), a variable amt. of the  $\text{NH}_4$  (up to 50%) is displaced by K, the same thing occurs, but to less extent and much more slowly, when the ppt. is washed with  $\text{Na}_2\text{SO}_4$ , as recommended by Scheffer, this reaction does not affect the results of titration in absence of  $\text{CH}_3\text{O}$ , but gives low results in its presence and gives high results if the phosphomolybdate ppt. is detd. gravimetrically. Accurate results can be obtained in both cases by washing first with  $\text{NH}_4\text{NO}_3$  in  $\text{HNO}_3$  and then with  $\text{H}_2\text{O}$ . Detailed directions are given for attacking natural Ca phosphates, fertilizers contg. org. matter (bone phosphates, guanos, etc.), superphosphates, activated phosphates and mixed fertilizers, basic slags, and alkali phosphates, and for pptg.  $\text{P}_2\text{O}_5$  in the soln. of the fertilizer both in the absence and in presence of citrate. A. Papineau-Couture

Spray program for fruit insect control in 1935. C. O. Liddy *Trans. Kentucky State Hort. Soc.* 1934, 134-40.—A mixt. of Bordeaux mixt. (1:5:1:5:100) and soap (1:100) was a satisfactory emulsifying agent for mineral oils, coal-tar oils and pine-tar oil. A satisfactory miscible dormant oil spray for scale insects was composed of (I) KOH (92% flakes) 2.5, oleic acid 8.5, H<sub>2</sub>O 1.5, Cellosolve 1.5 and phenol (85%) 1.0, and (II) dormant oil 82 and iso-amyl alc. 3 parts. I and II are mixed separately, in the order given, and II is then added to I until the mixt. becomes clear. The adverse effects of hard waters on Bordeaux mixt.-mineral oil emulsions were overcome by emulsifying the oil in 2:5:7 Bordeaux mixt. and then adding water and the remaining  $\text{CuSO}_4$  and lime to bring the mixt. up to the standard 0:6:100 ratio. The colloidal structure of Bordeaux mixt. was preserved by the addn. of 2:5-5:0 oz. sugar per 100 gal. of 0:6:100 mixt. The period of effectiveness of Bordeaux mixt. in controlling leaf curl of peaches was increased by the addn. of sugar. The H<sub>2</sub>O-sol. As in Pb arsenate sprays was not increased by the addn. of 0.33-0.50 lb. of specially prep'd tar soap per 100 gal., larger quantities increased the H<sub>2</sub>O-sol. As. The smallest amts. of H<sub>2</sub>O-sol. As were obtained with neutral or slightly acid soaps. The pine-tar oils in insecticidal soaps absorbed small amts. of sol. As and served as a buffer for arsenical injury. The surface tension of Pb arsenate sprays was reduced from 72 to 47-29 dynes per cm. by the addn. of 0.5-3.0 lb. soap per 100 gal. of spray; larger addns. caused no further decrease. The effect of soap in decreasing the surface tension of Pb arsenate sprays was reduced by the addn. of Bordeaux mixt. K. D. J.

Control of citrus insects with oil emulsions L. L. English Ala. Agr. Expt. Sta., 45th Ann. Rept. 1934, 26; cf. C. A. 29, 5533.—Oil emulsions showed marked residual effect in controlling the red spider on Satsuma oranges.



When applied in July and Sept. winter infestation was largely prevented. The tank mix oil spray was more effective than several proprietary emulsions against purple scale and white fly. Five polyhydric alcs. when applied as sprays were ineffective against white fly larvae.

C. R. Fellers  
Physiology of insects with reference to their control H. S. Swingle Ala Agr. Expt. Sta., 45th Ann. Rept. 1933-34, 26-7.—The speed of decomposition of the As insecticides depends on both the H-ion concn. of the soil and upon the anions present. Phosphates are present in the digestive juices of leaf-feeding insects, being especially abundant in those having an alk. reaction. At pH 2-5 the phosphate ion has little effect on As residue decomposition, but at pH 6-10 this ion increases both the speed and extent of decomposition.

C. R. Fellers  
Northern fowl mite and its control W. A. Maw, W. E. Whitehead and L. H. Bemont. Sci. Agr. Expt. Sta., Bull. 430, 23-31 (1935).—The northern fowl mite, *Liponyssus sylvarum* C. & F., which causes severe economic losses on mature fowls and other birds is satisfactorily controlled by painting roosts and the vent area of the fowls with an ointment consisting of 2 parts of petrolatum jelly and 1 part of naphthalene powder. *p*-Dichlorobenzene is preferred in cold weather because of its greater volatility as a substitute for naphthalene. S dust gave fair control of the mite.

C. R. Fellers  
Methods of controlling potato scab A. R. Albert, J. G. Milward and J. C. Walker. Wis. Agr. Expt. Sta., Bull. 430, 23-31 (1935).—Care must be taken not to lime soil reserved for potatoes as scab, *Actinomyces scabies*, seriously affects the value of the crop at soil reactions near pH 7.0. The use of such acid forming fertilizers as  $(\text{NH}_4)_2\text{SO}_4$  and crop rotations are also useful in controlling this disease.

C. R. Fellers  
The control of the root knot nematode, *Heterodera marioni* (Cornu) (Anguillulidae), on tuberoses by hot water and vapor heat Grace W. Sherman Proc. Helminthological Soc. Washington 2, 111 (1935).—The nematode was completely controlled by soaking the tubers in water at 116°F. for 1 hr., or at 118° or above for 0.5 hr. Exposure of the tubers to vapor heat at 124°F. for 0.5 hr. also gave complete control. None of the treatments was injurious to the plants.

K. D. Jacob  
Wild-onion control with creosote kerosene spray. E. V. Smith Ala Agr. Expt. Sta., 45th Ann. Rept. 1933-34, 24-5.—Spraying badly infested plots of wild onions with a 10.90 mixt. of creosote and kerosene at the rate of 218-652 gal per acre in Dec and Jan when the fewest bulbs were dormant markedly reduced the no. of onions the following year.

C. R. Fellers  
Report of the mycological section for the year 1933-34 M. J. Narasimhan Mysore Dept. Agr., Ann. Administration Rept. 1933-34, 19-22.—*Alternaria* on potatoes was effectively controlled by spraying the plants with 1 lb. Ca arsenate in 50 gal. water plus Ca caseinate as a spreader. Eradication of "Seegade Saitte" in paddy fields.—The weed, which forms a thick mat in paddy fields, was killed by application of  $\text{CuSO}_4$  at the rate of 1 lb./3000-6000 gal. water standing in the field. Paddy was not injured by contact with the  $\text{CuSO}_4$  soln. for 16-24 hrs. The weed roots put out new sprouts after about 2 weeks.

K. D. Jacob  
Report of work done in the entomological section for the year 1933-34 T. V. Subramaniam Mysore Dept. Agr., Ann. Administration Rept. 1933-34, 23-7, cf. C. A. 29, 535.—Promising control of the larvae of the tobacco stem borer (*Phthorimaea helioptera*) was obtained by spraying the tobacco seed beds twice weekly with honey oil-resin soap mixt. (1 lb./10 gal. water). The spray was also a very good contact insecticide against the green scale of coffee (*Coccus viridis*). Fish poisons as insecticides.—A 10% aq. ext. of the roots of *Randia demeterum* sprayed against the green scale of coffee gave an 80% mortality of the insects in 4 days. The ext. did not appear to have much toxic action either as a stomach or contact poison against surface grasshoppers. Crude oil emulsion, applied in irrigation water, gave partial control of earthworms in betel-rine gardens.

K. D. Jacob

Derris as an insecticide. David Hooper. *Chemist and Druggist* 123, 149-51 (1935).—The history, botanical and chem. characters and insecticidal properties of derris and its preps are discussed. S. Waldbott

Report of work done in the chemical section during 1933-34 B. Narasimha Iyengar, Mysore Dept. Agr., Ann. Administration Rept. 1933-34, 9-13.—Indigenous plant substances as insecticides.— $\text{CCl}_4$ ,  $\text{CHCl}_3$ , etc. and aq. exts. of *Mundulia suberosa* root bark and *Randia demeterum* (outer soft portion of semiprune fruit and root bark powder, resp.) gradually deteriorated in activity when they were kept in contact with the solvent. The activity was best preserved by pptg. the ext. by pouring it along with solvent into a large quantity of water, filtering the solid and drying and redissolving it in the desired solvent just before use. The aq. ext. of the root bark of *R. demeterum* possesses strong insecticidal properties. K. D. Jacob

The fungicidal action of sulfur. Frank Wilcoxon and S. E. A. McCallan. *J. Biol. Chem.* 109, 441-2 (1935).—S seems to owe its fungicidal power to the formation of  $\text{H}_2\text{S}$  rather than to traces of pentathionic acid in the S. J. T. M.

Household sprays R. R. Le G. Worsley. *East African Agr. J.* 1, 61-2 (1935).—Complete kills of cockroaches were obtained in 24, 48 and 72 hrs. by dusting with powd. *Derris elliptica*, *Tephrosia vogelii* (seeds of leaves) and pyrethrum, resp. Similar results were obtained with sprays prepd. by extg. 0.5 lb. of the resp. powders with 1 gal. kerosene. K. D. Jacob

Physiological problems connected with the use of sodium chlorate in weed control A. S. Crafts. *Plant Physiol.* 10, 699-711 (1935); cf. Aslander, C. A. 21, 792.—A crit. review and discussion of the physiol. responses of plants to chlorates from the standpoint of the practice of weed control. There can be no universally successful method for using chlorates owing to the extreme variations in susceptibility of species and the no. of factors (climatic, etc.) affecting toxicity and absorption. Practical suggestions are given for the successful use of chlorates in (a) humid and (b) arid regions. Twenty-nine references. Walter Thomas

Sheep dips Harold Silman. *Soap* (Sanitary Products Sect.) 11, No. 11, 93-5, 117 (1935).—The specifications, preps., advantages and disadvantages of arsenical, phenolic, sulfur and nicotine dips are reviewed.

Henry H. Richardson

Research and the rubber industry [soil problems] (Eaton) 30 Colorimetric detn. of phosphoric acid in grass (Greenhill, Pollard) 7 Colorimetric method for detg. nitrate N in grass (Ashton) 7 Free flowing powd. S (U. S. pat. 2,022,796) 18

Automatic soil irrigation. Lorenzo A. Richards. U. S. 2,023,490, Dec. 10. App. is described by which water is fed to the soil from a source of supply maintained under slightly less than atm. pressure to provide a slight pressure differential.

Soil disinfectants I. G. Farberindustrie A-G. Brit. 433,944, Aug. 21, 1935. An agent for application to the soil, intended to prevent the attack of plants by rust, consists of a mixt. of an aromatic compd. that contains 1 or more halogen atoms and also 1 or more  $\text{NO}_2$  groups in the nucleus with a fertilizer, other than  $\text{NH}_4\text{NO}_3$ , or an agent that improves the soil. In an example, 5 parts of 1,2,4-trichloro-3,5-dinitrobenzene is mixed with 95 of  $\text{NaNO}_3$ .

Fertilizer. Walter H. Kniskern and Charles K. Lawrence (to Atmospheric Nitrogen Corp.) U. S. 2,022,672, Dec. 3. A fertilizer is prepd. by treating an acidic fertilizer material such as superphosphate material with a soln. of  $\text{NH}_4\text{NO}_3$  and urea in an ammoniacal liquid contg. a substantial proportion of  $\text{NH}_3$  and in which at least one of the constituents  $\text{NH}_4\text{NO}_3$  and urea is dissolved in excess of the quantity necessary for satn. of the ammoniacal liquid in the absence of the other of said materials. U. S. 2,022,673 relates to a liquid fertilizer comprising urea, a fertilizer salt of a strong inorg. acid such as  $\text{K}_2\text{SO}_4$  or  $\text{KCl}$  and a substantial proportion of  $\text{NH}_3$ , at least one of

Transformation of nitrogenous matter during fermentation C Mestre Artigas and A Mestre Jané *Bol. Inst. Investigaciones Agronomicas* 1, No 2 (July, 1935); *Ann. fals.* 28, 563—A study of the harmful influence of ale on the properties of wines obtained by fermentation of musts to which ale. has been added. Conclusion: The cause of the troubles lies in the different manners in which nitrogenous compds are transformed by yeast when the latter commence their vegetative life in presence of ale.

A Papineau-Couture  
The influence of frost on the structure of barley and its behavior in the steep K G Schulz *Wochschr. Brau* 52, 118-19(1935)—Barley samples of various protein content (10.09-14.67%) were tested for mellowness by cutting the kernels both lengthwise and crosswise. The results obtained on the crosscut did not correspond with the length cut. The mellowness obtained on the length cut was higher and gives a more precise idea of the structure. There is no correlation between protein content and mellowness. Samples of very glassy barleys showed distinct improvements after being exposed to a temp. below freezing. The water was taken up somewhat faster in the steep by barley which was exposed to frost. S J.

Influence of moisture and temperature upon the change of substances during kiln drying (of malt) P. Kolbach and E. Schud. *Wochschr. Brau* 52, 129-34, 137-41, 147-50(1935)—The kiln drying process is divided into 3 phases: (1) while the green malt is still growing, (2) when growth ceases and enzymic reactions discontinue, (3) when no more enzymic reactions are taking place and merely chem and physicochem changes take place. While a degradation takes place in the kernel (endosperm) during the first phase, a synthesis is taking place in the acrospire. The correct moisture content required for growth is not ascertained precisely and various figures are given for the crit temp at which growth ceases. The second phase is overlapped by the first and no distinct line can be drawn between the second and third phase. The changes can be divided into 3 groups, i. e., (1) of enzymic nature during growth, (2) those which take place when growth ceases and (3) changes which take place without the action of enzymes. Various changes required for certain "type" malts (pale, Munich, Vienna) are discussed. The technic of the expts is described. The degradation of starch is first studied on green malt of an original moisture content of 43.6% for 8 hrs at 30°, 40°, 50° and 60°, resp. Fermentable carbohydrates and attenuation of aq ext are tabulated. The same expt was repeated with 34.6 and 24.9% moisture. In another expt. one sample contg 41.9% moisture was dried at 20°, 30°, 40° and 50°, a second with 33.5% moisture dried at 30°, 40°, 50° and 60°, a third with 23.2% moisture at 40°, 50°, 60° and 70° and a fourth sample with 15% moisture was dried at 50°, 60°, 70° and 80°. A finished malt with 5.2% moisture and the same sample exposed to air until 11.5% moisture was attained were dried for 8 hrs at 60°, 70°, 80° and 90°. All results are tabulated and discussed. The same scope of expts was carried out to det. the degradation of proteins. Sol N, permanently sol N, coagulable N and formol N were detd in each expt and their increase is graphically represented. Under identical conditions as described above the changes in  $pH$ , titratable acidity and titratable alkali are detd and discussed. S Józsa.

History of hot couch malting H Fink *Wochschr. Brau* 52, 97-100(1935) S Józsa

The interpretation of malt analyses H Lloyd Hind and E N Hammett *J. Inst. Brewing* 41, 429-38(1935), cf C. A. 28, 2119<sup>1</sup>—By modifying Bishop's equation ( $C A 28, 2118$ ) to  $EI = E + 10.6 N - 0.22 G$ , where  $E$  is the ext. of malt per quarter, an "Extract Index" is obtained, which, compared with the varietal factor  $A$ , gives information valuable in interpretation. Index of modification (I. M.) usually is taken as permanently sol N expressed as percentage of total N, it should be designated as such, other I. M. values, as ext I. M., pentosan I. M., etc., should be distinguished. Detn of each adds to the value of the analysis for interpretation. For typical 2-row and 6 row well-modified malts the

permanently sol N. I. M. values are about 36 and 30, the C. I.'s 108 and 103, resp. High cold-water ext cannot always be taken to indicate forcing, since synthetic reactions may compensate for enzymic hydrolysis. Thus total N and permanently sol N should be detd in addn to the standard analyses, these last being inadequate to assess the brewing value of the malt. Quick Landis.

Deterioration of hops during storage observations and preliminary experiments A. H. Burgess *J. Inst. Brewing* 41, 467-81(1935)— $N_2$  and  $CO_2$  do not retard the deterioration of hops,  $O_2$  accelerates it and cold storage retards it to a moderate degree only. Heating to 100° before kilning to destroy enzymes is not effective. Inoculation and sterilization expts show microorganisms to be the cause. *Mucor spinescens*, *Penicillium*, *Aspergillus niger* Van Tieghem rapidly reduce the amt of  $\alpha$ -acid. Q L.

The estimation of extract H Lüers and P. V. Miller *Wochschr. Brau* 52, 153-4(1935)—Bacterial enzyme (Superclastase) is used to liquefy the starch, which is subsequently saccharified by diastase. Results on 36 samples are compared with those obtained by the methods of Graf and Rothenbach-Schultze (*C A 29, 6693*). S J.

Difficult (wine) clarifications I Camplonch Romeu *Bol. Inst. Investigaciones Agronomicas* 1, No. 2 (July, 1935), *Ann. fals.* 28, 563—There exist turbid wines of abnormal compn which are very difficult to clarify by the usual methods. R. studied more particularly wines obtained from incompletely matured grapes, the juice of which was prepd by a continuous pressing process and which was concd slightly to 11°. Such wines contain large amts of malic acid, pectic and albuminoid matter, and even cellulosic compds, which are refractory to filtration or clarification. From a large no. of expts R. concludes that, in order to obtain perfectly clear wines, the  $pH$  must be raised to about 4 by decacidifying with  $CaCO_3$ , and using large amts of gelatin for clarification. Contrary to usual procedure, the gelatin should be added before the tannin. A Papineau-Couture.

Defecation with lime and determination of the volatile acidity of wines P. Jaumes *Ann. fals.* 28, 540-5 (1935), cf C. A. 29, 2294<sup>1</sup>—Defecation with  $CaO$  prior to distn in the Fr. official method for the detn of volatile acidity is shown to give high results owing to partial decompn of the sugars with formation of  $HCO_2H$  and  $AcOH$  (in proportions of approx. 80 and 20%, resp.). This decompn can be avoided by carrying out the  $CaO$  treatment in the cold, but under these conditions practically no defecation occurs. A Papineau-Couture.

Identification of sorbitol in sweet wines C. von der Heide and W. Zeisset *Z. Untersuch. Lebensm.* 70, 353-8(1935)—It is possible to ppt the sugar in sweet wines as  $Ca$  saccharate and then identify the sorbitol in the filtrate. There is no notable loss of sorbitol when this method is followed. A new formation of sorbitol in the course of the sepn is not to be feared. F. L. Dunlap.

Cherry brandy III The bouquet substances of cherry brandy. H. Mohler and W. Hammett *Z. Untersuch. Lebensm.* 70, 329-44(1935), cf C. A. 29, 1934<sup>1</sup>—Both the phys and chem methods employed in these expts are detailed. Biacetyl and benzyl alc have been identified. F. L. Dunlap.

Report on beer samples analyzed during the months of January and February, 1935 F. Ancker *Wochschr. Brau* 52, 119-20(1935)—Summary of 83 bottom-fermenting and 6 top fermenting beers. S Józsa.

Cold turbidity of beer K. Kretschmer and E. Emslander *Wochschr. Brau* 52, 113-17(1935)—The presence of air during racking is injurious for protein stability and taste. Color of glass bottles did not affect turbidity. The same conclusions are drawn for natural and synthetic pitches. Metals, however, show a deleterious effect. Filtration in case of suitable filter material improves the chill-proof qualities but affects the taste adversely. The quality of beer is greatly improved when the husks are not boiled with the mash. However, the chill-proof qualities are adversely affected by this procedure. Resistance toward low temps is decreased by substances

which cause turbidity, are sol. in alc. and have a similar mental effect on the delicate hop flavor. S. Józsa

Vitamin content of beer. A. Scheunert and M. Schieblich. *Z. Vitaminforsch.* 4, 294-9(1935).—Beer manuf. in the usual way contains only vitamins of the B group, and these in small amt; 100 cc. of a dark beer with a high ext. content contained 3 international units of B<sub>1</sub> and approx. 7.1 biol units of B<sub>2</sub>. During manuf. the yeast removes the vitamins, especially B<sub>1</sub> from the beer and stores them. If vitamins B<sub>1</sub> and B<sub>2</sub> are added to the final product prior to its storage, they remain unaltered.

Joseph S. Hefburn

Identification of saccharin and dulcin in beer. Olli Ant-Wuorinen. *Z. Untersuchs Lebensm.* 70, 389-91 (1935).—A simple and workable method is given for sepg these sweetening substances from drinks having a high ext. content, especially beer. The method is based on the adsorption capacity of bone charcoal. The best results are obtained by extg. the adsorbed substances with 94 wt % alc. The methods are described in full. In case the beer contains benzoic acid, it should be removed by extn. with a solvent. Five mg. of saccharin and 10 mg. of dulcin can be identified in 1 l. of beer. Smaller amts. of these sweetening substances have no practical significance. A special advantage of this method is that the possible presence of salicylic and benzoic acids, the most common of beer preservatives, can be detected at the same time.

F. L. Dunlap

The resistance of beer toward *Saccharobacillus pastorianus* J. L. Shumwell. *J. Inst. Brewing* 41, 245-58 (1935).—*S. pastorianus* produces most of the examples of souring or "turning" of beer by further attenuation of the residual sugars in equal with the yeast. A lag phase exists, the value of which depends only on the concn. of the inhibitors, H ion, hop antiseptic and alc., after which acid formation accompanied by sudden increase in turbidity proceeds substantially independently of the inhibitors, and dependent only on the equal residual sugar level.

Quick Landis

Annual report of the fiber expert to the government of Bengal for the year 1933-34. Nirmal Chandra Basu. *Ann. Rept. Dept. Agr. Bengal* 1933-34, II, 51-6.—A new strain of yeast was isolated and found to be quite suitable for the fermentation of waste juice of sugarcane where other kinds of yeast failed. Production of alc. amounting to 2.5% by vol was obtained.

K. D. Jacob

Yeast gum. F. Stockhausen and K. Silberstein. *Wechschr. Brau.* 52, 145-7(1935).—Methods of various investigators are discussed and directions are given for a quant. method with a formula for calcg. the glycogen content.

S. Józsa

Removing beer scale (Ger. pat. 617,555) 12. Citrates [from fermentation mash] (Ger. pat. 620,152) 10. App. for cleaning beer pipes (U. S. pat. 2,022,882) 1

Alcohol. Festi Patendi Aktiaseis. Ger. 617,868, Aug. 27, 1935 (Cl. 68, 29). Alc. is denatured by adding a small amt. of the C<sub>12</sub>H<sub>11</sub> obtained by the fractional distn. of bituminous shale, with or without addn. of other denaturing agents.

Butyl alcohol by fermentation. Winfred N. McCutchan and Cornelius F. Arzberger (to Commercial Solvents Corp.). U. S. 2,023,087, Dec. 3. Filtered distn. slop from a previous fermentation is incorporated in a carbohydrate mash used for the production of BuOH by bacterial fermentation.

Butyl alcohol production by fermentation. David A. Legg (to Commercial Solvents Corp.). U. S. 2,023,368,

Dec. 3. A distn. slop derived from a previous similar fermentation is incorporated with a fermentable carbohydrate mash to be fermented by butyl alc.-producing bacteria, and serves to reduce foaming and facilitate use of a mash of high concn.

Butyl-acetic fermentation process. Hugh R. Stiles (to Commercial Solvents Corp.). U. S. 2,023,374, Dec. 3. A starch carbohydrate mash such as one prepd. from corn meal and contg. 20-40% of hydrol. is fermented by organisms of the type of *Clostridium acetobutylicum* (Weizmann) at a temp. of about 35-37° and with addn. to the mash 0.01-0.1% of an alk. compd. of an alkali metal such as Na<sub>2</sub>CO<sub>3</sub>, capable of forming fermentable salts of acetic and butyric acids, which serve to facilitate the process.

Denaturing hydrocarbons, alcohols and white-spirits. Bozel-Maleta (Soc. industrielle de produits chimiques). Fr. 786,510, Sept. 5, 1935. These are rendered unsuitable as fuels by adding about 3-5% of perchloroethylene.

Bacterial preparations. Hauser & Sobotka A.-G. Austrian 143,291, Oct. 23, 1935 (Cl. 2c). A culture of acid-producing bacteria is treated with, or treated so as to produce therein, a nonhygroscopic, hydrated, sparingly sol. salt of the acid produced by the bacteria. Thus, a culture of lactic acid bacteria may be treated with CaCO<sub>3</sub> to form Ca lactate. The mixt. is then concd. *in vacuo* to such a degree that a solid compn. is obtained on cooling. Stable compns. which neither dry up nor absorb water are obtained.

Apparatus for vinegar manufacture. Hubert L. K. Meynen (to Standard Brands Inc.). U. S. 2,022,970, Dec. 3. A vinegar generator arranged for continuous circulation of vinegar mix through it and for introducing air into the generator during normal operation is constructed so as to exclude air and prevent discharge of the mix on failure of the circulation. Various structural and operative details are described.

Fermentations. Karoline von Gebetattel (nee von Falkenhay). Fr. 786,504, Sept. 5, 1935. Fermentation is favored by adding to the wort ground soybeans from which only the bitter constituents have been removed.

"Breather" or liquid seal for fermentation kegs, vats, etc. Emidio Stella (one-half to Marino De Lorenzo). U. S. 2,023,153, Dec. 3. Various structural details.

Malt. Robert Hoffmann. Ger. 617,602, Aug. 22, 1935 (Cl. 6b 1.01). Malt is pre-treated by wholly or partly removing the husks and subjecting it to a steeping process; it is then added to mash.

Straining apparatus for separating hop residues from beer wort. Gottfried Jakob. Ger. 620,356, Oct. 21, 1935 (Cl. 6b 14).

Brewing kettle for beer, etc. Hans Zirngibl. Ger. 617,781, Aug. 26, 1935 (Cl. 6b 12).

Yeast. Aktiebolaget Båsta. Ger. 617,780, Aug. 26, 1935 (Cl. 6b 1.02). Sulfite waste lye for use as the nutrient in yeast manuf. is pre-treated by neutralizing it with one or more basic substances. The liquid is made sufficiently alk. to neutralize the acid loosely bound to the lignin. In the example, the lye is first treated with finely ground limestone and then with alkali carbonate till an alkalinity of 0.2-0.5 or more is reached. The liquid is then aerated, sepd. from solid matter, and allowed to stand for 6-24 hrs. before use as the culture medium for yeast. Cf. C. A. 29, 5985<sup>1</sup>.

Yeast. Nikolaus Balla. Fr. 786,658, Sept. 7, 1935. Animal proteins are decomposed by hydrolysis in acid medium and the decompn. products obtained are used as nutritive substances for yeast. The animal proteins, may be replaced by keratin, gluten, chondrin, collagen or ossein.

## 17—PHARMACEUTICALS, COSMETICS AND PERFUMES

W. O. EMBRY

A method of the standardization of digitalis. I. A method for clinical standardization. F. R. Dieuaide,

C. L. Tung and C. W. Bien. *J. Clin. Investigation* 14, 725-31(1935).—Details of a method of clinical standardi-

zation of digitals are described. II. The relationship between laboratory methods of assay and potency as determined by experimental cumulative poisoning and clinical standardization. H. B. Van Dyke and R. C. La. *Ibid* 733-7.—Two samples of digitals prepns were found to be equally potent when assayed by dogs and cats but of different potency when assayed by frogs. Cumulative expts with dogs and clinical assays agreed with assays in mammals. J. B. Brown.

Hibiscus as a source of oil. D. Vakulin. *Mashobino Zhivoe Delo* 11, 490(1935).—Seeds of Russian and German *Hibiscus trionum* L. produced 23.8 and 21.77% of oil, resp. The related varieties *Hibiscus cannabinus* and *Abutilon avicennae* gave 17-19% oil. Chas. Blanc.

New procedures in the chemistry of perfumes. A. Lewinson. *Rieschstoff-Ind.* 10, 201-5(1935); cf. *C* A 29, 7579<sup>1</sup>.—A review dealing with ketones. H. M. B.

A colloidal form of quinine ("quinasol"). S. M. Gollandskij. *J. Physiol. (U. S. S. R.)* 19, 563-70(1935).—One vol. of a boiling satd. soln. of quinine sulfate in alc. is added to 1/2 vol. of a 0.25% boiling aq. soln. of a protective colloid, e. g., egg white or a vegetable protein. The mixt. is boiled for 5 min. to remove the alc. Another 1/2 vol. of H<sub>2</sub>O, cooled to 10°, is gradually added to the mixt., which is then set aside to cool. After filtration through a Schleicher and Schüll No. 597 filter, the hydrosol is ready for use. The max. concn. of quinasol is 0.5% of pure quinine in the dispersed phase. The hydrosol is stable for 5-7 days. The therapeutic value of the prepns is being investigated. H. Cohen.

Injections of barbitals. J. J. L. Zwicker. *Pharm. Weekblad* 72, 1273-4(1935).—A non-alk. soln. suitable for injection consists of 20 g. phenobarbital (or other barbitals), 38 g. EtMe<sub>2</sub>COH, 35 g. H<sub>2</sub>NCO<sub>2</sub>Et and 7 g. H<sub>2</sub>O. A. W. Dox.

Estimation of tragacanth. Hans Will. *Apoth. Ztg* 50, 1620(1935), cf. H. Schrader. (*C. A.* 29, 6360<sup>1</sup>). It is shown that viscosity of properly treated and stored tragacanth powder decreases very little over a yr. period, and heating at 110° does not effect sterilization but on the contrary damages the product appreciably. The viscosity and  $\eta_{sp}$  values of good tragacanth gums are not impaired over a 2 month period. W. O. E.

Investigations on drugs, nostrums and cosmetics. C. Griebel. *Apoth. Ztg* 50, 1649-52(1935); cf. *C* A 29, 6359<sup>1</sup>. W. O. E.

Horse-radish. A. Kuhn and G. Schäfer. *Pharm. Zentralhalle* 76, 629-30(1935), cf. Noetzel. (*C. A.* 29, 4518<sup>1</sup>). W. O. E.

Hypericum perforatum. Ernst Drafehn. *Pharm. Zentralhalle* 76, 701-4(1935).—An illustrated article descriptive of the histology and use of drugs. Twelve references. W. O. E.

Occurrence of caffeine in Brazilian drug plants. Friedr. W. Freyse. *Pharm. Zentralhalle* 76, 704-6(1935).—Of the numerous genera and species of caffeine-bearing plants cited, by far the greatest no. belong to *Sterculia* (9 genera and 23 species). The caffeine contents of some 16 different plants are reported. W. O. E.

Relationship of pharmacy to medicine. F. W. Crossley-Holland. *Quart. J. Pharm. Pharmacol.* 8, 321-30(1935).—See *C* A 29, 7018<sup>1</sup>. W. O. E.

Thyroid standardization and dosage. R. F. Corran, J. Pritchard and F. E. Rymill. *Quart. J. Pharm. Pharmacol.* 8, 331-6(1935).—The variation in the total I and thyroxine I contents of fresh and dried defatted glands is so great that there can be no satisfactory correlation between thyroid Brit. Pharm. 1932 and preps. expressed in terms of fresh gland or unstandardized dried defatted gland. The relative activities of thyroid U. S. Pharm. X and thyroid Brit. Pharm. 1932 are approx. 74 = 12-100. The direct detn. of I on the acid insol. ppt. contg. thyroxine yields uniform results giving a useful check on the Brit. Pharm. method. W. O. E.

Determination of the mercury content of mercurochrome. R. F. Corran and F. E. Rymill. *Quart. J. Pharm. Pharmacol.* 8, 340-3(1935).—By the Brit. Pharm. Codex method of assay no. manuf.'s sample examd.

1 conformed to the required standard. Furthermore, the results obtained by the Codex procedure show an appreciable variation. Assay by alk. KMnO<sub>4</sub> oxidation for Hg appears to yield more reliable and consistent values than the Codex method. W. O. E.

Determination of ferrous iron in presence of organic matter by Heisig's method. G. J. W. Ferrey. *Quart. J. Pharm. Pharmacol.* 8, 344-50(1935).—Attention is directed to the suitability of the iodate method for the assay of the saccharated Fe compds. of the Brit. Pharm. and the Brit. Pharm. Codex, and of ferrous lactate. Ferrous Fe can be titrated with accuracy by iodate in the presence of liquid glucose, acacia, tragacanth, sucrose, invert sugar in small amts., levulose, dextrose, lactose, glycerol, lactic and citric acids. Invert sugar in great excess produces a small error. The method is unsatisfactory in the presence of liquorice, marshmallow, gumme, and aq. cochineal ext. W. O. E.

Preparation and properties of Iodoform and thymol iodide. Norman Glass. *Quart. J. Pharm. Pharmacol.* 8, 351-60(1935).—Comments are made on the various requirements of the Pharmacopoeia. The prepns of CHI<sub>3</sub> from Me<sub>2</sub>CO, KI and NaClO<sub>2</sub> is considered in some detail. Micrographs of CHI<sub>3</sub> from various solvents are shown. Two methods of prepns. thymol iodide were studied. The substance resulting from direct iodination differs from that produced when NaClO<sub>2</sub> is the intermediary, since by this procedure a certain amt. of chlorination is effected. W. O. E.

Assay of glyceryl trinitrate tablets. Wilfred Smith. *Quart. J. Pharm. Pharmacol.* 8, 370-4(1935).—Previously published methods are reviewed in connection with a procedure depending on distn., hydrolysis, reduction with reduced Fe, and colorimetric detn. of the NH<sub>4</sub>, whereby only 5 tablets are required for analysis. W. O. E.

Assay of glyceryl trinitrate tablets. H. O. Meek. *Quart. J. Pharm. Pharmacol.* 8, 375-7(1935).—The conclusions formed from a study of colorimetric methods for detg. small quantities of glyceryl trinitrate in tablets are briefly presented, in connection with a procedure depending on the colorimetric detn. of nitrate by means of phenol-disulfonic acid, which has proved reliable and not difficult of control. W. O. E.

Percolation of cinchona and belladonna root—rate of alkaloid extraction and effect of degree of comminution. A. W. Bull. *Quart. J. Pharm. Pharmacol.* 8, 378-85(1935).—Percolation of moderately fine powd. cinchona (44/85) gave better extn. of both alkaloids and total solids, when compared with percolation of either a fine powder (85) or a moderately coarse powder (22/60), thus indicating that an optimum degree of comminution exists for percolation of this drug. The relative percentages of alkaloids to other solids increase in successive fractions of percolate. When the powders were graded and packed in the percolators in progressive sizes, it developed that the most satisfactory extn. occurs when that portion of drug most difficult of extn. (i. e., the finest powder) is packed on top and thus comes into contact with fresh menstruum. With belladonna root similar results were obtained with respect to an optimum degree of comminution. When the ratio of alkaloids is considered it is seen that the relative alkaloidal contents of successive fractions diminish, showing that the alkaloids of this root are more easily extd. than the other extractable material. W. O. E.

Isopropyl nitrite. C. L. M. Brown. *Quart. J. Pharm. Pharmacol.* 8, 380-9(1935).—Isopropyl nitrite can be conveniently prepns. in a state of purity at normal room temp. without serious loss. The yield appears to vary with the area of exposure, hence the best yield is obtained by using a narrow-mouth container. If excess of alc. is used a dild. product results containing approx. 38% by vol. of alc. and having a boiling-range of 42-42.5° and d. 0.8445. These figures agree with those of Kissel (43-44°), and of Silva (45°, d. 0.856 at 0° and 0.844 at 25°). The purified product obtained by B. b. 39.5-40° and had d<sub>4</sub> 0.8634. These values agree closely with those

of Sidgwick, b. 39°; and Bewad, b. 39-39.5°. A soln. of isopropyl nitrite in iso-PrOH is more stable than sweet spirits of niter. W O E

Melting point of chloral formamide. C. T. Bennett and N. R. Campbell. *Quart. J. Pharm. Pharmacol.* 8, 398-400 (1935).—In view of results obtained on some 20 com. samples and others specially prepd. in the lab., it appears that the m. p. of chloral formamide, as given in the Brit. Pharm. Codex 1934, is not in accord with expt. and that it would be more correctly recorded as 124-0°, with pharmaceutical limits between 122° and 126°. W O E

Alcohol content and specific gravities of the tinctures of the British Pharmaceutical Codex, 1934. C. T. Bennett and F. C. L. Bateman. *Quart. J. Pharm. Pharmacol.* 8, 406-8 (1935).—The const. and percentages of the tinctures in question (70 preps.) are presented in classified form for purposes of comparison. W O E

Use of rabbits in the assay of digitalis, strophanthus and squill. G. N. Rapson and S. W. F. Underhill. *Quart. J. Pharm. Pharmacol.* 8, 409-23 (1935).—The rabbit is less sensitive than the cat to ouabain, strophanthus or squill, but more sensitive than the dog or guinea pig to the 2 former. To digitalis it is the most resistant of these 4 species. The advantages in the use of rabbits lie in the short duration of each expt., and the ease with which a stock of animals can be obtained, the disadvantage is the fact that to attain the same degree of accuracy more animals must be used for each test. Where comparison has been made results obtained on rabbits agree with those obtained with the use of cats. W O E

Deterioration of atropine eye ointments on storage. Noel L. Alport. *Quart. J. Pharm. Pharmacol.* 8, 429-34 (1935).—All the atropine eye ointments examd. became weaker in atropine after storage. Ointments contg. atropine base, with or without HgO, deteriorated more rapidly when stored in glycerol-gelatin capsules, which, however, do not accelerate the deterioration of ointments made with atropine sulfate. Yellow eye ointment with atropine Bnt. Pharm. Codex, 1923 loses alkaloidal strength rapidly and is an unsatisfactory prepn. The alkaloidal content of eye ointment of atropine with HgO Bnt. Pharm., 1932 falls to about 1/2 of its original value in about a month and then remains nearly const. for a considerable time. The alkaloidal strength of CH<sub>2</sub>I<sub>2</sub> and atropine eye ointment Bnt. Pharm. Codex, 1934 was well maintained during the period of the observations. W O E

Improved method for the estimation of the essential oil content of drugs. T. Tusting Cocking and G. Middleton. *Quart. J. Pharm. Pharmacol.* 8, 435-42 (1935), cf. C. A. 29, 7014.—The app. previously described (C. A. 27, 564) for the direct detn. of essential oils in drugs and spices has been simplified (illustrated). The effect of powdering on the yields of oil obtained is shown. Details of the conditions of distn. are given for a no. of drugs and spices. Results of the examn. of a no. of samples of drugs and spices are also recorded. W O E

Relative merits of maceration and percolation for the preparation of tincture of digitalis. H. Berry and H. Davis. *Quart. J. Pharm. Pharmacol.* 8, 443-6 (1935).—The processes of percolation and maceration have been compared on a sample of standardized digitalis leaf. The total solids extd. and the potency of the tincture have been detd. for a variety of conditions. Maceration for 2 days, with occasional shaking, exts. the active principle as effectively as percolation, and should give more uniform results. W O E

Spectrographic absorption of ergometrine in relation to the British Pharmacopoeia color test. Noel L. Alport and Sydney K. Crews. *Quart. J. Pharm. Pharmacol.* 8, 447-52 (1935).—Samples of ergometrine have been examd. by the *p*-dimethylaminobenzaldehyde color test and the results compared with the coeffs. of extinction at 316 mμ. The purest specimen of ergometrine obtained, exhibiting an E value of 155 and m. 164°, gave a color by the chem. test equiv. to that produced by 1.78 times its wt. of ergotamine base. Ergometrine in aq. tartaric acid

soln. shows an absorption band in the ultraviolet region with a max. at 316 mμ. The same band is exhibited by solns of ergotamine. The color produced when ergometrine is submitted to the *p*-dimethylaminobenzaldehyde test is spectroscopically identical with that produced by the ergotamine under like conditions. W O E

Determination of lead in its official compounds and preparations. S. Wetherell. *Quart. J. Pharm. Pharmacol.* 8, 453-63 (1935).—The Brit. Pharm. 1932 methods for the assay of Pb preps. have been studied and found to be inaccurate. Alternate methods of assay, which combine sufficient accuracy with easy and rapid manipulation, are suggested as substitutes for the present official methods. Assay procedures for the suppository and plaster are considered and methods of estn. proposed. W O E

Stability of aqueous solutions of ouabain and *k*-strophanthin. H. Berry. *Quart. J. Pharm. Pharmacol.* 8, 464-71 (1935).—Variation in pH affects the stability of aq. solns of *k*-strophanthin and ouabain, but the latter is stable over a wider range of pH than the former. In order to insure perfect stability of *k*-strophanthin solns under ordinary dispensing conditions they should be buffered at about pH 6.5. With ouabain this is unnecessary. *k*-Strophanthin solns buffered at 6.5 can be sterilized by the official process of autoclaving (115° for 1 1/2 hr.) without loss of activity and should be very stable during storage. Unbuffered ouabain solns can be similarly sterilized, and should be very stable during storage. Glass containers for *k*-strophanthin solns should comply with the tests for limit of alkali. W O E

Analysis of some complex ointments of the Brit. Pharm. Codex. D. C. Garratt. *Quart. J. Pharm. Pharmacol.* 8, 472-8 (1935).—Methods are given for the complete analysis of compound benzoic acid, Me salicylate, and simple and compound resorcinol ointments. W O E

Examination of the extractives of capsicum. H. Berry. *Quart. J. Pharm. Pharmacol.* 8, 479-83 (1935).—The so-called oleoresins of capsicum vary in appearance, solv. and degree of pungency according to the solvent used for extn. Oleoresins of capsicum extd. with Et<sub>2</sub>O or Me<sub>2</sub>CO are sol. in Et<sub>2</sub>O, Me<sub>2</sub>CO, fixed oils and turpentine, but insol. in alc., while those extd. with alc. (90%) are insol. in Et<sub>2</sub>O, Me<sub>2</sub>CO, fixed oils and turpentine, but sol. in alc. (90%). When extd. with 60% alc. the oleoresin is insol. in 90% alc. and any of the above solvents. Both Et<sub>2</sub>O and alc. ext. much nonpungent matter from capsicum. Oleoresin of capsicum Bnt. Pharm. Codex 1934 is prepd. by extg. an Et<sub>2</sub>O oleoresin with 90% alc. and is sol. in Et<sub>2</sub>O, 90% alc., Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, petr. spirit, fixed oils and turpentine. It has a greater pungency value than any of the other oleoresins of capsicum, the pungency being about 3 or 4 times that of the Codex of 1923, which was extd. with 60% alc. W O E

The microscopy of powdered desiccated endocrine glands. Heber W. Youngken. *Am. J. Pharm.* 107, 463-71 (1935).—The more important results of studies made by Y. upon the microscopy of powd. desiccated endocrine glands obtained from cattle and hogs and, in the case of thyroid and pituitary from sheep also, are presented with a view toward providing microscopical standards for these prod. products which are becoming more and more extensively employed in modern organo-therapy. Descriptive microscopical standards are presented for powd. desiccated thyroid, suprarenal, whole pituitary, anterior pituitary, posterior pituitary, ovary, ovarian residue and corpus luteum. W. G. Gaessler

The manufacture of bath crystals and bath cubes. M. Lovat Hewitt. *Perfumery Essenc. Oil Recd.* 26, 400-12 (1935). E. H.

Vanishing cream. A. G. Arend. *Perfumery Essenc. Oil Recd.* 26, 423-5 (1935). E. H.

The usefulness of anesthetic agents. L. S. Lundy. *Can. Med. Assoc. J.* 33, 490-4 (1935).—A review. G. H. W. Lucas

The stabilization of plants in relation to phytochemical study and its application in galenic pharmacy. Em. Perrot and A. Goss. *Bull. sci. pharm.* 42, 513-19

(1935)—The fresh plants are heated in an autoclave in vapors of 95% EtOH to 0.5 kg pressure. This destroys enzymes which cause chemical changes during the usual drying process. Exts and alc tinctures prepd. from stabilized drugs are more uniform than such as prepd. from dry material. The active constituents are preserved as present in the fresh plant. A. E. Meyer

International congress of medicinal and aromatic plants. Propositions of modifications to No 1 of C. Lagneau. Analysis of essential oils and unification of its methods. R. Delaby. *Bull sci pharmacol* 42, 520-3 (1935), cf C A 30, 567<sup>4</sup>. A. E. Meyer

Sulfur ointments and their assay. Charles E. Brady and Henry M. Burlage. *J. Am Pharm Assoc* 24, 945-8 (1935).—Schulek's method (C. A. 28, 853<sup>4</sup>) and his iodometric titration of the KCNS (C. A. 17, 3465) is applicable to the assay of the official S ointments and was applied with promising results to samples of the 3 official ointments carefully prepd in the lab and to 2 com. samples of which one was stated to be twice the official strength. A. Papineau-Couture

Note on the assay of mass of ferrous carbonate. John C. Krantz, Jr., and C. Jelleff Carr. *J. Am Pharm Assoc* 24, 958-9 (1935).—The U. S. P. XI prescribes titration of mass of FeCO<sub>3</sub> with 0.1 N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, with diphenylamine as inside indicator. Titration with ceric sulfate, instead of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, gives results that are closer to the theoretical, and the end point is much sharper. When pure FeSO<sub>4</sub> is titrated in the absence of honey and sugar the results with the 2 reagents are identical. A. Papineau-Couture

Studies on cudbear. E. H. Wirth, L. E. Martin and P. G. Soderdahl. *J. Am Pharm Assoc* 24, 949-59 (1935).—Sixteen samples obtained from various sources representing in a fair degree the av. cudbear on the U. S. market were exam'd to ascertain its uniformity, quality and purity. While the com. samples exam'd. exhibited a surprising uniformity in tinctorial value, several of them had been intentionally dild., the most common diluent being potato starch. A colorimetric method for the detn of quality and a color standard are advised. Solv. tests are of questionable value in estg quality. The discussion of purity involves (1) studies on ash content which reveal that many of the com. samples run well under the N. F. max requirements, (2) microscopic studies which indicate considerable adulteration, both intentional (addn. of potato starch as a diluent) and unintentional (presence of woody, bark and leaf tissues). The usual methods for the detection of dye woods do not give satisfactory results, the presence of dye woods as adulterants of cudbear is, however, probably exceedingly rare. The As content varied from 0.25 to 4325 p. p. m., those produced in 1933 and 1934 had smaller As contents, indicating that the presence of As is probably due to carelessness. An As limit of 10 p. p. m. is tentatively suggested. A. P. C.

Assay of liniment of camphor. D. A. Overbye and R. E. Schoetzel. *J. Am Pharm Assoc* 24, 961-3 (1935).—A lab.-prepd sample of liniment of camphor contg 20.00% camphor gave 20.01% when assayed polarimetrically (correction is made for the rotation of the oil), 19.89% when assayed by the U. S. P. XI method (heating 2 hrs. at 110° in a stream of CO<sub>2</sub> and applying a correction of -0.13% obtained by running a blank on the oil), and 19.92% by heating in air (applying a correction of +0.62% obtained by running a blank on the oil). In order to obtain accurate results a blank should be run on the particular oil used in any given liniment of camphor, especially when the assay is carried out by heating in air. A. Papineau-Couture

Tincture of opium. P. L. Burrin and F. E. Bibbins. *J. Am Pharm Assoc* 24, 964-6 (1935).—Tincture of opium prepd according to the U. S. P. contains a very large amt of light, muddy ppt. in a short time. It is suggested that the directions be modified so as to obtain a product which will remain clear for a considerable length of time, and the following method has been found satisfactory. Dissolve 100 g granulated opium in about 200 cc of hot water, let stand overnight, filter, wash to a total

vol. of about 250 cc, boil for 15 min, let stand overnight, add 50 g. of paraffin, heat to melting, beat thoroughly, let cool, puncture the paraffin pellicle and drain off the liquid, wash the paraffin with water to a total vol. of about 800 cc, filter and add 200 cc alc. The amt of ppt in tincture prepd in this way was materially reduced and at the end of 18 months had not shown any further sedimentation, an assay of the clear supernatant liquid showed no loss in potency during this period. Use of caramel as a coloring agent causes an addnl ppt which is undesirable. A. Papineau-Couture

Study of compound cresol solution. K. L. Kaufman and C. O. Lee. *J. Am Pharm Assoc* 24, 959-70 (1935).—The U. S. P. X formula for compd. cresol soln. is objectionable because the time required for prepn is too long, 2 alkalies are used where 1 is sufficient, there is possible a loss of cresol and changes in the product due to continued heating. The following formula has been found free of these objections and is proposed. Place 80 g. KOH in 80 cc H<sub>2</sub>O, when soln. is about 75% complete add 10 cc. alc. and stir till soln. is complete, add to 300 g. of oil which has been previously warmed to about 60° and stir gently, when sapon is complete add 500 cc of cresol in small portions with stirring, remove from the water bath, cool and adjust to 1000 cc. with distd H<sub>2</sub>O. The following oils were studied and were found to be equally suitable for making this prepn: soybean, linseed, olive, peanut, corn, almond, sunflower and sesame. A special study of the suitability of soybean oil showed that the objections which have been raised to its use are unfounded, the problem of gelatinization, which has been reported by several workers, was encountered at times, but in every case the difficulty was traced to the use of inferior grades of cresol, U. S. P. grade cresol giving no trouble. Relative surface tension measurements, det'd by means of a Cenco-du Noüy precision tensiometer, of 2 different dilns of 8 samples of cresol soln. are given. A. P. C.

A history of dentifrices. Martha E. Foull and Elizabeth Pickering. *J. Am Pharm Assoc* 24, 975-81 (1935).

Evaluation of the bactericidal power of essential oils by determination of their phenol coefficients. Y. R. Naves. *Parfums de France* 13, 273-84 (1935) (in French and English).—A description of the technic of the detn. of phenol coeff., more particularly by the Hygienic Lab. and by the U. S. Food & Drug Administration methods (the latter being considered the better of the two), with a discussion of its application to essential oils. A. Papineau-Couture

Essential oil from the rhizomes of *Cyperus rotundus*. Linn. B. Jagannath Hegde and B. Sanjiva Rao. *J. Soc. Chem. Ind. Ind* 54, 387-8T (1935).—The low-boiling fraction was found to be mainly 1- $\alpha$  pinene with traces of cineole. The camphoraceous odor of the oil is due to these substances. In Indian tubers there is less than 0.5% The higher-boiling hydrocarbons are a mixt of tri- and bicyclic sesquiterpenes, the latter predominating, and belong to the eudalene group. A good yield of eudalene is obtained from them on dehydrogenation. A mixt of 2 secondary bicyclic alcs forms about 10% of the oil and has a delicate rose odor. Most of the alcs are tertiary and combine with H<sub>2</sub>BO<sub>3</sub>. A new alc., called *isocyperyl* (C<sub>15</sub>H<sub>24</sub>O), is bicyclic and belongs to the eudalene group. E. R. Rushton

Air conditioning in a pharmaceutical manufacturing plant. C. W. Swenson. *Heating and Ventilating* 32, 31-2 (1935).—Air in the tablet room has 92° dry bulb, 34% relative humidity. M. W. Schwarz

Pharmacy in the Ionian islands. I'm F. Emmanuel. *Chemist and Druggist* 123, 158-9 (1935).—A historical review of pharmacy in the Ionian islands since the time of Venetian dominion. S. Waldbott

Cod-liver oil. John F. Ward and H. E. F. Nottton. *Chemist and Druggist* 123, 246-7 (1935).—The Brit. Pharm. should define the oil as being "obtained from various species of the *Gadus* family"; this would exclude admixts with shark oil (high in unsapon. matter, often high in vitamin A; almost no vitamin D), herring oil and

seal oil (both devoid of unsaponifiable matter and vitamins A or D). Detn. of the vitamin value, notably that of D will disclose an inferior oil. Vitamin standardizing by color tests, the bioassays of A and D and the spectrometric detn. of A and D (cf. Morton, Heilbron, *et al.*, C. A. 21, 3037; 24, 8801; 25, 3380; 26, 5903, 29, 4791) are explained and discussed S. Waldbott

Cologne and lavender waters, modern small scale manufacture O. W. Jarvis *Chemist and Druggist* 123, 373(1935).—Formulas are suggested, based on the use of (British) denatured alc S. Waldbott

Rapid identification of cinchona preparations by the erythroquinine and the thalloquinine reactions R. Monnet, *J. pharm. chim.* 21, 450-6(1935).—A list of quantities of alkaline cinchona preps. to be used in testing is given. Mix or dissolve each in 10 cc.  $H_2O$ , render alk. with 10 drops  $NH_4OH$  or 5 drops  $NaOH$ , add 5 cc.  $CHCl_3$  and shake well, filter over anhyd.  $Na_2CO_3$  through cotton and collect about 4 cc.  $CHCl_3$  ext.; test 2 cc. for erythroquinine (A) (cf. C. 27, 5149, 5362) and 2 cc. for thalloquinine (B) (cf. Christensen, C. A. 11, 1253; French Codex, 1927, 566). A is preferred to B, for exts. and powders it may serve in an approx. detn. of alkaloids S. Waldbott

Determination of silver in organic medicaments, application to pomade of colloidal silver, Georges Antoine, *J. pharm. chim.* 21, 457-63(1935).—The Kahne method of destroying org. substances by the  $HClO_4$  technique (cf. C. A. 28, 1954; 5283, 7420) permits rapid and accurate detn. of Ag in medicinal org. compds. By greasing the walls of the mortar with vasolanolin mixt., before prep. and incorporating the aq. colloidal soln. of Ag, a homogeneous pomade contg. 10.5% Ag (Codex) is obtained, otherwise, results may vary from 7.15 to 8.80%. Twenty-two references. S. Waldbott

The Yugoslavian pharmacopoeia, 1933, Ivan Kalituchanatz, *J. pharm. chim.* 21, 554-8(1935).—Descriptive, with a few crit. notes. S. Waldbott

Hygroscopic properties of the dry extracts of the new (Swiss) pharmacopoeia, Ch. Régün, *Pharm. Acta Helv.* 10, 151-5(1935).—On definite wts. of 7 typical dry exts. the percentage increase of wt. was detd. after 1, 3 and 10 min. and then upon longer exposure under different conditions of humidity in air. Increase during the 1st min. was strongest for digitalis and opium exts. (1.02 and 0.58%). Incorporation of other ingredients must be done rapidly to obtain homogeneous mixts., and the containers must be securely stoppered. S. Waldbott

Oil content (petroleum ether extract) of the cotyledons of *Laurus nobilis* L. L. Rosenthaler, *Pharm. Acta Helv.* 10, 135-7(1935); cf. C. A. 17, 850.—The wt. of 100 cotyledons varied from 0.132 to 0.403 g.; av. 0.249 g., petr. ether ext. heated to const. wt. at 100°, 17.44%, 34.95%; av. 23.32%; the oil content of both cotyledons of the same seed is practically the same; differences hardly exceed 2%. S. Waldbott

Karkade, K. Leupin, *Pharm. Acta Helv.* 10, 138-42(1935).—Karkade consisting of the dried, fleshy, red calyces of *Hibiscus saffordii* L., is recommended as a mild laxative in the form of an acidulous drink. The acid taste of the drug was found by fractional microsublimation to be owing to large quantities of oxalic, malic, citric and tartaric acids. A botanical description of the drug, and 16 references are given. S. Waldbott

An incompatible shaking mixture containing zinc oxide, bismuth nitrate and glycerol, P. Casparis, P. Kämpf and Helene Mitter, *Pharm. Acta Helv.* 10, 143-6(1935).—The mixt. turns black in sunlight, more slowly in diffused light, also in that of the quartz lamp. The following hypothesis is completely verified by expt.:  $ZnO$  (but no other oxide) converts glycerol into its aldehyde when exposed to light and air; the aldehyde then reduces the Bi salt to Bi, again only when exposed to light. In the presence of  $H_2O$ , aldehyde and the Bi salt, blackening in the light did not at first take place, owing to  $HNO_3$  being set free by hydrolysis of the Bi salt; but addn. of  $ZnO$  or other basic oxide caused the mixt. to become black. The black substance was identified as Bi after sepp. it in

aq. mixt. by centrifuging; finely divided Bi proved sol. in weak  $AcOH$ . Darkening on exposure to light also occurs when glycerol is replaced by glycol or mannitol. S. Waldbott

The British Pharmaceutical Codex. A systematic list of vegetable drugs. T. A. Sprague, *Pharm. J.* 135, 214, 232-3(1935).—A botanical classification of all the vegetable drugs of the Codex is given, with frequent brief indications of their functions, e. g., whether an adulterant or admixt., a host plant of a fungus or insect, a substitute, a pathogenic microorganism S. Waldbott

Strong anticonvulsant of mercuric nitrate, Brit. Pharm. 1932, I. H. Giltait and J. C. Jameson, *Pharm. J.* 135, 230(1935).—A pale-yellow ointment of the correct consistency is obtained by following the Brit. Pharm. directions in prep. the Hg soln. and adding it to the mixt. of lard and olive oil, except that this mixt. is heated to 95°, not 150° as directed. Heating with const. stirring was continued for 45 min. S. Waldbott

Nonstaining iodine ointments C. L. M. Brown, *Pharm. J.* 135, 271-2(1935). cf. Richardson, C. A. 29, 4000.—Terbene seems to be a suitable base for the prep. of a nonstaining I ointment, or for stabilizing the pre-v types made with fixed oils. Two types of ointment are possible, lemon-yellow or deep green, depending on the relative quantities of terbene and I used. When I and a fixed oil are heated together in a closed system (reflux), the constituents tend to set up an equal so that a small percentage of I is always present. A formula and method can be used in the prep. of a standard "iodized terbene", from this the ointment can be made by simple diln. An analogous method is suggested for manifold purposes. For dispensing purposes, a mixt. of 1 (22 grains),  $CHCl_3$  and peanut oil (120, 180 minims) was heated on a boiling water bath for 30 min. (with a test-tube cooler suspended in the vapor); terbene (60 minims) and soft paraffin (to make 1 oz.) were added and the mixt. was heated again for 15 min. The product contained no free I; it was deep green and nonstaining to the skin. S. Waldbott

Acriflavine emulsions I. A. Lum, *Pharm. J.* 135, 457(1935).—The oil-in- $H_2O$  type of this emulsion (cf. Woodard, C. A. 27, 4020) is clinically more efficient than the  $H_2O$ -in-oil type (cf. Brit. Pharm. Codex; or Clarke, C. A. 26, 1304). The use of solns. of acriflavine (I) in normal saline solns. instead of emulsions is urged, as the oil present seems to detract from its bactericidal efficiency. Alk. fluid, e. g.,  $Ca(OH)_2$  soln., increase the bactericidal action of I (J. W. Tomb, *The Prescriber*, June 1935, 207). Aromatic acriflavine emulsion, W. Trillwood, *Brit. J.* 457.—An antiseptic desludant in wound dressings is prep. by adding 3 ml. of a mixt. of guaiacol 12.5 ml., eucalyptus oil 25.0 ml., thymol 6.25 g., Me. salicylate 25.0 ml., oil of lemon to make 100 ml., to 100 ml. of T's emulsion of I, prep. from 1 g.  $H_2O$  100 ml., olive oil 100 ml., oleic acid a few drops, sufficient saccharated soln. of lime (Brit. Pharm. Codex) (about 10 ml.) and olive oil or liquid paraffin to make 1 l. S. Waldbott

Digitalis leaf, T. Dewar and T. E. Wallis, *Pharm. J.* 135, 565-6(1935).—The macroscopical and microscopical characters, pharmacol. potencies and constituents of *Digitalis purpurea* L., *D. lanata* Ehrh., *D. hirsuta* L. and *D. thapsus* L. are tabulated and discussed. The botanical characters which differentiate the nonofficial from the official species are emphasized. For the chem. constituents, 15 references are given. S. Waldbott

Ferrous sulfate in mixtures T. O. Martin, *Pharm. J.* 135, 571(1935).—Neither sirup nor  $D$ -glucose seems to prevent oxidation of  $FeSO_4$  in mixts. but addn. of dil.  $H_2SO_4$  Brit. Pharm. (1 minum) to  $FeSO_4$  (5 grains) in  $H_2O$  (2 fluid drachms) will prevent it. S. W.

Properties and uses of some homologs of  $\alpha$ -arylcinnamaldehyde (in perfumery) (Forné) 10. Mineral or tar oil compounds, [for cosmetic or medicinal purposes] (Ger. pat. 620,760) 22. Alkylated phenols [as intermediates for prep. of drugs] (Ger. pat. 616,783) 10.

**Medicinal preparations** I G Farbenindustrie A-G, Brit 433,938, Aug 23, 1935 Polyglycols and their ethers are used as liquefying agents in the production of preps. of stable sterilizable substances such as are suitable for injection and comprise barbituric acid derivs., vitamins or hormones, trihalogenated Et alcs and their urethans, therapeutically active glucosides and alkaloid or synthetic alkaloid like substances. Glycol mono- and di hydroxy-, methoxy- and ethoxy-ethyl ethers are suitable.

**Medicinal substances** Oswald Silberrad Brit 433,062, Aug 8, 1935 Nonfreezing oily substances, suitable for the treatment of rheumatism by rubbing on the skin, are obtained by heating an anhyd salt of salicylic acid with esters of chloroacetic acid in which the ester group consists of 1 or more open chain alc. residues having not less than 3 or more than 8 C atoms, the products being salicylyl glycolic esters. Iso-Pr, Bu, Am, hexyl and octyl esters are thus obtainable. By mixing these compds with the known Me and Et esters, the tendency of the latter to crystallize is inhibited.

**Pure lecithin** I G Farbenind A-G (Gustav Klein and Karl Taubock, inventors). Ger. 617,508, Aug 20, 1935 (Cl. 53: 110) Crude lecithin or materials contg. lecithin are extd. with acetone or a similar solvent and the lecithin pptd. from the ext. by a sol. of an oxide of a trivalent metal or of Si. Thus, crude soybean lecithin or egg yolk is extd. with acetone or CCl<sub>4</sub> and the pure lecithin pptd. by Al<sub>2</sub>O<sub>3</sub> or SiO<sub>2</sub> sol. Cf. C. A. 29, 70214.

**Theretin** from the kernels of "exile nuts" Ku Kuei Chen and Amy Ling Chen (to Eli Lilly and Co.) U. S. 2,022,617, Nov 26, 1935 The kernels are defatted with ether, the residue is extd. with MeOH, the MeOH ext. is evapd. to dryness, the residue is dissolved in abs. EtOH, the soln. is allowed to stand until a sediment forms, the sediment is sep'd., and the supernatant liquid is subjected to fractional pptn. with ether to produce a ppt. of theretin.

**Amino alcohols.** Chemische Fabriken Joachim Wiernik & Co. A-G (Gustav Heilner, inventor). Ger. 615,412, July 3, 1935 (Cl. 12: 32.21) Addn. to 611,501 (C. A. 29, 60604) The alc. 1-phenyl-2-(diethylaminoethyl)-amino-1-propanol is prep'd. by treating 1-phenyl-2-amino-1-propanol with diethylaminoethyl halide or the corresponding sulfonic acid esters or their salts. The reaction takes place in the presence of an acid-binding agent. The alc. b. about 140° at 0.5 mm. and m. 182-183° with decomposition. It is used in therapy for bronchial ailments. Cf. C. A. 29, 58567.

**Amines** Knoll A-G Chemische Fabriken (Wilfried Klavehn, inventor). Ger. 617,506, Aug 26, 1935 (Cl. 12: 19.03) Secondary and tertiary unsatd. amines are prep'd. by treating 6-amino-2-methyl-2-heptene or its secondary derivs. with an alkylating agent. The prep'n. of the above amine or its derivs. and the alkylation may take place in a single operation. In an example, 6-amino-2-methyl-2-heptene is treated with EtBr to give 6-ethylamino-2-methyl-2-heptene, b. 186-188°. Prepn. is described of the following, in which the symbol [1] denotes 2-methyl-2-heptene, 6-methylamino-[1], b. 175-4°, 6-benzylamino-[1], b. 160-161° at 12 mm., 6-dimethylamino-[1], b. 183-5°, 6-methylbenzylamino-[1], b. 164° at 5 mm., and 6-methylethylamino-[1], b. 197-9°. The compds. are used in medicine.

**Stable colloidal dispersion of iodine** Wm. M. Malisoff (to Mackie-Henkels, Inc.) U. S. 2,022,729, Dec 3, 1935 A compn. suitable for use as a therapeutic and antiseptic is prep'd. by agitating free I with a dispersion of  $\alpha$ -amylose and starch residues substantially free from  $\beta$ -amylose.

**Acridinium compounds** I G Farbenindustrie A-G Brit 434,497, Sept 3, 1935 See Ger. 605,123 (C. A. 29, 12124).

**Guaiacol compounds** Salo Rosenzweig U. S. 2,023,551, Dec 10, 1935 Therapeutic compds. are formed by reaction of a phosphoric acid partially esterified with guaiacol such as diguaiacolphosphoric acid with an org. basic compd. such as triethanolamine, quinine or ethylenediamine.

**Alkyl chlorodihydroxybenzenes** Wm. E. Austin U. S. 2,023,160, Dec. 3, 1935 Germicidal and antiseptic

compds. of the general formula C<sub>6</sub>H<sub>2</sub>Cl(OH)<sub>2</sub>CR<sub>1</sub>R<sub>2</sub>R<sub>3</sub>, where R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> are alkyl groups, either straight-chained or branched, and the OH groups being either in ortho, in meta, or in para position to each other, such as the tertiary butyl, the tertiary amyl the tertiary hexyl, the tertiary heptyl, the tertiary octyl and the tertiary nonyl derivs. of chloropropylcatechol, of chlororesorcinol and of chlorohydroquinone, may be prep'd. by introducing the Cl into the corresponding tertiary alkyl dihydroxybenzene, or by introducing the desired tertiary alkyl group into the chlorodihydroxybenzene derivative. *tert*-Butylchlororesorcinol, *tert*-amylchlororesorcinol, *tert*-amylchlorohydroquinone, mono-*tert*-octylchlororesorcinol, *tert*-amylchloropropylcatechol and mono *tert*-octylchloropropylcatechol are all described, with details of their production.

**Colloidal sulfur solutions** The Drug Products Co., Inc. Brit. 433,833, Aug 21, 1935 An isotonic soln. for injection purposes is prep'd. by causing aq. solns. of a sulfide and SO<sub>2</sub> to react in the presence of a dispersing agent and in correct proportions to insure isotonicization.

**Common salt composition** Siegfried Michael Ger. 617,772, Aug 26, 1935 (Cl. 53k 1.01) A diet-therapeutic substance free from alkali and halogen for addn. to common salt consists of an aliphatic hydroxy acid, such as malic acid, neutralized with an aliphatic base contg. one or two NH<sub>2</sub> groups and two or more OH groups in the mol., such as 1-amino-2,3-propanediol.

**Sex hormones** Schering-Kahlbaum A-G Brit. 434,380, Aug 30, 1935 Alcs. contg. a tertiary OH, obtained from follicle hormones according to the process of specification No. 30297/8/34, in so far as they are of aromatic or unsatd. character, are hydrogenated to the satn. of the double bonds. In an example, 1.5 g. of the tertiary alc. of formula C<sub>17</sub>H<sub>25</sub>O<sub>2</sub> prep'd. by the action of CH<sub>3</sub>MgI on cryst. follicle hormone is dissolved in 150 cc. glacial AcOH and 200 mg. Pt oxide added according to the method of Adams Schringer, the mixt. is shaken for 18 hrs. in a H atm. and the catalyst removed, a product being obtained of formula C<sub>17</sub>H<sub>25</sub>O<sub>2</sub>.

**Whooping-cough vaccines** Schering-Kahlbaum A-G Brit. 433,910, Aug 22, 1935 Multivalent vaccines are prep'd. from whooping-cough bacilli by cultivating a no. of stocks of the bacillus Bordet-Gengou taken from different epidemics, selecting those stocks which after long continued cultivation upon artificial culture media show const. cultural properties and toxin formation, combining them and working them up together to form a vaccine. The bacteria may be disintegrated into an amorphous mass by repeated freezing and cooling, if desired while suspended in physiol. salt soln., and the product is then centrifuged to sep. the antigen-contg. liquid. By exposure to heat, e. g., at 100°, for 10 min., the poisonous antigens may be eliminated and a nonpoisonous vaccine produced. The culture media should contain a high proportion of blood.

**Antigangrene serum** Franz Schütz. Ger. 617,668, Aug. 23, 1935 (Cl. 304.6) A mixt. of toxic substances which produce skin gangrene are introduced into animals, blood serum is obtained from the animals and this is worked up in the usual way. The toxins for inoculation may be obtained from a gangrenous animal.

**Maggot extract** Stanton K. Livingston U. S. 2,022,890, Dec 3, 1935 A substantially physiol. salt soln. is used for extg. maggots to obtain an ext. which can be used for therapeutic purposes.

**Antiseptic and therapeutic aryl mercuric heterocyclic carboxylates.** Carl N. Andersen (to Lever Bros. Co.) U. S. 2,022,997, Dec 3, 1935 Compds. such as phenylmercuric furoate, m. 115°, phenylmercuric pyridine-2,3-dicarboxylate, m. 163-5°, phenylmercuric 2-phenylquinoline-4-carboxylate, m. 206-8°, phenylmercuric nicotinate, m. 193-5°, phenylmercuric 6-methyl-2-phenylquinoline-4-carboxylate, m. 162-3°, and phenylmercuric coumarin-carboxylate, m. 205-6°, are produced by the reaction together of the heterocyclic acid involved and a Hg compd. such as phenylmercuric hydride. Various examples with details of procedure are given.



**Fungicides suitable for killing ring-worm organisms.** Herman A. Bruson (to Resinous Products & Chemical Co.), U. S. 2,022,185, Nov. 26. Solns., ointments or dusting powders are used contg. active agents such as *p*-isopropylsahelylic acid, *p*-sec-butylsahelylic acid, *p*-tert-butylsahelylic acid or various other similar alkylated sahelylic acid derivs.

**Isinglass plasters.** Johnson & Johnson Brit. 433,460, Aug. 9, 1935. Dried fish sounds are immersed in  $H_2O$ , the mass is brought to the b. p. to render it sterile, an antiseptic, e. g.,  $PhOH$ , cresol, hydroxyquinoline,  $HgCl_2$ , or mercuriochrome, is added and the mass is immediately spread on fabric or other backing to form the plaster which is then dried, cut to shape and rolled on storage reels. The soln may be filtered and decolorized with animal charcoal before addn of the anti-septic.

**Cosmetics.** I. G. Iarbusindustrie A. G. Brit. 431,142, Aug. 9, 1935. Insol nonpoisonous metal salts of aliphatic acids contg. at least 7 C atoms and contg. an odd no. of C atoms are used as ingredients of cosmetics. Among examples, (1) a rice powder contains rice flour,  $Ti$  white,  $Zn$  or  $Mg$  undecylate and  $MgCO_3$ , and (2) a

face cream contains stearin saponid. in an aq. mixt. of borax, glycerol and  $K_2CO_3$ , and then mixed with  $Zn$  or  $Mg$  undecylate.

**Cosmetic pastes.** J. G. Mouson & Co. Ger. 620,843, Oct. 28, 1935 (Cl. 30h, 13). Addn to 601,475 (C. A. 28, 7434f). The mixt. of colloidal silicic acid and  $Al(OH)_3$  used according to Ger. 601,475 is replaced by other inorg. gels, e. g., hydrated  $MgO$  or  $TiO_2$ . Sp. compns. are described.

**Face cream.** Barney J. Dryfuss and Eugene F. Aubry, Jr. U. S. 2,023,125, Dec. 3. A face cream is formed chiefly of condensed buttermilk having its protein content in substantially unhardened form and unctuous material such as lanolin and liquid petrolatum emulsified with the condensed buttermilk.

**Face paints.** Hélène Winterstein (nee Viethaler). Fr. 787,071, Sept. 10, 1935. See Austran 112,033 (C. A. 29, 6366f).

**Rouge for lips.** Sébastien Sabatay. Fr. 780,828, Sept. 10, 1935. In addn to the ordinary constituents rouge contains fat acid salts of triethanolamine, e. g., the stearate, and glycerol or other liquid polyhydric alc.

## 18—ACIDS, ALKALIES, SALTS AND OTHER HEAVY CHEMICALS

### R. M. SYMMES

**The preparation of chemically pure hydrochloric acid.** T. V. Polyanski. *J. Chem. Ind. (Moscow)* 12, 921-3 (1935).—Crude  $HCl$  gas is passed slowly through a  $HCl$  soln and a column of cold, activated charcoal into a column of C heated to 200–300°. The gas leaving this column should not be below 250°. It then passes through a cond.  $HCl$  soln contg. suspended C, and another column of cold C, and is absorbed in distd  $H_2O$ . Conn. app. is described.

**Intensity of acid formation in the interaction of sulfur dioxide and nitroso acid.** I. N. Kuz'minikh and I. I. Surkov. *Khimstrol* 7, 473-9 (1935), cf. C. A. 29, 297f, Kuz'minikh and Yudin, C. A. 28, 7433f.—A discussion, with math. treatment, is based chiefly on the previous investigations.

**Calculation of the oxidation of nitrous oxide in the sulfuric acid systems.** A. M. Savinov. *Khimstrol* 7, 110-20 (1935).—Improvements of the Beskov nomograms (cf. C. A. 28, 4181f) are proposed.

**A most rational method of feeding sulfuric acid towers with nitrogen oxides.** A. Urinson. *Khimstrol* 7, 494-7 (1935).—A discussion, with math. treatment.

**Washing the Gay Lussac tower of the tower sulfuric acid system.** G. I. Iolova and I. A. Kuziak. *J. Chem. Ind. (Moscow)* 12, 921-3 (1935).—Details are given of washing such towers with  $H_2O$  and recovering the weak acid so formed.

**New type of acid sprayer.** M. A. Ilmich. *Khimstrol* 7, 431-5 (1935).—A device for acid spraying in the production of  $H_2SO_4$  is illustrated and described in detail.

**Handling and storing acids.** T. J. Dixon and I. Roffey. *Oil Colour Trades J.* 88, 1633-4 (1935).—A consideration of the materials of construction resistant to corrosion and a description of certain items of equipment. The materials considered include: cast Fe, mild steel, alloy steels, Al, Pb, Ag, Cu, rubber, glass, stoneware, vitreous enamel, and synthetic resin compds. Also in *Chemistry & Industry* 1935, 1104-9.

**Laboratory apparatus for ammonia synthesis.** V. A. Postnikov. *Trans. Inst. Chem. Tech. Ivanovo (U. S. S. R.)* 1, 66-7 (1935).—Illustration, with construction details.

**American potash mines prepared to produce over a million tons of crude salts annually.** Howard I. Smith. *Am. Inst. Mining Met. Engrs., Contribution No. 84*, 11 pp (1935).—Recent developments in the domestic potash industry are discussed.

**New method for barium chloride.** Alden H. I. mery. Decomposition of barium sulfate by calcium chloride in aqueous solution. R. Norris Shreve and Wm. N. Pritchard, Jr. *Ind. Eng.*

*Chem.* 27, 1488-91 (1935). In cond. aq. solns.,  $CaCl_2$  decomposes  $BaSO_4$ , forming  $BaCl_2$  and  $CaSO_4$ . Study of the reaction was possible only by the use of org. or non-ionizing solvents, e. g., ethylene glycol, or better a  $MeOH$ -ethylene glycol mixt., which is less viscous at high  $BaCl_2$  concns. Conversions up to 90%, even at 100° in 12 hrs., are obtained, and by fusion, up to 95%. The solvent may be recovered.

**The quality of the lime used in producing chloride of lime.** M. F. Pozin. *J. Chem. Ind. (Moscow)* 12, 926-32 (1935).—The fineness of particle size, rapidity of slaking and reactivity of lime toward  $Cl_2$  are increased when it is prepd. from  $CaCO_3$  by igniting at the lowest possible temp. In practice, this is 1000–1100°. No free  $CaO$  should be left in the  $Ca(OH)_2$ , since during chlorination this will be eluted, and will cause local overheating. A moisture content of 0–7% in the lime does not greatly affect chlorination, but the product keeps better if it is drier; hence the lime should contain about 0–0.5%  $H_2O$  before chlorination. The dispersity of the lime particles increases with standing, hence older lime chlorinates better.

**Production of sodium ferrocyanide.** T. I. Kunin. *Trans. Inst. Chem. Tech. Ivanovo (U. S. S. R.)* 1, 73-6 (1935).—In the production of  $Na_2Fe(CN)_6$  from animal waste, a considerable part of  $Na_2CO_3$  in the melt can be replaced by  $NaCl$ , without affecting the yield and crystn.

**The production of sodium ferrocyanide from calcium cyanamide.** V. F. Postnikov, T. I. Kunin and A. Kh. Iironnikov. *Trans. Inst. Chem. Tech. Ivanovo (U. S. S. R.)* 1, 77-86 (1935).—The effect of various factors in the production of  $Na_2Fe(CN)_6$  from  $CaCN_2$  by decomposing the charge in a gas-heated oven and converting the  $NaCN$  into  $Na_4Fe(CN)_6$  was investigated. The charge should be heated with a reducing flame and a gas with only a few tenths of 1% of  $O_2$ , because of the adverse effect of  $O_2$  on the yield of  $NaCN$ . In batches consisting of a mixt. of  $CaCN_2$ ,  $Na_2CO_3$  and C (coal) a large excess of  $Na_2CO_3$  (about 100%) gave increased yields of  $NaCN$ . The best results, with 27.65% N utilization, were obtained by heating the charge at 800° for 10 min. A 100% substitution of  $NaCl$  for  $Na_2CO_3$  in the charge produced only 5.6% N utilization. By replacing 30–50% of  $Na_2CO_3$  with  $NaCl$ , the yield of  $NaCN$  was increased. The best results were obtained with a mixt. of 43.1%  $CaCN_2$ , 21.11%  $Na_2CO_3$ , 21.11%  $NaCl$  and 8.6% C by heating it at 950° for 10 min. By a sudden cooling of the melt (plunging the baker into cold water) the reverse reaction was stopped and the N utilization was increased from 32.2 to 39.1%. Addn.

of Fe or  $\text{Fe}_2\text{O}_3$  to the mixt. gave neg. results. Freshly prep'd and unoxid.  $\text{CaCN}_2$  gave better results. In the conversion of  $\text{NaCN}$  to  $\text{NaFe}(\text{CN})_6$ , an excess of 5%  $\text{FeSO}_4$  improved the yields. After 1 recrystn., 98.5%  $\text{NaFe}(\text{CN})_6$ , free from  $\text{Na}_2\text{CO}_3$ ,  $\text{NaCl}$  and other impurities was obtained. Similar results were obtained with the use of recovered  $\text{Na}_2\text{CO}_3$  and with com.  $\text{Na}_2\text{CO}_3$  and  $\text{NaCl}$ .

Chas. Blanc

Trisodium phosphate and its commercial uses. Ernesto Turegano *Quimica* 12, 182-7 (1935); cf. C. A. 29, 7024<sup>1</sup>.—Com. uses of  $\text{Na}_3\text{PO}_4$  as a detergent are described.

L. E. Gilson

Interaction of sodium sulfate and pyrites. V. P. Postnikov, A. Kh. Bronnikov and I. P. Kamilov. *Trans. Inst. Chem. Tech. USSR* (U. S. S. R.) 1, 68-72 (1935).—Neg. results were obtained in the decompn. of a mixt. of botation pyrites and dry  $\text{Na}_2\text{SO}_4$  in a current of air at 1200°, 1300° and 1400° for the production of  $\text{SO}_2$  and  $\text{Na}_2\text{FeO}_4$ .

Chas. Blanc

Liquefaction of nitrogen dioxide under pressure. Direct production of concentrated nitric acid. P. I. Pronin. *Khimist* 7, 325-401 (1935).—For the direct production of 97-85%  $\text{HNO}_3$  by autoclaving a mixt. of dil.  $\text{HNO}_3$  and liquid  $\text{N}_2\text{O}_4$  at 70°, the  $\text{N}_2\text{O}_4$  was obtained from a mixt. of  $\text{NO}_2$  and  $\text{NO}$ , prep'd. from  $\text{NaNO}_2$  and  $\text{H}_2\text{SO}_4$ , with different proportions of O at 6, 9 and 10 atm. pressure and at various temps. and gas velocities. The app. and procedure are illustrated and described in detail. The optimum temp. of liquefaction is between -10° and -15°. The practical liquefaction effect is somewhat higher than the theoretical, because the water vapor in the system forms with N oxides  $\text{HNO}_3$ , which gives a mixt. with  $\text{N}_2\text{O}_4$  with slightly lower vapor tension than that of pure  $\text{N}_2\text{O}_4$ . The use of welded Al in the app. is practicable.  $\text{CaCl}_2$  cooling brine causes destruction of Al parts.

Chas. Blanc

Adsorption of nitrous oxides by activated carbon. V. T. Ruff. *Khimist* 7, 408-72 (1935).—Comparative lab. expts. with the recovery of  $\text{NO}_2$  from waste gases of the  $\text{HNO}_3$  production from  $\text{NH}_3$  showed that activated C is a better adsorbent than silica gel. The best results are obtained by cooling C with running water at 10-12°. The regeneration of the activated C is best effected at 150°. The C is unchanged after repeated operations of adsorption and desorption of  $\text{NO}_2$ .

Chas. Blanc

The purification of gases from hydrogen sulfide by oxidation on activated charcoal. S. Kazanovskii and K. Pisarev. *J. Chem. Ind. (Moscow)* 12, 913-21 (1931).—Gas contg. 25% excess O over the amt. required to oxidize the  $\text{H}_2\text{S}$  to  $\text{S}$  is heated with 3%  $\text{NH}_3$  to 70-85° and passed over C. The temp. of the reaction rises to 115-20°. If much  $\text{H}_2\text{O}$  is present, or is formed, higher temps. are required. The C is ext'd. for 1 hr. at room temp. with  $(\text{NH}_4)_2\text{S}$  soln. The soly. of the S is roughly proportional to the concn. of the soln. The ext. is heated to 95° to recover the S.

H. M. Leicester

Investigations in the preparation of iodine from seaweeds. I. Dry distillation of seaweeds. Chieh Ma. *Pharm. Chem. Research Reps.* (China) 1, No. 1, 51-9 (1935).—Dry distn. of a typical Chinese seaweed gives C 35.8, mixed salts 11.7, black alk. distillate ( $d_4^{20}$  1.021) 12.5, yellow alk. distillate ( $d_4^{20}$  1.027) 7.1, tar 1.0 and gases (by difference) 31.9%.

C. I. Tseng

A new method for reworking of animal waste into cyanogen compounds. V. P. Postnikov and T. I. Kunin. *Trans. Inst. Chem. Tech. USSR* (U. S. S. R.) 1, 87-9 (1935).—Destructive distn. of animal refuse (hooft and leather waste, contg. about 10% N) at 800° and conducting of the decompn. gases over C at 100° are described. By dilg. the decompn. gases with the waste gives a yield of 11-13.5%  $\text{HCN}$  and 50%  $\text{NH}_3$ , or about 60% utilization of the available N, was obtained. The waste gases are used as a fuel. About 4% of an oil was obtained as a by-product.

Chas. Blanc

Peat blacks. A. V. Pankov. *Trans. Inst. Chem. Tech. USSR* (U. S. S. R.) 1, 158-61 (1935).—Chem. and phys. properties of blacks obtained by carbonization of peat at 600-1000° are described.

Chas. Blanc

Removing  $\text{SO}_2$  from flue gases (U. S. pat. 2,021,936) 21.

Nitric acid. Giacomo Fauser. Fr. 787,053, Sept. 16, 1935. Gases obtained by the combustion of  $\text{NH}_3$  are cooled to a temp. at which a portion of the  $\text{NO}_2$  seps, as liquid, and washed with dil.  $\text{HNO}_3$  to absorb the remaining  $\text{NO}_2$ . A part of this soln. is heated to give off  $\text{NO}$ , which is caused to react, along with the  $\text{NO}_2$  from the other step, with O and a soln. of  $\text{N}_2\text{O}$  in dil. acid, under pressure. Conc'd  $\text{HNO}_3$  is obtained.

Bubble tower for use in the production of nitric acid. E. I. du Pont de Nemours & Co. Brit. 433,272, Aug. 6, 1935. Divided on 432,898 (C. A. 30, 578<sup>1</sup>). The tower comprises cooling coils and deflecting means supported on the trays and surrounding the bubbler caps, said deflecting means, e. g., of crushed quartz, ceramic rings, fins or metal grids, being limited in extent so that a considerable space exists between the upper surface of the material and the next higher tray.

Nitrogen oxides. Bama-Meguin A-G. Fr. 786,953, Sept. 14, 1935. Gases from the combustion of  $\text{NH}_3$  are submitted to a very rapid cooling during which the water seps; they are then submitted to oxidation and washed with very conc'd.  $\text{HNO}_3$ . Very conc'd.  $\text{HNO}_3$  is obtained.

Absorbing nitrogen oxides. N. V. de Bataafsche Petroleum Maatschappij. Brit. 434,302, Aug. 29, 1935. See Fr. 777,042 (C. A. 29, 3791<sup>1</sup>).

Sulfuric acid. Henry F. Merriam (to General Chemical Co.) U. S. 2,023,203, Dec. 3. A  $\text{SO}_2$ -contg. gas heated to at least about 650° is passed into contact with catalytic material to form  $\text{SO}_3$ , and during the conversion reaction heat is removed sufficient to permit formation of com. yields of  $\text{SO}_3$ . The catalytic reaction is carried out in a plurality of stages, vaporization of a heat-transfer liquid serving to cool the first stage and resulting vapor being used to cool catalytic material in a second stage. App. is described.

Sulfuric acid. Metallgesellschaft A-G. Brit. 433,676, Aug. 19, 1935. In producing highly conc'd.  $\text{H}_2\text{SO}_4$  by the condensation of gaseous mixts. of  $\text{SO}_2$  and  $\text{H}_2\text{O}$  vapor, the gases, at above 300°, are passed upwardly through an irrigation tower, whereby a portion of their  $\text{H}_2\text{SO}_4$  content is condensed, they are then subjected to elec. pptn. to remove the residual  $\text{H}_2\text{SO}_4$ , the acid recovered by the pptn. being used for irrigating the tower. Cf. C. A. 29, 3788<sup>1</sup>.

Conditioning hydrated alkali metal silicates. Edwin Cowles and The Electric Smelting and Aluminum Co. Brit. 434,635, Aug. 26, 1935. This corresponds to U. S. 1,975,407 (C. A. 28, 6954<sup>1</sup>). The treatment prevents caking during storage and is restricted to hydrated alkali metal silicates.

Synthetic ammonia. I. G. Farbennid A-G (Christian Schneider and Heinrich Butsch, inventors). Ger. 617,645, Aug. 26, 1935 (Cl. 12: 1.01). A N-H mixt. suitable for the synthesis of  $\text{NH}_3$  is obtained by dust dried bituminous fuel in a shaft furnace with a series of horizontal or slightly inclined grates fixed at intervals up the shaft. A conduit is arranged just above each grate to run back to the space below the grate. In the example, dried lignite gives CO 20.2, CO 22.1, H 36.0, N 17.9,  $\text{H}_2\text{S}$  1.0 and hydrocarbons 2.8%.

Lithium compounds from impure solutions. Henry S. Colton (to Gracelli Chemical Co.). U. S. 2,021,989, Nov. 26. A soln. contg. Li, Na and K comds. such as one from ore treatment is treated with Na metasilicate to ppt. Li silicate, and the ppt. is sepd. and treated with an acid such as  $\text{HCl}$  to form a desired sol. Li salt, the soln. of which is sepd. from the insol. ppt. U. S. 2,021,987 relates to a process in which Li, Na and K comds. are leached from an ore residue, Li is ppt'd. as carbonate from the hot soln., the ppt. is sepd., the soln. is cooled to sep. Na and K comds. from unrecovered Li values and the residual soln. is returned to the leaching step. An arrangement of app. is described. U. S. 2,021,988 involves a process in which Li carbonate and other carbonates are ppt'd. with a reagent such as  $\text{Na}_2\text{CO}_3$  and in which

a Li compd. is recovered from the ppt. by use of an acid such as HCl.  $\text{Li}_2\text{PO}_4$  also may be formed.

Alumina and alkali phosphates from aluminum phosphate. Chem. Werke vorm H. & L. Albert Ger. 620,901, Oct. 30, 1935 (Cl. 12, 31). Addn to 516,460 (C. A. 26, 3741). As the caustic alkali soln. required for the process of Ger. 516,460, use is made of the mother liquors, contg. caustic alkali and alkali aluminate, obtained in the known process wherein (1)  $\text{AlPO}_4$  is heated to 600–800° with caustic alkali, alkali carbonate or alkali sulfate and C, and (2) the melt is poured into water or into a part of the mother liquor from the treatment of a preceding batch of  $\text{AlPO}_4$ .

Basic alkali Kalunite Co. Ger. 620,171, Oct. 15, 1935 (Cl. 12m, 7). This corresponds to U. S. 1,914,176 (C. A. 27, 4355), but gives addnl. details.

Ammonium nitrate Thaddäus Hobler Ger. 614,324, June 6, 1935 (Cl. 12k, 6). Addn to 539,642 (C. A. 26, 2284). The method of 539,642 for prep.  $\text{NH}_4\text{NO}_3$  by neutralizing 50% or over  $\text{HNO}_3$  with  $\text{NH}_3$  is modified by treating the  $\text{NH}_4\text{NO}_3$  lye with  $\text{HNO}_3$  under pressure in several stages.

Ammonium sulfate Irederic M. Pyzel (to N. V. de Bataafsche Petroleum Maatschappij) Brit. 474,622, Sept. 5, 1935. This corresponds to Fr. 774,815 (C. A. 29, 2317). The  $\text{SO}_2$  content of the sludge being reduced to 2% or less of its  $\text{H}_2\text{SO}_4$  content before treatment with  $\text{NH}_3$ . App. is described.

Ammonium sulfate from acid sludge Jan D. Ruys (to Shell Development Co.) U. S. 2,022,813, Dec. 3. Acid sludge from treating oil or tar with  $\text{H}_2\text{SO}_4$  is neutralized with  $\text{NH}_3$  so that the resulting liquid contains less than 1% free  $\text{H}_2\text{SO}_4$  while maintaining a temp. below 95° during the neutralization and effecting the neutralization in a zone approx. satd. with  $(\text{NH}_4)_2\text{SO}_4$ . An arrangement of app. is described.

Barium hydroxide Chemische Fabrik Coswig Anhalt G. m. b. H. (Lduard von Drathen, inventor) Ger. 620,170, Oct. 15, 1935 (Cl. 12m, 1).  $\text{Ba}_3(\text{PO}_4)_2$ , obtained as a by-product in the manuf. of  $\text{H}_2\text{O}_2$  from  $\text{BaO}$  and  $\text{H}_2\text{PO}_4$ , is heated with C to yield  $\text{BaC}_2$  and P. The  $\text{BaC}_2$  is treated with water to yield  $\text{C}_2\text{H}_2$  and  $\text{Ba}(\text{OH})_2$ . The P may be reconverted into  $\text{H}_2\text{PO}_4$ .

Calcium hypochlorite pellets suitable for treating water, bleaching, etc. Walter S. Bachman U. S. 2,023,459, Dec. 10. Stable Ca hypochlorite pellets are produced by mixing a  $\text{CaSO}_4$  material such as plaster of Paris with a hypochlorite such as bleaching powder and with addn. of more than sufficient water to hydrate the sulfate, permitting the sulfate to hydrate at least partially and then subjecting the mass to rapid dehydration to set the hydrated sulfate and avoid hypochlorite decompos.

Anhydrous calcium sulfate. Sydney F. W. Crundall (to Peter Spence & Sons, Ltd.) U. S. 2,021,910, Nov. 26. For producing anhyd.  $\text{CaSO}_4$  in extremely finely divided and amorphous form, hydrated  $\text{CaSO}_4$  is treated with  $\text{H}_2\text{SO}_4$  for removing the combined water from and effecting disintegration of the material, under conditions such as to avoid dissolving of the  $\text{CaSO}_4$  or formation of sol. acid sulfate.

Tin oxide. Marcel Leroy and Henri Migout (trading as Migout Frères & Cie.) Brit. 434,150, Aug. 27, 1935. In the production of  $\text{SnO}_2$  by oxidation of pulverized molten Sn with hot air, the furnace chamber supplying the mixt. of molten metallic Sn and hot air to a combustion chamber and the combustion chamber itself are initially heated, e. g., to 1000°, to start the process, whereafter the temp. in the combustion chamber is maintained by the heat of combustion and a portion of this heat is recuperated to maintain the temp. in the furnace chamber. Cf. C. A. 29, 563<sup>3</sup>.

Stabilizing aqueous hydrogen peroxide solutions Albert Kunz. U. S. 2,022,940, Dec. 4. Antipyrine is used as a stabilizer.

Apparatus for production of carbon monoxide and hydrogen gas from hydrocarbons such as natural gas Charles V. Maier (to Thomas B. Swift). U. S. 2,022,778, Dec. 3. Various details are described of an app. with a porous

refractory catalytic mats, for effecting reaction of hydrocarbons such as natural gas with air for producing a gas mixt. consisting essentially of H and CO and which is substantially free from  $\text{CO}_2$  and water.

Reducing iron contamination of caustic soda solutions. Albert H. Hooker (to Hooker Electrochemical Co.) U. S. 2,023,271, Dec. 3. For reducing the contamination by Fe in 42–51% solns. of NaOH that have been concd. by evapn. from solns. initially satd. with respect to the combined soly. of NaOH, salts from which it was originally formed and any other salts assocd. therewith or present as contaminants and allowed to cool, the soln. is agitated in contact with its own natural sludge not less than 24 hrs. at a temp. of 15–35° and is clarified.

Lithium Hans Heinrich Hütte G. m. b. H. (Conway Friedrich von Giese and Hans Weidmann, inventors) Ger. 616,379, July 29, 1935 (Cl. 12f, 16). Addn. to 600,704 (C. A. 28, 743<sup>9</sup>). The method of 600,704 for obtaining Li by smelting Li compds. with  $\text{Na}_2\text{SO}_4$  and  $\text{K}_2\text{SO}_4$  is modified by replacing the  $\text{Na}_2\text{SO}_4$  wholly or partly by alk. earth sulfates, e. g.,  $\text{CaSO}_4$  or  $\text{MgSO}_4$  or a mixt. of these. An example is given.

Sulfur Raymond F. Bacon Ger. 620,159, Oct. 15, 1935 (Cl. 40a, 2 (a)) See Brit. 375,370 (C. A. 27, 4039).

Free flowing powdered sulfur Ferd W. Wieder (to San Francisco Sulphur Co.) U. S. 2,022,796, Dec. 3. A mineral oil, such as a light lubricating oil (suitably in a proportion of about 1% or less), is incorporated in molten S and the S is then cooled and ground, and is suitable for dusting on vines, etc.

Sulfur from hydrogen sulfide The Girdler Corp. Ger. 620,326, Oct. 19, 1935 (Cl. 12, 17). See Fr. 762,364 (C. A. 28, 4574<sup>1</sup>).

Sulfur and sulfur dioxide from pyrites Raymond F. Bacon Ger. 617,003, Aug. 22, 1935 (Cl. 12f, 21). Irony ground pyrites is heated as an air suspension to form S and  $\text{FeS}$ . The  $\text{FeS}$ , still as an air suspension, is burned in air to give  $\text{SO}_2$ . App. for carrying out the process and collecting the S and  $\text{SO}_2$  is described.

Apparatus for the continuous production of carbon black by the incomplete combustion of carbonaceous materials and deposition on a cooled surface Jan Dusi L. Brit. 434,391, Aug. 30, 1935.

Active carbon Franz Krezl Brit. 434,461, Sept. 2, 1935. See Fr. 785,427 (C. A. 30, 584<sup>9</sup>).

Active masses I. G. Farbenind. A.-G. (Fritz Stöwener, inventor) Ger. 617,591, Aug. 22, 1935 (Cl. 12g, 4 (1)). Active masses, especially for catalytic uses, are prepd. by drying irreversible colloids comprising a colloidal carrier substance and a catalyst. Thus,  $\text{H}_2\text{SO}_4$  is added to Na water-glass to give a clear  $\text{SiO}_2$  sol. This is allowed to form a jelly which is ground up with  $\text{UO}_2(\text{NO}_3)_2$ ,  $\text{HgNO}_3$  or  $(\text{NH}_4)_2\text{VO}_4$ . The mixt. is strongly heated to give a porous active mass. Other examples are given.

Catalysts. I. G. Farbenindustrie A.-G. Brit. 434,141, Aug. 27, 1935. Addn. to 379,335 (C. A. 27, 4041). Metal sulfide catalysts for destructive hydrogenation and other reactions are prepd. by the action of this salts on compds. of metals, other than alkali and alk. earth metals, while dispersed in a liquid medium. Thus salts of Cu, Sn, V, Sb, As, Mo, W, Cr, Re and Ge contg. an alkali or alk. earth metal or  $\text{NH}_4$  as cation are mentioned. The metal compd. may be an oxide, hydroxide or salt of a heavy metal of groups I to VIII. Among examples, a soln. of  $\text{TiCl}_4$  in  $\text{C}_2\text{H}_6$  is added to a soln. of  $\text{NiH}_2$  thiostungstate in cyclohexylamine, the ppt. is filtered, washed and treated with H at 300–400°.

Mineral wool. John L. Morrow and Jesse B. Barnitt (to Aluminum Co. of America) U. S. 2,022,811, Dec. 3. Residues such as those left in obtaining alumina from bauxite are mixed with an aluminate-forming material such as lime or Na oxide in the amt. necessary to produce a final mixt. contg. an amount of aluminate-forming material at least equal to that required to convert the alumina in the mixt. into aluminate, the mixt. is melted and the molten material is passed through a stream of gaseous fluid such as air or steam delivered at high pressure.

## 19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. E. BARTON AND C. H. KERR

Calculation of the melting temperature of a glass batch on the basis of the chemical composition V. F. Yulerev *Trans Inst Chem Tech Ivanovo (U S S R)* 1, 95-7 (1935)—The Bertrand formula (C. A. 20, 209) was used with fairly good results Chas Blanc

Melting glass with dust like sand A. N. Dauvalter *Keram. i Steklo* 11, No. 8, 15-18 (1935)—Comparative expts made with various kinds of sand used for melting glass are described Glass melted with dust-like sand was of higher quality than that melted with ordinary sand Tables show the results M. V. Kondoidy

Drying-ovens for glass-melting sand. F. Jochem. *Glasstech Ber* 13, 391-9 (1935) J. F. Hyde

Theory and practice of accelerated drawing of glass by the Fourcault method I. I. Kitagorodskii *Keram. i Steklo* 11, No. 7, 3-7 (1935)—The chief defects of the Fourcault glass produced in the U. S. S. R. are its low chem. resistance, great striation, and the presence of various inclusions, such as stones, cryst. formations and bubbles, these defects are due chiefly to an incorrect compn of the glass The latest investigations in Europe and the U. S. dealing with the compn., melting, coloring and cooling of glass are discussed, and the results of studies made by the Russian Inst. of Glass analyzed. Conclusions. (1) A coarse-grained batch melts much more slowly than a fine-grained batch, (2) during the melting of such a batch, the process of glass formation occurs very rapidly Constructional details to improve Fourcault machines are discussed M. V. Kondoidy

Selection of glasses with equal coefficients of expansion V. P. Uspenskii *Trans Inst Chem Tech Ivanovo (U S S R)* 1, 98-100 (1935)—Application of the Schott and Winkelmann formula (*Ann Phys Chem* 51, 735 (1894)) in the production of plate glass is discussed Chas Blanc

The time law of extraction of glasses E. Berger and W. Geßlein *Naturwissenschaften* 23, 817-18 (1935)—

Attack of glass by acids, alkalis, water, etc., follows the law.  $dx/dt = k e^{-kx}$ , in which  $x$  is the amt. of glass decomposed and  $t$  the time The same function applies to cond. change of water during the extn. of glass powder The law holds as long as the  $SiO_2$  skeleton remains intact, it is generally valid for activated adsorption (cf. Roginskii, C. A. 29, 10454) This view is supported by the fact that the dependence of  $k$  upon acid concn. follows the Langmuir adsorption equation The same time law also holds for oxidation of metals in air (Tammann), elastic after effect, vol. change, etc., of chilled glass, change of transparency with radiation and grain growth in cold-worked metals This is probably due to the fact that the equation represents a special soln. of the diffusion problem B. J. C. van der Hoeven

Extraction and analysis of gases from glass. R. H. Dalton *J. Am. Chem. Soc.* 57, 2150-3 (1935)—An app. is described for collecting and analyzing the gases evolved by glass when heated electrically in vacuum up to 1400°. By means of a Pyrex-to-porcelain seal all wax and grease joints are eliminated. One to 0.01-cc. samples of gas, are taken and analyzed for  $H_2O$ ,  $CO_2$ ,  $SO_2$ ,  $O_2$ ,  $H_2$ ,  $CO$  and by difference for  $N_2$  and other gases Results with some typical glasses show that  $H_2O$  is usually the most abundant constituent  $CO$  is also nearly always present, though often in small quantity  $CO$  and  $H_2$  are rarely present and there is never an appreciable residue of inert gas The amt. of  $SO_2$ ,  $O_2$  or  $CO_2$  is considerable in special glasses P. S. Roller

Application of highly refractory sillimanite materials in the glass industry A. A. Litvakovskii *Keram. i Steklo* 11, No. 8, 26-8 (1935)—Details of expts made with kyanite linings for pot and tank furnaces are given The results show that. (1) kyanite linings resist the attack of the glass mass well, and (2) the character of corrosion is different from that appearing in grog bricks while grog mixes acquire a "shell"-like appearance on their

surface, kyanite linings become only uniformly thinner. M. V. Kondoidy

Black residue waste of the potassium ferrocyanide production as a substitute for alkalis V. F. Yulerev and N. P. Podlopaev. *Trans Inst Chem Tech Ivanovo (U S S R)* 1, 90-4 (1935)—A good grade of glass suitable for the manuf. of bottles and other articles of dark color was obtained by a partial substitution (up to 75%) of the black residue for  $Na_2CO_3$  Chas Blanc

Effect of temperature on the resistance of bottles K. H. Borchard and H. H. Achmed. *Glashtüte* 67, 547 (1935)—Expts with dark bottles showed that their resistance diminishes by about 15% per degree on heating The resistance of light green bottles of the same compn. for pasteurizing diminishes by 13% between 23° and 71°. Resistance to thermal shock by heating from 23° to 80° yielded a breakage of 23% Progressive heating reduces breaking to 0 M. V. Kondoidy

Bottle glass V. F. Yulerev and V. P. Uspenskii *Trans Inst Chem Tech Ivanovo (U S S R)* 1, 101-9 (1935)—The use of various industrial alk. waste products, such as flotation nephelite tailings,  $NaHSO_4$ ,  $Na_2SO_4$ , slag, etc., as substitutes for  $Na$  and  $K$  salts in the production of bottle glass is discussed The practical application of the Teichsner formula with Keppeler's correction (*Glasfabrikation* 1, 100 (1911)) is recommended C. B.

Gem glass Rudolf Hohlbaum *Glashtüte* 65, 407-9, 420-2, 435-7, 436-7, 469-71, 531-3 (1935)—The production of imitation gem glass is described. This glass is manuf. in small plants and the methods of production and compn. are kept secret. The manuf. consists of 3 main parts (1) production of the basic or "composed" glass; (2) grinding, and (3) working the polished glass so that it will be similar to gems The basic glass or "Strass" (the name of a famous jeweler from Nuremberg of the 17th century) is a glass with a high lead content or is free from lead but contains boric acid This glass is colored with metallic oxides An account of the obtaining of imitation diamonds, topazes, rubies, emeralds, sapphires, amethysts, aquamarine, garnets, opals, etc., is given M. V. Kondoidy

Quality control [in ceramic manufacture] F. M. Thorman *Bull. Am. Ceram. Soc.* 14, 355-9 (1935) C. H. Kerr

Influence of grain of magnesite brick on its properties Hermann Salmang and Paul Nemitz *Spreerkal* 67, 717-19, 729-31, 745-7, 759-61, 775-8 (1934)—Compressive and transverse strengths are increased by an increase in forming pressure and of fine grain content Increase of pressure in prepressing raises the temp. of deformation, a certain content of coarse grain also raises the temp. of the beginning of deformation The apparent porosity can be substantially reduced by prepressing and suitable grain size. The firing shrinkage is decreased one-third with prepressure and suitable grain packing Microscopic examn. shows that increased fusion, increased prepressure and an increase of fine grains give a periclase aggregate and better knitting of the grains with each other C. B. Jenn

Kaolin and clay Their difference in shrinkage and porosity. Albert Vasel *Keram. Rundschau* 43, 543-4 (1935); cf. C. A. 30, 409 The greater shrinkage of clay is explained by its smaller mol. The greater porosity of kaolin after firing is explained by a liberation of  $SiO_2$  and formation of mullite, whereas with clay metakaolin is formed and no  $SiO_2$  is liberated. P. S. Roller

Increasing plasticity of kaolins and clays R. Angenitzkaya and I. Romankevich. *Keram. i Steklo* 11, No. 7, 27-32 (1935)—Various theories dealing with plasticity are discussed and studies made by the authors described. The increase of the capacity of absorbing through only "exchange" reactions with  $Na$  under the effect of  $NaOH$  is very limited, and the plasticity of clay material is, therefore, increased little The increase of the plasticity of clay

material by means of the exchange of cations can be obtained by a simultaneous increase of the capacity to absorb by introducing admixts. with a great absorption and high adsorbing properties. M. V. Kondoidy

Obtaining feldspar by means of flotation. G. Gerth. *Glashütte* 65, 610-17(1935).—Attempts to sep. feldspar and quartz from impurities present in pegmatites, granites, syenites and feldspar sands were successful. M. V. K.

Saggars H Tives *Keram Rundschau* 43, 493-4, 507-10, 510-22, 531-3(1935).—A discussion of compn., properties and prepn. P. S. Roller

Deterioration of refractory brick in melting furnaces due to cracking. E. T. Richards. *Feuerungstechnik* 23, 49-52 (1935).—This deterioration is due to thermal and mech causes or to a modification of the structure of the brick. Cracking is due chiefly to rapid variations in temp. The sensitivity of bricks depends on structure, d. and coeff. of dilatation. The more elastic and porous (up to a certain limit) the brick is, the better it resists temp. variations. The structure is modified during service because of the effect of temp. and chem. action, this change starts on the hot side of the brick and causes the two sides to have different structures, which may produce cracks. M. V. Kondoidy

Effect of insulation on the silica refractories of an open-hearth steel furnace roof. Lewis B. Miller. *Bull. Am. Ceram. Soc.* 14, 357-64(1935).—Silica refractories after service show 3 zones. The hottest zone consists chiefly of cristobalite, the middle zone chiefly tridymite and the outer zone chiefly quartz. The iron silicate liquid formed during service is less destructive when the roof is insulated. Insulating will prolong the life of the silica refractories. C. H. Kerr

Rammed mixes for cupola furnaces. Alfred Borsch. *Feuerungstechnik* 23, 41-3(1935).—Rammed mixes are usually mixes. of high-grade refractory quartzites with addn. of clay or kaolin as bond, having an acid character. The furnace should be heated slowly. The critical temp. of highly siliceous materials lies between 10° and 900°. If this span is passed too rapidly, the mix may crack, spill or its structure becomes loose. M. V. Kondoidy

Laboratory control in dry-process enameling. R. D. Cooke. *Bull. Am. Ceram. Soc.* 14, 373-4(1935).

Evaluation of clay for enameling. L. Vielhaber. *Emailwaren-Ind.* 12, 273-5(1935).—The properties of clay required for enamels are discussed. The ratio of  $Al_2O_3$  to  $SiO_2$  in clay is not determinative. Settling tests do not show the suspending power of the enamel and are not comparable as the amt. of water used is not known and the varying amt. of alkalies from the enamel when dissolved changes the results. The effect of alkalies may be considered as const., however, in ordinary enamels, and the clay can be evaluated on its action alone. Expts with various German clays are described. M. V. Kondoidy

Mixing and melting enamel. C. A. Ottersbach. *Glashütte* 65, 520-1(1935).—Directions for working and mixing the raw materials, and firing the enamel are given. Various types of furnaces are discussed. The melting temp. varies between 800° and 1000°. An overmelted white enamel becomes translucent, has a vitreous appearance and has needle tappings. M. V. Kondoidy

A method for testing the adherence of ground coats to metal. C. J. Kinzie and J. B. Miller. *Bull. Am. Ceram. Soc.* 14, 371-3(1935).—The machine is a modification of the Frichsen ductility testing machine and is essentially a No. 3 Arbor press with necessary accessories. C. H. Kerr

One cover-coat enamel finishes. P. R. Porter and G. L. Bryant. *Bull. Am. Ceram. Soc.* 14, 367-9(1935).—A single cover-coat reduces the thickness of the enamel and lessens chipping and cost. Precautions in manuf. are stressed. C. H. Kerr

Enamel thickness gage. M. A. Rusher. *Bull. Am. Ceram. Soc.* 14, 365-7(1935).—The gage depends for its operation upon the magnetic circuit passing through the coil of the gage head and through the sheet of steel directly

under the enamel where the gage head is placed. C. H. Kerr

Measuring temperature during enameling and its application in practice. Oskar Kerstan. *Glashütte* 65, 617-20, 635-7(1935).—The advantages of automatic control of temp. are pointed out. The temp. should be measured continuously in enamel ovens, muffle kilns, flue pipes and drying ovens. The temp. of the air-steam for gas generators, that leaving it, and that entering the burner should be measured periodically. The control of the chem. compn. of gas and flues, pressure in the gas collector, steam and air pressure, are very important. Diagrams are given and analyzed, and defects due to excessive or insufficient firing are discussed. M. V. K.

Minimum radius of curvature for enameled parts. H. H. Holscher. *Bull. Am. Ceram. Soc.* 14, 369-71 (1935).—The min. radius of curvature of iron to be enameled is  $1/16$  in. C. H. Kerr

Powdered enamel for bath tubs. H. J. Karmaus. *Sprechsaal* 67, 778-80, 792 7(1935).—Prepn. of castings for enameling is discussed. Compn., analysis, charge to the wet mill and prepn. of the ground enamel and frit are outlined. The analyses and compn. of different powd. enamels are tabulated and their characteristics explained. C. B. Jenni

Significance of plastic masses, ceramic materials and glass for the development of domestic raw materials (Spielvogel) 13 Kjeldahl flasks and other vessels from As-free glass (Lockemann) 1 Kaolin and clay (Vasel) 8 Trays for use in heat-treating ceramic articles (U. S. pat. 2,022,283) 9

Glass I G Farlennd A.-G. (Paul Huppert and Hans Wolf, inventors). Ger. 620,317, Oct. 10, 1935 (Cl. 32b 2). In the manuf. of phosphatic glass transparent to ultraviolet rays, a salt of  $NH_4$  is included in the melt, whereby the tendency of the glass to change in compn. under the action of the rays is restrained. A phosphate of  $NH_4$  or a salt of  $NH_4$  having a volatile anion, e. g.,  $(NH_4)_2CO_3$  or  $NH_4NO_3$ , may be used. The formation of blisters may be avoided by addn. of an oxidizing agent, e. g., an alkali nitrate or  $BaO$ . In one example, a mixt. comprising  $K_2CO_3$  13.77,  $KNO_3$  6.71,  $CaCO_3$  8.03,  $BaCO_3$  3.22,  $MgCO_3$  of 84.75 purity 18.53,  $H_2BO_3$  31.04,  $Al_2O_3$  28.8 and  $(NH_4)_2HPO_4$  48.7 parts by wt. is fused in a quartz crucible to yield a glass of the compn.  $K_2O$  12.5,  $CaO$  8,  $BaO$  2.6,  $MgO$  7.5,  $B_2O_3$  17.5,  $Al_2O_3$  28.8 and  $P_2O_5$  26.2%. Other examples are given.

Continuous renewal of glass in a drawing-off basin attached to a glass furnace. Emile Roissant (to Soc. anon. d'études et de constructions d'appareils mécaniques pour la verrerie). U. S. 2,023,745, Dec. 10. In drawing off glass in a rotating basin supplied by a glass furnace and covered for the larger part by the hood of the furnace, the drawing off is effected at a fixed station by means of molds successively immobilized at this fixed point and there is imparted to the basin an intermittent movement of rotation such that a mass of glass cooled by a drawing-off operation effects several revolutions before again coming to rest at the fixed drawing-off station. App. is described. U. S. 2,023,746 also relates to details of drawing off glass in a rotary basin of generally similar character.

Stirring molten glass in a continuous tank furnace. Wm. C. Taylor (to Corning Glass Works). U. S. 2,022,223, Nov. 20. A stirring rod is mounted intermediate the ends of the melting chamber for rotation about a substantially horizontal axis and carries a bent stirring thimble on its end for contact with the glass.

Cooling large masses of molten glass. Geo. V. McCauley (to Corning Glass Works). U. S. 2,022,215, Nov. 20. Glass such as a large telescope disk of high silica glass is rapidly cooled through its crystn. range to a temp. below its softening point but above its annealing point, and the exposed surface of the mass is reheated to permit the exterior to yield and compensate the shrinkage of the interior, and annealing is effected. App. is described.

**Apparatus for manufacture of glassware such as bottles** Edward Miller (to Lynch Corp.). U. S. 2,022,939, Dec. 3 Mech features

**Apparatus for forming and glazing glassware.** John C E Schwab (to Hazel-Atlas Glass Co.) U. S. 2,022,780, Dec 3 Mech features

**Apparatus for forming glass lens blanks** Albert N Cramer (to Alice B Cramer) U. S. 2,023,781, Dec 10 Mech and operative details

**Finishing the edges of glass sheets** Gerald White (to Libbey-Owens-Ford Glass Co.). U. S. 2,022,530, Nov 26 In finishing the edges of a sheet of laminated safety glass which have been previously sealed, an abrasive tool is used while supplied with a solvent for the sealing material such as kerosene and a diluent for the solvent such as water when pitch or the like has been used as a sealing material App is described

**Polishing glass by brushing with metallic bristles** carrying a moist abrasive Bernard Long (to Soc anon des manufactures des glaces & produits chimiques de Saint-Gobain, Chauny & Crey). U. S. 2,023,275, Dec 3 Various details of app and operation

**Apparatus for tempering sheets of glass** Raymond Meer Fr 786,514, Sept 5, 1935

**Step by-step conveyors for glass-annealing leers** Otto Maetz Brit 434,513, Sept 3, 1935

**Partitioned glass containers such as ink wells** Carl G Hilgenberg and Charles B Garwood (to Carr-Lowrey Glass Co.). U. S. 2,023,474, Dec 10 Various operative details

**Designs or lettering on bottles or other glass articles** Alex Kneip (to Winthrop Chemical Co.). U. S. 2,023,175, Dec 3 Designs are formed with an artificial resin hardened to an insol and infusible state, a soft filling agent such as asbestos powder and a dyestuff pigment

**Preventing blurring of glass** Feldmuehle A G vorm Loeb, Schoenfeld & Co., Rorschach Fr 786,726, Sept. 9, 1935 The glass is coated with a layer of transparent material which will absorb water without modification, e g, viscose, casein or gelatin

**Multicellular glass** Soc anon des manufactures des glaces et produits chimiques de Saint-Gobain, Chauny & Crey Fr 786,818, Sept 10, 1935 A mixt of ground glass or fritted and ground vitrifiable materials with one or more powd substances capable of liberating gases or vapors either by reaction between the added substances or with the glass or by simple decompn is heated to the temp at which the glass welds together by simple contact A mixt of C, a carbide, Carborundum, Si or Fe-Si and a glass contg an excess of alkali or alk earth sulfate, or a mixt of glass, white SiO<sub>2</sub>, Na<sub>2</sub>SO<sub>4</sub> and C, or a mixt of glass and talc or kieselsaure may be used.

**Compound glass** Wm L Monro (to American Window Glass Co) Brit 434,314, Aug 29, 1935 Laminated glass comprises a sheet of glass, the inner surface of which is fire polished and the outer surface ground and polished, united to a 2nd sheet, both surfaces of which are fire polished, by a cementing layer having preferably the same refractive index The cementing layer may be formed by applying a soln of polyacrylic acid ester to the surface of each sheet, drying and uniting; or it may consist of a sheet of cellulose acetate or nitrate united by adhesive

**Safety glass** Wm H Simmons, Henry Tumms and Plastic Safeglass Syndicate Ltd Brit 434,229, Aug 28, 1935 Compound glass is made by applying to a glass sheet a soln of partly polymerized vinyl acetate and a plasticizer, e g, benzyl alc, in a volatile solvent, e g, AcOBU, evap the solvent at below 100°F, applying a 2nd glass sheet to the coated surface, pressing the assembly while raising the temp gradually to 150°F, and removing the compu extruded at the edges

**Safety glass** Adolf Kampfer Fr 786,555, Sept 5, 1935 App for removing solvent from the intermediate layers

**Laminated glass** James W H Randall (to Libbey-Owens-Ford Glass Co) U. S. 2,022,479, Nov 26 Each sheet of glass to be joined is provided with a skin of resin material such as a polyhydric alc-polybasic acid

resin and the sheets are joined by an intervening transparent material such as a sheet of a cellulose ester compn placed between the coated surfaces and treated with a high-boiling low-vapor-pressure solvent such as dibutyl phthalate

**Apparatus (with rolls and bonding agent supply) for laminated glass manufacture** George B Watkins (to Libbey-Owens-Ford Glass Co.) U. S. 2,022,484, Nov 26 Various structural, mech and operative details

**Endless-chain tamping machines for molding bricks, etc.** Wm Geo Chapman and Dunns' Engineering Works Ltd Brit 433,761, Aug 20, 1935

**Title for use in the construction of furnace arches and walls, having a head portion formed to engage an anchoring member.** M H Detrick Co Brit, 432,526, July 29, 1935 The title has on at least 1 face series of projections with intervening depressions, the ribs and grooves alternating both along the length of the face and transversely across it.

**Ball mills with ceramic ware jars or linings** Theodore J Volkmer (to Vitro Mfg Co.) U. S. 2,022,552-3, Nov 26 Various details are described of mills suitable for grinding glazes, enamels, etc.

**Purifying granular sand** Rob R McGregor and Edwin W Tilton (to Macbeth-Evans Glass Co) U. S. 2,023,278, Dec 3 Sand contg impurities such as Fe oxides, etc, which have a lower softening temp than that of the sand is heated to 1000-1600° for a time sufficient to form agglomerates contg impurities, and the agglomerates are removed from the sand

**Tunnel ovens, primarily for pottery.** Bernard J Moore Brit 433,637, Aug 19, 1935 The temp. of the main heating zone, which is at a substantial distance from the entrance to the tunnel, is maintained mainly by combustion of fuel, e g, in muffles, while in the secondary or entry heating zone the heat is maintained mainly by elec. resistances adapted to heat this zone throughout its entire cross-section and situated at a relatively low level on the walls of the tunnel

**Electric furnace for baking porcelain with means for modifying the atmosphere** Siemens-Schuckertwerke A-G. Ir 787,009, Sept 16, 1935

**Trucks for conveying pottery ware through kilns** Bernard Joseph Moore. Brit 433,473, Aug 15, 1935

**Trucks for conveying pottery ware through kilns** Bernard Joseph Moore and Christopher Clifford Brit 433,497, Aug 15, 1935

**Checker brickwork structure for regenerator furnaces** Bonnybridge Silica and Fireclay Co Ltd and Walter McBryde Brit 433,762, Aug 20, 1935

**Building and refractory blocks, etc** Leslie E Hamson and The Brinworth Brick & Tile Co Ltd Brit 434,127, Aug 23, 1935 Bricks, etc, for ovens, kilns, furnaces, etc, are formed by extrusion with a large no of relatively small and closely spaced apertures arranged so that the sepg walls are uniform in thickness

**Casting hollow refractory articles such as those from fused alumina** Geo J Laster (to Carborundum Co) U. S. 2,023,044, Dec 3 In a mold which may be formed of suitably refractory material, a core is used of low-thermal capacity which may be formed of chromite or magnesia and which has a higher coeff of thermal expansion than the material being cast

**Apparatus for sintering refractory material such as in the manufacture of cemented carbides** George P Taylor (to General Elec Co) U. S. 2,022,528, Nov 26 Structural details comprising a hollow tube of metal such as Mo within which is placed a contacting hollow refractory heat-conducting C cylinder which serves to hold a charge under treatment

**Abrasive compositions** Philippe Vogel Jaggi and Diamantschleiferi Voegel & Wurz A-G Brit, 434,402, Aug 30, 1935 The compns comprise diamond, particles, graphite, Fe<sub>2</sub>O<sub>3</sub> as English red or colcothar and a bond of PhOH-aldehyde resin In an example, a compn comprises diamond 26, charcoal powder 50, graphite 50, colcothar 75 parts, a little powd PhOH aldehyde resin and sufficient liquid PhOH-aldehyde resin to make a paste

SiC in amt. equal to that of the diamond may be added. The compn. may be molded onto a base of PhOH-aldehyde resin and the whole may be fixed to a metallic support.

**Abrasive articles.** The Carborundum Co. Brit. 433,725, Aug. 20, 1935. A bonded polishing article, e. g., a wheel, comprises a polishing powder, e. g., pptd.  $Al_2O_3$ , green chromic oxide, rouge, crocus and a bonding material, e. g., resin, in the form of substantially discrete particles. The preferred method of making the articles comprises intimately mixing polishing powder and dry powder, resin of approx. the same fineness, compacting the mixt. without the simultaneous application of heat under a pressure only high enough to form the article and curing at an elevated temp. without the application of such pressure as to compact the article further to any substantial extent.

**Rubber bonded abrasive articles** Richard H. Martin (to Norton Co.). U. S. 2,022,893, Dec. 3. An article such as an abrasive wheel comprises grains of abrasive such as cryst.  $Al_2O_3$  or SiC united by a bond of hard vulcanized rubber contg. an insol. fluoride such as  $CaF_2$ , which improves the heat resistance and strength of the rubber at normal grinding temps. Cf. C. A. 29, 312.

**Abrasive layers suitable for emery paper** Gerhard Balke, Franz Grom and Karl Wolf (to I. G. Farbenind. A.-G.). U. S. 2,023,191, Dec. 3. Abrasive particles are used with a bond including a hardened condensation product of maleic acid or its anhydride with castor oil, etc.

**Abradant device suitable for use on rollers of floor**

1 abrading machines. Ludwig K. Heinrich (one-half to Edmund R. Aller). U. S. 2,022,199, Nov. 26. A sheet of flexible metal such as steel carries a flat layer of fluffy steel wool, fibers of which are fused to the metal sheet.

**Glasses.** Rolf Vassel. Ger. 620,166, Oct. 15, 1935 (Cl. 80b 23.03). In the manuf. of glasses contg. B, the latter is added as a mixt. of two or more unfused naturally occurring minerals contg. B, at least one of the minerals contg. an alkali compd. of B and at least one other of the minerals contg. an alk. earth compd. of B. Synthetically prep'd compds. of B may be added to the mineral mixt. if desired. If the glaze so prep'd. contains an excess of alkali, this may be eliminated by addn. of an alk. earth chloride, whereby an alkali chloride, which vaporizes at the working temp., is formed. If the minerals contain sulfates as impurities, the mixt. may include also a reducing agent, which may be an agent which is eliminated at the working temp., e. g., graphite, or an agent useful as a component of the glaze, e. g., Pb. A sp. compn. is described.

**Roller apparatus for dry-cooling fused enamels** Firmatana Metallkeramik G m b H Ger. 620,271, Oct. 17, 1935 (Cl. 48c 1).

**Steel enamel-coated ware** Jacob E. Rosenberg (to O. Hommel Co.). U. S. 2,022,434, Nov. 26. The surface to be enameled is treated with an ammoniacal soln. of a Co compd. and the soln. is evap'd with escape of  $NH_3$  and pptn. of Co hydroxide, the coated surface is then covered with a slip of prep'd. enamel rendered fluid with water, which is dried and then fired.

## 20—CEMENT AND OTHER BUILDING MATERIALS

J. C. WITT

**Cement-water ratio a simpler method.** Geo. R. Wernisch. *Concrete* 43, No. 12, 7-8(1935).—Diagrams and computations are presented which show that the cement-water ratio is more easily used than the water-cement ratio in designing concrete mixts. R. W.

**Clay-gypsum cement—composition and manufacture** Peter P. Budnikoff. *Concrete* 43, No. 7, 37-8, 42 (1935).—Compressive strengths of mixts. of clay, anhydrite and hydrated lime are given. R. W.

**A study of the preparation of a specification for high-strength portland cement.** C. Rupert Gause. *J. Research Natl. Bur. Standards* 15, 421-39(1935)(Research Paper No. 859) P. S. Roller

**Lime modulus, lime saturation and lime standard of portland cement.** Hans Kuhl. *Tonind-Zig.* 59, 1221-4 (1935).—Different formulas are compared. P. S. R.

**Productivity of rotary portland cement kilns** V. I. Serdyukov. *Trans. Inst. Chem. Tech. Ivanovo* (U. S. S. R.) 1, 110-15(1935).—A crit. discussion of the performance of Danish, American and German kilns. C. B.

**The "blown" cement kiln.** A. W. Catlin. *Pit & Quarry* 28, No. 5, 23-4, 36(1935).—A "blown" kiln is one in which all the secondary air is blown under pressure through the load immediately prior to its discharge at the end of the shell. It has the following advantages. Considerable fuel saving, initial cooling of the clinker, greatly improved combustion with higher flame temp., steadier operation, pos. control of combustion air, greatly improved quality of product, better grindability of the clinker, less dust in the clinker and exit gases with greater steaming capacity of waste-heat boilers, and reduced maintenance costs. Each advantage is discussed. One plant reports savings of 23 lb. of coal per bbl., another 19 lb. Alden H. Emery

**Addition of peat ash to lime in the formation of hydraulic solutions** V. F. Yuferev and V. I. Serdyukov. *Trans. Inst. Chem. Tech. Ivanovo* (U. S. S. R.) 1, 116-31(1935).—The use of fresh and calcined peat ash as an ingredient in a hydraulic mortar, concrete and building stones is discussed. Chas. Blane

**The expansion and corrosion of hardened mortar of various cements.** H. Shochiro Nagai, Kuma Matsuoka

and Kenji Nomi. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 500-3(1935), cf. C. A. 29, 5245<sup>1</sup>.—Sulfate solns., especially  $Na_2SO_4$  and  $MgSO_4$ , attacked all mortars considerably, particularly those made from portland cements. Chloride solns., as  $NaCl$  and  $MgCl_2$ , do not attack the mortars so much. Cement mixts., such as blast-furnace slag cement and high-alica portland cement, showed good resistant properties to the salt solns. High-alumina cement was very stable toward the action of the solns., but its binding and compressive strength were greatly affected. Exptl. data are given. Karl Kammermeyer

**Flexural strength predetermined** E. W. Bauman. *Concrete* 43, No. 11, 9-10, No. 12, 11-15(1935).—A description of methods for designing concrete for a specified modulus of rupture. Raymond Wilson

**The protection of concrete in sea water.** Richard Grün. *Tonind-Zig.* 59, 1185-6, 1202-3, 1213-15(1935).—A lecture. P. S. Roller

**Increasing the fire resistance of masonry by suitable mortar composition of the plaster.** Kristen. *Tonind-Zig.* 59, 1209-13(1935).—Masonry was surfaced with a mortar consisting of cement, lime and sand with different addns. of gypsum, calves' hair and grog. After 3 months the masonry was tested for fire resistance. Ten % gypsum proved to be the best addn. both from the standpoint of resistance and of strength. P. S. Roller

**Light-weight building materials with foamy structure.** Hans Wolf and Hans Welsch. *Angew. Chem.* 48, 642-4 (1935).—A discussion covering the available types, mfg. processes, mixing proportions and phys. properties. Five references. Karl Kammermeyer

**Preservatives and antitermite protection of timber.** C. Demere. *Ind. Eng. Chem.* 27, 1303-5(1935).—Three preservatives developed by the E. L. Bruce Co. for the protection of wood against decay and insect attack are described.  $\beta$ -Naphthol is the toxic constituent. 5-A consists of  $\beta$ -naphthol in soln. in a combination of 2 grades of black fuel oil; 5-B uses as a carrier a colorless petroleum distillate; 5-C same as 5-B but contains asphalt. A. L. K.

**Decay in structural timbers.** Ernest A. Rudge and Harold Lewis. *J. Soc. Chem. Ind.* 54, 385-77(1935);

cf C. A. 29, 3488<sup>1</sup>—A skirting board removed from a modern house showed dry rot, and the extent of decay as previously defined by R. and L. (cf C. A. 29, 8276<sup>1</sup>) was not correlated with the more advanced stages of fungal attack. This was shown by making chem. analyses of various portions of the wood (i. e., detg. ash, FeO, CaO, hot-H<sub>2</sub>O-sol matter, alkali-sol matter, cellulose, lignin and pentosans) and comparing these with mycological data obtained from the Forest Products Research Lab. Those parts of the board which showed the highest mech. strength, the highest cellulose content and the lowest alkali-sol matter, showed the most advanced stages of fungal attack, by mycological examn. In these findings, R. and L. claim further confirmation of their inorg. infiltration theory. The following precautions are given for wooden structures: (a) careful attention should be paid to damp-coursing, to remove the source of ions concerned in inorg. decay, (b) unprotected wood should not be fixed to lime plaster surfaces, (c) fixing timbers to wooden plugs with iron nails is to be discouraged.

Louis E. Wise  
"Inorganic infiltration" theory of wood decay. W. G. Campbell. *J. Soc. Chem. Ind.* 54, 372-4T (1935), cf C. A. 29, 4542<sup>1</sup>, 8276<sup>1</sup>.—Polemical. Reply Ernest A. Rudge. *Ibid.* 374T.

Louis E. Wise  
Minimizing wood shrinkage and swelling. Alfred J. Stamm and L. A. Hansen. *Ind. Eng. Chem.* 27, 1480-4 (1935), cf C. A. 29, 3487<sup>1</sup>.—Cellulose as an intermediary has been used for impregnating green or dry wood with water-insol. waxes, oils and resins, this prevents to a large extent subsequent dimension changes and shrinkage. The treatment serves as a combined seasoning and anti-shrink impregnation particularly applicable to refractory species of wood.  
J. W. Shipley

Pitch [for road construction] (Brit. pat. 433,826) 21  
Plastic compn. [in prepn. of floor and wall coverings] (Brit. pat. 433,994) 13. Tar [for roadmaking] (Brit. pat. 433,248) 21. Viscous compn. [for use on roads] (Fr. pat. 780,533) 13

Cement. Chemische Fabrik Grünau Landshoff & Meyer A.-G. Fr. 780,997, Sept. 14, 1935. The early or rapid resistances and the process of setting of cement are regulated by adding a mixt. of salts contg. the cations Ca and Al and the anions Cl, NO<sub>3</sub>, CNS, CH<sub>3</sub>COO and HCOO, and basic substances such as CaO, Al<sub>2</sub>O<sub>3</sub> or salts having a basic reaction. The Al may be partly replaced by Sn or Ti or other metals, salts easily decomposed by hydrolysis are formed. Cf. following abstr.

Cements. Chemische Fabrik Grünau Landshoff & Meyer A.-G. (Walter Kirschner, inventor). Ger. 620,393, Oct. 21, 1935 (Cl. 806.1 05). The hardening of hydraulic cements is accelerated by adding a mixt. contg. AlCl<sub>3</sub>, CaCl<sub>2</sub>, and either a third metal chloride or HCl. The third chloride is preferably an easily hydrolyzed chloride, e. g., a chloride of Ti, Sn or Fe. Alternatively, FeCl<sub>3</sub> or a chloride of a metal of the 4th periodic group may replace AlCl<sub>3</sub> or CaCl<sub>2</sub> in the mixt. No proportions are specified. Cf. preceding abstr.

Cements, etc. Emilio Polzinetti. Fr. 786,631, Sept. 6, 1935. The capillary holes between the grains of cements, mortar, etc., are stopped up by a mixt. of ppts. of different sp. wts. and mol. sizes. This effect is obtained by adding to the water used substances which will cause the pptn. of stearates of Ca, Fe, Zn and Al.

Sorel-cement mixture containing bituminous material. Hiram S. Lukens (to Magnesium Cement Laboratories, Inc.). U. S. reissue 19,779, Dec. 3. A reissue of original pat. No. 1,724,945 (C. A. 23, 4792).

Apparatus for molding tubular articles from cementitious and other materials. Harry E. Dunn and Wm. E. Dunn. Brit. 433,591, Aug. 10, 1935.

Asbestos-cement sheets, etc. Eric R. Hartarp. Brit. 433,977, Aug. 23, 1935. A hard glossy surface, which will not effloresce, is produced on the sheets, tiles, etc., by treating the surface with 1 or more oxides, hydroxides or insol. salts of Zn, Ca and Mg and then either with an alk.

silicate and a chloride soln. or with 1 or more solns. of a chloride, sulfate or phosphate that will react to yield the corresponding oxychloride, oxy sulfate or oxyphosphate. In 1 example, a base mixt. of ZnO 5, Ti white 2, SiO<sub>2</sub> 1.5 and H<sub>2</sub>O 20 parts is applied to the moist sheets and smoothed, a 2nd layer of soap 1, ZnO 2 and H<sub>2</sub>O 320 parts is then applied and the surface is brought to a gloss by hot irons, when the cement has set hard, the surface is treated with a soln. of Na or K silicate and the sheet is dried and stored at about 300°F., after which it is treated with a ZnCl<sub>2</sub> soln. (20-30° Twaddle) and dried again. Cf. C. A. 30, 599<sup>1</sup>.

Bags suitable for holding cement, etc. Joseph D. Cramer (to Chase Bag Co.). U. S. 2,022,501, Nov. 26. Various details are described of a cloth bag lined with crepe-paper plies and with a layer of asphaltic material securing together the plies of paper and with a binder of other material, less readily softened by heat than asphalt, uniting the cloth and paper plies. A latex adhesive may be used.

Rotary drum for pretreating cement meal or like materials with furnace gases. Fried Krupp Grusonwerk A.-G. (Georg Ruppert, inventor). Ger. 620,440, Oct. 21, 1935 (Cl. 806.1 10).

Rotary kilns. Johan S. Fastang. Brit. 433,224, Aug. 12, 1935. In a cement kiln having in the drying and preheating zones groups of heat-transmitting elements, e. g., chains, in contact with which the gases flow in series, the charge is divided into sep. streams, each stream coming into contact with 1 group only of said elements, and all the streams are united to flow through the hotter zones of the kiln.

Rotary kilns and coolers for the manufacture of cement, etc. Mikael Vogel-Jørgensen. Brit. 432,892, Aug. 6, 1935. The chains suspended therein have circular links. Concrete. J. G. Farberman A.-G. Ger. 617,867, Aug. 27, 1935 (Cl. 806.1 05). A concrete resistant to chemicals and of great hardness, suitable for constructional work, is made by mixing sand and cement with an Fe oxide mixt. obtained by reducing org. nitro compds. with Fe. PhNO<sub>2</sub> slurry reduced by Fe is given as an example of the Fe oxide mixt.

Concrete containing powdered aluminum. Karl P. Billner (to Indurated Concrete, Inc.). U. S. 2,023,001, Dec. 3. To a concrete mixt. comprising cement, sand and aggregate, there is added about 0.06% (or less) the wt. of the dry cement powder of powd. Al which serves to produce a concrete of high strength.

Treating fresh concrete. Orla E. Hood. U. S. 2,023,887, Dec. 10. An exposed cement concrete surface such as that of a concrete pavement is treated with a blanket of heat-insulating foam such as may be formed of sulfite cellulose liquor, glycerol foams and Ca-Mg chloride, following initial set and within the period of final set, which serves as a protecting and water-retaining material.

Light cellular aggregate suitable for use in concrete. Andrew L. Gladney (one half to Marshall S. Hanrahan). U. S. 2,021,956, Nov. 26. Broken pieces of well-consolidated tuff or pumice are subjected to a temp. to cause incipient fusion and generation of gas within the pieces substantially to increase their bulk, while tumbling the pieces about to prevent agglutination.

Apparatus for centrifugally molding hollow concrete pipe sections. Geo. E. Mitchell. Brit. 434,538, Sept. 4, 1935.

Concrete-block-making machines. Thomas Malone. Brit. 434,963, Sept. 12, 1935.

Road materials. J. R. Geigy A.-G. Brit. 434,150, Aug. 27, 1935. See 1r. 778,033 (C. A. 29, 4917<sup>1</sup>). The bituminous substances may be omitted.

Material for road surfaces and paving blocks. Bernard W. D. Lacey and Percy J. Bawcutt (to Dunlop Rubber Co. Ltd.). U. S. 2,023,688, Dec. 10. To a base such as concrete blocks, successive layers of rubber crumb are applied in admixt. with a relatively highly concd. aq. dispersion of coagulated rubber, the successive layers differing in proportion of crumb and dispersion and contg. differing portions of compounding ingredients such as



NH<sub>4</sub> oleate, latex, cement, etc., to give different degrees of hardness and resiliency.

**Bituminous paving material** Walter H. Hood, U. S. 2,023,068, Dec. 3 Mineral aggregate such as sand, stone or gravel is coated with bituminous material such as an asphalt cement, and powder Al stearate or the like is added to sep. the coated particles. Oil may be used with Al stearate, etc., in a modification of the process.

**Aqueous bituminous emulsions suitable for use in road building, etc.** Ulric B. Bray and Lawton B. Beckwith (to Union Oil Co. of Calif.) U. S. 2,022,229, Nov. 26 An emulsion of the slow-breaking type is prep'd by commingling melted bitumen with an aq. soln. contg. an emulsifying agent such as NaOH or a soap and after cooling to below about 38° adding a small proportion of a stabilizing agent such as Na caseinate, the stabilizing effect of which is lessened when added at higher temps.

**Macadamized roads** I. G. Farbenindustrie A.-G. Brit. 433,957, Aug. 23, 1935 Solid particles of MgCl<sub>2</sub>, which has a thawing and dust-laying action, are incorporated in a road structure, each particle being embedded in or enveloped by a solid or plastic medium adapted to allow gradual access of moisture thereto. Wax, paraffin, tar, pitch or bituminous substances may be used to envelop the particles.

**Expansion-joint filler suitable for use in highway construction** Herbert C. Jussen (to Philip Carey Mfg. Co.) U. S. 2,023,529, Dec. 10 A slab-like product comprises a core of resilient rubber material interspersed with sep. air cells and surface layers of weather-resisting plastic material such as asphalt compn. adapted to be displaced by pressure so that it may be carried over and form a shell on the edge of the core.

**Rubber compositions** I. G. Farbenindustrie A.-G. Brit. 433,741, Aug. 20, 1935 Building mortars are prep'd by treating an aq. emulsion of vulcanized or unvulcanized rubber with an alkali silicate and a powder contg. a substance sparingly sol. in H<sub>2</sub>O and capable of reacting with alkalis. The basic character of the alkali silicate is thus destroyed so that hardening and coagulation take place. The H<sub>2</sub>O of the mortar may be absorbed by the addn. of, e. g., CaSO<sub>4</sub>, cement, kieselguhr. Emulsions, e. g., of asphalt or C<sub>18</sub>H<sub>36</sub>, may be incorporated. In examples, mixts. of borax, pulverized quartz and *p*-toluenesulfonate or Na<sub>2</sub>SiF<sub>6</sub> are added to a rubber emulsion contg. Na silicate.

**Building material** Ernst Köppe, Ger. 620,293, Oct. 18, 1935 (Cl. 806 21 04) Waste Ca compds from the ammonia-soda process are mixed with cement and a filler of low sp. gr. and after addn. of aq. waterglass soln. the mixt. is compressed into bricks or plates and then dried. A specified compn. contains dried waste Ca compds 50, portland cement 30, wood shavings 15 and waterglass 5%.

**Building slabs.** Evan O. Williams and Arthur P. Laurie, Brit. 434,837, Sept. 10, 1935. The slabs are formed of granulated cork mixed with a binding agent comprising Na silicate and reinforced with a flexible fibrous material embedded in the slab and (or) secured to at least 1 surface thereto. The cork may be treated with a soln. of Na silicate and molded into a slab as described in Brit. 290,042 and 346,516. After drying, the slab may be treated with CaCl<sub>2</sub> soln.

**Filling cracks, etc., in masonry structures** Perry M. Moore, U. S. 2,022,547, Nov. 26. A filler for cracks, fissures, etc., in masonry comprises a waxy substance such as ceresin wax and a smaller quantity of a solid inert mineral matter such as diatomaceous earth which has a greater affinity for the waxy material than does the masonry structure to be treated. Cumar resin also may be added.

**Slabs for floors or walls** Richard Lattorf (to The United Lumber and Vorwöhle Rock Asphalt Co. Ltd.) Brit. 433,017, Aug. 7, 1935 A slab having sound and heat insulating properties is provided with an upper or wearing surface of artificial stone, formed of granular natural stone of rough texture with a matrix such as portland cement mortar, united with an underlayer of asphalt with or without other ingredients.

**Asphalted felt-base roofing material** Charles J. Merriam (to Stephen G. Wright) U. S. 2,022,429, Nov. 26. An asphalt-sat'd felt base is provided with an air-blown asphalt coating over an intervening layer of dusted-on inert powder such as talc or mica or Zn stearate.

**Apparatus for saturating roofing felt with molten asphalt** Geo. P. Heppes (to Patent and Licensing Corp.) U. S. 2,023,019, Dec. 3 Various structural, mech. and operative details.

**Facing floors and walls** Diespeker and Co. Ltd. and Leonard T. Rackham, Brit. 433,913, Aug. 22, 1935 Floors, etc., are faced with a mixt. of cement and granulated cork, e. g., portland cement 1 and cork 2 parts, which is troweled off, rolled and, when set, rubbed smooth with a stone.

**Preserving vegetable-fiber materials such as wall-boards, etc.** Francis L. Cislak (to Peter C. Reilly), U. S. 2,022,231, Nov. 26. The material is impregnated with a fungicidal water-insol. borate such as that of Zn or Cd and also with acenaphthene, fluorene, anthracene, phenanthrene, carbazole, a tar acid which is solid at 40° and which may be chlorinated or nitrated, or a metal salt of such a tar acid.

**Paper for plaster boards, etc.** Wm. A. Richardson, Brit. 434,778, Sept. 9, 1935. In the manuf. of paper to be used in making gypsum laths, etc., anhyd. hydrated or partly hydrated CaSO<sub>4</sub> is included in sufficient amt., e. g., 1-10%, to insure that when such paper is applied to a mixt. of plaster of Paris and H<sub>2</sub>O there is no retardation of the crystal bond between the paper and the plaster.

## 21—FUELS, GAS, TAR AND COKE

A. C. FIELDNER AND ALDEN H. EMERY

**The manufacture of liquid fuel from vegetable oils.** Eugene C. Koo and Shu-Ming Cheng, *Chinese Industry* 1, 2021-39 (1935).—The vapor-phase and catalytic cracking of vegetable oils are studied. By carrying out the vapor-phase cracking of rapeseed oil at 1000°F. and feeding the oil at the rate of \$30 cc./min., a 76% yield of crude oil (sp. gr. 0.837) is obtained. The slower the rate of addn. of the vegetable oil, however, the higher is the percentage of gasoline fraction in the crude oil. Too slow a rate of addn. causes, on the other hand, excessive decomposition and hence lowering in the yield of crude oil. The highest yield of gasoline fraction is obtained by adding the vegetable oil at the rate of 2.15 cc./min., when the cracking is carried out at 1000°F., the yield of crude oil (sp. gr. 0.817) in that case being 70%, of which about 40% distills below 200° (gasoline fraction). Similar expts. with soybean oil, cottonseed oil and China wood oil (tung oil) gave similar results. In the study of catalytic

cracking, NaOH or CaO is used as the catalyst, better results being obtained with the stronger alkali (NaOH). At 800°F. rapeseed oil yields 56-25% of crude oil, of which 52-7% belongs to the gasoline fraction. In either of these processes, a higher fraction obtained on redist. the crude oil resembles kerosene and can be used in its place. By mixing the crude oil with the vegetable oil itself, an oil suitable for use as fuel oil in Diesel engines is obtained.

**Motor fuels of high-octane-blending value produced by thermal process.** P. C. Keith, Jr., and J. T. Ward, *Oil and Gas J.* 34, No. 28, 36, 37, 39 (1935). J. R. Strong. Sulfur compounds present in motor fuels affect lead responsiveness. W. A. Schulze and A. E. Buell, *Oil and Gas J.* 34, No. 21, 22-4 (1935).—See C. A. 29, 83064. J. R. Strong.

**Petroleum refiners view development of high-speed Diesel-engine fuel.** Wm. Mendrus, W. Anslee and C. H.

Schlesman *Oil and Gas J.* 34, No. 26, 76, 70-80(1935).—The specifications for Diesel fuels are given. Two grades of Diesel fuel are thought to be sufficient if the engine builder would cooperate with the refiners. J. R. Strong

Characteristics of internal-combustion engines using butane-air mixtures C J Vogt *Oil and Gas J.* 34, No. 26, 52, 54, 56(1935). J. R. Strong

Cost of operating automotive Diesels compared with gasoline motors C G Anthony *Oil and Gas J.* 34, No. 27, 35-36(1935).—A shows graphically that operation of Diesel-powered trucks costs 72% as much as gasoline trucks. J. R. Strong

Fuel for high-speed Diesels G C Wilson *Power* 79, 553(1935), cf C A 29, 7609<sup>1</sup>.—Typical Diesel-fuel specifications are tabulated. Viscosity and ignition characteristics are the 2 most important properties. Cetene no. for fuels from Am. crude oils are 33-75 and 35-55 for gas oils from cracked stocks. Alden H. Emery

The qualities of fuels for Diesel motors I J. Mesurier and R. Stanfield *Rev. Combustibles liquides* 13, 291-8 (1935), cf C A 28, 7495<sup>1</sup>.—The construction of very sensitive motors has led to the adoption of too restrictive specifications for fuel, based on incomplete theory. From the conclusions of the committees of the various societies, the readiness of combustion and viscosity is the most important. Too much importance is placed on the distn. curves. Lab. tests for C residue are not sufficiently indicative, a 4% (Conradson method) residue fuel having functioned satisfactorily for 7000 hrs. The corrosion effects of S are negligible, especially when contrasted with other elements of wear, up to 2% S. For ash and sediment, ordinary filtration is insufficient. For hard asphalt, 1% is the max. Halsey E. Sullivan

Factors of regulation of Diesel motors. René Rataec. *Rev. Combustibles liquides* 13, 338-51(1935).—Photomicrographs are shown of sprays of liquid fuels in which different types of injectors are used. H. E. S.

Characteristic regulations of various countries concerning dissolved cylinder acetylene. Bloch-Sée and Pierre Rosenberg *J. acetylene* No. 95, 980-93(1935).—A summary of the cylinder acetylene regulations in the following countries: Germany, England, Australia, Austria, Belgium, Canada, Spain, U. S., Egypt, France, Greece, Holland, Hungary, Italy, Japan, Morocco, Norway, Poland, Portugal, Switzerland, Syria, Tunis, Czechoslovakia, Yugoslavia, U. S. S. R. and International (Convention of Berne, 1924). Albert S. Carter

Conversion of methane to acetylene. M. C. Simon *J. acetylene* No. 94, 973-77(1935).—A review. Albert S. Carter

Use of calcium carbide in low-temperature fractional distillation. J. Arnoul de Grey. *J. acetylene* No. 96, 1005(1935).—In the destructive distn. of low-grade fuels, e. g., French lignites, CaC<sub>2</sub> was added up to 5%. In proportions up to 2%, improvement was observed; a 57% increase was obtained in oil b. below 220° with 100% increase in gasoline cut (essence) after phenol removal and refining. Albert S. Carter

Some coal researches—their significance to the coal industry A McCulloch *Chemistry & Industry* 1935, 997-1004.—A review of the work done in pure coal research on the action of solvents, oxidizing agents and Cl on coal and coal carbonization. Joseph H. Wells

The "Chance" sand-flotation system of washing coal A B Price *Iron & Coal Trades Rev.* 131, 719-21, 762-3(1935). D. S.

The clarification of washery water and the recovery of coal slurry J L. Lewis *Colliery Guardian* 151, 895-7(1935). E. H.

"Sensitization" and other observations on slurry flocculation E T. Wilkins *J. Soc. Chem. Ind.* 54, 391-37(1935).—Starch and gelatin are good flocculators for coal washery slurries. Starch is capable of flocculating suspensions of closely graded coal particles up to 0.3 mm diam. Negatively charged slurries appear to be flocculated by either negatively or positively charged gelatin. Wetting agents had little effect on the settling rate of a slurry. Certain difficult slurries could be sensitized by

the addn. of wetting agents so that they became more easily flocculated by starch or gelatin. J. H. W.

The determination of nitrogen in coal by the Kjeldahl method, using selenium as a catalyst H E. Crossley, *J. Soc. Chem. Ind.* 54, 397-9T(1935).—The effects of the rate of heating, moisture, temp. of the boiling soln. and the after-boiling period were investigated. The following modified Kjeldahl method was developed: Thirty cc. of concd. H<sub>2</sub>SO<sub>4</sub> is added to a mixt. of 1 g. coal, 20 g. K<sub>2</sub>SO<sub>4</sub> and 0.2 g. Se. The flask is loosely stoppered with a glass bulb and heated at a predetd. rate. This rate shall be the min. one of the min. time of clearing. Digestion is carried out for 1 hr. after clearing and the N detd. as usual. Joseph H. Wells

Dust in coal mines, its formation and control. A. M. Bryan. *Iron & Coal Trades Rev.* 131, 807-10(1935). D. S.

Selection of steam-generating equipment to preparation, selection and burning of bituminous coal E G Bailey *Am. Inst. Mining Met. Engrs. Contribution* No. 93, 10 pp(1935). Alden H. Emery

Purified steam economics. Frederick C. Straub *Paper Ind.* 17, 572-4(1935).—A discussion of the difficulties which may be caused by impurities in steam, and of the economies effected by purifying steam. A. P. C.

Marshland utilization and peat cooking W. Wielandt. *Angew. Chem.* 48, 632-6(1935); cf. Boyle, C. A. 29, 5251<sup>1</sup>.—A comprehensive discussion of the economic and tech. aspects of the problem. Karl Kammermeyer

Further experiments upon the water-gas process J. G. King, B. H. Williams and R. V. Thomas *Depts. Sci. Ind. Research, Fuel Research Tech. Paper* No. 43, 58 pp(1935), cf. C. A. 26, 2302<sup>1</sup>.—Clunker accumulation in the generator is eliminated by keeping the temp. of the hottest part of the fuel bed sufficiently low to avoid fusion of the coke ash into hard conglomerate masses and by keeping the zones of high temp. away from the walls of the generator by inducing the air currents to flow mainly in the center of the coke charge. Under these conditions the output may be reduced by about 50% but the thermal efficiency increases about 20%. The higher the fusion temp. of the ash the less is the reduction in output. For greater efficiency during the blow cycle, the rate of blowing should be as great as practical limitations allow, the period of blowing being reduced so that the optimum temp. is not exceeded. In this way the percentage of CO and other thermal losses are reduced. Alteration of the size of the coke between 2- and 3 in. pieces and from 0 to 1-in. breeze has only a slight effect upon the thermal efficiency of the process. A greater output can be obtained with the smaller coke by increasing the vol. of air per blow. Gas cokes made in horizontal and vertical retorts from the same coal behave nearly alike. The thermal treatment to which the coke is subjected in the upper part of the generator levels out any differences that may originally exist. Low-temp. coke behaves differently owing to its greater activity to CO<sub>2</sub> during the blow and its higher content of volatile matter. Joseph H. Wells

Dew point of flue products from manufactured gas combustion. Jesse S. Yeaw and Louis Shindman *Ind. Eng. Chem.* 27, 1476-9(1935).—Expts. were carried out to det. the actual condensation temp. (dew point) of flue gases from combustion of manifold gas as compared with the calcd. temps. The true dew points were found to be about 10° higher than the calcd. temps. with no excess air. The difference is attributed to the effect of SO<sub>2</sub> derived from the combustion of 9 grains of S per 100 cu. ft. of the manifold gas. J. W. Shipley

Improved low-temperature analysis for measuring gas residue content W. W. Robinson, Jr. and F. N. Laird *Oil and Gas J.* 34, No. 28, 26-27(1935).—R. and L. describe improved app. for measuring residue on low-temp. analysis of gas. This eliminates the inaccuracy of empirically calcd. the residue as hexane in the case of wet gas and pentane in the case of dry gas. J. R. S.

Rubber in the gas industry C. R. Austen *Trans. Inst. Rubber Ind.* 11, 264-82(1935).—See C. A. 29, 2693<sup>1</sup>. C. C. Davis

Tar products for water- and damp-proofing. R. O. Rhodes and E. H. Hyde. *Ind. Eng. Chem.* 27, 1409-10 (1935).—The discussion relates particularly to the building trades.

Review of developments in heavy oil tar and emulsion handling. P. T. Dashiell. *Am. Gas Assoc. Monthly* 17, 426-30 (1935). E. H.

Rubber in the gas industry (Austen) 30 Pulverized coal for blast-furnace use (Campbell) 9 System for fractionating natural gas and other gas mixtures by liquefaction (Ger. pat. 620,077) 1  $\text{NH}_4$  sulfate from acid sludge [from treating tar with  $\text{H}_2\text{SO}_4$ ] (U. S. pat. 2,022,813) 18 Catalysts [for hydrogenation] (Brit. pat. 434,141) 18

Motor fuel Rudolf Pawlikowski. Ger. 620,259, Oct. 17, 1935 (Cl. 23b 4 02) In the manufacture of powdered coal or coal-oil mixtures for use as motor fuel, the powdered coal is pretreated by sepg. it by a known wet sepg. process into fractions of high- and low-ash content, the latter fraction being then reground in a liquid. Various details are given.

Liquid fuel Arthur A. Roberts. Brit. 434,109, Aug. 27, 1935 Fuel for burners or Diesel engines is obtained by emulsifying a minor proportion of  $\text{H}_2\text{O}$  in oil fuel by means of casein or a carbohydrate colloid, e. g., starch. Agents controlling combustion, e. g., B compounds and (or)  $\text{NaCl}$ , and  $\text{C}_2\text{H}_4$  to reduce the viscosity, may be added. Cf. C. A. 30, 598<sup>2</sup>

Liquid fuel Arthur A. Roberts and Clifford W. Smith (legal representatives of Arthur A. Roberts, deceased). Brit. 434,180, Aug. 27, 1935. Divided on 433,348 (C. A. 30, 598<sup>2</sup>) Liquid fuel, e. g., oil or coal-oil suspensions, has a B compound insol. in  $\text{H}_2\text{O}$  or oil distributed therein to control combustion or increase the effective temp. of combustion. Up to 10% of  $\text{H}_2\text{O}$  may be added; the viscosity may be reduced by a "cutback," e. g.,  $\text{C}_2\text{H}_4$ . In an example, 40 lb. of coal passing a 200-mesh screen is stabilized in 55 lb. of petroleum oil by 0.2 g. casein in 0.6 g.  $\text{H}_2\text{O}$ , together with 1% of a borosilicate.

Atomizer for liquid fuels. John G. McKean and Robert F. Jones. U. S. 2,023,767, Dec. 10 Structural and operative details.

Solid fuel. Wilhelm Groth. Brit. 433,239, Aug. 12, 1935. See I. r. 775,143 (C. A. 29, 2338<sup>9</sup>)

Hydrogenating carbonaceous materials International Hydrogenation Patents Co. Ltd. Brit. 434,624, Sept. 5, 1935 This corresponds to Fr. 775,202 (C. A. 29, 2066<sup>4</sup>), all parts of the app. coming into contact with the hot reagents being so coated.

Hydrogenating carbonaceous materials International Hydrogenation Patents Co. Ltd. Fr. 780,937, Sept. 14, 1935. In treating carbonaceous materials with hydrogenating gases in the presence of halogen, halohydric acids or compounds, capable of liberating halogen or halohydric acids under the conditions of working, e. g.,  $\text{PHCl}$ ,  $\text{CCl}_4$ ,  $\text{NH}_4\text{Cl}$  or  $\text{HCl}$ , the reaction is carried out in the presence of finely powdered metals disseminated in the materials treated. Fe, Ni, Co, Zn, Al, Sn, Pb, Cd, Cr, Mn, Ti, Ag, Cu, Mg, Be, Ge, or alloys such as bronze, magnalium or Al bronze may be used.

Destructive hydrogenation. International Hydrogenation Patents Co. Ltd. Brit. 434,307, Aug. 29, 1935 In the destructive hydrogenation of coal dispersed in oil, the dispersion is heated in a preheater so that the time of sojourn therein at 200–410° is less than 200 sec., preferably less than 100 sec. The time of sojourn in the range 370–410° is preferably more than 20–40 sec. The time of sojourn is adjusted by adding H at different points in the preheater or by adjusting the dimensions of the preheater.

Vertical retorts. Frederick J. West, Ernest West and West's Gas Improvement Co. Ltd. Brit. 434,562, Sept. 4, 1935 A carbonizing retort with a minor axis of normal length has its major axis dimension considerably increased and is provided, at the lower end, with 2 vertical helical

coke extractors arranged side by side in the direction of the major axis and driven in unison from a common shaft.

Vertical retort settings John Roberts. Brit. 433,702, Aug. 19, 1935 The setting comprises superposed sets of intercommunicating horizontal heating flues supplied by burners which are so set and spaced that a general circulation of heating gas is maintained at high velocity.

Gas-fired horizontal retort settings. Samuel Moore. Brit. 433,961, Aug. 23, 1935

Crushing-mill for coal, coke, etc. Thomas A. Long and Wm. W. Potts. Brit. 433,738, Aug. 20, 1935. Addn. to 387,508 (C. A. 27, 4909<sup>9</sup>)

Coal, coke and other crushers British Jeffrey-Diamond Ltd. and James W. Pickering. Brit. 433,950, Aug. 20, 1935

Apparatus for separating materials such as coal from slate or cinders by their different frictional resistance Frank Pardee and Frank Pardee, Jr. (to Anthracite Separator Co.) U. S. 2,023,286, Dec. 3. Structural, mech. and operative details. U. S. 2,023,287 also relates to app. for effecting similar sepgs.

Pneumatic cleaning of raw coal, etc. James H. Dickerson. U. S. 2,022,588, Nov. 26 Various details of app. and operation.

Apparatus for classifying coal and schist The Birtley Co. Ltd., Kenelm C. Appleyard and Alfred Thnt. Fr. 787,029, Sept. 10, 1935

Pneumatic process for refining powdered coal or ores Carlshütte A.-G. für Eisengießerei und Maschinenbau. Ger. 620,489, Oct. 22, 1935 (Cl. 1a 28 10)

Stabilizing bituminous coals Francis S. Woidech. I. r. 786,845, Sept. 10, 1935 Coal is treated with a hot (100°) inert gas, e. g., a mixt. of  $\text{CO}_2$  and  $\text{N}_2$ , and the gaseous or volatile products removed thereby are collected by washing with an oil or by passing through charcoal. The remaining product is treated with acid tars or phenolic oils which combine with the basic constituents of the ash and resinify. Each particle of coal is thus covered with a film which prevents oxidation during storage.

Apparatus for carbonizing subdivided coal, brown coal, etc., by direct treatment with hot gases August Streppel. Ger. 620,400, Oct. 21, 1935 (Cl. 10a 33 01).

Briquetting brown coal Karl Schmidt G. m. b. H. Ger. 620,059, Oct. 12, 1935 (Cl. 80a 25 01). The molds of briquet presses for brown coal are lined with Al (or an alloy of high Al content) of which the side in contact with the mold has been oxidized by anodic treatment to a depth of several mm.

Multiple-hearth apparatus for drying brown coal Zeitzer Eisengießerei und Maschinenbau A.-G. Ger. 620,151, Oct. 16, 1935 (Cl. 82a. 13).

System for carbonizing compressed peat, brown coal and like bulky fuels by direct treatment with hot gases A. Riebeck'sche Montanwerke A.-G. (Theodor Limberg, inventor). Ger. 620,399, Oct. 29, 1935 (Cl. 10a. 24 01).

High-pressure water-tube steam generator. Hans C. Fglöf (to Sulzer Gebrüder Soc. anon.) U. S. 2,023,012, Dec. 3. Various structural, mech. and operative details.

Device adapted for feeding water to steam generators, etc. John J. Harman (to Walworth Patents Inc.). U. S. 2,023,073, Dec. 3. Structural, mech., and operative details of a float-controlled app.

Sterling-type boiler and compensating steam superheater Charles W. Gordon (to Superheater Co.). U. S. 2,023,223, Dec. 3. Structural details.

Apparatus for controlling the temperature of a fluid such as in superheating steam Carl M. Underwood and Emerson Pugh (to Western Elec. Co.). U. S. 2,023,627, Dec. 10. Various structural and operative details.

Steam desuperheaters The Superheater Co. Ltd. and John E. Wood. Brit. 433,807, Aug. 21, 1935

Deconcentrator for steam. Erich Henkel and Howard G. Nehrbas (to Superheater Co.). U. S. 2,023,227, Dec. 3. Structural details.

Steam traps Gustav F. Gerdtis. Brit. 433,934, Aug. 22, 1935

Fuel gas. Rudolf Drawe. Ger. 620,392, Oct. 21, 1935 (Cl. 24a 1 07). A column of fuel in a gas-generator shaft

is gasified partly by O supplied at the top of the shaft and partly by a mixt. of O and steam supplied at the bottom of the shaft. The gas is withdrawn from a point near the middle of the shaft. Gas of low CO<sub>2</sub> content is obtained.

Municipal gas Harald Kemmer Ger 617,565, Aug 21, 1935 (Cl 26a 3 02) Gas, free from CO<sub>2</sub>, is obtained by subjecting coal gas contg CO to successive oxidation by steam and reduction by H. The calorific value of the original gas contg CO is raised by oxidizing in the first stage till only 4-5% of CO remains, the H freed in this process is used for the reducing stage. Oxidizing agents such as active C may be employed in the oxidizing stage, and contact agents such as Ni, ThO<sub>2</sub>, and active C may be used in the reducing stage.

Gas mixtures containing hydrogen from gaseous hydrocarbons, steam and fuel dust Allgemeine Staubvergasungs Ges m b H Ger 619,638, Oct 4, 1935 (Cl 24e 1 07) Addn to 616,466 (C A 29, 76223) Plant for use in the process of Ger 616,466 is described.

Condensing vapors from gas mixtures such as coal distillation gases Franz Pollitzer and Paul Schulten U S 2,022,782, Dec 3 Various details of app employing a refrigerating machine, etc., are described.

Automatic apparatus for regulating the calorific value of coal gas Gerhard Verste Ger 620,838, Oct 28, 1935 (Cl 26a 17) Addn to 557,010 (C A 27, 402)

Gas producer Vereinigte Westdeutsche Waggonfabriken A-G Ger 617,572, Aug 21, 1935 (Cl 24e 3 02)

Gas producer Karl Bergfeld Ger 617,574, Aug 21, 1935 (Cl 24e 12)

Water-gas producer Bamag-Meguin A-G Ger 617,709, Aug 27, 1935 (Cl 24e 1 02)

Apparatus for producing coal gas Edmund Kropiwinski Ger 617,575, Aug 21, 1935 (Cl 26a 1 01)

Shaft oven for obtaining coal gas Jean Lefèvre Ger 617,850, Aug 27, 1935 (Cl 26a 5)

Oven for the production of gas and coke Didier-Werke A-G Ger 617,789, Aug 20, 1935 (Cl 26a 1 02)

Revivifying gas purifier waste Gastechnik G. m b H Brit 433,823, Aug 21, 1935 In the removal of S compounds from gases by passage of the gas through layers of porous bodies, comprising mainly Fe(OH)<sub>3</sub>, and subsequent regeneration of the porous bodies by aeration, only completely sulfided layers are so treated. The formation of S shells around the porous bodies as a result of the rapid alternation of sulfation and aeration is thus avoided.

Removing sulfur dioxide from flue gases Henry I. Johnston (to Board of Trustees of the Univ of Ill) U S 2,021,936, Nov 26 The gases, contg free O, are washed with a soln. contg ferric and Mn ions in soln as catalysts and promoters to increase the oxidation of the SO<sub>2</sub> into H<sub>2</sub>SO<sub>4</sub> by the free O in the gases, the concn of the ferric ions being greater than that of the Mn ions. App is described and such app is claimed in U. S. 2,021,937.

Tar Lonza Elektrizitätswerke und Chemische Fabriken A-G Brit 433,248, Aug. 12, 1935 In treating

tars for obtaining binders suitable for roadmaking, insulation purposes, etc., by subjecting them, contg thickening agents, to an oxidizing treatment at elevated temp, while stirring the mass, the lighter oils, boiling up to 170°, are distd. off and the middle and heavy oils, of b p 170-350°, are retained in the reaction mass during the oxidation process or returned to the same after the oxidation. Bitumen, asphalt, artificial or natural resins or PhOH-aldehyde condensation products, which last may be formed *in situ*, are used as thickening agents. App is described.

Fractional distillation of tar, etc Thermal Industrial and Chemical (T. I. C.) Research Co Ltd, Wm. J. Chadder and Wm. H. Miller Brit 434,541, Sept 4, 1935, Fr. 786,783, Sept. 9, 1935 In the fractionation of tar, C<sub>12</sub>H<sub>6</sub>, etc., the condensate and the vapor leave the dephlegmator through a common outlet and flow to a separator. The app. comprises a distn. column integral with a dephlegmator chamber communicating through 1 or more ducts extending within the chamber of the upper part thereof, a condensing worm being arranged in the chamber so that condensate cannot enter the ducts.

Pitch Alexander Wacker Gesellschaft für elektrochemische Industrie G m b H Brit 433,826, Aug 21, 1935 See Fr. 773,577 (C A 29, 16147)

Pitch and tar Thermal Industrial and Chemical (T. I. C.) Research Co Ltd. and Carl O. Condurp. Brit. 432,998, Aug. 7, 1935 Gums, i. e., sticky or resinous materials, are obtained by distg. tar down to a hard pitch residue, removing an oil fraction boiling above 325°, treating the fraction with a reagent, e. g., an acid or alkali, sepg. the gum-contg. layer from the oily layer obtained, and recovering the gums therefrom.

Coke Eduard Deisenhammer and "Salvus" A-G für Nahrungsmittel und chem. Ind. Austrian 143,090, Oct 19, 1935 (Cl 10c) Coal of low-bitumen content, from which coke of poor quality only is ordinarily obtained, is mixed with about 8-10% of natural or artificial bitumen or like material and then coked at above 500° and a pressure above 2 atm. Hard coke is obtained. Sp. processes are described.

Means for compressing coal in coke-oven chambers Gustav Hilger. Ger. 620,398, Oct 21, 1935 (Cl 10a 15)

Addn to 610,659 (C A 29, 56367).

Means for compressing coal in discontinuously operated coke ovens Gustav Hilger Ger 618,970-1, Sept 19, 1935 (Cl 10a 15) Addns to 610,658 (C A 29, 56367).

Coke ovens Gwynne Cellan-Jones Brit 432,777, Aug 1, 1935 A heating-wall comprises vertical up burning combustion flues interspersed between, and connected at their upper ends to, downcast flues adjacent to the oven walls.

Coke ovens Carl Stull G m b H. Brit. 434,403, Aug 30, 1935. In the production of low- or medium-temp coke, the oven chamber is divided along its length into a no. of radiation spaces between which the fuel is contained and carbonized.

Coke ovens of the retort type The Koppers Co of Delaware. Fr. 788,585, Sept. 5, 1935

## 22—PETROLEUM, LUBRICANTS, ASPHALT AND WOOD PRODUCTS

W. F. PARAGHER

Laboratory investigations on acid treatment of oil sands F. B. Plummer and R. B. Newcome, Jr. *Am Inst Mining Met Engrs Contributions No 88*, 13 pp. (1935)—Acid treatment of fine-grained, impure limestone (common in reservoir rocks) results in large loss in permeability, because of clogging of pores by very fine insol. residues of acid-insol. silicates mixed with a little colloidal SiO<sub>2</sub> and traces of colloidal org. matter.

Alden H. Emery The development of torbanite in South Africa. W. Duff S. African Mining Eng. J. 46, Pt. II, 103-7 (1935)—The origin of oil shale and torbanite, mining methods and various types of retort for the recovery of oil are reviewed.

The bleaching action of fuller's earth E. Erdheim *Petroleum Z* 31, No 29, 7-8 (1935); cf. C. A. 29, 8303—Decolorizing tests were made with a natural bleaching agent, fuller's earth; an activated bleaching agent, Montana Z; and an activated charcoal, Nuchar. After decolorization, the bleaching agents were extd. with alc. The color of the ext. and the residue after evapn. indicated that the decolorization with fuller's earth gives a color lake.

Modern contact filtration and pressure filters C. E. Mossman *Petroleum Z* 31, No 23, 1-7 (1935)—Lubricating oil is clarified by a combination H<sub>2</sub>SO<sub>4</sub> treatment and contact filtration with fuller's earth. The process and equipment used are shown in the form of a flow-sheet.

The method of prep. and activating the fuller's earth are discussed. A Turbo-mixer is used to agitate the fuller's earth and the acidified oil. Pressure filters of the Sweetland or Kelly types are more frequently used to sep. the finely divided fuller's earth from the oil. Characteristics of the filters and accessory equipment of the process are discussed. C. B. Jenni

Gasoline survey for 1934. H. McD. Chantler. *Can. Dept. Mines, Mines Branch Rept. No. 764*, 22 pp (1935).—Detailed analyses of 117 samples of gasoline are given. The knock rating of the av. gasoline sold in 1934 was 66 octane no. The av. Reid vapor pressure was 7.5 lb per sq. in. Less than 5% of the samples contained more than 10 mg. of gum per 100 ml. All but 1 gave a neg. corrosion test by the Cu strip. There was an increase in volatility. Alden H. Emery

Hydrogenation at Billingham. Anon. *Steam Engr. 5*, 76-7, 80(1935).—The yield of gasoline from coal is 60-70% by weight; from low-temp. tar and creosote oil, 60-90%. Output is 100,000 tons of gasoline from coal per yr. and 50,000 tons from creosote oil and low-temp. tar. Four tons of coal is used to give 1 ton of gasoline. Raw coal is cleaned to less than 2 1/4% ash, and mixed with oil to give a 50% paste. This mixt. is treated at 450° and 230 atm. pressure. Alden H. Emery

Synthetic gasoline from carbon monoxide and hydrogen. I. S. Tsutsumi. *Rept. Imp. Fuel Research Inst. Japan No. 31, J. Fuel Soc. Japan 14*, 110-16 (in English) (1935).—The paper includes: (1) general procedure; (2) effect of conditions upon the gasoline yield, such as effect of reaction temp., gas velocity and compn. of raw gas; (3) research on catalysts, such as effect of carrier (catalysts used are Ni-Mn, Ni-U, Co-Cu, Co-Ni-Cu and Co promoted by Ag; and (4) mechanism of gasoline formation from CO and H<sub>2</sub>. Summary: An optimum reaction temp. and gas velocity for the formation of gasoline exist. It is preferable to use a gas mixt. contg. an excess of H over CO; H<sub>2</sub> = 1:2. The catalyst prep. from acetate showed the same activity as that from nitrate. Ag was found to be superior to Cu as a promoter. F. I. N.

Continental methods of testing lubricating oils. A. Kufferath and Edmund R. Thews. *Can. Chem. Met. 19*, 305, 321(1935).—A brief review. W. H. Boynton

Pressure resistance of lubricating oils. Theodor Rabunovich. *Petroleum Z. 31*, No. 28, 9-14(1935).—Theories of lubrication are reviewed. The moment of rotation of an expl. shaft was measured by a torsion dynamometer. The temp. of the lubricating layer was measured by a thermometer and the load by a lever transmission. The chief advantage of the machine is its adaptability. Test procedure and data are discussed in detail, along with the calcn. and expression of results. Relations between temp. increase and av. surface pressure for oils of different viscosities are plotted. C. B. Jenni

The determination of the bromine or iodine number of mineral lubricating oils. Emil E. Casimer and M. Dimitriu. *Petroleum Z. 31*, No. 33, 1-4(1935).—The McIlhenny and Wijs methods are used to det. the degree of unsat. of an oil. In the McIlhenny Br method, secondary reactions may produce HBr and cause erroneous results. Moisture in the reagents, instability of the Br derivs. of some hydrocarbons, relation of time of reaction and excess Br, and the effect of the compn. of the solvent are contributing factors to the irregularities mentioned. Data are given for the addn. and substitution indices for a few Roumanian, French and American lubricating oils, as detd. by the McIlhenny method. Significance of the time of reaction and excess halogen by the Wijs method is also noted. C. B. Jenni

Lubricating grease—a technical material—methods of manufacture. R. N. Smith. *Can. Chem. Met. 19*, 302, 304(1935).—Methods are outlined of prep. several types of grease, such as lime-base cup-grease, soda-base grease, semisolid grease, Al-base and Pb-base grease, solid lubricant and high-pressure grease. W. H. Boynton

Graphite lubrication and bearing metal. W. Lenx. *Petroleum Z. 31*, No. 33, 14(1935).—A brief review of the

literature on the use of graphite as a lubricant. The kind of bearing material is no more important in graphite lubrication than in oil lubrication. C. B. Jenni

The use of oils and fats and lubrication. Rudolf Pläcker. *Svensk Papperst.-Tidn. 62*, 791-2(1935).—Factors detg. the selection of lubricants for various purposes are discussed. J. W. Perry

Selection and use of lubricants for mining plant and machinery. Wilfred E. Gooday. *S. African Mining Eng. J. 46*, Pt. I, 803-4(1935). Alden H. Emery

Color testing of bitumen. D. M. Wilson. *Chemistry & Industry 1935*, 1040-2.—The prepn. of solns. in different solvents, and an app. with a photoelectric cell for measuring the light transmission is described. Data on various bitumens are given and the value of the method in testing and control is discussed. E. R. Rushton

Analysis of bitumen- and tar-mineral mixtures. H. Nussel and H. Henneke. *Bitumen 5*, 143-9(1935).—The detn. of free C in tar and insol. org. matter in bitumen is considered. Insol. matter from the extd. mixt. can be analyzed either by combustion or by oxidation with H<sub>2</sub>SO<sub>4</sub> satd. with CrO<sub>3</sub>. Practical examples and results are given. C. B. Jenni

The economic-technological importance of bitumens to German industry. Th. Temme. *Bitumen 5*, 161-6(1935).—Methods of obtaining bitumens from petroleum are briefly reviewed. Applications in road construction are described. Other uses discussed include bituminous paints, roofing paper, binding agents, insulating papers, cable coatings, insulating materials and rubber. C. B. Jenni

Plastic properties of asphalt bitumen. R. N. J. Saal. *Chem. Weekblad 32*, 435-41(1935).—A short review of the properties of asphalt bitumens, particularly dealing with methods for detn. of  $\tau$ -D curves (Couette, Pochettino, torsion and capillary viscometers). For low shear stresses the Couette app. is used, for high stresses the pressure capillary meter is preferred and the results are interpreted in  $\tau$ -D terms (S. and Koenig, C. A. 27, 2795). The temp. sensitivity, elasticity and thixotropy of several asphalt species were studied and curves for their behavior are given. The general conclusions are: asphaltic bitumens have differences in hardness as well as in type. Usually, high-temp. sensitivity, low plasticity, elasticity and thixotropy go together and vice versa. The result as applied to routine detns. show that for nonplastic asphalts, penetration and viscosity  $\eta$  are related:  $\eta = (5.1 \times 10^4) / \text{penetration}^{1.1}$  poises. The ring and ball temp. corresponds to a 10-20 000 poise viscosity. High plasticity usually accompanies a flat ductility-temp. curve. B. J. C. van der Hoeven

Resin extraction in German forests by a new process. M. Hesseland. *Angew. Chem. 48*, 636-9(1935).—Exptl. large-scale results on *Pinus sylvestris* are reported. The outer rough bark of the tree is first removed for 1/2 of the diam., care being taken not to injure the cambium. Then 2 incisions at right angles are made and a vertical incision is made downward from the apex (further pairs of incisions are added at intervals of 10 to 14 days). The incisions are then stimulated with some inorg. acid, alkali or salt; e.g., 3-5 drops of 25% HCl. Stimulation resulted in increased yields without impairing the quality of the sap, which had an approx. compn. of 23.4% of oil of turpentine, 70.4% of resin and 5.7% of H<sub>2</sub>O. The oil of turpentine b. 160-200° (50% at 163°) and had a d<sub>20</sub> of 0.863, and a sp. rotation of +20.6° at 20° [p]. Cost calcns. are appended. Four references. K. K.

Petroleum refiners view development of high-speed Diesel engine fuel (Mendius, et al.) 21. Waxes—mineral (Mellan) 13. Origin of petroleum (Krejci-Graft) 8. App. for treatment of substantially immiscible liquids as in treating oil fractions with H<sub>2</sub>SO<sub>4</sub> (U. S. pat. 2,022,205) 1. Emulsifying and dispersing agents (Brit. pat. 434,424) 13. Torpedo for shooting oil wells (U. S. pat. 2,023,831) 24. NH<sub>4</sub> sulfate from acid sludge [from treating oil with H<sub>2</sub>SO<sub>4</sub>] (U. S. pat. 2,022,813) 18.

**Solvent extraction of petroleum** John V. Starr (to Standard Oil Development Co.). U. S. 2,022,259, Nov. 26. Various details of app. and operation are described for countercurrent treatment as in the treatment of petroleum oils with a solvent such as phenol,  $\text{SO}_2$ , etc.

**Sweetening petroleum oils** Walter A. Schulze and Lovell V. Chaney (to Phillips Petroleum Co.). U. S. 2,022,942, Dec. 3. For improving petroleum oil contg. S compds with oxidizing properties which have been formed during the sweetening of the oil with an alk. hypochlorite soln., a mercaptan is added to the oil while it is out of contact with the hypochlorite soln. so that such S compds are converted into disulfides.

**Chlorinating petroleum hydrocarbons** Benjamin Galls-worthy (to Texas Co.). U. S. 2,022,619, Nov. 26. A lubricating oil or paraffin wax is treated with an aq. soln. of HCl and HOCl at a temp. of about 65-93° and under a pressure of 25-500 lb per sq in. App. is described.

**Stabilizing cracked petroleum distillates** LeRoy G. Story (to Texas Co.). U. S. 2,023,385, Dec. 3. Deterioration and gum formation on storage are inhibited by adding about 0.01-0.1% of a diaminophenol such as 2,4-diaminophenol.

**Cracking petroleum oils** Charles B. Buerger (to Gulf Refining Co.). U. S. 2,023,063, Dec. 3. Oil to be cracked is passed through a serially arranged tubular heater, and is heated to cracking temps. in one zone of the heater subjected to the hottest gases of a furnace, and is further heated in a 2nd zone of the heater subjected to furnace gases already used in the first-mentioned zone (the oil and gases passing countercurrentwise in the 2nd zone) and the heated oil is delivered to other app. for further treatment. Various features of app. arrangement are described.

**Cracking oils** Fritz Uhlmann (to Aktis A.-G.). U. S. 2,023,754, Dec. 10. Oil or oil vapor is passed over coke within a high-frequency field, and at the same time water is "cracked" by electrolysis within the high frequency field to liberate  $\text{H}_2$  for combining with the oil vapors. App. is described.

**Cracking hydrocarbon oils** Wm. L. Gornory (to Standard Oil Development Co.). U. S. 2,022,280, Nov. 26. Oil is heated to cracking temp. in a coil, passed to a vaporizing zone from which a reflux condensate is collected which is subjected to distn. in a sep. reflux stripping zone, an unvaporized liquid fraction from the latter being passed to the heating coil. Uncondensed vapors from the vaporizing zone are fractionated in a sep. fractionating zone, and the vapor fraction evolved in the reflux stripping zone is in part conducted to the vaporizing zone and in part to the fractionating zone, vapors from the fractionating zone are removed and condensed. App. is described. Cf. C. A. 29, 5907.

**Cracking hydrocarbon oils** Gustav Egloff (to Universal Oil Products Co.). U. S. 2,022,803, Dec. 3. A vaporous mixt. of hydrocarbons such as vapors from a fuel oil is fractionated to obtain relatively heavier and lighter cuts of higher b. p. than gasoline, a portion of the heavier cut is heated to a cracking temp. while maintained under sufficient pressure to retain a substantial portion in the liquid phase, the lighter cut is separately cracked in the vapor phase by heating it to a higher temp. than the heavier cut; another portion of the heavier cut is diverted from the liquid phase heating step and introduced to the lighter cut after the vapor-phase cracking of the latter, and the commingled vapors are subjected to dephlegmation and condensation. App. is described.

**Cracking hydrocarbon oils** Lev A. Mekler (to Universal Oil Products Co.). U. S. 2,022,810, Dec. 3. Vapors formed in another stage of the process are passed to a fractionating zone where they are fractionated in contact with charging oil to form a mixt. of charging oil and primary reflux, and the vapors are further fractionated out of contact with charging oil to form a relatively light secondary reflux, the mixt. mentioned is passed through a heating zone and heated to a cracking temp. under sufficient pressure to maintain a substantial portion in the liquid phase, and, simultaneously, a substantial portion of the secondary reflux is vaporized and the resultant

vapors are heated independently of and to a higher cracking temp. than the mixt. mentioned; the thus heated vapors and the heated mixt. mentioned are introduced into an enlarged reaction zone maintained under cracking conditions of temp. and pressure, the vaporous and liquid conversion products are removed as a mixt. from the reaction zone and the pressure on them is lowered; vapors and residue are sep'd. under the reduced pressure, and the vapors are fed to the fractionating zone as first mentioned, the fractionated vapors being finally condensed. App. is described.

**Cracking hydrocarbon oils** I. G. Farbenmnd. A.-G. (Georg Stern and Robert Hasenclever, inventors.). Ger. 617,710, Aug. 24, 1935 (Cl. 23b. 1.04). Heavy oils are vaporized and heated with a large amt. of the vapor of oils of lower b. p.

**Cracking heavy hydrocarbon oils** Neal S. Olsen (to Universal Oil Products Co.). U. S. 2,023,205, Dec. 3. A mixt. of oil such as an asphaltic base oil and a finely divided hydrogenating catalyst such as finely divided Fe is heated to a cracking temp. under pressure, atomized with H and the atomized mixt. is subjected to a temp. of about 425-605° under a pressure in excess of 1000 lb per sq in. App. is described.

**Distilling and cracking hydrocarbon oils** Guy O. Shefstad (to Texas Co.). U. S. 2,023,619, Dec. 10. App. and procedure are described involving passing charging stock as a crude petroleum contg. crude residual constituents, lower-boiling constituents and natural gasoline fractions through a heating coil to heat the oil to a temp. to vaporize a given fraction, passing the heated oil into a distg. and fractionating zone under approx. atm. pressure, fractionating vapors thus evolved to sep. a distillate contg. natural gasoline fractions, taking off the stripped oil and passing it, under superatm. pressure, through a coil heated to a cracking temp. and thence into an enlarged chamber maintained under a relatively high pressure and at a cracking temp. and thence passing vapors to a fractionating column under pressure materially greater than that in the distg. and fractionating zone mentioned, to form an overhead vapor fraction and a reflux condensate. The reflux condensate is passed into the distg. and fractionating zone to strip from it the lower-boiling constituents and combine the heavier portions with the liquid in such zone, and the combined liquid is thence passed to the cracking coil as charging stock.

**Cracking heavy naphtha** Arthur E. Pew, Jr. (to Sun Oil Co.). U. S. 2,021,923, Nov. 26. A heavy naphtha having a crit. temp. within its cracking range but below the temp. at which it can be effectively cracked is heated, as a continuously flowing stream, to a temp. within its cracking range and so close to but below its crit. temp. as to effect a rise in its crit. temp. and the temp. of the oil in the flowing stream is increased, while still maintaining it below its rising crit. temp., and a suitable pressure is maintained.

**Apparatus for cracking hydrocarbon oils** Universal Oil Products Co. Ger. 617,950, Aug. 29, 1935 (Cl. 23b. 1.04).

**Polymerizing olefins in an oil-cracking process** Frederick W. Sullivan, Jr. (to Standard Oil Co. of Ind.). U. S. 2,022,221, Nov. 26. Various details of app. are described and of a process in which a stream of oil is cracked in the presence of recycled normally gaseous hydrocarbons formed in the process and from which substantial amts. of H and  $\text{CH}_4$  have been removed and in which unsat'd. hydrocarbons are polymerized and included in the final product to obtain a gasoline of relatively high antiknock properties. Various details of sepn., fractionation, etc., are described.

**Return bend suitable for hydrocarbon-oil-conversion apparatus** Andrew T. Dudley (to Texas Co.). U. S. 2,023,805, Dec. 10.

**Hydrocarbon-oil conversion** Richard F. Trow (to Texas Co.). U. S. 2,023,775, Dec. 10. A flowing stream of clean oil such as a clean gas oil is heated to a high temp. of conversion, transferred to enlarged zones whence evolved vapors are transferred to a coil in which they are heated to a temp. higher than that to which the stream of

oil is heated; a depositing residue is withdrawn from the enlarged zones, highly heated vapors are removed from the coil and commingled with a cooler portion of the charge oil to heat the latter to a conversion temp.; the residue withdrawn from the enlarged zones is commingled with the vapors and admixed oil, the commingled products are passed into a reaction and distn. vessel, a stream of charge oil is delivered into the upper portion of this vessel, resultant total vapors are scrubbed in the distn. zone, and the scrubbed vapors are transferred and fractionated to develop an overhead ultimate motor fuel and the clean oil first mentioned while withdrawing residue from the reaction and distn. vessel. App. is described.

**Purifying acid-treated light hydrocarbon oils to remove sulfonic esters** Francis M. Archibald and Philip Janssen (to Standard Oil Development Co.). U. S. 2,022,208, Nov. 26. The oil is subjected to flash distn. in the presence of previously sepd. sulfonic esters which catalytically promote the liberation of further quantities of sulfonic esters, and the purified oil distillate and sulfonic esters are separately removed. App. is described.

**Hydrocarbon oxidation products** Standard Oil Development Co. Brit. 434,311, Aug. 29, 1935. Petroleum or slop wax is treated to reduce its oil content to 25% or less and is then oxidized, either directly or after distn., which may be accompanied or followed by cracking or dehydrogenation. The reduction of the oil content may be effected by extrn. with a selective solvent for the oil. The wax may then be distd. without decompn. at 600°F., or it may be cracked at 850–900°F., at atm. or reduced pressure. Dehydrogenation may be effected at 850–900°F. at atm. or slightly raised pressure in the presence of a catalyst, H may be present during dehydrogenation. Oxidation is then effected in the liquid phase at below 400°F. with air or other gas contg. free O. Al-lined vessels may be used and a catalyst may be present.

**Hydrogenating oils** I. G. Farbenindustrie A.-G. (to International Hydrogenation Patents Co. Ltd.). Brit. 433,593, Aug. 16, 1935. Addn. to 424,531 (C. A. 29, 5239). In refining hydrocarbons contg. unsatd. compds., by a 1st stage hydrogenation at below 300°, followed by a 2nd stage treatment at higher temps. and pressures, as described in 424,531, the 2nd stage is carried out under atm. or low pressures up to 20 atm. A fraction, boiling at 100–200°, of the products obtained by cracking gas oil at 100° is led with H under 50 atm. and at 200° over WC. The products are led with H at atm. pressure and 400° over a catalyst produced by treating Ni molybdate with H<sub>2</sub>S at 450°.

**Refining mineral oils** Clarence R. McKay and John H. Smith (to Sinclair Refining Co.). U. S. 2,022,338, Nov. 26. For refining and stabilizing of color, a metal hydrosulfide such as Ca(OH)<sub>2</sub> is added to an acid-treated oil so as to effect consolidation of the sludge without fully neutralizing the morg. acid in the oil, the sludge is settled, and the clarified oil is treated with a solid adsorbent material such as infusorial earth while heated, and then sepd. App. is described.

**Refining mineral oils** Willem J. D. van Dyck (to Shell Development Co.). U. S. 2,023,375, Dec. 3. An oil such as one already treated with liquid SO<sub>2</sub> is extrd. with a solvent comprising an org. compd. other than a nitrated monocyclic aromatic hydrocarbon, which contains a nitro group, such as nitromethane, an org. compd. other than a thiocyanate which contains a —CN group, such as propionitrile or benzonitrile or with methyl thiocyanate, which serve to sep. paraffinic and naphthenic compds. Several examples are given.

**Fractionating mineral oils by solvent treatment** Willem J. D. van Dyck (to Shell Development Co.). U. S. 2,023,109, Dec. 3. An oil such as a lubricating fraction and at least partially immiscible solvents such as benzene and liquid SO<sub>2</sub> or MeOH and CS<sub>2</sub> are brought into contact with each other in counterflow. App. is described.

**Apparatus for refining mineral oils** Aktiebolaget Separator-Nobel. Brit. 433,584, Aug. 16, 1935. In the continuous refining of oils by acid, the tanks, pipes, mixers and other app. are so vented that gas pockets

are not formed, any gases being carried off by the mixt. or escaping at suitable points of the system. App. is described.

**Mineral- or tar-oil compositions** Kerasin A.-G. Ger. 620,260, Oct. 17, 1935 (Cl. 23c. 3). Comps. which are solid at atm. temp. are obtained by mixing mineral or tar oils with small proportions of soap (3–6%) and beeswax (3–10%) at a moderately raised temp. The soap is preferably a Na soap, and is advantageously added in an org. solvent, e. g., alc. The beeswax may be replaced in part by paraffin wax, but at least 1% of beeswax must still be used. Appropriate addns., e. g., perfumes or therapeutic substances, may be included in the compns., which may be used, e. g., for cosmetic or medicinal purposes or as lubricant greases.

**Pour-point reduction of mineral oils** Hyman Limburg (to Shell Development Co.). U. S. 2,023,309, Dec. 3. For obtaining a soln. of concd. pour-point-reducing substances from a mixt. contg. them such as wool fat, the substances desired are sepd. from the mixt. with an adsorbing agent such as paraffin or silica gel and the latter, with adsorbed substances, is sepd. and treated with a solvent such as pyridine in which the pour-point-reducing substances readily dissolve.

**Low-pour-point mineral-oil composition suitable for lubrication** Adrianus J. van Peski (to Shell Development Co.). U. S. 2,022,900, Dec. 3. A wax-contg. mineral oil is used with an admixt. of a relatively small proportion (suitably about 0.5%) of a condensation product (such as that of sucrose and stearic acid) which serves to lower the pour point of the oil and which is derived from a sugar having at least one cyclic nucleus and not more than 2 monosaccharide groups in its mol. and an aliphatic acid.

**Hydrometer suitable for use with a stream of hydrocarbon distillate** Oscar H. Fairchild (to Universal Oil Products Co.). U. S. 2,023,196, Dec. 3. Structural details.

**Countercurrent system suitable for treating oils with sulfonic acid** Evert T. Pummil (to Socony-Vacuum Oil Co.). U. S. 2,023,545–6, Dec. 10. Various details of app. and operation.

**Removing deposits from conduits of oil-conversion apparatus** Harold R. Snow and Russell L. Zimmerman (to Standard Oil Co. of Ind.). U. S. 2,022,935, Dec. 3. Structural and operative details are described of an app., including a plunger, etc.

**Protective device for holding casinghead drips** Orf B. Zeek (to Magnolia Petroleum Co.). U. S. 2,022,267, Nov. 26. Various structural details.

**Antiknock motor fuel** Nicolaas Max (to Shell Development Co.). U. S. 2,023,142, Dec. 3. A hydrocarbon motor fuel mixt. is prepd. contg. a small proportion (suitably about 0.02–0.06% of the metal) of salicylal-methyl (or allyl) amine-Cu or -Co or other suitable heavy metal compd. of the general formula R<sup>1</sup>R<sup>2</sup>C:NR<sup>3</sup>, in which R<sup>1</sup> represents an alkyl group of H, R<sup>2</sup> represents a hydrocarbon grouping directly linked to at least one hydroxyl group other than that contained in a carboxylic acid, and R<sup>3</sup> represents an alkyl group, the heavy metal present being one capable of forming a complex compd. with NH<sub>2</sub>.

**Stabilizing the color of cracked hydrocarbon motor fuels** Robert E. Wilson (to Gasoline Antioxidant Co.). U. S. 2,023,110, Dec. 3. Cracked motor fuel treated with a gum-formation-inhibiting agent such as  $\alpha$ -naphthol,  $\alpha$ -naphthylamine or  $p$ -phenylenediamine, tending to cause color degradation of the fuel, is also treated with a small proportion of a polyhydroxy benzene compd. such as pyrogallol, hydroquinone, catechol or gallic acid to retard the color degradation.

**Regenerating spent sodium plumbite solution used for treating gasoline or kerosene**, etc. Frederick W. Stone and James N. Garrison. U. S. 2,022,550, Nov. 26. A body of the spent soln. is heated and subjected to relatively violent and then to relatively gentle agitation to break up the emulsion so that the oil rises and is skimmed off; air is discharged into the remaining spent soln. and intimately mixed with it and the treatment is continued until the regenerating reaction is complete. App. is described.

Inhibiting gum formation in cracked motor fuels Louis A. Clarke and Charles C. Towne (to Texas Co.) U. S. 2,023,871, Dec. 10. About 0.01% of monochloro-hydroquinone or the like is added to a material such as a cracked gasoline or kerosene to retard deterioration and gum formation.

Sweetening light naphthas Arthur F. Endres (to Standard Oil Co. of Ind.). U. S. 2,022,847, Dec. 3. In prep. light naphthas comprising mostly butanes and pentanes from crude light naphtha, the latter is sweetened with an alk. soln. of Na plumbite before it is stabilized. App. is described.

Treating acid sludge from sulfuric acid treatment of cracked oils and naphthas Arthur B. Brown and David W. Bransky (to Standard Oil Co. of Ind.). U. S. 2,022,800, Dec. 3. "Oleum liver" is added to the sludge and the mixt. is dild. with water until an acid concn. of about 30% is reached, the mixt. is heated to about 93° and agitated, then allowed to settle, and the acid layer is withdrawn and concd.

Dewaxing oils Leo D. Jones (to The Sharples Specialty Co.) Brit. 434,579, Sept. 4, 1935. In dewaxing oils by a succession of dewaxing stages consisting of chilling and sepg. the wax, the partially dewaxed oil is heated between each stage to bring the residual wax into soln. A crude oil is mixed with a diluent, e. g., naphtha,  $C_{10}H_8$ ,  $C_{12}H_{10}$ ,  $C_6H_6$ , and the mixt. cooled slowly to -20°F., the wax is removed by centrifuging or filtration with the use of kieselguhr or similar filter aid, the oil contg. fine particles of wax is heated to above 30°F. and again chilled, preferably to a lower temp., e. g., from -30° to -60°F., than in the 1st stage, to yield a further amt. of wax which is sepd., a low cold test lubricating oil being left. Cf. C. A. 30, 280.

Dewaxing hydrocarbon oils Earl Petty (to Alco Products Inc.) U. S. 2,023,181, Dec. 3. For dewaxing hydrocarbon oils bearing a wax-cryst. retarder in the form of coloring material, the oil is blended with a diluent such as naphtha and propane and the soln. thus formed is chilled to a temp. at which the retarding effect of the coloring material is overcome, such a portion of the coloring material is removed (suitably by use of  $H_2SO_4$  or  $SO_2$ ) that the wax-cryst. retarding effect of the remainder will not prevent substantially complete wax pptn. on further chilling, and the soln. is "sub-chilled" and wax thus pptd. is sepd. Various details of procedure are described. Cf. C. A. 29, 6043.

Doctor solutions for sweetening sour oils Byron F. Dooley, Jr. (to Texas Co.). U. S. 2,022,558, Nov. 26. Impure Pb sludge in spent doctor soln. is sepd. and extd. with an org. solvent such as naphtha or kerosene to remove org. impurities and the purified sludge is dissolved in a caustic soln. maintained at an elevated temp. while blowing with air, to obtain a regenerated soln. substantially free from substances tending to impart color and gum instability to naphthas treated with the soln.

Lubricating oils I. G. Farbenindustrie A.-G. Brit. 434,295, Aug. 29, 1935. The oils are improved as regards turbidity, setting point and color by an addn., e. g., 0.05-3%, of an oil-sol. vacuum residue, solid at ordinary temps., derived from de-asphalted hydrocarbon products contg. cyclic constituents, e. g., tar, asphalt-basic or mixed basic mineral oils, especially from hydrogenation products or from the destructive hydrogenation of coals or coal tars. The initial material is treated with flocculents, e. g., benzene, gas oil or liquefied normally gaseous hydrocarbons, to ppt. asphalts, and, after sepn., the oil is subjected to vacuum distn., preferably up to 300-350° and under 78 mm. Hg pressure. In an example, a destructive hydrogenation product boiling above 350° is treated with  $C_6H_6$  and centrifuged to remove solid constituents, then with a light benzene contg. petr. ether to sepd. asphalt, after which the larger portion of the precipitant is distd. off and the mixt. is then refined with  $H_2SO_4$  and then NaOH, the washed and dried oil is distd. up to 350° under 8 mm. Hg pressure to yield a red-brown resin as residue.

Lubricating oils Texaco Development Corp. Fr.

1786,581, Sept. 5, 1935. Oils contg. paraffin are dissolved in a mixt. of solvents contg. liquid  $SO_2$  and an agent modifying the properties of the solvent, e. g.,  $PhCl$  and  $C_6H_5Cl$ , in proportions such that at below -18° the mixt. has a practically complete solvent action on the liquid hydrocarbons and practically no solvent action on the solid hydrocarbons of the oils. The mixt. is cooled to -18° or lower and the solid hydrocarbons are sepd. Thus, a solvent contg.  $SO_2$ , 30 and  $PhCl$  70% may be used.

Lubricating compositions; electrical insulations Standard Oil Development Co. Brit. 434,626, Sept. 5, 1935. An elec. insulation or lubricating compn., especially for use at high temps., consists of a high-boiling aryl ester of an inorg. acid, e. g., triphenyl or tritolyl phosphate or borate, and an oxidation inhibitor, e. g., resorcinol, pyrogallol,  $\alpha$ -naphthol, naphthylamines, thiophenols, thio-naphthols, sol. thereon. The ester may be mixed with thickening or thinning agents, e. g.,  $Pb$ , diphenyl oxide, dimethylnaphthalene, glycol phthalate, Et. oleate, sol.  $PhOH$ - $CH_2O$  resins, volatilized oils. Na, Ca or Pb soaps may also be added.

Lubricants, motor fuel Carl M. Clementson and Sven O. H. Clementson (trading as C. & S. Clementson). Brit. 434,240, Aug. 28, 1935. Lubricants that inhibit corrosion consist of mineral, vegetable or animal oils mixed with the soln. resulting from the mixing of an alkali peroxide with  $MeOH$ , or other alc., and a homogenizing agent, e. g., hydrogenated  $PhOH$  or cresol. The lubricant may be added to the fuel for an internal-combustion engine. Lubricants Standard Oil Development Co. Brit. 434,058, Aug. 20, 1935. See Fr. 779,896 (C. A. 29, 5648).

Lubricants Pierre A. Deloche Fr. 787,075, Sept. 16, 1935. App. is described for mixing by opposing jets and circulation by pump, mineral oil with castor oil (1-20) and halides, stearic acid or stearates (1-3%).

Reconditioning used lubricating oils Geo. R. Pierce and Leslie B. Denning, Sr. Brit. 433,570, Aug. 22, 1935. See Fr. 769,519 (C. A. 29, 5969). App. is described.

Filtering and regenerating apparatus for used oil Samuel Levine Fr. 786,925, Sept. 14, 1935.

Rectifying polluted oil such as that from automobile engines Robert P. Moyer and Fredella H. Moyer U. S. 2,022,731, Dec. 3. Various details of app. and operation are described, the oil being dild. with a volatile solvent such as naphtha or  $CCl_4$  and centrifugally filtered and solvent vapors being sepd. and condensed.

Removing viscous hydrocarbon deposits from the interior surfaces of tanks, etc. Verne J. Todd U. S. 2,023,496, Dec. 10. Steam is used to apply to a detergent mixt. including a saponified fat,  $Na_2PO_4$ ,  $Na_2CO_3$ , Na silicate and a hydrocarbon solvent such as furnace oil. Various details of app. and operation are described.

Asphalt emulsion Robert K. Franke, Henry W. Moreton and Charles W. Hill. U. S. 2,023,540, Dec. 10. Asphalt about 36 gal. is emulsified with a colloidal soln. formed of colloidal hydrous Mg silicate 10 lb. and water 20 gal., and 420 cc. of a 0.1 N HOAc soln., together with addl. water. App. is described.

Bituminous dispersions I. G. Farbenindustrie A.-G. Brit. 433,550, Aug. 16, 1935. Coarse dispersions, where  $H_2O$ -insol. org. substances, e. g., humic acid, lignic acid, acids of high mol. wt.,  $PhOH$ -like substances, soot, finely ground resins, waxes or pitches, are used as emulsifying agents, are obtained by using such proportions of bitumen and  $H_2O$  that a stiff magna that is difficult to stir is obtained. Fused bitumen and  $H_2O$  are added alternately, preferably with cooling, to a paste of the emulsifying agent contg. little  $H_2O$ . After each addn. of bitumen, stirring becomes difficult.  $H_2O$  is then added until the mass can just be stirred again. Alternatively, the components are added at 2 or more parts of the dispersing vessel so that at some points stirring is impossible, because of addn. of bitumen, and at other points the mass can be stirred, because of addn. of  $H_2O$ . The product is a stiff mass, which is dild., after the final addn. of bitumen, to form a dispersion contg. about 50% bitumen.



**Bituminous compositions.** I. G. Farbenindustrie A.-G. Brit. 433,874, Aug. 22, 1935. The compns. contain Fe oxide obtained by the reduction of aromatic nitro compds with the aid of Fe. In compns. contg. bituminous emulsion and (1) sand, (2) Rhine sand (I) and basalt chips (II), or (3) I, II and portland cement, increased compressive strength is obtained when part of the sand is replaced by the Fe oxide.

**Rubber and bitumen compositions.** Internationale Vereniging voor de Rubber- en Andere Cultures in Nederlandsch-Indie Brit. 434,317, Aug. 20, 1935. This corresponds to Fr 783,041 (C. A. 29, 7068<sup>9</sup>).

**Bituminous compositions.** Planktoll chemische Fabrik G m b H and Johannes B. Carpozow Brit. 434,926, Sept. 11, 1935. Sea-water mud, in a moist state and after drying at a low temp. in the absence of air, is mixed with

bituminous substances, e. g., natural or artificial asphalts, or coal tars, if desired in emulsified form, and the whole is heated *in vacuo* at 180° until the constitutionally bound H<sub>2</sub>O of the mud is completely expelled. Before use, the mud may be sepd. from cryst. matter.

**Apparatus and method for carbonizing wood.** Paul Guillaume. Brit. 433,256, Aug. 12, 1935. In the production of wood charcoal, during a period of slow carbonization the gaseous hydrocarbons and distillates of tar or oils suspended in the gases are caused to remain in contact with, and are recirculated through, the mass of charcoal while the latter is cooling.

**Wood oil.** Sommer-Schmidding-Werke Vertriebs G. m b H Ger. 617,849, Aug. 27, 1935 (Cl. 22A. 2). Addn. to 594,920 (C. A. 28, 4594<sup>9</sup>) App. for obtaining pure heavy oil from wood oil is described.

## 23—CELLULOSE AND PAPER

CARLETON E. CURRAN

**The nature of cellulose.** E. Heuser. *Paper Trade J.* 101, No. 21, 39-46, No. 22, 35-41, No. 23, 39-42 (1935).—A historical review with bibliography of 59 references.

A. Papmeue-Couture

**Separation of cellulose with chlorine.** V. I. Minaev and S. S. Frolow. *Trans. Inst. Chem. Tech. Lianoto* (U. S. S. R.) 1, 196-203 (1935).—In the preliminary communication lab. expts. are described for the sepn. of cellulose from various cellular waste materials by steeping them in H<sub>2</sub>O or Ca(OH)<sub>2</sub> soln. and treating the products with Cl by the Pomilio-Cataldi method. Chas. Blanc.

**The application of the modified one-step process as a general method of determination of cellulose.** Yuan-Chi Tang and Hsiob-Li Wang. *Naturwissenschaften* 23, 577 (1935); cf. C. A. 29, 8333<sup>9</sup>.—The "one step" method (Schmidt, et al., C. A. 26, 1772) was modified and shortened: A 1.5% ClO<sub>2</sub> soln. and 1 to 1.5% pyridine soln. are now used. The phloroglucinol reaction disappears some time before the destruction is finished.

B. J. C. van der Hoeven

**Ammonia-cellulose (preliminary communication).** K. Hess and C. Trogus. *Ber.* 68B, 1986-8 (1935).—This report is published because of the appearance of a brief abstr. (*Cellulose-Chem.* 16, 6 (1935)) of a paper by Peterson, King and Barry presented at the New York meeting of the Am. Chem. Soc. in April, 1935. It had been shown in an earlier paper (C. A. 26, 591) that cellulose (I) reacts with aq. solns. of diamines to form a new type of double compds. with characteristic Röntgen diagrams. The study has been extended to NH<sub>3</sub>, whose aq. solns. have hitherto been considered as being indifferent toward I, and it has been found that liquid NH<sub>3</sub> markedly alters the lattice of I. When NH<sub>3</sub> gas, carefully dried over KOH, is allowed to act on dry I at -70° to -80°, the NH<sub>3</sub> condenses over the fibers without producing any perceptible alteration (swelling) in their external appearance. If the NH<sub>3</sub> is distd. off (again in the absence of moisture), there remain the apparently unaltered fibers which, however, show a Röntgen diagram entirely different from that of the original material (photograph given). A table of the equatorial interferences of this product (II), which is possibly an NH<sub>3</sub>-poor ammonia-cellulose or a 3rd modification of I and is provisionally called *cellulose III*, shows that the new diagram cannot be confused with those of the 2 hitherto known forms of I (natural cellulose and hydrate cellulose). If, after evapn. of the NH<sub>3</sub>, the fibers are transferred to water, MeOH or concd. aq. NH<sub>4</sub>OH, the diagram of the original fibers is in no case restored. Photographs and interference positions of the products treated with MeOH and aq. NH<sub>4</sub>OH are given. Whether these diagrams represent further modifications of I or addn. compds. of I with the liquids can be detd. only by further expts., but the ease with which the Röntgen picture can be changed shows that II is a very reactive form of I. C. A. R.

**Cellulose triacetate.** Jean Grand. *Compt. rend.* 201,

678 (1935).—The following procedure yields a cellulose triacetate of good quality, more resistant to sapon. by H<sub>2</sub>O than the commercial acetates, completely sol. in methylene chloride-alc. mixt. The cellulose is treated for 15 min. with a mixt. of equal parts of HOAc and 0.5 N HClO<sub>4</sub>, dried completely and immersed in a bath of 1 part Ac<sub>2</sub>O and 5 parts of CCl<sub>4</sub> maintained at a temp. below 10° and agitated.

Rachel Brown

**Benrylcellulose.** A review of properties and uses. R. C. Bickmore. *Chem. Industries* 37, 445-7 (1935).

E. H.

**Nitration of cellulose with nitrogen pentoxide vapor.** René Dalmon. *Compt. rend.* 201, 1123-4 (1935), cf. C. A. 30, 282<sup>9</sup>.—Pure N<sub>2</sub>O<sub>5</sub> was passed over dried cotton in a tube by a current of dry air or O<sub>2</sub> at the ordinary temp. The reaction proceeds according to C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>(OH) + 3N<sub>2</sub>O<sub>5</sub> = C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>(NO<sub>3</sub>)<sub>3</sub> + 3HNO<sub>3</sub>, the HNO<sub>3</sub> appearing as dew on the cotton. After washing, etc., the product is pure trimtrate (14.12% N).

C. A. Silberrad

**The decomposition of cellulose in the nitrating and denitrating processes.** Z. Rogovin and M. Shlyakover. *Angew. Chem.* 48, 647-50 (1935).—See C. A. 29, 6754<sup>9</sup>.

Karl Kammermeyer

**Mechanical properties of mixed nitrocellulose films.** A. I. Medvedev. *Trans. Inst. Chem. Tech. Ivanovo* (U. S. S. R.) 1, 204-13 (1935), cf. C. A. 28, 3892<sup>9</sup>.—Films were obtained by mixing 3% acetone solns. of nitrocellulose and its fractions with 18, 30, 46 and 66% of Glyptal. The addn. of Glyptal to nitrocellulose solns. greatly improves the mech. properties of films. Elongation curves for the mixed films run somewhat higher than those for pure films. The general elongation of mixed nitrocellulose films is slightly decreased, while that of denitrated films is increased, but to a less degree than that of films with polyvinyl acetate. In denitrated films the favorable action of Glyptal on the elastic properties is greater than that of polyvinyl acetate. The mech. properties of heated films, with and without the addn. of Glyptal and polyvinyl acetate, change with a considerable decrease of the strength, increase of general elongation and reduction of the time of deformation. At 50° the elastic properties of films with polyvinyl acetate are lower and with Glyptal are higher than those of pure nitrocellulose.

Chas. Blanc

**Advances in colloid chemistry (Cellulose and paper.)** Rudolf Lorenz. *Zellstoff u. Papier* 15, 193-6, 235-6, 276-9, 313-19 (1935).—A detailed review with 99 references.

S. I. Aronovsky

**Literature of lignin.** Bror Holmberg. *Scensk Pappers-Tid.* 38, 556-7 (1935); cf. C. A. 17, 3783, 23, 5575; 24, 3358, 6007; 26, 6132; 27, 5099, 28, 1529<sup>9</sup>, 5854<sup>9</sup>; 29, 5151<sup>9</sup>, 5651<sup>9</sup>, 7634<sup>9</sup>.—By means of numerous references and quotations H. shows that the objections raised by Klason (cf. C. A. 29, 7396<sup>9</sup>) in regard to the chem. compn. of lignin are not well founded.

W. S.

**Action of formaldehyde and acetic anhydride on viscose**

rayon V I Minaev and S S Frolov. *Trans. Inst. Chem. Tech. Ivanovo* (U. S. S. R.) 1, 166-73(1935).—Tests with viscose rayon confirmed essentially the expts of other investigators (cf. Schenk, *C. A.* 26, 296, 27, 3602; Wood, *C. A.* 26, 1254, 27, 3602) on the action of  $\text{CH}_3\text{O}$  and  $\text{Ac}_2\text{O}$  on cellulose. Better results were obtained by treating viscose rayon with  $\text{CH}_3\text{O}$  vapors and with solns of  $\text{CH}_3\text{O}$  in  $\text{Me}_2\text{CO}$  than in  $\text{H}_2\text{O}$ . The action of  $\text{Ac}_2\text{O}$  is considerably weaker, the results depending more on the conditions of treatment than with  $\text{CH}_3\text{O}$ . C. B.

The acid content of rayons and its determination. Max Ludtke. *Angew. Chem.* 48, 650-1(1935).—The acid in rayons was detd by means of the reaction  $(\text{CH}_3\text{CO}_2)_2\text{Ca} + 2\text{RCO}_2\text{H} = (\text{RCO}_2)_2\text{Ca} + 2\text{H}_3\text{CO}_2\text{H}$  with a 1 N acetate soln and 0.01 N NaOH soln for the titration. Exptl results on 14 kinds of rayon are presented. The acid no., e.g., the amt. of acid present in 1 g of dried solid, expressed as cc of 0.01 N soln (relative values), was lowest (100) for an acetate rayon prepd from linters, and also quite low for 2 Cu rayons likewise prepd from cotton linters (1.53 and 1.78). These results confirm previous observations that products prepd. from bleached cotton possess a much lower acid no. than those prepd. from cellulose. A nitro-rayon gave a value of 3.66. Viscose rayons which were known to have been subjected to a long ripening period showed high values (4.23 to 5.31), while the other viscose rayons varied from 1.87 to 4.97. Acid nos. are also reported for a viscose rayon which had been dyed with various substantive dyes. While the original material showed a value of about 4.37, the values of the dyed rayons were all higher (4.54-6.14), because of the presence of sulfonic acid residues, and these differences can be used as a comparative scale for the amt of fixed dye. Eight references. Karl Kammermeyer.

Rayon from egg white G Salzmänn. *Melhandl. Textilber.* 16, 705-7(1935).—A review of processes for making rayon from egg white and other proteins.

The Southern States as a future center for American pulp and paper industry. Erland Waldenström. *Swensk Pappers-Tidn.* 38, 588-603(1935).—Descriptive. Wilhelm Segerblom.

Technical advances in the paper pulp industry of the northern countries (Europe) in 1933-1934. C. A. Ernst. *Zellstoff u. Papier* 15, 141-6, 241-3, 282-4, 357-8(1935).—A discussion of the mech and chem advances in the manuf. of ground wood pulp, sulfite pulp and in bleaching. S. I. Aronovsky.

Advances in paper and pulp testing in 1934. B. Korn. *Zellstoff u. Papier* 15, 353-6(1935).—A brief review of the newer microscopic, chem., mech and phys. methods of testing pulp and paper, including 56 references. S. I. Aronovsky.

Modern heat insulation in the pulp and paper industry. G. E. Grimshaw. *Paper Ind.* 17, 566-71(1935).—A brief discussion of modern heat insulation, showing how improvement in lab. equipment for the measurement of heat transfer has rendered it possible to judge of the efficiency of insulation and hence permitted improving heat-insulating materials. A. Papineau-Couture.

Arc welding builds longer life into pulp and paper mill equipment. R. R. Kondal. *Paper Trade J.* 101, No. 18, 50, 52(1935).—A brief outline of the merits of arc welding for sulfite digesters. A. Papineau-Couture.

Pulping treatment and fiber properties. Geo. A. Richter. *Paper Trade J.* 101, No. 19, 36-40(1935).—A review of the important properties of sulfite, soda and kraft pulps and the improvements that have been made in these pulps in recent yrs. The contributions of new paper properties that have been made by new pulps are outlined. A. Papineau-Couture.

The morphology of cellulose fibers as related to the manufacture of paper. Geo. J. Ritter. *Paper Trade J.* 101, No. 18, 92-100(1935).—A description of the morphology of cellulose fibers as depicted by different investigators, together with a brief discussion of both generally accepted and controversial views regarding the morphology and

some of the attendant phys. properties of fibers.

A. Papineau-Couture. Some effects of mechanical treatment of fibers on sheet structure. R. H. Doughty. *Paper Trade J.* 101, No. 18, 31-3(1935).—An outline of material properties and the operation of unit processes, together with a review of semiquantitative data on the influence of fiber treatment on fiber properties and thence on sheet structure to show how one part of this science can be developed.

A. Papineau-Couture. Wood handling and preparing. J. W. Patterson. *Pulp Paper Can.* 36, 551-4(1935).—A description of the layout and equipment of the wood room of the mill of the E. B. Eddy Co. Ltd., Hull, Que. A. Papineau-Couture.

The influence of hydrogen ion concentration with pretreatment of wood on its subsequent delignification. A. J. Corey and O. Maass. *Can. J. Research* 13B, 289-93(1935), cf. *C. A.* 30, 2837.—The results obtained by the authors in a previous investigation have been checked. The influence of an acid, a salt and a base on the pretreatment of wood at 130° has been detd. It is shown that pretreatment of wood meal decreases the rate of subsequent delignification. Pretreatment was carried out over the temp. range 100° to 140° and the effect of temp. is shown to be very marked. As it was found that the  $p_H$  of the water after pretreatment decreased, pretreatment was carried out on a chip in which the  $p_H$  was kept more const. by a forced flow of water through the chip. It is shown that the rate of delignification is increased. Pretreatment was carried out in buffer solns and it is shown that there is a  $p_H$  value for which the influence of pretreatment is a min. The inferences drawn in connection with sulfite cooking are briefly discussed. J. W. S.

Investigations of methods for determining the strength of chemical wood pulp. Sigurd Köhler. *Statens Provingsanstalt, Medd.* No. 47, 1-38(1930).—By the strength of chem. wood pulp is meant the strength properties of sheets made from the pulp after the latter has been disintegrated. Any suitable arrangement can be adopted to disintegrate the pulp in water, provided that the pulp is not subjected to a noticeable degree of beating. Comparative tests have been made to det. the beating degree ("Slowness") by use of 2 different Schopper-Riegler "beating degree testers" and a Green "freeness tester." It was found that the values of the 2 Schopper-Riegler testers did not tally. To obtain check results when operating one and the same Schopper-Riegler app., it is necessary to standardize the technic. Systematic tests have been made concerning the effect of different pressing processes and of the uniformity of the sheets in regard to weight, breaking length, etc. It was found that the breaking length of paper from different sections of the same sheet varies owing to the impossibility of obtaining uniform distribution of fibers in all portions of the papers. If the no. of fibers lying lengthways is greater at a certain place in a sheet of paper than at another, then the sheet is stronger in that one direction. Duplex sheets are found to be 8% stronger than that of the single sheets. Stirring the pulp in the dezzle-box by means of a perforated plate showed a decided improvement in uniformity in the tensile strength of different parts of the sheet. J. W. Holst.

Some recent developments in bleaching chemical wood pulps. John D. Rue. *Paper Trade J.* 101, No. 18, 87-90(1935). A. Papineau-Couture.

Fungal attack on wet groundwood pulp. W. Schmid. *Papierfabr.* 33 (Tech. Teil), 380-2, 387-9(1935); cf. *C. A.* 29, 4937.—The various sources of primary and secondary infection and methods of inhibiting fungal attack are discussed. Special emphasis is placed on conditions existing in open and closed white water systems. No new exptl. data, but 10 references, most of them to the recent Scandinavian literature are included. Louis E. Wise.

The chemistry of sulfite pulping. XXXII. Production of sulfite pulp with digesting acid containing selenium. Erik Hägglund, C. H. Bäckström, Mladen Karanovic, Lars Runquist and Olaf Vincent. *Swensk Pappers-Tidn.*

38, 650-66, 695-701(1935); cf. C. A. 28, 1528<sup>4</sup>.—When sulfite digesting acid is heated alone its stability rises with the increasing base content. The formation of  $H_2SO_4$  decreases with increasing base content. This applies to pure digesting acid as well as to that containing Se. This is probably due to the fact that the change of thiosulfate to pentasulfate in the formation of sulfate decreases with the concentration of the  $H$  ions. In the production of sulfite pulp with chemically pure digesting acid either with or without the presence of Se it has turned out that the limit of injurious Se concentration is distinctly higher than has been thought. A Se content of 0.75 mg per l. does not diminish the strength of the pulp. Only when the Se concentration rises to 1 or 2 mg per l. does any evident impairing of pulp yield appear. When the digesting acid contains other substances besides Se which can occur in fluctuating amounts in the cooking acid the permissible limit of Se content decreases materially.

Standardization of the method for determining the whiteness of sulfite pulp. A. M. Katz. *Bumazhnyaya Prom.* 14, No. 7, 33-8(1935).—The Kondratik differential photometer is better suited for the determination of the whiteness of sulfite pulp than are the Ostwald and Puffinrich photometers.

Paulson process for spent sulfite liquor utilization. Sidney D. Wells. *Paper Trade J.* 101, No. 19, 40-2(1935). *Paper Ind.* 17, 659-60(1935).—A progress report on the Paulson process of evap. and burning sulfite waste liquor (U. S. pat. 1,393,241, C. A. 16, 495). A heat balance is given. The introduction of indirect heating of the liquor and the use of screw presses to remove liquor from the pulp from the blow pits are the most recent refinements to the process.

Sulfate pulp quality control—sampling and testing of individual digester cooks. Carl I. Ahlstrom. *Paper Trade J.* 101, No. 21, 37-8(1935).—A description of the procedure developed at the Longview, Wash., mill of Longview Fibre Co. for sampling each cook individually as it is being blown to the diffusers and for the rapid determination of the bleachability (degree of cooking) by Wiles'  $KMnO_4$  method (C. A. 28, 2896<sup>5</sup>) in order to control the cooking operation at reasonably short notice. By the use of this procedure, modification of the cooking conditions to obtain a desired change in pulp quality is effected in a much shorter period than previously.

Pulp from sawdust. I. A. Il'inski. *Bumazhnyaya Prom.* 14, No. 8, 86-90(1935).—Spruce and pine sawdusts cooked, with stirring, with 16° Bé sulfate liquor at 8 atm. for 4 hrs. and then bleached with a consumption of 3% of active Cl gave 35-2% of pulp suitable for the production of good grades of paper.

Use of waste chips resulting from alkaline extraction of rosin in paper production. P. D. Zotov. *Bumazhnyaya Prom.* 14, No. 9, 45-54(1935).—Preliminary lab. expts. showed that the waste chips obtained from the extraction of rosin with NaOH can be used in the production of sulfate and mech. pulp comparable in quality to regular stocks.

Chemical composition of flax chaff. B. P. Osnov and A. G. Suova. *Bumazhnyaya Prom.* 14, No. 7, 72-3(1935).—Four samples of flax chaff were sifted through a nest of 4 sieves of different mesh, giving 2-18.8% bast fiber, 21-4-62.6% coarse and 21.1-60.7% fine chaff and 5.5-23.1% dust. The samples and their fractions were analyzed and cooked. The coarse and fine fractions of chaff are of similar composition. The bast fraction contains the greatest percentage of cellulose. The greater part of the dust consists of inorganic matter. To obtain uniform pulp, it is necessary to remove the dust from the chaff and to separate the bast fibers.

Belt conveyors prove efficient means of handling black ash in Pacific Coast pulp mill recovery room. D. L. Shurley. *Paper Trade J.* 101, No. 18, 34-5(1935).—A description of the Sandvik steel conveyor-belt system recently installed at the St. Helens Pulp & Paper Co., St. Helens, Ore.

Forced circulation in pulping digesters. S. A. Fotiev. *Bumazhnyaya Prom.* 14, No. 10, 7-21(1935).—A discussion

of various forms of forced circulation and types of digesters used in sulfite pulping.

The centrifugal (pulp) screen. Egon Strobach. *Wochbl. Papierfabr.* 66 (Tech. Teil) 852-4(1935).—A detailed description of the "Erfkensator" without new experimental data.

United States patents on paper making. Third quarter, 1935. Clarence J. West. *Paper Trade J.* 101, No. 17, 39-41(1935); cf. C. A. 29, 7649<sup>4</sup>.—A list in numerical sequence of patents relating to pulp and paper making issued at Washington during July, Aug. and Sept., 1935.

Utilization of *Pinus insignis* in paper making. L. Vidal, M. Brot and M. Aribert. *Bull. inst. pap.* 1935, 181-5.—See C. A. 29, 7650<sup>4</sup>.

Production of fine papers from cotton waste and flax-hemp stuffings. A. Ya. Goncharov. *Bumazhnyaya Prom.* 14, No. 7, 66-71(1935). cf. C. A. 29, 339<sup>4</sup>.—A good grade of pulp, suitable for reworking into copying and cigarette paper, was obtained from various cotton waste and flax-hemp screenings by cooking with 18% NaOH + Na<sub>2</sub>S at 3-5 atm. pressure for 12 hrs. and subsequent 2-stage bleaching with 5-6% Ca(ClO)<sub>2</sub>.

The optical characteristics of paper. II. A precision opacimeter. F. A. Steele. *Paper Trade J.* 101, No. 17, 31-5(1935). cf. C. A. 29, 3832<sup>4</sup>.—A description is given of the technical details of the design, construction and calibration of the instrument used for measuring paper opacity that was used for establishing the previously reported mathematical relationships in regard to the optical characteristics of paper. The sample is placed over an opening in the top of a sphere constructed of thin Al. and is diffusely illuminated by the walls of the sphere, which in turn are illuminated by means of elec. bulbs mounted inside the sphere, the bulbs being shielded so that they do not shine directly on the sample. Light reflected from the sample passes down through a vertical collimator tube, at the bottom of which is a sensitive photoelectric cell. Provision is made for inserting color filters above the cell if desired. The current generated in the cell is measured by means of a suitable galvanometer set up. In measuring the opacity of paper, the reflectance of the sample is determined when covered with a black and with a white background, resp., so that the problem of measuring opacity is essentially one of measuring reflectance.

Sulfur dyestuffs in paper making. Maurice Dérénb. *Papeterie* 57, 870-4(1935).—A brief outline of the preparation of S dyestuffs and of the manner of using them in the coloring of paper.

Factors of importance in the drying of paper. F. C. Clark. *Paper Trade J.* 101, No. 10, 33-5(1935).—The importance of preventing overdrying of paper at any stage in its manufacture and the importance and difficulty of maintaining the correct moisture content are discussed.

Study of the drying of paper and of the effect of hot-air blasts. Paul Vidal. *Papeterie* 57, 829-33, 874-81, 918-26(1935).—A mathematical discussion, based on observations on 2 paper machines 2 m. wide and running at 60 to 100 m. per min. on ordinary and medium grades, showed that the advantages and disadvantages of ventilation by means of hot air blasts just about balance.

A forward step in the control of paper drying. A. H. Stanton. *Paper Trade J.* 101, No. 18, 62, 64(1935).—A description of the principle and advantages of the "Tenso-Temp" system in which the steam-admission valve supplying steam to the driers is operated by a single air-controlled system under the joint action of a tension-control unit and a temp. control unit.

Effect of drier drainage on the drying of paper and its control. H. G. Cram. *Paper Trade J.* 101, No. 17, 36-9(1935).

Presence of lumps in paper. D. C. M. Pappay. *Papeterie* 57, 834, 837(1935).—Presence of fiber lumps in the best prepd. of fine papers is due to agglomeration and adhesion of fibers caused by agitation in the chests or by rolling together of the fibers by friction in passing through

pipes. It is suggested that the trouble can be appreciably reduced by diminishing as far as possible unnecessary agitation and formation of eddies, and more particularly by dropping the beaters singly into the chests, or at most 2 at a time.

A Papineau-Couture  
New physicochemical method of determining sizing [in paper] I I Koralevskii *Pulp Paper Can.* 36, 546-51 (1935).—See C. A. 29, 2333.

A. P. C.  
Progress in the production of water-resistant bitumen papers Friedrich Huth *Wochl. Papierfabr.* 66 (Tech. Teil) 887-8 (1935).—Deals largely with the application of asphalt emulsions used in impregnating paper.

Louis C. Wise  
Consumption of sulfuric acid in parchmentization of paper N A Laskovich *Bumashnaya Prom.* 14, No. 7, 14-25 (1935).—A considerable economy in the consumption of  $H_2SO_4$  can be effected by parchmentizing paper with 42.5-47%  $H_2SO_4$  (65.5-65.9° B $\phi$ ) and passing the paper through 2 wash baths. The waste acid should be directly coned to 31-4° B $\phi$ .

Chas. Blanc  
New methods for measuring the waterproofness or impermeability of paper Maurice Dénéré *Papeterie* 57, 966-70 (1935).—J. Grant's method (C. A. 29, 1633) has been modified to render it quicker for papers that exhibit high impermeability or resistance to water. The sample is placed over a small jar provided with a screw top which holds the sample in place and provided with a central opening for placing the indicator on the paper. The bottom of the jar is provided with a side tube connected with a vertical graduated tube, so that by using a suitable liquid (e. g., Hg in the tube and bottom of the jar and water or any desired soln. in the top of the jar), the test can be carried out under a suitably hydrostatic head and thus accelerated. In the case of the original Grant test, "creeping" of the water or soln. over the edges of the paper can be prevented or minimized by dipping the latter over a width of 3-5 mm. in molten paraffin.

A Papineau-Couture  
Determination of water vapor permeability of cellulose wrapping materials W. H. Charch and A. G. Scroggie. *Paper Trade J.* 101, No. 14, 31-9 (1935).—A detailed description is given of a test that has been found satisfactory for determining the permeability to water vapor of the most highly moistureproof wrappers, such as moistureproof Cellophane. It is based on the exposure of a small dish containing  $H_2O$  and sealed tightly with the membrane to be tested, to a practically dry atm. for a period of 24 to 30 hrs. at const. temp. The dish is weighed before and after this exposure and the loss in wt. is calculated to a standard basis for comparison. It is suggested that the method should give even more accurate results when used with wrappers exhibiting a high rate of moisture transmission. The factors influencing its accuracy are discussed, and a special thermostat is described which has a capacity for over 500 test dishes, will hold a given temp. const. within  $\pm 0.2^\circ$  at any one point, and will maintain an atm. of less than 2% relative humidity over periods of days.

A Papineau-Couture  
Can the coated paper industry advantageously replace casein by other adhesives? Fritz Hoyer *Gelatine, Leim, Klebstoffe* 3, 174-6 (1935).—The German casein industry is not in position to meet the demands of the coated paper industry. Of the possible substitutes starch can be obtained domestically but it has disadvantages (lack of water resistance and difficulties in operating technique) that limit its use. Animal glues have been used but the domestic industry is not self-supporting.

D. B.  
Modernized coating equipment Frank W. Egan *Paper Trade J.* 101, No. 18, 90-1 (1935).—An outline of recent developments in paper coating equipment.

A Papineau-Couture  
Improvements in the Fourdrinier machine Karl Bachmann *Papierfabr.* 33 (Tech. Teil) 361-5 (1935).—Mech. innovations in the wet end of the paper machine are fully described and illustrated. These are covered by German patents 606,174 (C. A. 29, 1633<sup>a</sup>); 583,291 (C. A. 28, 324<sup>a</sup>); 583,034 (C. A. 28, 323<sup>a</sup>); 585,508 (C. A. 29, 1537<sup>a</sup>) and 585,525.

Louis E. Wise  
Degree of polymerization of cellulose and its application in characterizing pulps August Noll. *Papierfabr.* 33 (Tech. Teil) 377-80 (1935).—Using the technique and applying the definitions of Staudinger and his coworkers (cf. C. A. 29, 6753<sup>a</sup>) N. assumes that the sp. viscosity of a cellulose sol is a function of the length of the thread-like cellulose mol. (i. e., the mol. wt.), and that the degree of polymerization (I) is the mol. wt./162. The  $\alpha$ -cellulose content, alkali-sol. matter, Cu no., viscosity of xanthate and cuprammonium sols, "mol. wts." and I, were determined in the case of 20 pulps. The following list gives, resp., the source of the pulp, the no. of samples examined (in parentheses), and the ranges for I in each type of pulp: aspen (2) 275, beech (2) 340-410, spruce (11) 295-625, pine (1) 295, cotton linters (3) 350-445; cotton wadding (1) 500. On beating an individual pulp in a *Jokro* mill for 2 hrs., from 14<sup>a</sup> Schopper Riegler to 94<sup>a</sup> Schopper Riegler, no appreciable change in I occurred, although the initial tearing length of 1800 mm. had increased to 8640 mm. This is evidence that even protracted beating does not rupture the macromols. The relationships between xanthate viscosities and I, and between cuprammonium viscosities and I for all pulps examined are shown graphically. These curves are similar, and show that in control tests nothing is gained by detg. I of a sp. pulp sample. Viscosity detns. are sufficient. There is no obvious relationship between I of a pulp and its Cu no. or  $\alpha$ -cellulose content.

Louis C. Wise  
Innovations in paper-machine construction Wintermeyer. *Wochl. Papierfabr.* 66 (Tech. Teil) 806-9, 829-31 (1935).—An address.

Louis E. Wise  
The autonomy electrode in paper making Maurice Dénéré *Papeterie* 57, 933-41 (1935).—An explanation of the principles of the Sb electrode and of the technique of its use in the measurement of pH.

A Papineau-Couture  
Stock control apparatus Geo W. Dodge. *Paper Trade J.* 101, No. 18, 70-1 (1935).—A brief description of the Precision Regulator and the Precision Regulating and Metering Device, manu'd by Paper & Industrial Appliances, Inc., New York.

A Papineau-Couture  
"Stainless" (steel) in the paper industry. J. C. C. Holding. *Paper Trade J.* 101, No. 18, 65 (1935).—Though there is no known commercially practicable metal or alloy that will stand up forever under the severe chem. punishment to which most paper mill equipment is subjected, various stainless steels have amply demonstrated sufficient superiority over ordinary C steel and other alloys to justify their adoption.

A Papineau-Couture  
Refuse disposal at the New Brunswick International Paper Co., Dalhousie, N. B. F. L. Allen. *Pulp Paper Can.* 36, 555-9 (1935).—Descriptive.

A. P. C.  
Refuse disposal. W. T. Bennett. *Pulp Paper Can.* 36, 489-90 (1935).—A description of the wood refuse-disposal plant at the mill of Can. International Paper Co., Gatineau, Que.

A Papineau-Couture  
Experiments in printing P. H. Prior. *Paper Trade J.* 101, No. 15, 39-44 (1935).—See C. A. 29, 4570<sup>a</sup>.

A Papineau-Couture  
Fluorescence of colored papers Maurice Dénéré *Papeterie* 57, 797-800 (1935).—A table is given showing the fluorescence of a no. of com. dyestuffs on paper (bleached and unbleached sulfite, with or without admixt. of ground-wood) when exposed to a Hg arc lamp. The fluorescences of the various dyes are not as bright when fixed on paper as on other usual fibers (rayon, cotton, wool, silk). Interesting distinctions can be made in the case of yellow, orange and red papers, but not with the other colors. Dyestuffs in mixts. can be identified in spite of the fact that they frequently are partially masked by one another.

A Papineau-Couture  
The tanning of gill licks of the bronze-powder type when in contact with paper. O. J. Schierholtz. *Pulp Paper Can.* 36, 609-13 (1935).—Acidity in paper originating in the hydrolysis of papermakers' alum is shown to be a primary cause of the tanning of gill gums which are frequently used to decorate wallpapers, etc. Treatment of the paper, prior to gilding, with a soln. of alk. earth bicarbonates prevents tanning due to alum or other

tarnish-producing constituents normally present in com. papers. An effective alternative method is to apply an alk. earth hydroxide soln. followed immediately by a soln. of  $\text{CO}_2$  in water. During tarnishing of gilt papers a gas (probably  $\text{SO}_2$ ) is produced which itself has a tarnishing effect on gilt. Comparatively few types of com. papers are free from tarnish-producing ingredients. The tarnishing of gilt gum is an extremely delicate test for  $\text{SO}_2$ , being much more sensitive than the usual qualitative reagents.

**A. Papineau-Couture**  
**Suffness testing of paper and cardboard.** Kurt Berndt *Papierfabr* 33 (Tech. Teil) 397-8 (1935).—A new instrument is described, which measures the force in g. required to bend (i. e., to cause the deflection of) a clamped sample strip (15 mm. wide and of any convenient length up to 250 mm.) to any definite convenient angle (usually  $5-20^\circ$ ). The results of stiffness tests with a no. of board samples are shown graphically. The advantages claimed for the new tester are: (1) detn. of abs. instead of comparative values, (2) paper as well as heavy board may be tested without modification in the instrument, (3) the latter is simple in construction, and is easily and rapidly regulated and operated, (4) very short as well as long sample strips may be used.

**Making fiberboard elastic and resistant to water.** I. Ettingof and E. Kormer. *Bumashnaya Prom.* 14, No. 7, 54-8 (1935), cf. Ettingof, *Iskusstvennaya Kosha* Nos. 7-8 (1933), Nos. 11-12 (1934).—Good elasticity and water-resistance were imparted to fiberboard by treating it with petrolatum jelly and Ca, Mg, Al, Zn and Mn naphthenates.

**Notes on the testing of corrugated cardboard.** L. H. M. et Argy. *Pulp Paper Can.* 36, 607-8 (1935).—See C. A. 29, 3834.

**Boxboard.** W. Höppler. *Wochbl. Papierfabr* 66 (Tech. Teil) 850-2 (1935).—No new data are given but the mfg. processes and properties of the finished product are outlined.

**Starch [and its use in the paper industry] (Holt) 13**  
 Treatment of boiler feed water and water used in the [paper] plant (List) 14. Lignin and related compds. (Brauns, Hibbert) 10. System  $\text{CaO-SO}_2\text{-H}_2\text{O}$  [delignification of wood] (Gishler, Maass) 2. App. for sifting or straining materials suspended in liquid, such as wood pulp (Brit. pat. 434,069) 1. App. for testing the permeability of porous materials such as paper (U. S. pat. 2,021,048) 1. Yeast [sulfite waste lye as nutrient in yeast manuf.] (Ger. pat. 617,780) 16. Viscous compn. [for waterproofing paper] (Fr. pat. 786,553) 13.

**Cellulose.** British Celanese Ltd., Walter H. Groombridge and Eric V. Mellers. Brit. 433,783, Aug. 16, 1935. Cellulose suitable for the manuf. of paper or cellulose derivs. is made from wood, straw, grass, etc., by extg. the lignin by soln. in a solvent mixt. comprising at least 20%  $\text{H}_2\text{O}$  and an org. solvent from at least 2 of the following classes: monohydric alcs. of the fatty series, simple aliphatic ketones; cyclic diethers; aromatic hydrocarbons. Resins may be extd. from the materials with  $\text{CaH}_2$  or  $\text{CaH}_2\text{-alc}$  before the process. In 1 example, wood chips are sprayed with an equal wt. of a mixt. of concd.  $\text{HCl}$  30 and glacial  $\text{AcOH}$  70 parts, are left at atm. temp. for 6 hrs. and then washed and mixed with 8-10 times as much of a mixt. of  $\text{Me}_2\text{CO}$  15,  $\text{H}_2\text{O}$  35 and alc. 50 parts in an autoclave and heated to  $150-200^\circ$  for 2-3 hrs. The solvent is withdrawn, preferably without reduction of pressure and further solvent is introduced. In the 2nd or later stages of the extn. the temp. may be  $210-220^\circ$ .

**Cellulose from lignocellulose materials.** Henry Dreyfus U. S. 2,022,654, Dec. 3. Lignin is extd. from materials such as wood, straw or grass with use of an org. solvent medium such as dioxane, etc., at an elevated temp. (suitably about  $170-200^\circ$ ) under a pressure in excess of that developed by the solvent medium at the temp. employed. Numerous examples are given.

**Cellulose from lignocellulose materials.** Walter H.

Groombridge and Eric V. Mellers (to Celanese Corp. of America). U. S. 2,022,664, Dec. 3. Lignin is extd. from materials such as spruce chips by use of org. solvents such as an  $\text{EtOH}$  soln. contg. inorg. basic substances such as  $\text{NaOH}$  0.5-8.0%.

**Bleaching cellulosic fibers.** La Société des produits peroxydés. Fr. 786,938, Sept. 14, 1935. Fibers such as cotton are bleached by solns. of per. compds. under a pressure of 1.5-4 atm. and at temps. of at least  $80^\circ$ . The temp. may be increased to  $115^\circ$  as the operation progresses. In the case of a preliminary boiling, a bleaching bath is used to which no substance of alk. reaction need be added or a bath is used contg. no  $\text{NaOH}$  but to which Na silicate, an alkali ortho- or pyro-phosphate or alkali oxalate is added. Cf. C. A. 29, 3534.

**Cellulose esters.** Deutsche Kunstseiden-Studenges. m. B. H. Ger. 620,300, Oct. 18, 1935 (Cl. 12a, 0). A cellulose ester of a lower aliphatic acid, after sepn. from the reaction mixt. in which it was prepd., is treated in a solvent with a halogen compd. of P, e. g.,  $\text{POCl}_3$ ,  $\text{PCl}_5$  or  $\text{PCl}_3$ . The temp. during this treatment must not exceed  $50^\circ$ , and the proportion of P compd. must not exceed about 3%, calcd. on the ester. A small proportion of an org. base may be added. Esters of high viscosity, which retain their soly in acetone, are obtained. An example is given of the treatment of cellulose acetate.

**Cellulose organic acid esters.** Wilhelm Walter and Rudolf Hofmann (to I. G. Farbenind. A.-G.). U. S. 2,022,446, Nov. 26. In prep. an ester such as cellulose acetate, the  $\text{H}_2\text{SO}_4$  catalyst in a crude ester soln. such as obtained from esterifying cellulose with an aliphatic acid anhydride such as  $\text{Ac}_2\text{O}$  in the presence of  $\text{H}_2\text{SO}_4$  and with addn. of a solvent (such as  $\text{HOAc}$  and ethylene chloride) for the ester formed is neutralized (suitably with  $\text{NaOAc}$ ) and water is added in such quantity that the aliphatic acid present is dild. to an aq. soln. of about 50-80% strength, and the reaction mass is heated to about  $60-100^\circ$  for 1-3 hrs. to effect stabilization.

**Cellulose mixed ester compositions for sheets, films, etc.** Hans T. Clarke and Carl J. Malm (to Eastman Kodak Co.) U. S. 2,021,887, Nov. 26. A mixed org. ester of cellulose such as cellulose acetate-propionate or -butyrate is used with an amt. of a triaryl phosphate such as triphenyl phosphate sufficient to increase the flexibility of a sheet or film substantially beyond the inherent flexibility of an unplasticized sheet or film of the mixed ester, without substantial impairment of transparency.

**Cellulose ester sheets.** Kodak Ltd. Brit. 434,265, Aug. 28, 1935. Cellulose ester sheets are superficially saponified on 1 or both faces and are subjected to a depolishing treatment to render them capable of carrying impressions, writings, designs and tracings. The saponification may be caustic alkali,  $\text{NH}_4\text{OH}$  or an amine. For depolishing, a swelling agent, e. g.,  $\text{EtOH}$ , a mixt. of alc. and  $\text{Me}_2\text{CO}$ , is applied to the sheet which may then be pressed into contact with a roughened cylinder, which may be heated. Resin or wax may be added to the saponified liquid to avoid any tendency of ink to smudge when applied to the finished sheet.

**Cellulose acetate.** Clifford I. Haney (to Celanese Corp. of America) U. S. 2,022,856, Dec. 3. For producing cellulose acetate of relatively high-viscosity characteristics, a cellulose of relatively low-viscosity characteristics, having a viscosity of 25-100 centipoise, is treated with an acetylating material comprising  $\text{H}_2\text{SO}_4$ ,  $\text{Ac}_2\text{O}$  and an amt. of  $\text{HOAc}$  2.0-2.5 times the wt. of the cellulose, under such conditions as to produce a cellulose acetate which when hydrolyzed to acetone soly. has a viscosity of over 10. Various examples are given. Cf. C. A. 29, 3167.

**Cellulose derivative compositions suitable for films, filaments, lacquers, etc.** Henry B. Smith (to Eastman Kodak Co.). U. S. 2,021,901, Nov. 26. An org. cellulose deriv. such as cellulose acetate is used with tributyl phosphate as a plasticizer. U. S. 2,021,902 relates to the use of tributyl phosphate with cellulose org. esters such as cellulose acetate-propionate.

**Artificial filaments, etc.** British Celanese Ltd. and Percy F. C. Sowter. Brit. 434,426, Sept. 2, 1935. Cellu-

lose acetate soln. is extruded into a coagulating bath contg at least 40% Me<sub>2</sub>CO and which may have as non-solvent H<sub>2</sub>O, MeOH or EtOH and may contain salts, sugars, mannitol, sorbitol,  $\alpha$ -cellulose and other substances which reduce the soly of the solvent in the non-solvent. Cf C. A. 30, 614<sup>1</sup>

Artificial filaments, etc., of cellulose derivatives such as cellulose acetate. Henry Dreyfus U. S. 2,022,411, Nov. 26. Artificial filaments, ribbons, etc., are formed of an org. deriv. of cellulose such as cellulose acetate, formate or benzyl cellulose with which about 1-15% of an alkyl borate such as triethyl borate is incorporated.

Dry spinning artificial filaments from material such as cellulose acetate. Ettore Viviani (to Ruth-Aldo Co.). U. S. 2,022,260, Nov. 26. Various structural and operative details of a spinning cell are described.

Artificial threads from cellulose acetate or the like. Hans Altwegg and Armin Eichler (to DuPont Rayon Co.). U. S. 2,022,838, Dec. 3. A thread of normal luster is formed by the dry spinning of a soln. of the cellulose deriv. contg. a normally nonvolatile org. acid or anhydride such as stearic acid and the luster is subsequently reduced by treating the thread with a substance such as BaCl<sub>2</sub> in soln. which reacts with the acid or anhydride to form a substantially insol. compd. Cf C. A. 29, 1984<sup>1</sup>

Fabrics containing cellulose acetate. Camille Dreyfus U. S. 2,022,410, Nov. 26. Filaments or the like are formed by extrusion of a soln. of cellulose acetate into a setting medium, and the material is withdrawn from this medium while still contg. at least 0.1% of volatile solvent, and in such condition formed into a fabric such as a knitted fabric (suitably while contg. about 0.1-4.0% of a solvent such as acetone).

Viscose fibers. Deutsche Hydrierwerke A.-G. (Walther Schrauth and Richard Hueter, inventors). Ger. 617,851, Sept. 5, 1935 (Cl. 295 3 02). The viscose is spun in a bath contg. the water-sol. reaction products of H<sub>2</sub>SO<sub>4</sub> and high-mol. said or unsatd. aliphatic alcs with at least 10 C atoms, and, optionally, pptg. acids or salts. Higher alcs. such as cyclohexanol or other fat solvents may be added to the bath. In an example, viscose is spun in a bath comprising an aq. soln. of H<sub>2</sub>SO<sub>4</sub> and sulfonated oleyl alc.

Rayon. Edward G. Budd Manufacturing Co. Fr. 786,561, Sept. 5, 1935. App. for making filaments of high denier no. and high resistance.

Modified rayon. I. G. Farbenindustrie A.-G. Fr. 786,659, Sept. 7, 1935. Albuminous substances such as casein or albumin along with substances which fix the albuminous substances or give condensation products therewith, such as choleic acid, formamide or acetamide are incorporated in the spinning soln.

Device for receiving freshly spun rayon. Feldmuehle A.-G. vorm. Loeb, Schoenfeld & Co., Rorschach. Ger. 617,791, Aug. 20, 1935 (Cl. 29a. 6 13).

Spinning nozzle for rayon. Werkstätten für Präzisionsmechanik G. m. b. H. Ger. 617,790, Aug. 26, 1935 (Cl. 29a 6 10); Fr. 786,665, Sept. 7, 1935

Pot-spinning machines for rayon. Paul H. Minck. Ger. 620,091, Oct. 15, 1935 (Cl. 29a 6 06). Means is described for recommencing spinning after a pot has been changed. Cf C. A. 29, 927<sup>1</sup>

Treating cellulose material such as wood pulp, paper or cotton linters. Alma Dobry (to Compagnie de produits chimiques et électrométallurgiques Alais, Forges et Camargue). U. S. 2,022,589, Nov. 26. Cellulose material is treated with a concd. soln. of a perchlorate such as a satd. soln. of Be or Al perchlorate and with a liquid such as water adapted to effect the sepn. of the cellulose from the perchlorate soln. by pptn.

Cellulose pulp from wood. Clinton K. Textor (to Northwest Paper Co.). U. S. 2,022,872, Dec. 3. For producing a substantially lignin-free cellulose, wood is digested with a soln. contg. alkali metal salts of sulfurous and carbonic acids as the 2 essential chem. ingredients, sufficient sulfite being used, in excess of the amt. required to release the cellulose, to prevent staining. U. S. 2,022,873 relates to a process in which so-called hardwood

or short-fibered wood is digested, until cellulose is released, with an alk. Na<sub>2</sub>SO<sub>3</sub> liquor, contg. sufficient excess of Na<sub>2</sub>SO<sub>3</sub> to prevent staining and sufficient of a Na carbonate to give a pH of 7.5-9.5, at a temp. of 175-185° and at the corresponding satd. pressure for 3-7 hrs., of which the first 1-2 hrs. are used in attaining the specified temp. and pressure. Numerous details are given.

Cellulose digester. Friedrich Schulte. Austrian 142,580, Aug. 10, 1935 (Cl. 55c). Correction of patent no. 2 (see C. A. 29, 8311<sup>1</sup>).

Cellulose digester. Jens W. A. Elling. Ger. 616,156, July 20, 1935 (Cl. 555 2 01). Addn. to 689,751 (C. A. 28, 2530<sup>1</sup>). A treble digester for fibrous materials is described.

Sulfite pulp digester. Paul B. Lacy, Edwin F. Libby and Ralph V. Metz (to Industrial Chemical Sales Co.). U. S. 2,022,378, Nov. 26. A steel shell carries a firmly adherent, impervious, sprayed lining of a corrosion resisting metal such as Cd, monel metal, stainless steel, Sn, Pb or bronze.

Pulps containing hard binder substances and suitable for forming sheets on a paper machine. Harry C. Fisher (to Richardson Co.). U. S. 2,022,311, Nov. 26. A substantially homogeneous plastic mass is formed of a binder such as gilsonite or asphalt and fibrous material in which the fibers are included within the body of the binder; more fibrous material and water are then added and the product is beaten to form a pulp so that the product contains coated and uncoated fibers.

Sulfite solutions. Siemens Lurgi-Cottrell Elektrofilter-Ges. m. b. H. für Forschung und Patentverwertung (Friedrich Wilhelm Hoss, inventor). Ger. 620,401, Oct. 21, 1935 (Cl. 12a 21). In the manuf. of sulfite solns., particularly for cellulose manuf., from crude gases contg. SO<sub>2</sub> and impurities such as As and Se, the gases are purified by passing them in direct succession first through a washing tower and then through an electrofilter constructed as an indirect cooler.

Forming webs such as those of felted fibers and impregnating them with latex, etc. Izador J. Novak (to Raybestos-Manhattan, Inc.). U. S. 2,022,687, Dec. 3. Various details of app. and operation are described.

Utilizing waste wood-pulp liquor for combustion in a boiler furnace with recovery of inorganic constituents. Josiah H. Rohrer (to Badeniahausen Corp.). U. S. 2,023,031, Dec. 3. Various details of app. and operation.

Paper. Paul Lindner and Hugo Rumppe. Ger. 617,684, Aug. 23, 1935 (Cl. 55c 11 10). App. for sifting, washing and disintegrating vegetable-fiber waste, especially old paper, is described.

Paper making apparatus. Maurice O. Neilson U. S. 2,022,298, Nov. 26. Structural and mech. features.

Felts of paper-making machines. Tom Milnes and Ayres Ltd. Brit. 434,521, Sept. 3, 1935.

Apparatus for straining fibrous suspensions for making paper, etc. Erich Winkler and Franz Schönemann. Ger. 620,278, Oct. 17, 1935 (Cl. 55d 2 10).

Drying machines for paper. Samuel Milne. Brit. 433,544, Aug. 15, 1935. The felts of a paper-making machine are dried by steam-heated cylinders to a low moisture content and raised to a higher temp. than that of the paper web passing about drying-cylinders.

Hollander. Aldo Galliani. Austrian 143,159, Oct. 25, 1935 (Cl. 55a).

Treating card or paper stock for use in duplicating processes. Raymond S. Jones U. S. 2,021,938, Nov. 26. The material is superficially treated with a compn. (such as specified glycerol mixts.) which imparts hygroscopicity to the faces only of the material.

Waterproof printing or writing paper. Heinrich Kraut and Hermann Burger. Ger. 615,348, Nov. 1, 1935 (Cl. 55f 15 20). A mixt. of spermaceti and a resin, e. g. colophony, is used as the waterproofing agent.

Apparatus suitable for coating paper or cloth by friction calendaring. Geo. E. Pelton U. S. 2,022,322, Nov. 26. Structural, mech. and operative details.

Coated parchmentized paper suitable for use as an

imitation leather, etc. Ervin E. Strawn (to Paterson Parchment Paper Co.), U. S. 2,027,711, Dec. 10. One surface of a porous cellulosic material such as waterleaf paper is provided with a very thin coating of material such as "film scrap liquor" which is not affected by chemicals used for parchmentizing and which is anchored in the pores of the material underneath and the latter is then parchmentized as by treatment with  $H_2SO_4$  to form a firmly bonded product.

Cigarette wrappers Nathan Sulzberger. Brit. 433,228, Aug. 12, 1935. The wrapper is impregnated with a dil. soln. of a  $H_2O$ -resisting cellulosic material, e. g., cellulose esters or ethers, collodion, celluloid, pyroxylin, cellulose acetate or nitrocellulose in  $Et_2O$ , alc.,  $Me_2CO$  or other solvent, that will evaporate readily without leaving a perceptible

odor or flavor and without forming a surface film or coating. The soln. may contain coloring matter, a "flattening" agent, e. g., paraffin, plasticizers, e. g., di-Bu or di-Am phthalate, medicinal and aromatic substances, e. g., menthol, oil of cloves, tobacco ext.

Treatment of effluents of paper manufacture, etc. John Dickinson & Co. Ltd., Julius Grant, Dorr-Oliver Co. Ltd., Robert I. Stewart and Philip Evans. Brit. 434,225, Aug. 28, 1935. Paper mill and like wastes containing or other washings having high  $O$ -absorbing characteristics are mixed with effluents from other parts of the paper mill, etc., which contain oxidizing agents or substances capable of forming oxidizing agents, e. g., effluents or sludges from causticizing, or bleaching plant, to form a harmless effluent for direct discharge into a river. App. is described.

## 24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE AND C. G. STORM

Deflagration of explosive substances by ultrasonic waves Neda Marinescu. *Compt. rend.* 201, 1187-9 (1935).—Calc. shows that the temp. of a small air bubble in a liquid subjected to ultrasonic waves by means of a piezo-quartz oscillator operating under a p. d. of 10,000 v at a frequency of  $10^4$  would be raised adiabatically to about  $234^\circ$ . This is confirmed by the fact that certain explosives which deflagrate at a temp. below that, e. g.,  $NI_3$ , Ag fulminate and some peroxide mixts., when placed in a liquid that does not wet them, i. e., so as to be surrounded by air, deflagrate when the liquid is subjected to such waves, or in some cases, e. g.,  $NI_3$ , without intervention of any liquid. The temp. is given by  $T = A(aF)^{1/3}$ , where  $a$  is the amplitude,  $F$  the frequency of the waves, and  $A$  a const. No increase in  $a$  or  $F$  sufficed to explode any nitro deriv.

C. A. Silbrade. Liberation of nitrous oxide during the decomposition of nitrocelluloses Robert Vandoni. *Compt. rend.* 201, 674-5 (1935).—The gaseous products of decompn were detd. for the following: (1) gun cotton heated at  $108.5^\circ$ ; (2) a fiber nitrated to 12% N heated at  $60^\circ$  with 25%  $HNO_3$ ; (3) a fiber nitrated to 14% N heated in a sealed tube at  $130^\circ$  with  $H_2O$ .

	CO%	NO%	N <sub>2</sub> O%	CO <sub>2</sub> %	CH <sub>4</sub> %	H <sub>2</sub> %	N <sub>2</sub> %
(1)	63	61	1	89	10	16	1
(2)	42	13	32	23	15	39	5
(3)	64	43	1	05	26	57	2

Rachel Brown

Brisance and its determination A Majrich and I. Sorm. *Z. ges. Schiess- Sprengstoffw.* 30, 295-9, 337-40 (1935); cf. *C. A.* 28, 3901\*, 5085\*, 29, 4585\*, 4941\*, 5271\*.—The brisances of various explosives were compared by means of a fragmentation test. The explosives (1-2.6 g.) were compressed at 2000 kg./sq. cm. to approx. equal heights in steel cylinders 40 mm. high  $\times$  25 mm. diam., with central cavity 30 mm. deep  $\times$  6 mm. diam., and fired with a detonator consisting of 0.1 g.  $PbN_3$  and 0.1 g. pentaerythritol tetranitrate pressed at 500 kg./sq. cm. in a Cu shell  $25 \times 6$  mm. The fragments of the steel cylinder were collected, screened, counted and weighed. The results were compared with those obtained by various other methods proposed for measurement of brisance, the Trauzl test, Pb-plate test and compression test. The relative brisances of various explosives vary according to the method used in detg. this factor. Each method has its definite limitations. Among the various formulas proposed for expressing brisance, that of Kast (*C. A.* 15, 1217) is favored.

C. G. Storm. Detonating fuse without metal covering Anon. *Z. ges. Schiess- Sprengstoffw.* 30, 333-4 (1935).—A detonating fuse manuf. at the Bofors (Sweden) plant of the Nobel Co. contains a core of pentaerythritol tetranitrate with a covering of cotton tape and cord impregnated with gum. It withstands a water-resistance test of 48 hrs. under 25 m. of water, and is not detonated by either flame or impact.

C. G. Storm

Induction period in gaseous thermal explosions O. K. Rice, Augustine O. Allen and Hallock C. Campbell. *J. Am. Chem. Soc.* 57, 2212-22 (1935).—By integrating the differential equation involving temp. and time in an exothermic gas reaction, a sudden temp. rise, corresponding to an explosion, is noted above a definite pressure. From such curves an approx. method for calcg. the induction period in thermal explosions was devised and applied to explosions of azomethane and ethylazide (*C. A.* 29, 2359\*, 5273\*). Approx. values for the heat of decompn of these compds. were thereby obtained.

C. G. Storm. Explosion regions XXVI. Shape of explosion regions and their diameters I. W. P. Jorissen. *Rec. trav. chim.* 54, 888-92 (1935), cf. *C. A.* 27, 3875.—A mathematical discussion and analysis of explosion limits for combustible gas or vapor diluted by the addn. of a non (or difficultly) combustible gas.

B. E. Anderson. Why compressor explosions A. F. Rieber. *Power* 79, 522-3 (1935).—The main causes of compressor explosions are excessive oil for cylinder lubrication and failure of intake or discharge valves to function properly.

Alden H. Emery. Dust explosions. A. A. Van der Dussen. *Rec. trav. chim.* 54, 873-84 (1935).—See *C. A.* 29, 3162\*.

C. G. Storm. Sensitized explosions I. The hydrogen-oxygen reaction catalyzed by nitrogen peroxide S. G. Iord and R. G. W. Norrish. *Proc. Roy. Soc. (London)* A152, 196-220 (1935).—The region of ignition of H and O which occurs between narrow concn. limits for the catalyst,  $NO_2$ , is bounded by regions of easily measurable slow reaction. Light, which generates O atoms from the  $NO_2$ , increases the velocity of the slow reaction but does not affect the ignition limits. Induction periods occur for both slow and ignition reactions, those for the slow reaction are terminated by a sharp pressure increase, followed by a reaction whose velocity rapidly decreases to a steady value. At const. temp. a curve of these induction periods runs continuously from the region of slow reaction to that of ignition, passing through a min. corresponding to the max. of catalytic effect. Irradiation reduces the induction period from values of 20-200 sec. to less than 1 sec. It is concluded that branching initially exceeds the extinction of chains over the whole catalysis region, that an equil. concn. of reaction chains is ultimately imposed on the slow reaction, that ignition occurs as a thermal effect when the no. of chains exceeds a critical value, and that  $NO_2$  affects the origin, branching and extinction of the chains.

L. E. Steiner. Explosion at Reinsdorf on June 13, 1935 Anon. *Z. ges. Schiess- Sprengstoffw.* 30, 332 (1935).—Fire, followed by several explosions, occurred in a building where TNT residues from spent acid were purified by washing with hot water and soda soln. Flying missiles caused the explosion of a dryhouse 660 m. distant, where the drying of a wet mixt. of nitrocellulose and nitroglycerin for use in the manuf. of smokeless powder was conducted. Another

nearby dryhouse merely burned. The nitroglycerin plant, 200 m distant from the TNT residue plant, also exploded, presumably as a result of damage caused by the first explosion. Other explosions occurred in the TNT nitrating plant. Structural damage to buildings was caused at a distance of 1400 m. Eighty-two were killed and over 800 injured. C. G. Storm

Explosive blasting charges Wm R. Farren and Joseph Smith, Jr. (to Atlas Powder Co.) U. S. 2,023,784, Dec. 10. A charge of blasting explosive is held in a container of flexible and extensible material formed of 2 laminae of creped paper cemented together by an asphaltic compn.

Blasting apparatus John W. Sanford (to Sullivan Machinery Co.) U. S. 2,021,898, Nov. 26. Structural details of app. carrying a blasting charge in the lower end of a loading tube associated with a concentric wash-water tube.

Torpedo for shooting oil wells Alfred E. Ellis U. S. 2,023,831, Dec. 10. An explosive-contg. vessel which may be formed of Sn inner and outer shells is proportioned to float in liquid in the well, in an inclined position adjacent the surface of the liquid.

Toy torpedo. Wm L. Kann U. S. 2,022,770, Dec. 3. A capsule of readily deformable material contains an explosive mixt. and an impact igniter and is held under compression to prevent shifting of the impact igniter.

Shot-gun shells Edwin L. Johnson (to E. I. du Pont de Nemours & Co.) U. S. 2,023,888, Dec. 10. Paper shells are first coated with a cellulose ester compn. such as one of cellulose nitrate or acetate, and the wall of the paper contained between the inner and outer surfaces is then impregnated with a paraffin compn.

Ignition compositions Oswald F. Wyss Brit. 433,117, Aug. 6, 1935. This corresponds to Ger. 595,068 (C. A. 29, 1252).

Semipyrrophoric metals Oswald F. Wyss Brit. 433,174, Aug. 6, 1935. Divided on 433,117 (preceding abstr.). Metals in semipyrrophoric form, i. e., in such state that their ignition temp. is between 50° and 300°, are manufactured by the reduction of metal compds. by thermal decompn. in the presence of inert gas or gases or by chem. reduction with H at raised temp., the temp. and reaction conditions being maintained throughout the reaction intermediate those required to produce nonpyrrophoric metals and those to produce pyrrophoric metals. For chem. reduction, oxides, nitrates, carbonates, sulfates, oxalates, tartrates or citrates may be used, for thermal decompn., oxalates, tartrates or citrates. Alternatively, metal compds. may be reduced so as to produce pyrrophoric metals, the latter being then cooled in a current of H and when cold treated in inert gases, air being excluded. Cf. C. A. 29, 1252.

Gas-detecting miners' lamps Wm M. Thornton and Wm Maurice Brit. 433,570, Aug. 16, 1935

## 25-DYES AND TEXTILE CHEMISTRY

L. A. OLNEY

Analysis of azo dyes with hyposulfite V. I. Minaev and V. N. Kisel'nikov. *Trans. Inst. Chem. Tech. Ivanovo* (U. S. S. R.) 1, 59-65 (1935); cf. C. A. 26, 1543; 27, 4084. The method was applied with good results to the analysis of a no. of azo dyes. Chas. Blanc

The vat dyes of the benzanthrone series XIII. Some 6,6'-derivatives of isovanilanthrone. Toshio Maki and Yoshio Sago *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 560-4 (1935), cf. C. A. 29, 8337. The 6,8,2,1-dichlorobenzanthrone (I) condensed in the PhOH-alc.-KOH melt at 143° mainly to 6,6'-dichloroisovanilanthrone (II) which dyed cotton blue-violet. The Cl atoms in 6- and 8'-positions were completely inert toward KOH and remained unchanged. The yield of pure II (purified with glacial AcOH, hyposulfite and PhCl) was 54.5% of the theoretical. A hyposulfite-insol., PhCl-sol., gray substance also formed was called "B-dye"; it changed to a vat dye when heated with concd. H<sub>2</sub>SO<sub>4</sub>, coloring cotton a dark violet-gray. The yield of "B-dye" was 23.9% of the theoretical. On account of the great stability of the Cl atoms in 6,6'-positions, their methoxylation was difficult. Long and repeated heating with MeOH-KOH and subsequent recryst. gave a small amt. of pure 6,6'-dimethoxyisovanilanthrone, which dyed cotton dark blue in the hyposulfite vat. The Cl atoms of II were hardly hydrolyzed at all in the PhOH-KOH melt at 183°, but at 200° the corresponding 6,6'-dihydroxyisovanilanthrone (III) was formed, which gave an acid-sensitive, blue-violet cotton dye. Boiling III with p-O<sub>2</sub>NCH<sub>2</sub>COCl in PhNO<sub>2</sub> gave the bis(p-nitrobenzoate) which dyed cotton dark blue-violet. The 6,6'-substituents of isovanilanthrone in general exert a moderate bathochromic action. Structural formulas are given. The expts. are described in considerable detail. XIV. Some 8,8'-derivatives of isovanilanthrone. Ibid., 564-7. The 8,8'-derivs. were prep'd in a like manner from 8,8,2,1-dichlorobenzanthrone (IV). The main product of the PhOH-alc.-KOH melt at 143° was 8,8'-dihydroxyisovanilanthrone (V), with a yield of the pure comp'd. (purified with glacial AcOH and PhCl) of about 24% of the theoretical. V gave a red-violet, but somewhat acid-sensitive cotton dye. A considerable amt. of a gray-violet, PhCl-sol., and hyposulfite-sol. dye was also formed, which was likewise called "B-dye." It may have been a mixt. of trihydroxy and chlorodihydroxy compds. Heating with concd. H<sub>2</sub>SO<sub>4</sub>,

changed the "B-dye" to a dark gray-violet vat dye, difficultly sol. in org. solvents. Methoxylation of V gave the corresponding 8,8'-dimethoxy deriv., which gave a rather brilliant red-violet and acid fast dye. With p-O<sub>2</sub>NCH<sub>2</sub>COCl the 8,8'-bis(p-nitrobenzoate) was prep'd from the dihydroxy deriv. The Cl atom of IV in 8-position is so labile that it is easily hydrolyzed by KOH at a relatively low temp. (143°) and exchanged for the OH group, while the Cl atom in 6-position of I is very stable. The 8,8'-derivs. of the isovanilanthrone, in contrast to the 6,6'-derivs. are easily sol. in alk. hyposulfite, even at a relatively low temp. The 8,8'-derivs. exert almost no bathochromic action and give mostly red-violet dyes. Structural formulas are given and the expts. are described in considerable detail.

Karl Kammermeyer

The theory of wool dyeing with particular reference to acid dyes in colloidal solution. F. L. Goodall *J. Soc. Dyers Colourists* 51, 405-14 (1935).—A summary of available information and some theoretical considerations tending to make clear the fundamental difference in behavior in relation to wool, between dyes colloiddally dispersed and those almost completely ionized in soln. Nineteen references.

W. H. Boynton

Dyeing acetate cotton unions, with special reference to mercerized fabrics. R. H. Bourdon. *Silk J.* 12, No. 137, 32-3 (1935).—Various operations are cited to overcome different difficulties in the processing of mercerized cotton and acetate rayon.

Halsey E. Siliman

Silk fabric printing with direct colors. J. M. Hill *Silk J.* 12, No. 137, 33-4 (1935); cf. C. A. 29, 6764.

Halsey E. Siliman

Printed sleeve linings II. N. N. Caruthers. *Textile Jfr.* 61, 469-9 (1935).—Printing formulas and procedure for some special cases are given. While many textbooks advocate for fast color silk effects steaming for 1 hr. at the usual pressure, practical experience has shown that sufficient fixation takes place on hot calendaring to coagulate the albumin and hold the ZnO.

H. E. S.

Printing plush and velours of rayon. F. Weber Schultze. *Rev. gen. mat. color* 39, 385-8 (1935).—Complete formulas for printing are given. Plush and velours are printed in a similar manner. Velours of regenerated cellulose are more fragile than those of acetate. Vat colors are used for acetates and direct colors for viscose.

H. E. S.

Bibliography on the physical testing of textiles (1909 to



1934). Ellery H. Harvey. *Am. Dyestuff Repr.* 24, 703-4, 714-15 (1935).

The determination of the degree of the porosity of (textile) goods. J. Geissler. *Spinner u. Weber* 53, No 50, 1 (1935)—The degree of porosity is a measure of the amt. of air enclosed within the fibers of a fabric and can be detd. from measurements of the wt., thickness and sp. gr. as follows. Place a weighed piece of the fabric (10 X 10 sq cm) between 2 glass plates of known thickness (6 mm) and measure accurately the total thickness. The various textile fabrics have these sp. grs.: cotton 1.47-1.5, flax 1.43-1.46, hemp 1.48, jute 1.44, bleached ramie 1.5, sheep wool 1.32, degummed natural silk 1.37, nitro, Cu and viscose rayons 1.52 and acetate rayon 1.33. For a sample of cotton weighing 4 g., with a thickness of 2 mm and having a sp. gr. of 1.5 the surface was 100 sq cm, the vol. 20 cc., the cotton content, therefore, 4/1.5 = 2.66 cc., the air content 17.34 cc., i. e., degree of porosity 86.70%. Similar examples are given for 3 other textile fabrics.

Leopold Scheffan. Leopold Scheffan. Degumming of real silk materials. A. J. Hall. *Textile Mercury* 93, 303, 307 (1935). cf. Mosher, C. A. 29, 7085.—With Japanese raw silk yarn max degumming was obtained at both pa 1.0-3.0 and 9.0-11.5 at 212°F. However, for max strength and max degumming a pa of 0.5-10.5 was required.

F. H. Moser. Accelerated aging test for weighted silk. Wm. D. Appel and Daniel A. Jessup. *Proc. Am. Assoc. Textile Chem. Colorists* 1935, 350-1, 334-5; *Am. Dyestuff Repr.* 24, 706-7, 710-11 (1935)—Specimens of the cloth suitable for warp and filling breaking strength tests are exposed for 20 hrs. to the radiation from a glass-enclosed C-arc lamp operated on d. c. of approx 13 amp with the voltage across the arc approx 140 v. The distance from the specimens to the center of the arc is 15 in. (38.1 cm). Specimens are enclosed in a cabinet glazed with window glass about 0.32 cm. thick between the specimens and the arc. The air of the cabinet is maintained at 75-77% relative humidity and 63° to 67°. The breaking strength of the cloth after exposure in the direction, warp or filling, showing the greater change in strength is taken to be indicative of the probable relative stability of the fabric with respect to exposure to light, heat and moist air. Relative breaking strengths of accelerated aged silk, a comparison of natural and accelerated aging, and accelerated aging of typical dress fabrics are shown in curves. Also in *J. Research Natl. Bur. Standards* 15, 601-8 (1935) (Research Paper No. 853).

W. H. B. Flax yarn qualities and types. V. S. A. G. Caldwell. *Textile Mfr.* 61, 402-3 (1935). cf. C. A. 29, 5664.—The methods of dealing with yarn test results and the effects of twists in flax yarns are explained.

H. E. S. Flax-yarn qualities. S. A. G. Caldwell. *Textile Mfr.* 61, 443 (1935); cf. preceding abstr.—The apparent densities of Courtra and Irish flax yarns and the effects of twist are described.

Halsey E. Sullivan. The valuation of surface water for the textile industry. H. Bauch. *Melliand Textilber.* 16, 757-60 (1935).—A review.

F. H. Moser. Bleaching procedure. Otto Mecheles, G. Dierkes, E. Feller and E. Rudolf. *Melliand Textilber.* 16, 725-9 (1935).—To obtain max. whiteness for cotton and rayon textiles, peroxide bleaches should be heated to 80° in about 30-45 min. and from 80° to 100° in about 3.5 hrs. Iron or rust in the bleach reduces its efficiency. Peroxide bleaches of mercerized cotton and of viscose rayon showed greater whiteness, lower luster and greater softness than similar Cl bleaches.

F. H. Moser. Rational utilization of the bleaching properties of sodium hypochlorite. V. I. Minaev. *Trans. Inst. Chem. Tech. Inyetro* (U. S. S. R.) 1, 174-80 (1935); cf. Minaev, et al., *Moskovsk. Tekst.-Ind.* 44, Nos. 2, 3 (1929).—White, boiled talco was bleached with NaOCl at 18° for 2.5 and 10 min. at a concn. of 1 g. NaOCl in 1 l. H<sub>2</sub>O, without and with the addition of 2, 4 and 8 g. (mol. of NaHCO<sub>3</sub>) to 1 g. (mol. of NaOCl) and (or) 2 g. of a wetting agent (α-nakal and org. acids) per 1 l. of the NaOCl soln. In all cases on addn. of NaHCO<sub>3</sub> or a wetting agent the bleaching

time was reduced 5 times and the formation of oxycellulose did not exceed 0.2%. The addn. of 2 and 4 mols. of NaHCO<sub>3</sub> produced after 2 min. of bleaching 79.5% and 80.94% whiteness, resp. with 40% reduction in the Cl consumption. The use of 8 mols. of NaHCO<sub>3</sub> showed no advantage. The addn. of wetting agents gave in 2 min. 87.76% of whiteness but at a considerably greater Cl consumption as compared with the bleaching in the presence of NaHCO<sub>3</sub>. With the addn. of NaHCO<sub>3</sub> and a wetting agent the degree of whiteness and the Cl consumption were still more increased without an increase in the oxycellulose formation. The acceleration of the bleaching reaction can be conceived either as a result of the liberation of HOCl from the NaOCl solns, or as a hydrolysis  $\text{ClO}^- + \text{H}_2\text{O} \rightleftharpoons \text{ClOH} + \text{OH}^-$ , or as a double decompn. of NaOCl and NaHCO<sub>3</sub>:  $\text{NaOCl} + \text{NaHCO}_3 = \text{Na}_2\text{CO}_3 + \text{HOCl}$  or  $\text{HOCl} + \text{NaOCl} + 2\text{NaHCO}_3 = \text{Na}_2\text{CO}_3 + \text{HOCl} + \text{NaHCO}_3$ . If the mechanism of the bleaching reaction is conceived according to the scheme:  $\text{NaOCl} + \text{HOH} \rightarrow \text{HOCl} + \text{NaOH}$ , then the addn. of NaHCO<sub>3</sub> is necessary to neutralize the gradually accumulating NaOH to prevent the reversion of the hydrolysis reaction. On the basis of the exptl. data and the theoretical consideration the addn. of NaHCO<sub>3</sub> to NaOCl solns. (obtained by satg. NaOH soln with Cl and contg. 0.5-1% excess of NaOH) for the stabilization of the system should be detd. stoichiometrically according to the formula  $[(n \text{ NaOCl} + m \text{ NaOH}) + \{(n + m) \text{ NaHCO}_3\}] = \{(n + m) \text{ Na}_2\text{CO}_3 + m \text{ H}_2\text{O}\} + n \text{ HOCl}$ , with the addn. of a slight excess of NaHCO<sub>3</sub>. The NaOCl solns. of the given concn. when treated with NaHCO<sub>3</sub> decompd. slowly, i. e., about 50% in 200-250 hrs.

Chas. Blanc. The behavior of starch and dextrin in solutions in relation to the usual additions to the finishing and sizing baths. F. Dittmar and H. Kober. *Melliand Textilber.* 16, 734-8 (1935).—Solns. of similar textile starches show great differences in turbidity. Addn. of large amts. of salts or colloids show only a slight increase in turbidity. However, addn. of small amts. shows a considerable change in the flocculation threshold. Clarity of the soln. does not insure clarity in the film. Differences in the turbidity of solns. of similar starches are due to different amts. of hydrolysis in soln. Those solns. that remain clear are from starches more homogeneous in compn.

F. H. Moser. The chemical examination of furs in relation to dermatitis. VI. Identification of vegetable and other dyes. H. E. Cox. *Analyst* 60, 793-801 (1935). cf. C. A. 29, 5664.—Dermatitis rarely results from irritation caused by the common vegetable dyes used on furs. The characteristics of logwood, quercitron, fustic, henna, turmeric, catechu, gall nuts and oak bark and pyrogallol are discussed and a simple scheme of analysis based on boiling the fur with HCl and then boiling for half a minute with Na formaldehydesulfoxylate soln. is outlined, by means of which the dyes used can be detected in most cases.

W. T. H. Dry-cleaning rayon fabrics. H. Roché. *Silk J.* 12, No. 137, 38 (1935).—Suitable methods for cleaning are outlined, in consideration of the more recent developments in chemicals and machinery. Trichloroethylene is fairly safe with the improved drying machinery at low temp. to prevent softening. It is also safer physiologically than most agents.

Halsey E. Sullivan. Factice and rubberized fabrics (Garach) 30. Acid-combining capacity of wool (Speakman, Stott) 2. Emulsifying and dispersing agents [in the textile industry] (Brit. pat. 434,424) 13. Anthrone derivs. (Ger. pat. 620,345) 10. Asym. ureas [products used as dye intermediates] (U. S. pat. 2,022,935) 10. Compn. for waterproofing fabrics of garments (U. S. pat. 2,022,405) 13. App. for coating cloth (U. S. pat. 2,022,322) 23. Liquid seal and vent for tanks, etc., contg. volatile solvents such as those used for "dry cleaning" (U. S. pat. 2,023,534) 1. Alkylated phenols [as intermediates for the prepn. of dyes] (Ger. pat. 616,786) 10. Viscous compn. for waterproofing cloth (Fr. pat. 786,553) 13.

**Dyes** I G Farbenind. A-G. (Ludwig Zeh and Claus Heuck, inventors). Ger. 617,727, Aug. 24, 1935 (Cl. 22e 7 02) Water-sol. derivs of vat dyes are obtained by converting acid  $H_2SO_4$  esters of leuco vat dyes into salts with amines which contain both etheral and OH groups in an aliphatic chain and which are optionally substituted at the N atom by alkyl, aralkyl, cycloalkyl or alkylene residues. The alkylene residue may be interrupted by a hetero atom such as O, S or N. In an example, the pyridine salt of the acid  $H_2SO_4$  ester of leuco-2,1-naphthothindigo is heated in water with a base formed by heating piperidine, water and  $CaH_2O$ . The product is a water-sol. basic salt of the above acid ester of the leuco vat dye. Other examples are given.

**Dyes** I G Farbenind. A-G. (Georg Kalischer and Werner Zerweck, inventors). Ger. 620,340, Oct. 19, 1935 (Cl. 22e 4) Dyes of the oxazine series are obtained by condensing  $\alpha$ -aminophenols contg a nitro group with  $p$ -quinones or halo  $p$ -quinones, e. g., chloranil. The reaction may be effected by stirring the reagents in alc. contg anhyd.  $NaOAc$ . The nitroaminophenol may contain addnl substituents, e. g., a  $SO_2H$  or another  $NO_2$  group. The products are useful as pigment dyes or, if they contain a keto group, as vat dyes for wool or, if a  $SO_2H$  group is present, as acid wool dyes. Examples are given.

**Fulling-fast dyes** Chemische Fabrik vorm. Sandoz Ger. 618,000, Aug. 30, 1935 (Cl. 22b 3 02). Arylamino anthraquinones with an unsubstituted 4'-position in the aryl nucleus are treated with aldehydes or substances yielding these in a neutral or acid medium, and further  $H_2SO_4$  groups are introduced into the product, if not sol. enough. Thus, 1-amino-4-phenylaminoanthraquinone-2-sulfonic acid is suspended in  $H_2SO_4$  and heated with  $BaH_2$ . The resulting dye colors wool from an acid bath in fulling- and light-fast greenish blue shades. Other examples are given. Cl. C. A. 30, 294<sup>1</sup>.

**Dyeing compositions** I G. Farbenind A-G (Ludwig Zeh and Erich Konrad, inventors). Ger. 617,718, Aug. 27, 1935 (Cl. 8m 3 02). A compn. contains salts of leuco  $H_2SO_4$  esters of dyestuffs obtained from benzanthrone and salts of compds contg the SH group. Thus, a 2% addn of Na dimethyldithiocarbamate to a printing paste contg salts of the leuco  $H_2SO_4$  ester of dimethoxydibenzanthrone renders the paste more stable.

**Azo dyes** Mordecai Mendoza (To Imperial Chemical Industries Ltd.). U. S. 2,021,917, Nov. 26. Dyes which dye wool various red shades are formed by coupling tetrazotized  $m$ -toluidine with one equiv. of 2-naphthol 6,8-disulfonic acid and one equiv. of 2-naphthol-3,6-disulfonic acid or the like.

**Azo dyes** Lmmet F. Hitch, Miles A. Dahlen and Martin L. Friedrich (to E. I. du Pont de Nemours & Co.). U. S. 2,023,590, Dec. 10. Water-insol. dyes of the ice color series which may be used to form insol. pigments or coupled on the fiber, are produced by coupling a diazotized arylamine with a fluorinated arylamide of 2,3-hydroxynaphthoic acid or the like. Numerous examples are given. U. S. 2,023,591, Dec. 10. Dyes (most of which dye red shades) which may be developed on the fiber, are produced by coupling a diazotized fluorinated arylamine with an ice-color coupling component. The fluorinated arylamine is preferably of the benzene or naphthalene series, and may have substituted thereon one or more of the well known nonwater-solubilizing groups. The ice-color coupling component is preferably an arylamide of 2,3-hydroxynaphthoic acid, the arylamide nucleus of which may have substituted thereon one or more nonwater-solubilizing groups. Numerous examples are given.

**Azo dyes** Gerhard Schrader (to General Aniline Works). U. S. 2,023,773, Dec. 10. Insol. azo dyes yielding, when produced on the fiber, generally yellow to red to blue to black shades of good fastness to light, are obtainable by coupling in substance or on the fiber a diazotized aromatic primary amine free from a substituent inducing soly. in water with  $\alpha$ -naphthothiazole substituted in the 1-position by alkyl or aryl and in the 6- or

7-position by OH and the 7- or 6-position by  $RNHC=O$ , in which R is a benzene or a naphthalene ring. The dyes produced in substance are generally yellow to red to blue to black powders, suitable for prep. lakes, while those produced on the fiber, especially cellulose fiber, according to the usual printing process or according to the method of prep. ice colors, generally yield yellow to red to blue to black shades of good fastness to light. Numerous examples are given.

**Azo dyes** I. G. Farbenindustrie A-G. Brit. 434,243, Aug. 28, 1935. This corresponds to Fr. 777,850 (C. A. 29, 4593<sup>1</sup>). Not only aromatic diazo but also diazoazo and tetraazo compds of the character described may be combined. The aromatic  $\alpha$ -hydroxycarboxylic acid amides must be free from  $COOH$  and  $SO_2H$  groups. The products are sol. in hydrocarbons, alcs, ethers, ketones, paraffin, fats, waxes and oils and may be used for coloring them and in the manuf. of colored nitrocellulose lacquers, films, candles, polishing-waxes and shoe polishes.

**Azo dyes** J. R. Geigy S. A. Fr. 789,775, Sept. 9, 1935. See Swiss 175,355 (C. A. 30, 296<sup>1</sup>).

**Azo dyes** J. R. Geigy S. A. Fr. 787,009, Sept. 16, 1935. Dyes insol. in water are made by coupling a negatively substituted diazo component free from solubilizing groups with a tertiary amine of the formula  $PhN(R)R'$ , (R is a dihydroxypropyl and R' an alkyl group and the position para to the amino group is free). Thus, 6-chloro-2,4-dinitroaniline  $\rightarrow$  3-ethyl(dihydroxypropyl)-amino-4-methoxytoluene dyes acetate silk blue shades and may be used for coloring lacquers. Other examples are given.

**Azo dyes** Imperial Chemical Industries Ltd. Fr. 786,733, Sept. 9, 1935. Yellow and orange dyes are prep. by coupling diazotized  $\alpha$ -mononitroaniline with a  $N$ -sulfatoethyl deriv. of an amine of the  $CaH_2$  series which may be coupled in the position para to the  $NH_2$  group. Thus,  $\alpha$ -mononitroaniline  $\rightarrow m$ -toluidine  $N$ -ethyl sulfate dyes wool, silk and rayon orange shades. Other examples are given. Cl. C. A. 29, 7087<sup>1</sup>.

**Azo dyes** I G Farbenind A-G. Ir. 786,608, Sept. 6, 1935. Dyes insol. in water are prep. by coupling the usual coupling components for insol. dyes with compds of the formula  $R^1COR^2(NH_2)COR^3$ , in which  $R^1$ ,  $R^2$  and  $R^3$  are aryl residues of the  $CaH_2$  or  $C_6H_5$  series which may contain substituents other than solubilizing groups. Thus, 1-amino-2,5-dibenzoylbenzene  $\rightarrow$  1-(2',3'-hydroxynaphthylamino)-4-chloro 2,5-dimethoxybenzene dyes cotton a fast scarlet shade. Other examples are given.

**Azo dyes** I G Farbenind A-G. Fr. 787,755, Sept. 9, 1935. Dyes are prep. by combining aromatic diazo compds. with hydroxyalkyl ethers of aromatic hydroxy-alkylamines, e. g., compds. obtained by the action of ethylene oxide on  $PhNH_2$ ,  $m$ -phenylenediamine,  $m$ -toluidine,  $m$ -chloroaniline, monobutylresorcinol or  $PhNHCH_3$ .

**Azo dyes** I G Farbenind A-G. Fr. 786,750, Sept. 9, 1935. Aromatic diazo compds. free from  $SO_2H$  or  $COOH$  groups are combined with tertiary aromatic amines of the formula  $XN(CH_2)_2R$  (X is an aromatic radical capable of combining in the para position and R is an alkyl group contg at least 2 C atoms, an aralkyl or cycloalkyl group). Thus,  $p$ -nitroaniline  $\rightarrow N$ -methyl- $N$ -benzylaniline gives an intense red on acetate silk. Several examples are given.

**Azo dyes** I. G. Farbenind. A-G. (Hermann Winkler, inventor). Ger. 620,344, Oct. 19, 1935 (Cl. 22a 1). The diazo compds. of aniline-3- or 4-sulfonic or -carboxylic acid anilides, substituted in the  $SO_2NHPh$  or  $CONHPh$  residue with a  $COOH$  and a OH group in  $\alpha$ -position to one another, but not substituted with an acylamino group, are coupled with 1-(2'-hydroxy-3'-carboxyphenyl)-3-methyl-5-pyrazolone or a deriv. thereof. The dyes so obtained are convertible into Cr compds. which generally yield fast yellow shades on animal fibers. The Cr compds. may be produced in substance or on the fiber. Examples are given.

**Azo dyes** I. G. Farbenind A-G. (Heinrich Chingstein, Hans Roos and Carl Heuser, inventors). Ger. 620,461, Oct. 22, 1935 (Cl. 22a 7). Diammononaphthalenesulfonic

acids, not contg.  $\text{NH}_2$  groups in *o*- or *para*-position to one another, are condensed with 2 mols. of a nitrobenzoyl chloride and the products are reduced. The diamines so obtained are tetrazotized and coupled with 2 mols of 1-(3'-aminophenyl)-3-methyl-5-pyrazolone or 1-(3'-aminophenyl)-5-pyrazolone-3-carboxylic acid or an alkyl ester thereof. Alternatively, 1 mol of one of the specified pyrazolone derivs and 1 mol of another yellow component not contg. a diazotizable  $\text{NH}_2$  group may be used. A modified process consists in condensing a monoacyldiaminonaphthalenesulfonic acid of the type specified with 1 mol of a nitrobenzoyl chloride, reducing and diazotizing the product, coupling with a yellow component not contg. a diazotizable  $\text{NH}_2$  group, splitting off the acyl group, diazotizing and coupling with one of the specified pyrazolone derivs. Yellow or orange dyes of good discharging properties are obtained. Examples are given.

**Azo dyes.** Soc pour l'ind chim. à Bâle. Ger 617,999, Aug 30, 1935 (Cl 22a 2). Mixts of chromable monoazo dyes of which at least one is a diazotized aminonaphtholmono-, -di- or -trisulfonate coupled with a naphthol residue, are treated with an alk. chroming agent and an org. compd. contg. a OH group. Thus the dye from diazotized 1-amino-2-naphthol-4-sulfonic acid and 1-naphthol is mixed with the dye from diazotized 1-amino-2-naphthol-4-sulfonic acid and 2-naphthol and the mixt. stirred with water. The product is treated with a chromite soln made from  $\text{Cr}(\text{OH})_3$  paste, glycerol and  $\text{KOH}$ . The resulting dye colors wool from an acid bath in fast marine-blue shades. Other examples are given.

**Azo dyes, dyeing.** I G Farbenind A-G (Friedrich Muth, inventor). Ger 620,459, Oct 22, 1935 (Cl 22a 1). Aromatic or heterocyclic diazo compds are coupled, in substance or on the fiber, with arylides of 2-hydroxycarbazole-*o*-carboxylic acids substituted on the nuclear N atom by a hydrocarbon radical. Water-insol. dyes of various colors are obtained. Numerous dyes are described.

**Azo dyes, dyeing.** I G Farbenind A-G (Gerhard Schrader, Albert Schmelzer and Fritz Ballauf, inventors). Ger 620,836, Oct 28, 1935 (Cl 22a 1). Addn to 481,445 (C A 24, 245). The diazo compds of unsulfonated 3-amino-2-alkoxycarbazoles are coupled, in substance or on the fiber, with arylides of 2,3-hydroxynaphthoic acid. Blue dyes are produced. Examples are given.

**Water-insoluble azo dyes, dyeing.** I G Farbenindustrie A-G. Brit 434,209, Aug 28, 1935. See Fr 768,884 (C A 29, 608').

**Water-insoluble azo dyes, dyeing.** I G Farbenindustrie A-G. Brit 434,433, Sept 2, 1935. See Fr 768,931 (C A 29, 609'). For "X and Y are H,  $\text{CH}_3$ , or alkoxy" read "X and Y are halogen,  $\text{CH}_3$ , or alkoxy."

**Green azo dyes.** Wm G Reid (to Imperial Chemical Industries Ltd.). U. S. 2,023,614, Dec. 10. Green dyeing is effected by impregnating cellulose material with a diacylacyldiamino compd. of the class consisting of diacylacyl-*o*-toluidide, diacylacyl-1,4,4'-diaminobenzophenone, diacylacyl-1,4,4'-diaminodiphenylurea, diacylacyl-1,4,4'-diaminodiphenylmethane and bis(benzoylacyl)-*o*-toluidide, and then treating the impregnated material with a diazotized 1-substituted amino-1-amino-anilinoanthraquinone. Several examples are given with details of procedure. U. S. 2,023,615 relates to the production of green dyes by coupling a diazotized amino-1-amino-anilinoanthraquinone compd. such as that of 1-methylamino-4-(4'-aminophenyl)aminoanthraquinone with a diacylacyldiamino compd. in which the acylacyl groups are substituted on sep. amino groups such as diacylacyl-*o*-toluidide. Several examples with details of procedure are given.

**Yellow chromable monoazo dyes.** Erich Fischer and Herbert Kracker (to General Aniline Works.). U. S. 2,023,933, Dec. 10. Diazotized 3-halo-2-aminobenzene-1-carboxylic acids are combined with pyrazolone derivs such as 1-(4'-sulfophenyl)-3-methyl-5-pyrazolone, 1-(2'-chloro-5'-sulfophenyl)-3-methyl-5-pyrazolone, 1-(2',5'-dichloro-4'-sulfophenyl)-3-methyl-5-pyrazolone,

1-(4'-chloro-2'-sulfophenyl)-3-methyl-5-pyrazolone, 1-(2'-hydroxy-3'-carboxy-5'-sulfophenyl)-3-methyl-5-pyrazolone, 1-(4'-methyl-2'-sulfophenyl)-3-methyl-5-pyrazolone, 1-(4'-sulfophenyl)-5-pyrazolone-3-carboxylic acid ethyl ester, 1-(4'-sulfophenyl)-3-methyl-5-pyrazolone, and the like. Cf. C A 29, 6433'.

**Chromicform azo dyes.** Soc pour l'ind chim. à Bâle. Brit 434,139, Aug 27, 1935. Cr compds of azo dyes are made by treating the azo dyes such as an *o*-aminonaphthol-sulfonic acid, which contains no  $\text{NO}_2$ , with a 2-hydroxynaphthalene with an alk. chroming agent and with an org. compd. contg. at least 1 OH at not above  $90^\circ$  or with a complex compd. obtained by the action of an org. compd. contg. at least 1 OH on an alk. suspension of Cr hydroxide. They yield navy blue shades, faster to rubbing than the dyes of Brit 186,635 (C A 17, 886). For an example, cf. Swiss 172,555 (C A 29, 5668').

**Azo dyes containing chromium.** Soc pour l'ind chim à Bâle. Ger 617,949, Aug 29, 1935 (Cl 22a 2). Chromed dyes are prepd. by the action of a chroming agent on a mixt. of chromable azo dyes contg. at least one diazotized aminonaphtholmono-, -di- or -trisulfonic acid coupled with a naphthol residue. An alk. soln. of  $\text{Cr}_2\text{O}_3$  in such amt. that there is 1 atom of Cr to each chromable azo dye, is used as the chroming agent. In an example, a paste of  $\text{Cr}_2\text{O}_3$ ,  $\text{KOH}$  and 2-hydroxynaphthalene is added to diazotized 1-amino-2-hydroxynaphthalene-4-sulfonic acid. On completion of the coupling, a concd. soln. of diazotized 1-hydroxy-2-amino-4-methylbenzenesulfonic acid is added. The resulting dye colors wool from a  $\text{H}_2\text{SO}_4$  bath in reddish blue shades. Other examples are given.

**Azo dyes containing chromium.** Soc pour l'ind chim à Bâle. Ger 620,200, Oct 17, 1935 (Cl 22a 2). A mixt. is prepd. contg. at least one Cr compd. of an azo dye and at least one chromable azo dye. The mixt. must contain at least one dye, chromed or unchromed, obtainable by coupling a diazotized *o*-aminonaphtholmono-, -di- or -trisulfonic acid with a naphthol. The mixt. is then heated in an aq. medium with a substantially undehydrolyzed alkali salt, e. g.,  $\text{Na}_2\text{SO}_3$ . The products are blue dyes applicable to animal, vegetable or artificial fibers or useful for coloring lacquers or as pigment dyes. Examples are given.

**Azo dyes containing chromium.** Soc pour l'ind chim à Bâle. Ger 620,257, Oct 17, 1935 (Cl 22a 2). The dyes obtained by coupling diazotized *o*-aminonaphthol-sulfonic acids with naphthols or their substitution products, are heated with any of the usual chroming agents in the presence of a water-sol. salt of an organic carboxylic or sulfonic acid. The proportion of chroming agent must be such that less than 1 atom of Cr is present for each chromable group in the dye. Blue dyes for animal fibers are obtained. The products are faster than the corresponding Cr-contg. dyes prepd. in the absence of a salt of an org. acid. Numerous examples are given. Cf. C A 29, 2306' and 2 preceding abstrs.

**Disazo dyes.** Hans Krukalit and Walter Limbacher (to General Aniline Works.). U. S. 2,023,176, Dec. 3. This corresponds to Ger 607,678 (C A 29, 4185'). The coupling compd. may also contain the group  $\text{RNH-SO}_2\text{R}'$ .

**Disazo dyes.** J R. Geigy A-G. Ger 620,258, Oct. 17, 1935 (Cl 22a 6). Tetrazotized 4,4'-diamino-2,2'-dimethylbiphenyl is coupled with components not contg. a COOH or  $\text{SO}_3\text{H}$  group. Water-insol. dyes of various colors are obtained. The dyes are sol. in all the usual org. solvents, and are useful for coloring cellulose ester or spirit lacquers, or as components of printing inks. Examples are given.

**Disazo dyes.** Durand & Huguenin S. A. Fr. 786,649, Sept. 6, 1935. Mordant dyes are prepd. by diazotizing a sulfonic amine of the  $\text{C}_6\text{H}_4$  series, coupling with a dialkyl ether of aminohydroquinone, rediazotizing and coupling with an *o*-hydroxybenzenecarboxylic acid. Thus, 1-amino-4-chlorobenzene-3-sulfonic acid  $\rightarrow$  aminohydroquinone dimethyl ether  $\rightarrow$  salicylic acid gives deep brown-

red shades on cotton mordanted with Cr Other examples are given

**Disazo dyes, dyeing** I G Farbenindustrie A-G. Brit 434,416, Sept 2, 1935 Dyes, insol in  $H_2O$ , are made in substance, on a substratum or on the fiber by diazotizing a substitution product of 2-amino-1,1'-azobenzene and coupling with an arylide of 2,3-hydroxynaphthoic acid, only such components being used as do not contain groups kindling sofly They are faster to boiling NaOH soln than the corresponding dyes from diazotized  $p$ -aminazo compds (cf Brit 378,555 (C A 27, 4089)). In examples, the following dyes are made on cotton: (1) 4-methyl 2-amino 2',4'-dichlorobenzene  $\rightarrow$  1-(2',3'-hydroxynaphthylamino) - 2-methyl - 4-chlorobenzene (Bordeaux-red), and (2) 6-methyl-4-methoxy-2-amino 2',4'-dichlorobenzene  $\rightarrow$  the amide (red-brown). Cf C A 29, 3172

**Brown disazo dyes** Francis H Smith (to E. I. du Pont de Nemours & Co.) U. S. 2,022,606, Nov. 25 A diazotizing acid such as HCl is added to a mixt of water,  $NaNO_2$  and a diamine such as *m*-phenylenediamine or the like in the continuously maintained presence of a salting-out agent such as NaCl in sufficient amount to salt out the dye substantially as fast as it is formed

**Dry compositions for producing azo dyes on the fiber** Compagnie nationale de matieres colorantes et manufactures de produits chim du Nord reumes etablissemets Kuhlmann Ger 620,247, Oct 17, 1935 (Cl 8m 13). See Fr 772,061 (C A 29, 1650)

**Vat dyes** I G Farbenind A-G (Norbert Stenger and Eduard Albrecht, inventors) Ger 617,726, Aug 27, 1935 (Cl 22a 7 01) Lysim indigoid dyes are obtained by condensing 4,7-dimethyl-6,7-dihydroxydihydroxythiophenes or their reactive 2-derivs, with different indigoid dye forming compds The products may be further halogenated In an example, 4,5-dimethyl-6,7-dichloro-dihydroxythiophene is condensed with 4-methyl-6-chlorohydroxythiophene-2- $p$ -dimethylamino in the presence of glacial AcOH to give a vat dye which colors cotton in fast bluish red shades Other examples are given

**Vat dyes** I G Farbenind A-G (Werner Zerweck and Ernst Honold, inventors) Ger 618,811, Sept. 16, 1935 (Cl 22b 3 03). Addn to 602,336 (C A 29, 6117) Dyes of various colors are obtained by forming amides from  $\alpha$ -amino compds of the anthraquinone series and arylmethazolo-mono- or -di-carboxylic acids substituted with an anthraquinonyl residue The reaction may be effected by heating  $\alpha$ -aminoanthraquinones with the acid chlorides, or a halo anthraquinones with the acid amides The acids used as starting materials are obtainable by condensing anthraquinonealdehydes with  $\alpha$ -aminomercaptoaryl carboxylic acids Examples are given Cf C A 30, 623

**Vat dyes of the dibenzanthrone series** Edward T. Howell (to E. I. du Pont de Nemours & Co.) U. S. 2,022,584, Dec 3 In prep a dibenzanthrone compd, such as 6,6'-dichlorodibenzanthrone, the corresponding 2,2'-dibenzanthronyl compd is heated with  $AlCl_3$  and an oxidizing agent such as  $KNO_3$  (suitably at a temp. of about 105-150° in a melt of NaCl and KCl). Several examples are given

**Vat dyes of the pyranthrone series** Georg Kranzlein, Heinrich Vollman and Werner Schultheis (to General Aniline Works) U. S. 2,022,926, Dec 10 Dyes which produce orange shades on cotton are formed by condensing pyrene with an aromatic carboxylic acid deriv. of the general formula aryl-COR in which R represents halogen or the group  $OCOAr_1$  (the 2 aryls being of the benzene or naphthalene series and being identical), such as benzoyl chloride, *m*-chlorobenzoyl chloride, *p*-toluyl chloride or *n*-naphthyl chloride, in the presence of  $AlCl_3$  and while introducing a dehydrogenating agent such as O or air or a solid oxidizing agent such as a heavy metal oxide, into the reaction mixt

**Halogenated vat dyes of the benzanthronylaminoanthraquinone series** Ernst Honold and Rudolf Müller (to General Aniline Works) U. S. 2,022,240, Nov. 20. In-

tial materials such as those obtained by the action of benzoyl chloride on an alk condensation product of benzanthronyl-1-aminoanthraquinone,  $Bz$ -1-benzanthronyl-1-amino-5-aminoanthraquinone or the like are converted into dichloro derivs. by use of a reagent such as suluryl chloride in  $PhNO_2$  to produce vat dyes generally dyeing cotton from a hyposulfite vat bluish green to olive green shades Several examples are given Cf. C. A. 29, 4185

**Vat dye printing pastes** Philip H. Stott and Earl E. Beard (to L. I. du Pont de Nemours & Co.) U. S. 2,022,748, Dec 3. The printing qualities of a vat dye of the algal series are improved by incorporating into the printing paste conig. the dye an acylaminoanthraquinone of high soly in the alk hyposulfite vat such as dibenzoyl-1,8-diaminoanthraquinone, etc. Various examples are given

**Metalized dyes** See pour l'ind chim à Bâle. Fr. 786,920, Sept 14, 1935 Dyes of the formula  $R \rightarrow R' - R$ , in which R represents diazo compds one at least of which contains groups capable of forming lakes, and  $R'$  is an aryl compd. conig. at least 2 amino groups capable of coupling twice, are treated with metallizing agents The 2nd coupling may take place after the metallization. Thus 4-nitro-2-aminophenol  $\rightarrow$  1,7-diaminobenzene  $\rightarrow$  4-nitro-2-amino-1-phenol-6-sulfonic acid is metallized with  $FeCl_3$ , giving a dye which gives fast deep olive-brown shades on Cr-tanned leather. Several examples are given Cf C. A. 29, 8348

**Dyes of the anthraquinone series** Henry Dreyfus U. S. 2,022,956, Dec 3. Dyes which are suitable for use with cellulose acetate, etc., are produced by the reaction of an amino alc. such as  $\beta$ -aminoethyl alc. on  $\alpha$ -negatively substituted anthraquinone, such as  $\alpha$ -chloroanthraquinone, in which the neg substituent is capable of being replaced by an amino group by the action of an amine.

**Acid wool anthraquinone dyes** I. G. Farbenindustrie A-G Brit 434,429, Sept 2, 1935 The dyes are obtained by causing a polynuclear aromatic base which is partially hydrogenated to react with an anthraquinone deriv. that contains no  $SO_2H$  group and either contains in 1,4-, 1,5-, or 1,8 position substituents known to be exchangeable for amino radicals by reaction with amino compds or contains such an exchangeable substituent in 1 of these positions besides other substituents in any position, and then sulfonating the products thus obtained Cf. C. A. 29, 935

**Azabenzanthroneacridine dyes** Max A. Kunz, Karl Koerberle and Gerd Kochendoerfer (to General Aniline Works) U. S. 2,023,470, Dec 10 Numerous examples are given of the production of dyes by treating with condensing agents azabenzanthrones which are connected in one per position with an org. radical by means of a linking member which may be a N, O, S, SS, Se or CO bridge. Hereby products are formed corresponding to the general formula  $X-Y-Z$ , in which X stands for an azabenzanthrone radical, Y for the bridge and Z for an org. radical, at least the linkage X-Y being attached to a peri-position of the azabenzanthrone radical, preferably the two linkages attached to X are in the two peri positions of the azabenzanthrone E.g. from  $Bz$ -1- $\alpha$ -anthraquinonylamino-8-azabenzanthrone by treatment with  $H_2SO_4$  or  $ClSO_3H$ , an orange-yellow dye is obtained By treatment with alk potash,  $Bz$ -1- $\alpha$ -anthraquinonylamino-8-azabenzanthrone yields an olive green dye  $Bz$ -1- $N$ -Pyrazolanthronyl-8-azabenzanthronyl is converted into 8-azabenzanthronepyrazolanthrone From the reaction product of thioglycolic acid or an anthraquinomercaptau on  $Bz$ -1-bromo-8-azabenzanthrone, condensation products conig. S are obtained by alk. condensation

**Dibenzanthrone dyes** E. I. du Pont de Nemours & Co Brit 434,132, Aug 27, 1935 This corresponds to Ger 613,179 (C A 29, 5670) but is restricted to the products obtained from the monoisopropyl or monoisobutyl ether of  $Bz$ -2,  $Bz$ -2'-dihydroxydibenzanthrone They are presumed to be dibenzanthrones wherein the  $Bz$ -2,  $Bz$ -2'-positions are linked as parts of a pyran ring

structure. Further dyes may be obtained not only by nitrating, sulfonating or halogenating but also by nitrating and reducing, by oxidizing to yield hydroxy derivatives or by condensing with an org. carboxylic acid in the presence of an AlCl<sub>3</sub> condensing agent. Among examples, (1) *Rs-2'-di-4'-hydroxydibenzanthrone monoisopropyl ether* is heated to 170–180° in CH2Cl2, CHCl3 or CCl4 in the presence of AcONa and, optionally in the presence of benzal chloride, BiH3, mono- or di-chlorohydrin, CH2Cl2, *p,p'*-dibromodibenzophenone dichloride, diphenyldichloromethane or dibromostilbene as catalysts, the product may be purified by treatment with H2O, for conversion to the sulfate, which is sep'd and decomp'd by H2O, or by heating in CH2Cl2 and filtering, it dyes cotton fast green shades, (2) the product of (1) is treated with MnO2 in H2SO4 at 18–20° and then with Na2SO4 to give a hydroxy deriv. (yellowish green), and (3) the product of (1) is condensed with  $\alpha$ -chloroanthraquinone- $\beta$ -carboxylic acid chloride in a melt of AlCl3 and SnCl4 at 110–120° (dyes greenish blue).

**Indigoid dyes** I G Farbindustrie A G Ger 780,737, Sept. 9, 1935 Violet to blue vat dyes are prep'd by condensing 4,7-dichloro-5-methyl-3-hydroxythiophthalic (1) to form thiophthalene-(2) indol (2) indigo, and, if necessary, subsequently halogenating I, in 160–161°, is obtained by converting 2,5-dichlorotoluene to the sulfonyl chloride to 2,5-dichloro-4-methylphenyl-1-thioglycolic acid, m 104°, and closing the ring 1 examples are given Cf C A 29, 2307<sup>1</sup>

**Pyrene dyes** I G Farbindustrie A G Brit 434,027, Aug. 26, 1935 This corresponds to Fr 769,837 (C A 29, 6139), the starting material being 2,5,7,10-tetrachloropyrene-3,8-quinone. Most of the products dye cotton and some may be converted into leuco-sulfur esters and used according to known methods.

**Triarylmethane dyes** I G Farbindustrie A G (Carl Winter and Paul Krebs, inventors) Ger 620,402, Oct. 22, 1935 (Cl 226 10) As components in the manuf. of triarylmethane dyes by standard processes, use is made of comp'ds. of the formula  $R'N(R'')CN$ , in which R is an aryl group, R' is H or an alkyl, hydroxyalkyl or aralkyl group, and n is a whole no. The substitution or sapon products of these comp'ds. may also be used, or the CN group may be sapon'd after the dye has been produced. A typical product is obtained by heating 4,4'-tetrachlorodibromobenzophenone with  $\alpha$ -naphthylamino- $\beta$ -propionitrile (obtainable from  $\alpha$ -naphthylamine and acrylonitrile) to 55° in toluene in the presence of POCl3, the product is a water-sol. blue dye applicable to paper, rayon mordanted cotton, and silk. Other examples are given.

**Rendering water soluble dyes soluble in organic solvents and insoluble in water** Wolf Krutichsky, Carl J. Beckert and Jack Braver U S 2,022,678, Dec. 3 A dye such as Naphthol yellow S (C I No 10) is rendered sol. in org. solvents such as CH2Cl2, acetone, alc. and "mineral naphtha" by combining it with  $\alpha$ -hydroxyethanolamine hydrochloride or various suitable comp'ds. of the general formula  $ACX_nNR_n$  in which Ac is an acyl group linked to Y, Y represents O, N, S, P and I, X is a hydrocarbon radical which may be substituted by a hydroxy or alkoxy radical forming the chemical linkage between Y and N, n is a whole number and R represents H or an alkyl group which may be substituted by a hydroxy or alkoxy radical. Numerous examples are given.

**Diazoamino compounds, azo dyes.** I G Farbindustrie A G Brit 433,578, Aug. 22, 1935. See Fr. 769,838 (C. A. 29, 6099)

**Diazonium compounds.** Anton Osenbeck and Ernst Tietze (to General Aniline Works) U S 2,022,923, Dec. 3. Numerous examples are given of the production of diazonium salts of the general formula  $RN_2NSO_2R'NHX$  where R stands for the radical of a diazotized non-sulfonated diazotization component suitable for producing azo dyes, R' stands for an aromatic radical and X stands for the radical of a carboxylic acid which is attached with the C atom of the CO group to the N atom of the NH group. The comp'ds. are generally stable, yellow to orange,

cryst. substances sol. in water, and are useful as intermediates in the manuf. of dyes Cf C. A. 28, 3079<sup>1</sup>.

**Dyeing** Soc. pour l'ind. chim. à Bâle. Ger. 617,644, Aug. 23, 1935 (Cl 8m. 2.03) The absorptive capacity of animal fibers for dyes is diminished by employing dye baths contg. sulfonated condensation products of aromatic hydroxy comp'ds. and benzoin. By this means, two-color effects are produced on cotton and wool or like textile mixtures. Thus, a half wool and cotton fabric is dyed in a bath contg. chloraniline and 8 RN (Schultz Dye Tables 7 420), Na2SO4 and a small addn. of the product obtained by condensing Na 2-hydroxy naphthalene-6-sulfonate with benzoin in cond. H2SO4. After washing, rinsing and drying the fabric, the wool is scarcely colored while the cotton is dyed deep red. Without the addn. of the above condensation product, the wool is also dyed deep red. Other examples are given.

**Dyeing** I G Farbindustrie A G (Othmar Drapal, inventor) Ger 620,422 (Oct. 19, 1935 (Cl 8m 12). In producing water-insol. azo dyes on the fiber, use is made of an aq. alk. bath which contains an arylide of a cyclic  $\alpha$ -hydroxy carboxylic acid and to which has been added a mixt. of Turkey red oil, sulfate cellulose lye, and Na2PO4. Addn. of this mixt. prevents the pptn. of Ca salts in the bath and leads to dyings of better fastness to rubbing than those ordinarily obtained. Other wetting agents and (or) solvents may also be added to the bath. An example is given.

**Dyeing with vat dyes** Richard G. Clarkson and Frank W. Johnson (to I. du Pont de Nemours & Co.) U S 2,021,932, Nov. 26 A dye soln. is applied to fiber or fabric in the presence of a cellulose comp'd. such as Na cellulose glycolate which is adapted to be dispersed in alk. soln. and serves as a retardant.

**Dyeing textiles** Soc. pour l'ind. chim. à Bâle Fr 780,671, Sept. 7, 1935 Fast shades are obtained by transforming in known manner on the fiber or on other appropriate supports, the NH comp'ds. of aromatic hydrocarbons formed of more than 3 condensed rings. Examples are given of dyings obtained by oxidizing diaminopyrene, and by diazotizing it and coupling with 2,3-dihydroxy naphthoic acid.

**Dyeing wool** Irma C. F. Plouquet (Theodor Walthers, inventor) Ger 617,512, Aug. 20, 1935 (Cl 8m 1.01) Indigo blue effects are produced on wool by passing the fabric through an open jigger contg. the indigo or bromoindigo dye in a roller vat, to effect the basic dyeing. The fabric is then overdyed with bromoindigo. Examples are given.

**Dyeing skins, hair and feathers** I G Farbindustrie A G (Trichl chem. int., inventor) Ger 617,047, Aug. 30, 1935 (Cl 8m 10 02) The above substances are treated with an oxidizing bath contg. a salt of 1,5-dihydroxynaphthalene and aminodiphenylamine optionally substituted by alkyl, cycloalkyl, aralkyl, hydroxyalkyl or alkoxy groups. Thus, a salt of 1,5-dihydroxynaphthalene and 4-amino-diphenylamine is dissolved in water and a 3% soln. of H2O2 add'd. A white dog skin mordanted with Fe salt is soaked in the bath for 3 hrs. and the hair becomes blackish blue and the leather blue. Other examples are given.

**Dyeing and printing textile materials** Miles A. Dahlen (to I. du Pont de Nemours & Co.) U S 2,021,911, Nov. 26 Bright fast colors (brown, orange and red in examples given) are produced on goods such as cotton in a "one-bath" process by use of compas. comprising diazonium comp'ds. of the general formula:  $aryl-N-NR$  in which aryl represents an aryl nucleus of the benzene, azobenzene, biphenyl, diphenylamine, naphthalene, carbazole or anthraquinone series contg. no solubilizing group, but which may contain groups such as alkyl, alkoxy, halogen, nitro and benzoylamino, and R represents a pyrrolidine or piperidine nucleus contg. at least one solubilizing group; and any of the well-known ice-color coupling components such as the arylamides of 2-hydroxy-3-naphthoic acid, hydroxy carbazole, hydroxy naphtho-carbazole and hydroxyanthracene-carboxylic acids,  $\alpha$ - and  $\beta$ -naphthol, acetoacetyl derivs. of arylamines, and phenylmethylpyrazolone. The material is treated with

the mixt comprising one of the well-known ice-color coupling components and a diazomino compd., and developed by subjecting it to the action of a dil acid at elevated temps. Several examples with details of procedure are given.

**Printing textiles.** Imperial Chemical Industries Ltd., Samuel Howard and Alce Wormald. Brit. 433,803, Aug. 19, 1935. White resist effects are produced on cellulosic material by printing with a resist paste contg. a salt of a primary, secondary or tertiary amine or a quaternary NII, halide, overprinting or padding with a prepn. contg. a sulfonic ester of a leuco vat dye having affinity for cotton and further treating to develop the vat dyeing. Among 6 samples, cotton cloth is printed with an aq. paste contg. British gum and benzylpyridinium chloride, dried, overprinted with an aq. paste contg. Solodon jade green, neutral starch tragacanth thickening and  $\text{NaNO}_2$ , again dried, passed through dil.  $\text{H}_2\text{SO}_4$  at  $60^\circ$ , washed and soaped at the boil.

**Colored pattern effects on textile materials.** Geo. H. Ellis and John Allan (to Celanese Corp. of America). U. S. 2,022,413, Nov. 26, 1935. In forming colored pattern effects on textile materials such as a cotton or cellulose acetate fabric by discharge of ground color, there is applied to selected areas, before moist heat treatment, both a compn. comprising a discharging agent for the ground color and a coloring matter resistant to such discharging agent, and a compn. comprising an agent which will discharge the ground color and also the coloring matter of the other compn., the materials are then subjected to moist heat treatment to effect discharge and color fixation.

**Machine for dyeing hanks of yarn.** Smith, Drum & Co. Ger. 617,974, Aug. 30, 1935 (Cl. 8a 8 01).

**Piece-dyeing jigger.** Hans Geduldiger. Ger. 617,933, Aug. 29, 1935 (Cl. 8a 9 50).

**Apparatus for dyeing hat bodies.** Turner, Atherton and Co. and Albert Turner. Brit. 434,024, Aug. 26, 1935.

**Apparatus for applying dye solutions or other liquids to traveling yarns such as those of natural or artificial materials.** Ernest L. Greenwood (to Celanese Corp. of America). U. S. 2,022,854, Dec. 3, 1935. Structural details of a siphon wick device.

**Apparatus for producing azo dyes on knitted tubular fabrics.** I. G. Farbenind. A.-G. (Carl Vorck, inventor). Ger. 620,323, Oct. 19, 1935 (Cl. 8m 12).

**Printing machine for fabrics.** Radebeuler Maschinenfabrik August Koenig G. m. b. H. Ger. 617,875, Aug. 27, 1935 (Cl. 8c 7 01).

**Apparatus (with a photoelectric cell) for comparison of colors such as those of textile materials.** Harold H. Sheldon (to Sheldon Elec. Corp.). U. S. 2,022,327, Nov. 26, 1935. Structural features.

**Apparatus (with lamps and color filters) for matching colors such as those of dyed clothing, etc.** Harold H. Sheldon (to Sheldon Elec. Corp.). U. S. 2,022,326, Nov. 26, 1935. Structural features.

**Artificial threads.** Siemens-Schuckertwerke A.-G. I. r. 786,669, Sept. 7, 1935. Threads made from polymerization products of aryl olefins such as polystyrene, "vinifol," "vinilytes" and "chlorvin" are made up of a no. of individual very fine strands. The strands may be mixed and worked with animal or vegetable wool, silk or asbestos.

**Treating artificial filaments, fabrics, etc., dyeing.** British Celanese Ltd., Albert Mellor and Ralph J. Mann. Brit. 434,203, Aug. 28, 1935. Artificial threads, yarns, etc., are treated with a solvent, swelling or softening agent under such conditions that the agent does not completely penetrate the filaments, etc., and are then subjected to dyeing or other wet textile operation in rope or other folded form. In examples, (1) cellulose acetate fabric is padded with a 10% aq. soln. of  $\text{HCOOH}$ ,  $\text{AcOH}$  or ethylene glycol monoacetate, given an air run for 30 sec. and washed, and (2) similar fabric is passed through a bath contg.  $\text{C}_2\text{H}_5\text{Cl}$ , 60 and  $\text{PhMe}$  20% for about 2 min., washed with  $\text{PhMe}$  and dried, in each case before dyeing or other wet textile operation on the fabric in rope form.

**Apparatus for drying wound rayon threads.** Vereinigte

1 Glanzstoff-Fabriken A.-G. Ger. 619,778, Oct. 7, 1935 (Cl. 29a 6 08). Addn. to 600,236 (Cl. A 28, 65769).

**Textile fabric.** Frederick B. Smith (to Celanese Corp. of America). U. S. 2,022,391, Nov. 26, 1935. For producing a fabric having a full hand, a process is used of assoc. together yarns of cellulose acetate and yarns of regenerated cellulose which, after having reached the set condition, have been subjected to a stretching operation in alternating relationship in such manner that a fabric is formed wherein at least part of the cellulose acetate yarn floats, and then subjecting the fabric to a treatment which causes the regenerated cellulose yarn to shrink.

**Rayon.** British Bemberg Ltd. Brit. 434,320, Aug. 29, 1935. Freshly spun and washed cellulose hydrate rayon in wound form is treated with an aq. emulsion of a fat or oil at above  $70^\circ$ , freed from excess of liquid and dried without intermediate washing. Any mineral, animal or vegetable oil or fat may be used. In an example, freshly spun, decoppered and washed skeins of cuprammonium rayon are treated 30 min. in an aq. emulsion contg. 15% olive oil at  $99^\circ$ , centrifuged and dried.

**Crepe fabrics.** Heberlein & Co. A.-G. Brit. 433,902, Aug. 22, 1935. Figured effects are produced on crepe fabrics having overwristed threads by printing them locally with reserves that exert a cementing action on the threads and are resistant to subsequent wet treatments and then treating the fabric in a crepe-setting bath, followed by any further processing treatment. The reserving effect may be obtained by treatment with albuminous substances that are subsequently rendered insol. by coagulation by heat or tanning, or by the application of natural or artificial resins, cellulose esters or regenerated cellulose.

**Coated fabrics.** Sandura Co., Inc. Brit. 434,489, Aug. 26, 1935. Decorative sheet material for covering walls and floors is made by applying to 1 side of a fibrous sheet a liquid backing compn., e. g., clear or pigmented oil or lacquer, dye, glue, latex, casein, wax or resin, in controlled amt. to penetrate the sheet but not fill the pores and then applying to the other side decorative coloring material, e. g., paint, ink or dye, which penetrates well down into the body of the sheet. Before application of the decorative material, a moistening agent, e. g., a soln. of bodied linseed oil in a light hydrocarbon, may be applied to the face of the sheet as described in Brit. 434,490 (below). In 434,490, Aug. 26, 1935, the decorative material is applied 1st and a toughening agent, e. g., oil, latex or lacquer, then introduced so that the fibers are coated but the pores incompletely filled. The fibrous sheet may be moistened with a soln. of bodied linseed oil in naphtha or  $\text{C}_4\text{H}_8$  before decorating, e. g., by printing. A backing material may be applied to the sheet before moistening or decorating, as described in Brit. 434,489 (above).

**Coating textiles.** Julien Baude. Fr. 786,897, Sept. 11, 1935. Textiles are made less permeable to air and liquids and fireproof by a coating compn. contg.  $\text{CaCO}_3$ , powdered metal, Na silicate and a pigment.

**Apparatus for binding cut edges of pile fabrics.** Wm. A. Anderson (to Charles H. Davison). U. S. 2,023,378, Dec. 3, 1935. Numerous structural and operative details of an app. applying cement to the edges to be bound.

**Washing wool.** I. G. Farbenind. A.-G. (Hermann Schutte, inventor). Ger. 616,797, Aug. 5, 1935 (Cl. 29b 1). Crude wool is washed by solns. of acid  $\text{H}_2\text{SO}_4$  esters of the higher alcs. obtained by oxidizing paraffin hydrocarbons, or by water-sol. salts of such esters. Thus, a washing bath contains the neutral Na salt of the acid  $\text{H}_2\text{SO}_4$  esters obtained from a mixt. of higher alcs. obtained as above.

**Delustering regenerated cellulose filaments.** Franz Hoelckeskamp (to American Bemberg Corp.). U. S. 2,022,961, Dec. 3, 1935. A soln. of viscose or cuprammonium cellulose is spun, with stretching, to form filaments which are pptd. in the presence of a water-sol. metal salt such as  $\text{BaCl}_2$  and subsequently coagulated with a compd. contg. one or more acid H atoms such as  $\text{H}_2\text{SO}_4$ , which reacts with the metal salt to form a water-insol. ppt. App. is described.

**Apparatus for drying fabrics in the open width.** Sur

James F. Norton & Co. Ltd. and David G. Norton 1  
Brit. 432,982, Aug. 7, 1935

Apparatus for drying spools of textile materials Joseph  
Annic Brit. 431,346, Aug. 29, 1935

Centrifugal machines and textile-treating process  
André Miesse Brit. 434,379, Aug. 30, 1935 Textile  
materials are washed, dyed, dressed, etc., in a machine  
consisting of 2 rotary containers in a fixed casing, centri-  
fugal force being used only to produce pressure in the liquid  
in which the materials are immersed so that treatment is  
under high pressure without circulation

Apparatus for shrinking textile materials John H.  
Wrigley and Alexander Melville (to Cluett Peabody &  
Co.) U. S. 2,021,975, Nov. 26 Various structural,  
mechanical and operative details

Apparatus for heating liquids, such as bleaching liquor  
used in the textile industry Wm. I. Hobdell Brit.  
434,211, Aug. 28, 1935

Bleaching cellulose fibers Adolf Danzinger Austrian  
143,295, Oct. 25, 1935 (Cl. 8) Natural or artificial  
cellulose fibers, particularly cotton, are bleached with  
sols of per compounds at a temp. above 80°, preferably  
above 100°, under a pressure of at least 1.5 atm. Weekly  
alk. baths are preferably used. A bucking treatment in  
a relatively strong alk. bath may precede the bleaching  
processes are described

Treating textile materials with liquids as in washing,  
dyeing, bleaching, etc. Leon De R. Iaber and Charles J.  
Carroll (to Faber Engineering Co.) U. S. 2,023,013,  
Dec. 3 Liquid is withdrawn from a pool (in a described

app.) and aerated, used for treating textile material and  
then returned to the pool

Drying-oil derivatives suitable for sizing natural silk  
or rayon, etc. Karl Ott, Herbert Gensel and Herbert  
Schuster (to I. G. Farbenindustrie A.-G.) U. S. 2,023,768,  
Dec. 10 A drying oil such as linseed oil is treated with  
a water-sol. sulfuric acid salt such as  $\text{NaHSO}_4$  in the  
presence of a gaseous oxidizing agent such as air (suitably  
at a temp. of about 60-90° until a water-sol. product is  
obtained)

Mercerizing machine Zittauer Maschinenfabrik A.-G.  
Ger. 616,231, July 23, 1935 (Cl. 8) 24 01) Addn to  
509,505

Removing stains such as those of iron from cotton,  
rayon, etc. James F. White (to Matheson Alkali Works).  
U. S. 2,022,262, Nov. 26 The stain is treated with an  
alk. soln. comprising an alkali metal chloride or alk. earth  
metal chloride and oxalic acid or a water-sol. oxalate (suit-  
ably with an acidifying agent such as  $\text{HOAc}$ )

Fibrous sheet material such as cushioning material.  
Charles W. Leguillon (to B. F. Goodrich Co.) U. S.  
2,023,273, Dec. 3 Various details of app. and operation  
are described for forming material in sheet form from  
crossed fibers united by a flexible agglutinant such as a  
rubber compn.

Luminous paste Henri G. G. Desmaré Fr. 786,572,  
Sept. 3, 1935 A luminescent sulfide is incorporated in a  
transparent and white colloidal emulsion of rubber to make  
a paste suitable for coating and printing on fibers and  
cloth

## 26—PAINTS, VARNISHES AND NATURAL RESINS

A. H. Sabin and Carlton H. Rose

Bitumen paints and red lead primers. Hans Hebb-  
ling. *Farben-Ztg.* 40, 908 (1935) —A brief discussion of  
possible reactions between red lead primings and bitumen  
paints applied thereto J. W. Perry

Paints meeting unusual resistance requirements es-  
pecially for agricultural uses R. Scheifele *Farben-  
Ztg.* 40, 810-11, 830-7 (1935) —A no. of unusual resis-  
tance requirements are classified, the desirability of simulat-  
ing actual conditions of use in accelerated testing is dis-  
cussed and the usefulness of special paints on the farm is  
pointed out. J. W. Perry

Comparing the adhesion of a paint film on different  
materials H. L. Matthysen *Verfahrenstechnik* 8, 276-8  
(1935) —Results of abrasion tests with coarse  $\text{SiC}$  (cf.  
C. A. 28, 5686) revealed that the adhesion of 3 different  
oil paints applied to duralumin was approx. doubled by  
anodic oxidation of the latter Cf. C. A. 28, 2625

Progress in paints for ships' keels Manfred Ragg  
*Farben-Ztg.* 40, 1167-9 (1935) J. W. Perry

Railroads and paints. B. Scheifele *Farben-Ztg.* 40,  
988-9 (1935) —Color schemes and types of paints used by  
the German State Railways are discussed J. W. P.

Preventing (metal) corrosion and paint B. Scheifele  
*Farben-Ztg.* 40, 1164-5 (1935) —A discussion emphasizing  
(a) sealing the surface and (b) rendering the metal passive,  
as the important factors J. W. Perry

New products and problems in construction painting  
C. F. Rassweiler. *Ind. Eng. Chem.* 27, 1294-7 (1935) —  
Many problems arising from new construction methods and  
materials have been solved by use of synthetic resins such  
as glyceryl phthalates (I) and  $\text{PhOH-CH}_2\text{O}$  (II). The  
crit. properties of modern interior finishes are rapid drying  
(no longer incompatible with durability), good hiding and  
retention of appearance. Important quick-drying prod-  
ucts are architectural white enamels and floor varnishes,  
contg. I and II, resp., and wall paints, contg. processed  
oils and Ti pigments, the degree of penetration into porous  
surfaces being controlled by the state of dispersion of the  
ingredients. Crit. properties of modern exterior finishes  
are protection of metal, durability on wood and alk. sur-  
faces (cement, plaster and asbestos board), and retention of

appearance. I and II confer exceptional  $\text{H}_2\text{O}$  imper-  
meability and exposure resistance, which are further in-  
creased by "metal-protective pigments." Pre-finished  
construction units are of interest because max. resistance  
of paint films develops after aging. Marine paints con-  
tain I; acid- and alkali-resistant paints contain II or  
chlorinated rubber. The problem of adhesion on galvanized  
Fe may be solved by Zn dust as pigment. Impermeability  
of synthetic-resin house paints causes them to peel off as  
 $\text{H}_2\text{O}$  works its way out. This difficulty is increased by  
modern wall construction and air conditioning but may be  
solved by priming both sides of clapboard in the mill.  
Paints contg. I are easily cleaned and do not change color.  
Durable "self-cleaning paints," in which a slow chalking  
carries away the dirt, have now been formulated.

Arnold M. Collins  
Modern work on pigments. Lehren *Farbe u. Lack*  
1935, 547 —The importance of particle size and shape and  
methods for overcoming gelling and settling difficulties  
are briefly discussed J. W. Perry

Pigments and agents for extending pigments to be used  
in oil paints and enamels H. Rasquin. *Farben-Ztg.* 40,  
1038-9 (1935) —The place of pigment extenders in the  
dry color industry is discussed Cf. C. A. 29, 4189

J. W. Perry  
The nature and characteristics of lakes and pigments.  
H. Samuels. *J. Oil & Colour Chem. Assoc.* 18, 375-400  
(1935) —A review of org. and inorg. pigments, their  
manuf. and uses J. W. Perry

Processes of inspissation of mixtures of pigments in  
oil A. V. Pamfilov, E. N. Roslyakova, A. S. Khudyakov  
and A. A. Blagodarova. *Trans. Inst. Chem. Tech.  
Jenovo* (U. S. S. R.) 1, 143-57 (1935) —The processes  
of thickening (livering) of oil-pigment mixts. are highly  
complex and are, probably, produced by many causes.  
Examn. of mixts. of  $\text{ZnO}$ -linseed oil disclosed the in-  
adequacy of the conception of livering as a result of the  
formation of a metallic soap proposed by Fischer and Suer  
(C. A. 26, 5219). The process of soap formation proceed-  
very slowly and depends not on the reaction of a metallic  
base with a free acid constituent of the oil but on the  
decompn. of the glyceride. Chas. Blanc

Inks for printing currency and securities Karl H Bettner *Am Ink Maker* 13, No 11, 15-17 (1935) — A review L H

Oils of interest for enamels and lacquers H. Ulrich *Farben-Zig* 40, 1037 (1935) — A discussion of the possibility of using dehydrated castor oil, oiticica oil and specially treated oils (prepd by heat-treating linseed oil, fish oil or soybean oil with or without simultaneous air blowing) as components of protective coating compns J W Perry

Reduction of linseed oil consumption in the production of linoleum A V Pamsilov and E G Ivancheva. *Trans Inst Chem Tech Ivanovo (U S S R)* 1, 139-42 (1935) — Formulas are given for the substitution of 50% of Ca salts of naphthenic acids for linseed oil in the production of oilcloth Chas Blanc

Fish-oil stand oil as a substitute for linseed-oil stand oil Gottfried Kaempfe *Farben-Zig* 40, 1009 (1935) — Fish-oil stand oil, prepd by removing the saponifiable and less highly unsaturated components from fish oil and simultaneously heat-treating the remaining highly unsaturated constituents, has excellent drying properties and is well suited for use in paints and varnishes, either with or without admixt. of other stand oils J W Perry

The moisture sensitivity of linseed oil coats W. H. Droste. *Angew Chem* 48, 644-7 (1935) — Investigations were carried out on the weathering of coats shortly after application. The addn of diluents results in a loosening of the coat and an increase of the sol part. Addn of 20% of polymerized oil to 80% of linseed oil did not give any notable improvement. The sol substance of inert pigment contains free split acids. The amt of sol matter is influenced by sol salts which are probably present in the pigment. The metals contained in the drier also go partially into soln. The sol matter in basic pigments is not smaller than that in inert pigments. The acids going into soln are completely neutralized by the basic components of the pigment, except with white lead. Zinc white and its mixts behave alike, mainly Zn soaps being dissolved, and their soly does not change with increasing age of the coat. The sol part in a two-day-old coat of red lead, however, is very great, but decreases considerably after 28 days, and the soly of Pb soaps decreases parallel with it. White lead does not show this behavior in such a marked manner, as it cannot neutralize the resulting free acids as fast on account of its lower basicity. The test results have definitely shown that sol substances are formed in all linseed oil coats, independently of the choice of the pigment. The amt of sol substance increases if unfavorable weather conditions exist during the first drying period. After these substances are removed by becoming washed out, further exposure of the film to the air results in the renewed formation of sol substance, whose amt is again a function of the weather conditions. Exptl results are presented and theoretical considerations are discussed. Four references. Karl Kammermeyer

Heat treating oiticica oil. H Kemner. *Farbe u Lack* 1935, 545-6 — Heating oiticica oil to 250° for 15 min yielded a stand oil having optimum properties. The tendency to "wrinkling" may be further reduced by addn of resins, litharge or glycerol before or during the heat treatment. Oils treated with glycerol yielded films very sensitive to water J W Perry

The use of the "Höppler" viscometer for oils, lacquers and varnishes D Cannegieter *Verfahrenstechnik* 8, 252-4 (1935) — The construction, use and advantages of the Höppler viscometer are discussed J W Perry

Pressing of flaxseed with the use of Andersen forepress A. Tykin. *Maslobolno Zhirnoe Delo* 11, 470 81 (1935) — The Andersen press was found unsuitable for the first stage of pressing of flaxseed in the 2-stage process Chas Blanc

The cultivation and processing of flaxseed R Heublyum. *Seifensieder-Zig* 62, 689-90, 720-1 (1935) — A review of recent progress J W Perry

The purification and standardization of kauri gum J R Hosking. *New Zealand J Sci Tech* 17, 369-87 (1935) — Aside from the direct effects of the general eco-

nomic depression the decrease during later years in the consumption of kauri gum is a result of its high cost compared with that of other natural resins, nonstandardization, doubt by consumers as to the continuity of supply, competition with artificial resins and the demand for quick-drying varnishes. The main uses of kauri gum are discussed. The better grades are used for oil varnishes and the poorer grades in the manu of linoleum, the latter industry absorbing about 70% of the output. In regard to the conditions detg the future of the kauri gum industry the conclusions are drawn that (1) the continuity of the kauri gum industry depends on the utilization of the lower grades of gum, (2) a much purer product is demanded than has hitherto been produced; (3) the lower grades should be standardized and (4) the price of the standardized material should be kept low. Ests indicate that there is at least as much kauri gum remaining in the soil as has so far been produced. Present methods of purification are described (MacLaurin's salt vacuum process, the Hoyland process and the Buchan process). Solvent extrn produces a purer and more homogeneous product than flotation. An improved solvent process is described for the utilization of low-grade kauri in the production of gum of approx 93.5% resin content and possessing all the properties of a good-grade fossil gum. A mixt of 7 parts benzene and 3 parts acetone was found to be a rapid working solvent at 50° for kauri gum of most ages and allowed a quick settling of the impurities. A detailed diagram of the mfg process is given. The purified kauri gum is better suited to the requirements of the linoleum industry and has a wider field of utilization than unpurified grades D Thuesen

Mechanical properties of plastic films A Vila and F. Tesson. *Compt rend* 201, 1031-3 (1935) — An arrangement is described for testing the viscous flow on stretching and elasticity of films of paints, etc., as formed on surfaces for protection, after subjection to artificial aging (e.g., by V.'s method, *C. A.* 29, 6141). The results are shown by curves, and indicate a red ochre paint to give the most, tar contg Al the least, elastic film, while the tar and a red lead paint showed the greatest viscous flow

C A Suberrad  
Evaporation of lacquer solvents W. K. Lewis, Lombard Squires and C E Sanders. *Ind Eng Chem* 27, 1395-6 (1935) — Exptl results show the inaccuracy of the assumption that a solvent mixt of const b p. at the drying temp of the film is a const-evap mixt. The mechanism of the evapn. is studied W. H. Boynton

Various nitrocellulose lacquers. A Kraus. *Nitrocellulose* 6, 199-201 (1935) — A review. E. M. Symmes

Phenolic resin varnishes. Influence of linseed oil on film properties V. H. Turkington, Robert J. Moore, W. H. Butler and R. C. Shuey. *Ind Eng Chem*, 27, 1321-5 (1935), cf. *C. A.* 25, 423, 4419 — The properties of varnishes made under comparable conditions from varying proportions of Bakelite (I), China wood oil (II) and linseed oil (III) were systematically investigated and data presented in triangular diagrams. Increasing I increased drying rate, durability (as detd by both checking and loss of gloss), and resistance to NaOH and boiling H<sub>2</sub>O. III could be substituted for II without much change in durability or H<sub>2</sub>O resistance (blistering) but drying time increased (particularly in long oil formulas) and NaOH resistance decreased (particularly for short oil varnishes). Arnold M. Collins

Synthetic varnishes and enamels, their manufacture, working and uses Tr. Kolke. *Farben-Zig* 40, 1198-9, 1220-1 (1935) — A general discussion of the use of synthetic resins in protective coating compns J. W. P.

Electrochemical methods in varnish and pigment industry A V. Pamsilov. *Trans Inst Chem Tech Ivanovo (U. S. S. R.)* 1, 132-8 (1935) — The progress made in the application of electrochem methods in the lab control and in the production of mineral pigments is reviewed Chas Blanc

"Wrinkling" of wool-oil varnishes. The effect of the drier H. Wolff and G. Zeidler. *Farben-Zig* 40, 1010 (1935) — The wrinkling of a no. of varnishes, which con-



tained the "wrinkling"-preventing agent "Mittel 109 J," and which were prepd. from various resins and an improperly heat-treated China wood oil, was more severe with a Mn-Pb combination drier than with a Co drier.

**J. W. Perry**  
**Varnish-blending graph.** R. C. Shuey. *Ind. Eng. Chem.* 27, 1391-4 (1935)—A graphical method is described for detg. wt percent and wt.-vol relationships of the volatile and nonvolatile components of oleoresinous varnishes. See samples and their soln. are given.

**W. H. Boynton**  
**Natural resins for the varnish industry.** C. L. Mantell, C. H. Allen and K. M. Sprinkel. *Ind. Eng. Chem.* 27, 1369-73 (1935)—The natural varnish resins are classified, their grading is explained, and their properties are tabulated. Methods of pretreatment for varnish making are discussed, including mastication (cf. C. A. 28, 2550). Wider use is recommended because of uniformity (controlled by government supervision), steady supply, cheapness (av. 5-7 cents/lb.), freedom from patent domination, and performance equal to best synthetic resins, in China wood oil varnishes. Arnold M. Collins

**Corrosion problem—paints and anticorrosive coatings (Genin)** 9 **Tarnishing of gilt inks of the bronze-powder type when in contact with paper (Scherholtz)** 23 **Action of alk. solns. on ultramarine blue (Leschewski, Podschus)** 2 **Plastic compns. (in prepn. of linoleum and oil-cloth) (Brit. pat. 433,994)** 13. **Azo dyes [for coloring lacquers] (Fr. pat. 787,000)** 25. **Acetyl compds. [as starting materials for paints] (Ger. pat. 617,080)** 10. **Disazo dyes [for coloring lacquers] (Ger. pat. 620,258)** 25.

**Apparatus for filtering paints.** Pierre L. T. J. Petit. *Fr.* 780,543, Sept. 5, 1935

**Apparatus for demonstrating reflecting properties of surface finishes such as paints.** Earl L. Canfield. *U. S.* 2,022,402, Nov. 26. **Structural details**

**Pigment dispersion in nitrocellulose.** Harry E. Stone. *U. S.* 2,021,949, Nov. 26. A mixt. of a black carbon pigment, nitrocellulose, Cu butyl phthalate and a plasticizer such as tricresyl phosphate is subjected to a roll-grinding process to produce a mass which is hard and brittle at room temps. and is suitable for use in making lacquers.

**Titanium compounds; pigments.** **Titangesellschaft m. b. H.** *Brit.* 433,960, Aug. 23, 1935. See *Fr.* 753,585 (C. A. 29, 6261).

**Zinc pigments.** I. G. Farber and A. G. (Elkbert Lederle and Max Günther, inventors). *Ger.* 618,868, Sept. 17, 1935 (Cl. 22f 5). Addn. to 603,113 (C. A. 29, 5631). Solns. of Zn salts generally are treated simultaneously with an alkali sulfide (or (NH<sub>4</sub>)<sub>2</sub>S) and NaOH (or soda), and the pptts. are washed and dried.

**Zinc sulfide pigment.** Harlan A. Depew (to American Zinc, Lead & Smelting Co.). *U. S.* 2,021,690, Nov. 26. Steam is mixed with a ZnS pigment to be purified and the material is passed into a furnace and there treated with a current of inert gas and the pigment is compacted into a seal within the furnace at the point where it enters the furnace so that the volatilization of the volatile material creates a gas pressure which assists in the sealing effect. App. is described and the furnace treatment serves to remove S-O acid components. *U. S.* 2,021,691 relates to app. of the same character and use.

**Chrome yellows.** Elkbert Lederle and Max Guenther (to General Aniline Works). *U. S.* 2,023,928, Dec. 10. A chrome yellow stable to light is obtained by pptg. a

material comprising neutral Pb chromate and at least one salt such as Pb sulfate crystd. in rhombic crystals, capable of forming with Pb chromate an isomorphous series of mixed crystals and capable of being converted into a monoclinic modification, and heating the material suspended in an aq. medium at a temp. above the conversion point at which conversion into the monoclinic modification occurs until such conversion is substantially complete. A temp. of about 60° for 2 hrs. is suitable. Several examples are given.

**Drying oils.** N. V. Koninklijke Pharmaceutische Fabrieken v/h Brocades-Scheerman & Pharmacia. *Ger.* 617,728, Aug. 24, 1935 (Cl. 22h. 2). **Drying oils, together with asphalt-like products,** are obtained in the purification of mineral and tar oils with H<sub>2</sub>SO<sub>4</sub>. The oils are treated with excess of concd. H<sub>2</sub>SO<sub>4</sub> at about 50° and the resulting sulfonic acids converted to their salts. These are dry-distd., leaving an asphalt-like residue. The oils are purified by washing with NaOH lye and finally vacuum distd. to give a drying oil. An example is given.

**Rosin drying oil.** Robert C. Palmer and Paul O. Powers (to Newport Industries, Inc.). *U. S.* 2,022,978, Dec. 3. Partially esterified wood rosin is heated to about 260-325° with fuller's earth until the acid value of the mixt has been considerably reduced. The product can be used in printing inks and varnishes. *U. S.* 2,022,974 relates to the use of a product of like character in bronzing-ink vehicles by admixt. with about 10-30% of a high-viscosity refined petroleum oil, a liquid ester of phthalic or abietic acid, castor oil or tricresyl phosphate as a softening agent. Such mixts. form a nonbrittle film.

**Coating compositions containing drying oils.** Robert L. Sibley (to Rubber Service Laboratories Co.). *U. S.* 2,022,301, Nov. 26. A dimethylhydroquinoline or other alkyl substituted hydroquinoline is employed in varnishes or the like contg. drying oils such as tung oil to control oxidation. *U. S.* 2,022,302 relates to the similar use of a hydroacridine such as methylidihydroacridine.

**Nitrocellulose lacquers.** Charles Bogn and Vaughn Kelly (to Commercial Solvents Corp.). *U. S.* 2,023,363, Dec. 3. For preventing gelling, livering or agglomeration of nitrocellulose lacquers contg. basic Zn pigments and esters of lactic or hydroxybutyric acid, the lacquers are mixed with 0.1-1.5% of a hydroxy "polybasic" aliphatic org. acid such as citric, tartaric or malic acid or with an alkali metal salt of such an acid.

**Lacquer containing synthetic resin.** Melville M. Wilson (to Sharples Solvents Corp.). *U. S.* 2,022,331, Nov. 26. A lacquer which is free from bluish on drying contains an oil-sol. resinous condensation product formed from tertiary amyl phenol and a CH<sub>2</sub>-contg. substance, a vehicle comprising a hydrocarbon oil, a cellulose ester such as cellulose nitrate, a relatively small proportion of a dialkyl phthalate, and an ester gum or polyhydric alc. "polybasic" acid resin in a quantity at least two-thirds that of the first-mentioned condensation product. Several examples are given.

**Printers' varnishes.** Walter K. R. Hartmann and Carl P. L. Hartmann (trading as Chemische Fabrik Halle-Ammendorf Gebr. Hartmann). *Brit.* 434,450, Sept. 2, 1935. In a process for the production of high-gloss overprints, an overprinting varnish is used comprising a drying oil, an oil-sol. natural or synthetic resin and a small amt. of an oil-swellable org. salt of Mg, alk. earth or earth metal, e. g., soaps of these metals. Driers may be added and the varnishes may be colored by Victoria blue base, Rhodamine base, Sudan dyes, Miliori blue, Fanal green, Permanent red, etc. The org. salts may be formed in the varnish by adding separately, e. g., Al(OH)<sub>3</sub> and stearic acid.

## 27—FATS, FATTY OILS, WAXES AND SOAPS

E. SCHERUBEL

**Thermal properties of fats.** M. Ravich. *Maslobojno Zhivooe Delo* 11, 376-8 (1935).—Thermal properties of oils and fats for the production of margarine are discussed. Chas. Blanc

**New methods for producing animal fats.** R. Strauss *Fettechem. Umschau* 42, 190-3, 209-13 (1935).—A review of recent progress with 31 references. J. W. Perry  
**Saponification of fats with formation of concentrated**

glycerol waters M. Nikiforov. *Masloboino Zhirnoe Delo* 11, 355-7 (1935) — A procedure is described for a repeated use of glycerol waters in the sapon of fats with kerosene as a contact agent Chas Blanc

Rapid method for factory control of fat saponification G. Klein, N. Kaminski and P. Yunchman. *Masloboino Zhirnoe Delo* 11, 502-3 (1935) — Withdraw a sample of sapon fat at 70-90° with a 5-cc pipet, heated to 60-70°, into a 200-cc tared flask, allow to cool and weigh. Add 100 cc of satd NaCl at 60-70°, 30 cc. of refined sunflower oil at 60-70° and 3 drops of 1% methyl orange, and titrate H<sub>2</sub>SO<sub>4</sub> with 0.5 N NaOH. Introduce into the mixt. 10 drops of 10% phenolphthalein and titrate with 0.5 N NaOH with shaking. The percentage of sapon =  $(a - b) \times 0.141 \times 100/H$ , where  $H = g$  of sample,  $a = cc$  of 0.5 N NaOH consumed in the titration of fatty acids, 0.141 = titer of 0.5 N oleic acid (the mol wt. of oleic acid was accepted as equal to that of all the fatty acids);  $b$  = correction for the acidity of the 30 cc. of sunflower oil (determined by a similar procedure). The detn is made in 5 min with an accuracy to 1% as compared with the standard method Chas Blanc

The determination of the acetyl number of fats II. Carrying out the acetylation. K. Tafel, M. de Mingo and H. Thaler. *Fettechem Umschau* 42, 141-4 (1935); cf. C. A. 28, 7565<sup>1</sup> — In order to select the most suitable method for quantitatively and selectively acetylating the hydroxyl groups of fatty substances, the acetylation of tetrahydroxystearic acid (I) m 156°, hexahydroxystearic acid (II), dihydroxystearic acid (III), castor oil (IV) and grape seed oil (V) were studied by (Method A—cf. *Essentielle Untersuchungsmethoden für die Fett- und Wachstumsindustrie*, 2nd Ed. Stuttgart, 1930, page 90) refluxing with Ac<sub>2</sub>O and petr ether or, more preferably, Et<sub>2</sub>O, by (Method B—cf. Franchimont, *Compt rend* 89, 711-13 (1879)) warming the substance dissolved in Ac<sub>2</sub>O contg. a small amt of H<sub>2</sub>SO<sub>4</sub> to a temp not exceeding 45°, dissolving the reaction product in ether, washing the ether soln successively with an aq 50% AcOH soln, a satd NaCl soln contg. KHCO<sub>3</sub> and a satd NaCl soln, then drying the ether soln over anhyd Na<sub>2</sub>SO<sub>4</sub>, and finally distg off the ether and drying over H<sub>2</sub>SO<sub>4</sub> in a vacuum and (Method C—cf. Verley and Bolsing, *Ber* 34, 3351-62 (1901)) by heating the substance on the water bath with an excess of Ac<sub>2</sub>O and a small amt of pyridine, pouring the reaction mixt. into water, taking up the acetylated product with ether and proceeding as outlined under Method B. In all cases the bound AcOH was detd. by Freudenberg's method (C. A. 18, 234), which had been previously shown (C. A. 28, 7565<sup>1</sup>) to be best suited for this purpose. Method B gave the most readily reproducible and most nearly theoretical results with I, II and III, while methods A and C were distinctly inferior. The 3 methods gave fairly concordant results with IV but not with V. This was due, perhaps, to the relatively large amts of free fatty acids in V as indicated by an acid no. of 48.2. This surmise was supported by expts showing that free fatty acids, especially those which are not too high up in the homologous series, e. g., lauric, react with Ac<sub>2</sub>O under the conditions of methods A, B and C to form mixed anhydrides which do not decompose rapidly on contact with water. The pos. error caused thereby is apt to be more serious than is commonly realized. Method B is recommended as the best suited for fat investigations. An improved prepn of III based on Hilditch's work (C. A. 20, 3280) is described J. W. Perry

Oiticica fat and its fundamental difference from China wood oil C. P. A. Kappelmeier. *Fettechem Umschau* 42, 145-52; *Verftroniek* 8, 279-83 (1935) — Various samples of oiticica fat (I), obtained either direct from Brazil or extd from freshly ground nut kernels preferably with ether or benzene or, less suitably, with petr. ether, had the following consts: acid no. 2-8, sapon. no. 187-193, I no. (Wij's—after reacting 2 hrs.) 140-150, Ac no. 30-40, hexabromide test neg., gelatination temp. 260-70°,  $d_{4}^{20}$  0.9670-80,  $n_D^{20}$  1.5140-80,  $n_D^{25}$  =  $n_D$  (dispersion) 0.0170-0.0185. (Since I ordinarily has a buttery consistency, the last 3 consts were detd. on melted, supercooled samples—

a procedure which is not recommended.) Removing the break from I by rapidly heating to 200° and then centrifuging failed to prevent I from reassuming its buttery consistency at room temp, although this can be prevented by converting I into stand oil by heating to 225-50°.  $\beta$ -Couepic acid (II) was prepd by first adding a small amt of I to a benzene soln. of the free fatty acids of I and then exposing to direct sunlight. The fine leaflets of II, which sepd out, m 101-2° after recrystn from acetone and vacuum drying. The ketone group in II, which was revealed by the formation of a semicarbazone, m. 134-7°, seems to have a disturbing effect on acidimetric titrations and also on I no. detns. Whatever the cause, the apparent I no. of II (Kaufmann method) increased from 194 to 332 and a small amt of free acid appeared on increasing the time of halogenation from 2 to 190 hrs. Hydrogenation studies offer more promise in this field than halogenation expts.  $K$ - $\beta$ -couepate is a stable, cryst. salt, whose ready crystn from alc sapon mixts. aids greatly in detecting II. Ammonium- $\beta$ -couepate m 127° and gives no color with FeCl<sub>3</sub> soln. Catalytic hydrogenation converted II into hexahydrocouepic acid (III) which yielded a semicarbazone m. 135.5°. Me- $\beta$ -couepate (IV) m 41°, and behaved like II when halogenated. A comparison of the consts. of IV and of freshly prepd samples of the Me esters of  $\alpha$ - and  $\beta$ -eleostearic acid confirmed the non-identity of IV with either of the latter. Nevertheless I may contain varying amts of eleostearic acids (cf. van Loom and Steger, C. A. 25, 4843). A mixed m p indicated that IV is identical with the hexahydrocouepic acid of Brown and Farmer (C. A. 29, 5294<sup>1</sup>). K. criticizes Rossmann's theory (C. A. 26, 2416) that only 2 geometrically isomeric eleostearic acids can exist. J. W. Perry

Washing fats after refining M. Sandomurski and O. Mikhailovskaya. *Masloboino Zhirnoe Delo* 11, 357-9 (1935) — All traces of soapstock are removed from oils after neutralization by 2-3 washings with 0.25% H<sub>2</sub>SO<sub>4</sub>. The mixt. is easily sepd into 2 layers without forming an intermediate layer. The losses of oil are reduced as compared with water washing Chas Blanc

Water solubility and washing of sulfuric acid esters of unsaturated fats C. Steiner. *Fettechem Umschau* 42, 201-5 (1935) — Castor oil, olein (I), linseed oil (II), oleyl alc (III) and peanut oil were sulfonated by slowly adding varying amts of H<sub>2</sub>SO<sub>4</sub> to 100-g. portions of the fatty materials held at 28-30°. Except for a few samples of II which were sulfonated with a 92% acid, 98% H<sub>2</sub>SO<sub>4</sub> was used. As a general rule, increasing the amt of H<sub>2</sub>SO<sub>4</sub> used caused an increase in organically bound SO<sub>3</sub>. This increase, in turn, was paralleled by a tendency for the sulfonated fatty material to form, with hot water, less and less turbid emulsions. Clear solns were obtained only with highly sulfonated I and III. The neutralization nos. of the sulfonated fat samples were detd. and their behavior on washing with a 10% Na<sub>2</sub>SO<sub>4</sub> soln. was studied J. W. Perry

The spoiling of fats and biochemical reactions involving fats K. Tafel. *Fettechem Umschau* 42, 164-8 (1935) — This review covers a no. of recent developments which reveal possible relationships between the oxidation of unsatd fatty substances and the  $\beta$ -oxidation of fats *in vitro* on the one hand and the mechanism of fat metabolisms and similar biochem. reactions on the other. Glyceride hydrolysis is also discussed both as a purely chem. and as a biochem. process. Over 30 references. J. W. Perry

Acidic components of wool grease E. E. U. Abraham and T. P. Hilditch. *J. Soc. Chem. Ind.* 54, 393-404T (1935) — The acidic components of fresh neutral wool grease, wool grease from waste liquors and wool grease obtained by solvent extn. of merino wool have been sepd and, as Me esters, submitted to fractional distn. in vacuum. The acids present in the distd. Me ester fractions and residues were crystd. to obtain individual components. The results confirm those of earlier workers in showing that the chief component acid is a wax like solid, m 73-5°, which is not "cerotic" (hexacosic) acid, but is of similar compn (C<sub>26</sub>H<sub>52</sub>O<sub>2</sub> or C<sub>27</sub>H<sub>54</sub>O<sub>2</sub>, less probably C<sub>28</sub>H<sub>56</sub>O<sub>2</sub>).

There is also evidence of the presence of a series of acids represented by the formulas  $C_{15}H_{31}O_2$ ,  $C_{17}H_{33}O_2$ ,  $C_{19}H_{37}O_2$ ,  $C_{21}H_{39}O_2$  (or  $C_{21}H_{39}O_2$ ,  $C_{23}H_{41}O_2$ , and  $C_{25}H_{43}O_2$  for  $C_{21}H_{39}O_2$ ). The acid of lowest m. p. (about 22°) and greatest soly. was not susceptible to purification; in its impure condition it gave analytical figures close to those required by the formula  $C_{15}H_{31}O_2$ . The acids of formulas approx  $C_{17}H_{33}O_2$ ,  $C_{19}H_{37}O_2$ , and  $C_{21}H_{39}O_2$  were obviously identical, from their m. p. and other properties, with the "linopalnic acid," "lanocenic acid" and "lanocenic acid lactone" described by Darmstadter and Lischutz (Ber 31, 1122 (1898)). Evidence of "lactone" formation was observed when the wool grease acid fractions of high mol. wt. were boiled with dil. mineral acid. Acids of the normal aliphatic series are not present in wool grease. It is suggested that the acids of sterol waxes secreted in sebaceous and similar glands are probably derived from an isoprene or terpene foundation rather than from an unbranched chain of C atoms. E. Scherubel.

Hydrogenation of fats in presence of nickel carbonate and formate reduced in the oil. L. Eimburg and M. Popov. *Masloboino Zhirovoe Delo* 11, 363 (1935). Cf. C. A. 29, 8309, 8373.  $NiCO_3$  can be used in the hydrogenation of high-grade oils with low-pressure steam for heating the oil before hydrogenation. A mixt. of  $NiCO_3$  and  $Ni$  formate can be used for the hydrogenation of low-grade oils when effective refining of the oil is not feasible. Chas. Blanc.

Hydrogenation of fats with copper-nickel catalyst. V. Yasul'ev. *Masloboino Zhirovoe Delo* 11, 444-8 (1935). — Exptl. evidence of the advantages of using Cu-Ni catalyst in the hydrogenation of refined and crude oils confirms the results of U. S. practice. Chas. Blanc.

Selective hydrogenation (of vegetable fats). L. Eimburg, B. Sterlin and B. Krushchinskii. *Masloboino Zhirovoe Delo* 11, 471-3 (1935). — The selective process of hydrogenation of cottonseed and sunflower oils was studied by heating the oils in an autoclave at 160°, 180° and 200° with 0.1, 0.2 and 0.4% Ni pptd. on kieselguhr and a H current at a rate of 2, 3, 4 and 6 l./min. The samples taken at definite intervals were tested for the m. p., I and thiocyanate nos. and contents of solid acids. The selective process of hydrogenation was detd. by the contents of solid acids formed and the linoleic acid unchanged. Under all conditions of hydrogenation of cottonseed oil the selectivity was greater at a higher temp. (200°). With the increased ratio of either Ni or H the rate of hydrogenation increases and the degree of selectivity correspondingly declines. A selective hydrogenation can be effected not by any definite optimum system but by an optimum correlation of the single factors in the process. A min. of the catalyst and a max. of H are required; the converse is true. At a certain correlation of the catalyst and H the temp. of the reaction can be lowered without affecting the selectivity of hydrogenation: in the presence of 0.1% and 0.15% Ni (based on the wt. of oil) and H rate of 4 l./min. the selectivity is of the same character as that at 180° (for 0.1%) and 160° and 180° (for 0.15%). The same is true with 0.2% Ni and H rate of 3 l./min. For the selective hydrogenation of sunflower oil more active conditions are required. Thus, at a rate of 6 l./min. of H the selective character of hydrogenation is greater than that of cottonseed oil. Chas. Blanc.

Conversion of cottonseed oil into a lard-like fat mixture by the method of conjugated hydrogenation. V. Puranov and G. Ivanova. *Masloboino Zhirovoe Delo* 11, 365-8 (1935). — By the method of Lyubarskii (C. A. 27, 1533) of catalytic conjugated hydrogenation an appropriate mixt. of cottonseed oil and EtOH was autoclaved in the presence of Al-Ni (72% Al, 28% Ni) at 190-200° and 23-7 atm. for 30 min., giving a snow-white fat mixt. of normal taste with the compn. and consistency of lard, acidity 3.73 mg. m. 32.2° I no. 74.2 and Hehner no. 66. Chas. Blanc.

Effectiveness of hydrogenation with the use of nickel formate at the Kazansk works. A. Yasul'ev. *Masloboino Zhirovoe Delo* 11, 474-6 (1935). — The advantages of hydrogenation of vegetable oils in the presence of Ni formate are

discussed from the economical and tech. viewpoints.

Chas. Blanc.  
Continuous hydrogenation at the Gor'kov works. A. Panushev. *Masloboino Zhirovoe Delo* 11, 466-8 (1935). — Results of 3 years' experience of continuous hydrogenation of cottonseed and sunflower oils are tabulated and discussed. Chas. Blanc.

Production of oils without sediment. A. Raspopina. *Masloboino Zhirovoe Delo* 11, 482-3 (1935). — The method of Zinov'ev (C. A. 22, 2675, 24, 4408) for drying oils is suggested as a possible method for the production of sunflower oil with min. sedimentation. Chas. Blanc.

Hydration of sunflower oil. M. Bauman, I. Grabovskii, F. Vishnepol'skaya and G. Gushchin. *Masloboino Zhirovoe Delo* 11, 477-9 (1935). — The best results in clarifying sunflower oil were obtained by stirring the oil, pressed or non-pressed, at 70° first with 1% of 3%  $NH_4Cl$  for 20 min. and then with 1% of 0.25%  $HCl$ , followed by salting out with 2.5%  $NaCl$  at 68°, settling for 16 hrs. and filtering at 40°. The product after 8-10 months of storage formed only 1-1.5% of white sediment and showed no change in acidity. Chas. Blanc.

Hydrogenation of black oils. V. Chervyakov. *Masloboino Zhirovoe Delo* 11, 364-6 (1935). — Methods of refining and hydrogenation of hempseed, linseed, coriander and white mustard oils are described. Chas. Blanc.

Characteristics of the oils contained in different parts (skin, endosperm, cotyledons and embryos) of the seeds of *Strophanthus kombe*, *hispidus*, *gratus* and their importance in determining the time, state of conservation and quality of the seeds (pharmacognostic investigations). Luigi Tocco and Bruno Sanni. *Arch. intern. pharmacodynamie* 50, 332-8 (1935). — The oils contained in the endosperm and cotyledons of said seeds are clear, transparent, straw colored, with pungent odor and nauseating taste. Their m. p. (0-2) and the degree of acidity (2-7) are much lower than those known. In the skin one finds fatty oils; those of *S. kombe* and *hispidus* are green because of the chlorophyll. As the seeds are altered the oils become rancid. P. F. Mettild.

Polymerization of fatty oils. IX. The hydrogenation of polymerized ethyl linolenate. Alph. Steger and J. van Leen. *Rec. trav. chim.* 54, 750-6 (1935); cf. C. A. 29, 5203. — Et linolenate was prepd and then polymerized until further treatment produced no change. High-vacuum distn. gave the following fractions: (1) up to 93°, 1.4; (2) 95-100°, 30.7; (3) 100-180°, 7.2; (4) 180-200°, 21.8 and (5) residue 38.9%. Fraction 1 was composed of decompn. products. Fraction 2 did not contain polymers yet it could not be hydrogenated to Et stearate. The said acid derived from fraction 2 after hydrogenation is attacked by Bertram's  $K_2MnO_4$  soln. This indicates the acid is not a fat acid. From the hydrogenated product of fraction 2 an acid whose formula corresponded to  $C_{17}H_{33}O_2$  was sep'd. by the Twitchell Pb salt method. These properties together with physical characteristics indicate a compd. of cyclic structure. The fractions 4 and 5 contained the polymers. These could not be hydrogenated to an I. no. of 0. Oxidation of the hydrogenated product of these fractions according to the Bertram method yielded a residue of cyclic compds. Conclusion: By polymerization of Et linolenate at 300° in a current of  $CO_2$  2 reactions take place: (a) A polymerization and (b) a cyclization of linolenic acid into a nonpolymerizable compd. which after hydrogenation corresponds to the formula  $C_{17}H_{33}O_2$ . Both reactions occur simultaneously until no linolenate remains. This explains why linolenic acid cannot be completely converted to polymers. X. The polymerization of ethyl linolenate. *Ibid.* 756-9. — Et linolenate was polymerized at 300° for 6, 10 and 14 hrs. I. no. (Wijs and Br methods), thiocyanogen no. and  $\eta_{sp}^c$  are reported on each product. The Wijs I. no. with varying periods of contact, thiocyanogen no. and  $\eta_{sp}^c$  are reported on the high-vacuum distillate to 100° and the residue of each product. Substitution of I was evident in the residues and was greater with longer polymerization time; while in the distillate substitution of I was not detectable. Conclusion: High polymers and low mol.-wt. isomers of Et

linolenate are formed on polymerization. The high polymers increase with the time of heating. M. M. Piskur

Fatty acid standards. British Standards Institution *Chemist and Druggist* 123, 631 (1935).—Recently published specifications for coconut oil, peanut oil, olive oil, rapeseed oil and raw linseed oil are quoted. S. Waldbott

Factors determining color of cottonseed oil. A. Goldovskii, M. Podolskaya and L. Mirer. *Moskoboino Zhivoe Delo* 11, 442-4 (1935); cf. C. A. 29, 83764. C. B.

Refined olive oil from pulp, in comparison with natural and refined, pressed olive oils. Daniel Mangrane. *Analges soc. esp. fis. quim.* 33, 707-79 (1935).—Methods of detg the S content, physicochem. consts. and compn. of olive and S oils are reviewed. S is best controlled in refining of S oils by heating 20 min. 50 cc. of oil with 50 cc. of 25% aq. NaOH, with a Ag foil immersed therein, then observing the S stain on the Ag. This test is sensitive to 0.5 parts of S per 10,000. The development of H<sub>2</sub>S in some apparently well-refined S oils was investigated and confirmed in S oils contg. 3-4 parts of S per 10,000. Pure CS<sub>2</sub> does not attack olive oil. The S in S oils originates in the S dissolved in com. CS<sub>2</sub>, which should not exceed 8 parts per 10,000. By deodorizing by acid and superheated steam, the S content may be reduced to 1.5-2 parts per 10,000. H<sub>2</sub>S dissolved in CS<sub>2</sub>, and sulphydrates, also attack olive oils. Olive residues give a strong S test. Pressed oils and extd. oils have a S content not exceeding 1 part per 10,000. Intensity of the S test increases with ripening of the olives. Oils from wormy olives give a stronger test, and the S content of the worms is rather high. S can add to the unsatd. bonds of the fatty acid. The phys. and chem. const. of both oils are always within the same range. Various empirical tests for certain fat acids are described, and no appreciable difference is found between the 2 oils. Pressed oil shows a min. soap content; refined oil shows none. Peroxidation of some substances during the deodorizing process is studied. The resin content is higher in refined S oils. E. M. Symmes

A note on the spectroscopic determination of the vitamin A content of pichard oil. G. M. Shrum and Thomas G. How. *Can. J. Research* 13A, 93-8 (1935).—An attempt has been made to measure spectroscopically the vitamin A content of pichard oil. By use of the same conversion factor as for cod-liver oil, measurements of the extinction coeff. at 3280 Å. gave vitamin A values much larger than those obtained by biol. tests. It has been shown that this discrepancy cannot be wholly ascribed to either the coloring matter or the saponifiable material in the oil. A satisfactory check on the results can be obtained only when it is possible to remove completely the vitamin A from the oil, without otherwise modifying it. J. W. S.

Determination of color of sunflower oil. M. Sokolova and I. Engel. *Moskoboino Zhivoe Delo* 11, 481-2 (1935).—The method of Fomrobert and Pallaut (*Farben-Zig* No. 17, 967 (1926); cf. C. A. 20, 2421) for detg coloration of oils was applied to the estn. of the quality of sunflower oil. Chas. Blanc

Reconstruction of Krasnodarski oil works. A. Skupia, I. Morozov and A. Grashin. *Moskoboino Zhivoe Delo* 11, 435-42 (1935).—Construction and operation details of refining and reworking of vegetable oils are discussed. Chas. Blanc

Unsaturated acids of natural oils. IV. The highly unsaturated acid of *Telfaria occidentalis*. C. H. Farmer and E. S. Pace. *J. Chem. Soc.* 1935, 1630-2; cf. C. A. 29, 60851.—Freshly extd. oil from the kernels of *Telfaria occidentalis* contains  $\alpha$ -eleostearic acid, m. 48°, after exposure to diffused light for some weeks very small yields of the  $\beta$ -isomer are isolated, the yield being greatly increased after exposure to ultraviolet radiation for some hrs. A sample of oil after standing 7 months gives 10% of the  $\beta$ -isomer. The solid material pptd. from the oil on standing is  $\beta$ -eleostearin. V.  $\alpha$ - and  $\beta$ -Licanic acids. W. B. Brown and E. H. Farmer. *Ibid.* 1632-3.—Com. oiticica fat and the kernel oil of *Licania rigida*, both of which normally yield  $\alpha$ -licanic acid (I) on sapon, give after irradiation in the presence of a trace of I or of S the iso- or  $\beta$ -acid (II), m. 99.5°, identical with that form-

erly isolated from liquid (isomerized) oiticica oil; the *semicarbazones* of I and II m. 110-11° and 138° (slow heating), resp.; with boiling alc. the former is transformed into a compd. m. 127°. The said acids in the oil are palmitic and stearic, 1 and 1.4% being isolated (sepn. probably far from quant.). C. J. West

New method for determining acid number of oils. Fedchenko and Kashin. *Moskoboino Zhivoe Delo* 11, 498 (1935).—The method makes it possible to obtain accurate results without the use of alc. and Et<sub>2</sub>O. Weigh a sample of oil in a flask (15-18 cm. high, 2 cm. in diam., neck bore 1 cm. in diam.), add 15-20 cc. of 10% NaCl in H<sub>2</sub>O and titrate with 0.1 N KOH in the presence of phenolphthalein to a faint rose that does not disappear with energetic shaking for 1 min. The method gave good results with black cottonseed oil. Chas. Blanc

Influence of the nature of a carrier on catalyst activity. E. Botkovskaya and P. Artamonov. *Moskoboino Zhivoe Delo* 11, 468-51 (1935).—Expts. in comparative hydrogenation of vegetable oils in the presence of Ni catalyst pptd. on silica gel and on kieselguhr and tripoli of various origins, with and without preliminary treatment with H<sub>2</sub>SO<sub>4</sub> and Na<sub>2</sub>CO<sub>3</sub>, disclosed that contrary to the general theory the best carriers are those contg. a max. of Al and Fe oxides and a min. of SiO<sub>2</sub>. Further evidence was furnished by the use of new carriers, such as clays rich in Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. Thus by the use of a clay of the compn. SiO<sub>2</sub> 59.76, TiO<sub>2</sub> 0.51, Al<sub>2</sub>O<sub>3</sub> 18.5, Fe<sub>2</sub>O<sub>3</sub> 8.1, CaO 0.79, MgO 2.66, K<sub>2</sub>O 5.08, Na<sub>2</sub>O 0.16, P<sub>2</sub>O<sub>5</sub> 0.16 and SO<sub>2</sub> 0.19%, a fat mixt., m. 61°, was obtained, i. e., a considerably greater activity of Ni than when pptd. on kieselguhr. The practical interpretation of the results is that the catalytic activity of Ni decreases with the greater contents of SiO<sub>2</sub>, sol. in Na<sub>2</sub>CO<sub>3</sub>, in the carrier and increases with its increased sol. in H<sub>2</sub>SO<sub>4</sub>. The presence of P in a carrier has no poisonous effect on the catalyst. C. B.

Purification of contact glycerol waters with barium carbonate and iron shavings. P. Andronov. *Moskoboino Zhivoe Delo* 11, 353-5 (1935).—Treating acid glycerol waters with Fe shavings and a strong air current at 90° and neutralizing the filtrate with Ba(OH)<sub>2</sub>, gave the best results with glycerol, d<sub>20</sub> 1.244, 1.14% ash and org. contents. Treating glycerol waters with BaCO<sub>3</sub> before or after neutralization with Ca(OH)<sub>2</sub> is of no marked advantage. Chas. Blanc

Products from coconut-oil wax. Smeona Santiago Tanchico. *Philippine J. Sci.* 57, 423-6 (1935).—During the storage of coconut oil a sediment seps on the bottom of the tank; and when purified the sediment forms white crystals sol. in various org. solvents. The sediment is not a glyceride but appears to be a wax contg. the myricyl ester of cerotic acid. Com. products, such as floor wax and furniture and leather polishes, were prepd. from this wax. E. Scherubel

The bleaching of Japan wax. III. Iwao Sakuma and Iso Momose. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 503-4 (1935); cf. C. A. 29, 52961.—The extinction coeffs. of the portion of com. bleached Japan wax sol. in petr. ether are smaller than those of the original product. Sunbleached Japan wax undergoes a change in color when kept in the dark and the browning is accelerated when excessive amts. of alkali are present. Too long an exposure should be avoided in sunlight bleaching. Exptl. data are given. K. K.

The properties of alkyl alkali sulfates prepared from higher fatty alcohols as detergent and wetting agents. S. Ueno, S. Yokoyama and Y. Iwakura. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 603-8 (1935).—Lauryl, oleyl and cetyl alcs. were esterified with concd. H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) and neutralized with NH<sub>4</sub>OH, KOH and NaOH, and the resulting products were compared with com. products: Gardinol, Igepon a. p. and Igepon T. The properties observed were soly., surface tension, drop no. (surface tension against petroleum), lathering power and detergent power. The exptl. results are presented and show that good detergent activity of the alkyl alkali sulfates was developed only in a completely sol. condition. The optimum condition was about 0.1-

0.25% soln. at 50°. The cetyl compd., contrary to other reports, had a high detergent power and at moderate temps., all 3 alc. derivs. showed about the same detergent power.

From the notebook of the soap manufacturer Karl Pfaff. *Rieschstoff-Ind* 10, 200-1 (1935); cf *C A* 30, 6364.—Floating and veterinary soaps are discussed.

Producing smooth, transparent soft soap with 40% hardened fat-fatty acids. *Grann Seifensieder-Zig* 62, 456 (1935).—Methods for prepg soft soap using large proportions of hardened hsh oil are described. Deodorized fish oil is less suitable because of the danger of the development of a bad odor.

Curd soap and attempts at improving its quality. *R Krings, Seifensieder Zig* 62, 739-41 (1935).—Methods for making soap under present-day German conditions are discussed.

Centrifugal method of separation of soap from fats after neutralization. *A Zinov'ev and A Vilster, Maslobolno Zhivore Delo* 11, 476-7 (1935).—Good results are reported with the use of the Sharples centrifugal separator.

The process of water evaporation by soap. *A Klyuchevich, Maslobolno Zhivore Delo* 11, 448-50 (1935).—A preliminary discussion of the causes of cracking of soap cakes on drying in storage.

Variations of the  $pH$  of aqueous soap solutions as a function of the free alkali content. *Vizern and Guillot, Seifensieder-Zig* 62, 625-6 (1935).—See *C A* 29, 6453.

Use of liquid resin (tallol) in soap making. *P. Kupehinski and A. Yasniul, Maslobolno Zhivore Delo* 11, 492-4 (1935).—Liquid resin, obtained from black liquor of sulfate pulping, added up to 25% to fats gave a good grade of hard soap. The product was improved by the use of dist. liquid resin (tallol). A greater proportion of tallol results in soft or liquid stock.

Starch and animal glue in the soap industry. *Fritz Ohl, Gelatine, Leim, Klebstoffe* 3, 168-71 (1935).—Soaps can be improved by the addn. of starch, casein or hide glue. Nineteen patents and characteristics of the products are discussed.

Waxes (Mellan) 13. Manuf. of liquid fuel from vegetable waxes (Koo, Cheng) 21. Higher alcs from waxes (*U. S. pat.* 2,021,926) 10

Fats. Harburger Oelwerke Brinckman & Mergell. *Ger.* 617,520, Aug. 20, 1935 (*Cl* 23a. 2). Addn. to 612,716 (*C. A.* 29, 6454). Clear odorless fats are obtained by treating fatty materials with  $CH_3Cl$  and driving off the latter by steam in the pre-nce of water and an adsorbent such as fuller's earth.

Wool-fat alcohols. *Deutsche Hydrierwerke A-G* (Ludwig Prigge and Karl Hennig, inventors). *Ger.* 617,975, Aug. 30, 1935 (*Cl* 12o. 5 02). Alcs. incapable

of sapon. are obtained from wool fat and other high-mol. wax-like substances by sapon., the wool fat, etc., treating the product with at least 30% of a soap with no more than 18 C atoms in the mol., and finally distg. Thus, wool fat is sapon. with NaOH and water and palmic soap, obtained by treating palm oil with NaOH, added. The mixt. is distd. with superheated steam to give a product of wool fat alc.

Bleaching fats, oils and fat acids. *Balthasar E. Reuter* (to Mathieson Alkali Works). *U. S.* 2,022,738, Dec. 3. See *Fr* 778,882 (*C. A.* 29, 4062).

Purifying fatty oils. *Karl Merck, Louis Merck, Wilhelm Merck and Fritz Merck* (trading as (firm of) E. Merck). *Brit* 434,432, Sept. 2, 1935. Tunny-fish liver oil is freed from undesirable color, taste and odor by treating at ordinary temp with aq or alc alkali and then sepg the purified oil. The alkali removes the larger proportion of free fat acids without sapon., or loss of vitamin A.

Oils, vitamins. *Aktieselskabet Ferrosan, Brit.* 433,030, Aug. 22, 1935. The vitamin-contg unsaponifiable components of fats and oils are extd or concd. by sapon. the oil with a soln of  $NH_3$  or alkali metal hydroxide in a  $H_2O$ -sol. org solvent, e.g.,  $Me_2CO$ ,  $MeOH$ ,  $EtOH$ , cooling or standing to sep the soap and acidifying the residual soln to ppt. fat acids from the soap which remains dissolved, said acids collecting as a layer with the unsaponifiable components.

Refined hydrogenated sesame oil. *Guy W. Phelps and Raymond Bradley* (to Industrial Patents Corp.). *U. S.* 2,022,361, Nov. 20. See *Can.* 345,681 (*C. A.* 29, 2380).

Oil cakes. *Benjamin G. Leites, Fr.* 786,676, Sept. 7, 1935. Oil cakes of increased albumen content are obtained by carefully removing the envelope of the grains and, preferably, the cellulose of the core. The cake is ground and suspended in water and the particles of envelope swollen with water descend to the bottom. The remaining suspension is transferred to another vessel where, after prolonged rest, the particles of the core in suspension descend. The liquid is sepd. and used again.

Mono- and di-glycerides. *Carl W. Christensen* (to Armour and Co.). *U. S.* 2,022,493, Nov. 26. In forming a glyceride, a mixt. of a higher fatty acid such as stearic acid and a glycerate of an alkali-forming metal such as Na glycerate having at least one free hydroxyl H is heated to about 100-200°. *U. S.* 2,022,494 relates to a process in which glycerol is esterified, to form a mono- or di-glyceride, by heating with a higher fatty acid such as lard fatty acids in the presence of an alkali-forming metal glycerate, at 100-220°.

Soap. *Adolf Welter, Ger.* 620,391, Oct. 21, 1935 (*Cl* 23a. 1). In effecting such processes in the soap industry as are normally carried out at 180° or above, heating is effected by direct introduction of steam which has been compressed sufficiently to raise its condensation temp. above the process temp. Local overheating is thus avoided, and the danger of explosion is reduced.

## 28—SUGAR, STARCH AND GUMS

J K DALE

The acidity of starch. *Josef Mayrhofer, Oester. Chem. Zig* 33, 178-9 (1935).—The difficulties attendant upon the measurement of the acidity of samples of commercial starch are discussed and the advantages of electro-metric titration pointed out. For ordinary purposes, it is recommended to titrate 20-5 g. of starch in 100 cc. of water with 0.1 N NaOH till the soln. is red to phenolphthalein (4 cc. of 1% indicator soln. used).

Sugar. *Pedro Sanchez, Fr.* 787,028, Sept. 16, 1935. A sirup prepd. with previously refined sugar is decolorized by means of active *Cl* (hypochlorites of Ca, Mg, Ba, Sr or Al), dechlorinated by active C or  $H_2O_2$  and crystallized. The treatment with *Cl* may be carried out in the

presence of an acid or acid salt capable of eliminating practically all the ash-forming material in the sirup. *Cl* C. A. 29, 2014.

Evaporating pans for sugar refineries, etc. *Franz Pest, Ger.* 617,641, Aug. 22, 1935 (*Cl* 6b. 12).

Aqueous solutions of sugar sap, etc. *Carbo-Norit-Union Verwaltungs-G. m. b. H.* (Helmut Rüping, inventor). *Ger.* 617,706, Aug. 24, 1935 (*Cl* 12d. 1.03). The aq. solns. are decolorized and cleared by treatment with an adsorbent such as active C, kieselguhr, fuller's earth, etc., which has been mixed with a substance which forms a gel or a gelatinous ppt. in contact with the soln. An example of such a mixt. is  $CaCl_2$  and  $Na_2PO_4$ , which forms a Ca gel.

Apparatus for continuous clarification of sugar juices, etc., and scum removal Francis R. Fortier. U S 2,022,661, Dec. 3 Various structural, mech and operative details

Recovery of nonsugars from molasses, sugar sirups, sweet waters and sugar wastes, etc. Gustave T. Reich U S 2,022,824, Dec 3 A mixt of isopropyl alc and EtOH is used for treating the material, which seps into 2 layers, the upper layer contg the saccharin components and the lower layer the nonsugars (both layers being freely flowing liquid.) Cf C. A. 30, 612<sup>1</sup>

Dextrose International Patents Development Co Fr 787,069, Sept 16, 1935 Dextrose is crystallized from

solns of converted starch by a crysta in movement, in a crystallizer filled by successive fractions, and, at an intermediate stage of the crysta. operation, the supersatn. of the massecuite is reduced by adding to the charge a soln. having a lower d. than that of the successively added fractions Cf C. A. 30, 612<sup>2</sup>

Starch International Patents Development Co Fr 787,060, Sept. 16, 1935 In making starch from corn, the ground starch of high d. is treated to recover starch and gluten and the resulting water is used to steep the maize, while the ground starch of low d. is sent to the sepn operations without removing the starch and the gluten Cf C. A. 29, 1673<sup>3</sup>

## 29—LEATHER AND GLUE

ALLEN ROGERS

Evaporation of tan liquors Emil Kirschbaum and J. Schumunke Chem. Fabrik 1935, 490-2—Results of expts with a triple-effect evaporator on 3 mixts of liquor are plotted for. the relation of the sp. gr. to the total-solid content, the b-p rise in relation to the abs. boiling pressure and to the sp. gr. at different pressures, the sp. heat in relation to the sp. gr., the relation of the temp to the sp. gr., and the relation of the coeff. of heat transmission to the apparent liquor level and sp. gr. Eight references J. H. Moore

Influence of some sulfur-containing tanning compounds on the deterioration of vegetable tanned leather by sulfuric acid E. L. Wallace, J. R. Kanagy and C. L. Critchfield, J. Am. Leather Chem. Assoc. 30, 510-18 (1935)—Correction of journal name, cf C. A. 29, 8389<sup>1</sup>

Action of microorganisms on vegetable tanning materials II. General consideration of the factors influencing biological activity (in tan liquors). Wm. P. Doelger, J. Am. Leather Chem. Assoc. 30, 587-600 (1935), cf. C. A. 29, 2015<sup>1</sup>—Tan liquor is a relatively poor medium for microorganisms because of the prevalently high acidity and inhibitory influence of tannin. Acid-producing bacteria are invariably present; putrefactive organisms occur only in weak tan liquors. Yeasts abound, including both those that form wrinkled and smooth colonies on Sabouraud's medium; pseudo-yeasts, nonsugar fermenting, predominate. Molds are scarce, the scum fanthar to tanners consists of yeasts. Mold most frequently encountered are *A. niger* and *P. glaucum*, both of which convert "tannic acid" to gallic acid. Microorganisms were counted in tail, medium and head rocker liquors from many tanneries, with both Sabouraud's medium and a "tannin medium," prep'd by using filtered, detannized oak-bark ext. together with dextrose, beef ext., peptone,  $\text{NH}_4\text{NO}_3$  and  $\text{K}_2\text{HPO}_4$ . Counts on Sabouraud's medium were much higher (sometimes 1000 times) than on tannin medium. The latter varied between 2000 and 160,000 organisms per cc. Counts on a complete rocker series from one tannery showed that bacteria decrease and yeasts increase in going from tail to head liquors, i. e., with increasing tannin and acidity. Differences between counts from different tanneries may easily be due to differences in the blends employed. Solns of oak-bark ext. and sulfated quebracho, each contg. 3% tannin, sterilized and inoculated with tail rocker liquor, gave counts of 60 million and 2 thousand, resp., after 5 days. When rocker liquors were allowed to stand idle for 23 days, the no. of organisms increased 50 to 100 times. Lab. expts. show that an inactive rocker liquor is gradually depleted of all fermentable nontannin and acid, while if 25% of the liquor is renewed daily, the percentage of lactic acid remains nearly const. after 4 days. Effect of temp. on lactic acid formation was studied in detannized oak-bark ext. solns contg. added dextrose. The acid is more rapidly formed initially and destroyed subsequently at 25° and 30° than at 20° or 35°; below 17° no lactic acid is formed. Acetic acid formation also has an optimum temp. range of 25° to 30°, but the temp. effect is much more marked than with lactic

acid, almost no acetic acid is formed either at 20° or 35°. The no. of microorganisms present rises and falls with lactic acid concn. After the latter reaches its max., growth is largely restricted to those organisms utilizing lactic acid or other fermentation product. H. B. Merrill

Chrome liquors V. Effect of various factors in manufacture of sucrose-reduced liquors upon the chrome oxide and oil take-up of pickled hide powder. Edwin R. Theis and E. J. Serfass, J. Am. Leather Chem. Assoc. 30, 600-14 (1935); cf. C. A. 29, 7114<sup>1</sup>—Hide powder samples were treated as follows: pickled to equil with soln. contg. 10% NaCl + HCl, final  $\text{pH} = 3.6$  (or 1.8 in one series); 80 g. pickled powder contg. 50% liquid tanned 4 hrs. at 27° to 31° and const. agitation with 200 cc. liquor contg. 1%  $\text{Cr}_2\text{O}_3$ ; washed; fat-liquored with 5% highly sulfonated oil in 100%  $\text{H}_2\text{O}$ , 1 hr., at 55°; dried and analyzed for  $\text{Cr}_2\text{O}_3$ ,  $\text{SO}_2$ , free and combined oil. Results—In Cr liquors reduced with natural heat of reaction only,  $\text{Cr}_2\text{O}_3$  fixation increases with concn. of dichromate prevailing during the reduction, still higher fixations are given by all liquors boiled during reduction; in the latter, fixation increases only slightly with increasing concn. during manuf., and passes through a max. at a mfg. concn. of 175 g.  $\text{Cr}_2\text{O}_3$  per l. Basicity of the Cr compd. fixed by the hide powder is slightly erratic, and shows no distinct trend with variation of liquor mfg. conditions. Presence of excess sucrose, above the amt. required for complete reduction, has no effect on  $\text{Cr}_2\text{O}_3$  fixation, contrary to popular belief. Oil take-up during fat liquoring decreases with increasing  $\text{Cr}_2\text{O}_3$  content of the hide powder.

H. B. Merrill  
Deterioration of vegetable-tanned leathers containing sulfonic acid and glucose Everett L. Wallace and Joseph R. Kanagy, J. Am. Leather Chem. Assoc. 30, 614-20 (1935); cf. C. A. 29, 8389<sup>1</sup>—The presence of about 5% glucose has no effect on the rate of deterioration. Also in J. Research Natl. Bur. Standards 15, 523-7 (1935) (Research Paper No. 846)

H. B. Merrill  
Effect of acid on leather—a summary. Warren E. Emley, J. Am. Leather Chem. Assoc. 30, 620-6 (1935)

H. B. Merrill  
Liquid glues Willy Hacker. Kunststoffe 25, 315-17 (1935)—A brief review of raw materials, recipes and processes of manuf. J. W. Perry

Metallized dyes [for leather] (Fr. pat. 786,920) 25  
Alkylated phenols [as tanning agents] (Ger. pat. 616,786) 10

Hides and skins A. Th. Böhme Chem. Fabrik and Paul Pfeiffer, Ger. 617,957, Aug. 29, 1935 (Cl. 28a 9)  
Chrome-tanned leather is treated with a soln. of a mixt. of  $\text{CaCl}_2$  or  $\text{MgCl}_2$  and an antiseptic on removal from the tanning bath, and stored in a moist, undried state. The leather may be sprinkled with moist sawdust treated with an antiseptic

Preparing skins for tanning J. R. Geigy A.-G. Brit. 433,815, Aug. 21, 1935 This corresponds to U S 1,974,861 (C. A. 28, 7589<sup>1</sup>) The skins are first limed

**Tanning depulped hides.** René Botson and Julien Kamp Ger 617,956, Aug 29, 1935 (Cl. 28a. 3). Hides are tanned by a vegetable, mineral or synthetic tanning bath to which is added 5% of rubber and the distillate going over at 70–230° in the distn of old rubber. Examples are given.

**Tanning I. G. Farbenind. A.-G.** (Gustav Mauthe and Walter Pense, inventors) Ger 615,150, June 28, 1935 (Cl. 28a. 9). Addn to 613,782 (C. A. 29, 8392). The method of 613,782 for fixing tanning agents by water-sol salts of aldehyde condensation products of aromatic amine or mixts of these with phenols, is modified by adding substances of the pyridinium type to the fixing bath. Protective colloids such as size may also be added. In the example  $\text{CH}_3\text{O}$  and methylaniline are condensed in the presence of  $\text{HCl}$  and the product neutralized with  $\text{NaOH}$ .  $N$ -Methylpyridinium chloride and pyridine- $\text{HCl}$  are added to form a fixing bath for leather tanned with ext. of quebracho wood and valonea.

**Leather.** Ritter Chemical Co. Brit 434,745, Sept 9, 1935. Skins that have been pickled in strong acid, e. g., the usual soln of  $\text{H}_2\text{SO}_4$  and  $\text{NaCl}$ , are treated with a salt of a nonsoap-forming nontanning org. acid to bring the pH to about 5.0–6.0 and then chrom-tanned. Borax and  $\text{NaCl}$  and (or)  $\text{Na}_2\text{SO}_4$  is preferably added to the org. acid salt, e. g.,  $\text{AcONa}$ ,  $\text{Na}$  formate,  $\text{Na}$  lactate. Basic Cr sulfate, with added  $\text{NaCl}$  or  $\text{Na}_2\text{SO}_4$ , is used for the tanning. In 1 example skins so tanned are further treated with an agent made from sulfonated  $\alpha$ -cresol condensed with  $\text{CH}_3\text{O}$  and fat-liquored with sulfonated sperm and coconut oils.

**Pressure roller apparatus and method for expressing moisture from leather.** John Krause G. m. b. H. Brit. 433,564, Aug. 16, 1935.

**Emulsifiable fatty mixture suitable for stuffing leather.** Karl Ott and Gustav Mauthe (to General Aniline Works). U. S. 2,023,769, Dec. 10. A mixt emulsifiable with water is prep'd by partially sulfiting one mol proportion of train oil with about 0.33–0.67 mol. of aq.  $\text{NaHSO}_3$  in 40° Bé soln at a temp. of about 60–80° with simultaneous oxidation with air.

**Cellulose ether finishes for leather.** Imperial Chemical Industries Ltd., Archibald A. Harrison and Geo. S. J. White. Brit 434,423, Aug. 27, 1935. A finish of good fastness to wet and dry rubbing is produced on leather by applying a compn comprising a soln in a lower aliphatic alc. of Et cellulose and, as plastizer, a long-chain aliphatic alc. or acid having 8 or more C atoms or an oil such as castor or neatfoot or a wax such as carnauba or an ester such as triresyl phosphate or di-Bu phthalate, and, if desired, as an adhesive, a natural or synthetic gum or resin that is sol in the lower aliphatic alc., with or without coloring matter or pigment. A small proportion of an alc., ester or ether of h. p. 90–160° may be added to reduce the rate of evapn. Among examples, Ti white 30, is dispersed by milling into a mixt. of bleached shellac 12, in methylated spirit (I) 100, with Et cellulose 5, in 125 parts, the dispersion is mixed with a soln of cetyl alc. 5, in I 25, Rotor red B 10, dissolved in I and with Et cellulose 10, in 1100 parts. Brushed onto sumach-tanned grained sheepskin splits and polished with a soft brush, the compn yields a moderately flexible bright bordeaux finish.

### 30—RUBBER AND ALLIED SUBSTANCES

C. C. Davis

**Research and the rubber industry.** B. J. Eaton *India-Rubber J.* 90, 527–9, 536–9 (1935).—A discussion, dealing especially with soil problems, botanical problems and the quality of raw rubber.

**The rubber industry in Japan.** E. A. Hauser. *Kautschuk* 11, 186–8 (1935).—An illustrated description.

**A new method of surface examination for the rubber industry.** A. Salmony-Karsten. *Caoutchouc & gutta-percha* 32, 17303–5 (1935).—An illustrated description of a comparison microscope of special construction and special illumination. Typical applications are included.

**Patent literature in the field of latex and of rubber in 1933 and 1934.** St. Reiner and O. Dudlitz. *Caoutchouc & gutta-percha* 32, 17308–10 (1935), cf. C. A. 29, 8394<sup>1</sup>.—Forty-four U. S., 2 Australian, 5 Canadian, 2 Danish and 9 German patents are summarized.

**Latex comes of age.** M. P. Williams. *Rubber Age* (N. Y.) 38, 81–3 (1935).—An illustrated review of the rapid advance in the industrial use of rubber latex, with special attention to the advantages and disadvantages of latex and the selection of a method of manifold (electrodeposition, spraying, dipping, etc.).

**The importance of latex in the economic use of substitutes and in the use of economical materials.** Hermann Miedel. *Kautschuk* 11, 181–5 (1935).—The discussion deals with various uses, as in the manuf. of plush, velour, fleece, artificial leather, paper, cork, upholstery, etc.

**Raw rubber and latex in America.** Edgar Rhodes. *Bull. Rubber Growers' Assoc.* 17, 492–502 (1935).—A critical discussion, based on R.'s recent extensive trip through the rubber-mfg. plants of the U. S. A. and dealing with various features relating to ordinary raw rubber and latex.

**The technical evaluation of raw rubber in rubber manufacture.** H. C. Riebl. *Kautschuk* 11, 164–7 (1935), cf. C. A. 29, 8396<sup>1</sup>.—A review and discussion of various features.

C. C. Davis

**The recovery of solvents in the rubber industry.** H. III. E. Reismann. *Kautschuk* 11, 168–72, 183–6 (1935), cf. C. A. 29, 8399<sup>1</sup>.—The removal of solvent vapors from air by absorption in a liquid, the evapn. and recovery of the solvent, the losses involved and modern equipment are discussed and illustrated.

**Progress of chlorinated rubber.** Geo. C. Clark. *Rubber Age* (N. Y.) 38, 139–41 (1935).—An illustrated review of several mfg. processes adopted in the past few years, with a description of the properties and uses of chlorinated rubber.

**Factice and rubberized fabrics.** Garach. *Caoutchouc & gutta-percha* 32, 17305–7 (1935).—A discussion of the value of factice.

**The history of factice manufacture.** J. F. E. Russell. *Trans. Inst. Rubber Ind.* 11, 289–94 (1935).—A review and discussion.

**The testing of substitute [factice].** J. H. Carrington. *Trans. Inst. Rubber Ind.* 11, 303–11 (1935); cf. preceding abstr.—The paper describes tests which are, or may be, applied to evaluating factice, points out errors in the literature and describes an investigation of the nature of the acetone ext. and S. The routine tests include color, fineness, odor, d., acidity, ash, mineral content, saponifiable and unsaponifiable substances, and acetone,  $\text{CHCl}_3$  and petr.-ether exts. The acetone ext. detn. is very sensitive to the type of extractor and other conditions, as was proved by comparative tests, and none of the usual methods gives complete extn. in less than 24 hrs.

Part of the S in the acetone ext. is combined; hence ordinary oxidation methods do not give the true free S, and cold soln. methods give low values because part of the free S is dissolved in the free oil. The method of Bolonnikov and Gurova (C. A. 28, 4034<sup>1</sup>; 29, 2020<sup>1</sup>) is highly recommended as accurate and rapid. Oxidation of factice with  $\text{HNO}_3$  and  $\text{KClO}_4$  gives low total S values, and it is recommended to heat with a mixt. of concd. and fuming  $\text{HNO}_3$  for 4 hrs. at 100°, evap. with  $\text{NaNO}_3$ , again evap. with aq.  $\text{Na}_2\text{CO}_3$ , fuse the residue for 30 min.

and det sulfate. The expts of Stamberger (C. A. 23, 1526) and Knight and S. (C. A. 23, 749) give a clue to the nature of the substances extd by acetone and the differences between the various methods of extn. A series of expts showed that hot extn. (1) removes increasing quantities of free oil and free S but never removes all of these, (2) causes depolymerization (or disaggregation) by the peptizing action of the hot acetone itself and (3) causes polymerization (or aggregation) by heat. The rates of (2) and (3) differ, and their combined effect is shown by the crossing of the extn curves of the Soxhlet and Knoefler methods. Cold acetone has little peptizing effect, for otherwise in the absence of heat polymerization (or heat aggregation) during extn., the cold acetone ext. should increase rapidly with time, whereas actually the rate of increase is small. Factice should be tested in a rubber mixt., with special attention to its (1) cost; (2) effect on processing, plasticity, extrusion, scorching, etc.; (3) effect on rate of vulcanization, tensile modulus, abrasion resistance, etc. and (4) effect on aging.

C. C. Davis  
Shoe machinery and accessories in rubber goods manufacture. G. Donaldson. *Trans Inst Rubber Ind* 11, 283-8 (1935).

The latest developments in the field of rubber flooring manufacture. W. H. Reece. *Kautschuk* 11, 161-4 (1935).—A review and discussion.

C. C. Davis  
The strength of vulcanized rubber. W. H. Reece. *Trans Inst Rubber Ind* 11, 212-35 (1935).—A crit. review and discussion of the work of Wiegand and Braendle (C. A. 23, 4372), Frey (C. A. 22, 4870; 23, 4098), Cotton and Barron (C. A. 23, 623) and the Phys. Testing Comm. of the Am. Chem. Soc. (C. A. 19, 1792, 23, 1306), in which considerable attention is devoted to elaborating and interpreting mathematically, statistically and logically the results of these investigations.

C. C. Davis  
Determination of true free sulfur in vulcanized rubber. A modification of the thiocyanate method. Syukusaburo Miyamoto, Ichirō Aoe and Idemasa Nagai. *Ind. Eng. Chem., Anal. Ed.* 7, 414-10 (1935).—The method of Davis and Foucar (C. A. 6, 1114, Caspari, India-Rubber Laboratory Practice, London 1914, 116; C. A. 8, 2072) for detg. true free S by treatment of the acetone ext. with boiling EtOH, and KCN and titration of the KSCN with AgNO<sub>3</sub>, always involved difficulties because excess KCN goes into soln. and renders the end point of the final titration indistinct. The following method overcomes this difficulty much better than does that of Castiglioni (C. A. 27, 681, 5678), by sepn. of KCN from KSCN by means of their difference in soly. in acetone. *Procedure*.—Reflux the dry acetone ext. with alk. EtOH and KCN, evap., ext. with acetone, evap. the ext., dissolve in water, add HNO<sub>3</sub> and a little Fe(NO<sub>3</sub>)<sub>3</sub> and titrate with 0.1 N AgNO<sub>3</sub>.

C. C. Davis  
Industrial gas masks [use of rubber] (Davis) 13  
Ebonite as a construction material in chem. industry (Salmony-Karsten) 1  
Rubber in the gas industry (Austen) 21  
Rubber-bonded abrasive articles (U. S. Pat. 2,022,893) 19  
Puncture-sealing compn. (Brit. Pat. 434,630) 13  
Active C (Brit. Pat. 434,461) 18  
Rubber compns. (Brit. Pat. 433,741) 20

Rubber latex. Metallgesellschaft A.-G. and Johannes Jaenicke. Brit. 434,214, Aug. 28, 1935. Dark coloration is prevented or removed from latexes and their stability is increased by treatment with alk. stabilizers and small amts. of Zn compds. Concn. of the latex may take place before, or after, the addn. In an example, 2.5 kg. of ZnO is made into a paste and added to 18.2 kg. of KOH in its own wt. of H<sub>2</sub>O; this is added to 5000 kg. of latex of 28% solid content, to which has been added 30 kg. of potash-coconut oil soap and the mixt. is concd. to 75% solids as described in Brit. 382,235 (C. A. 27, 4439) and 393,742 (C. A. 27, 6019).

Thickening latex. Metallgesellschaft A.-G. Ger. 616,317, July 26, 1935 (Cl. 396.1). Addn. to 595,727

(C. A. 28, 5285). Evap. app. for thickening latex is described.

Rubber. Imperial Chemical Industries Ltd. and Wm. Baird. Brit. 434,051, Sept. 6, 1935. 4,4'-Dimethoxydiphenylamine is included in rubber mixes to check oxidation and flux-cracking.

Rubber. Hansa-Mühle A.-G. (Bruno Rewald, inventor) Ger. 617,732, Aug. 24, 1935 (Cl. 396.5).  
A method of mixing powd. substances such as filling material, vulcanizing agents or accelerators, into plastic materials such as rubber mixes., consists in pre-mixing the powd. materials with practically oil-free phosphatides of animal or vegetable origin, and then adding the mass to the plastic materials. Thus, soybean phosphatide contg. 30-70% of oil is vacuum dried three times and washed with acetoacetic ester, to remove the oil. The product is kneaded with flows of S. The resulting mixt. is used for softening rubber. Other examples describe the prepn. of linoleum, asphalt, wax masses, etc.

Concentrating rubber liquids. Metallgesellschaft A.-G. Fr. 786,757, Sept. 9, 1935. Latex is concd. by evapn. while avoiding formation of steam and when it has reached a degree of concn. of about 80-85%, further evapn. takes place under reduced pressure over a large surface and with the introduction of NH<sub>3</sub>.

Rubber compositions. Ernst Fröhlich. Brit. 433,396, Aug. 14, 1935. Chlorinated rubber is added to rubber compns. to reduce their resiliency and allow embossing, particularly in the manuf. of a leather substitute, the mixt. obtained being vulcanized. Articles may be made from the compn. or an outer film which may be of different color from the base may be applied by vulcanization. The surface may be coated with a cellulose lacquer, in which case a softener, e. g., castor oil, linseed oil, spalin or an increased proportion of chlorinated rubber, should be added to the rubber compn. A specific compn. is rubber 100, S 3, accelerator 1, oxidizable stand oil 30, chlorinated rubber 20 and coloring matter 10 parts (C. A. 29, 2203).

Rubber compositions. International Latex Processes Ltd. Brit. 433,418, Aug. 14, 1935. Compns. for the production of soft rubber goods are made by masticating a mixt. of compounded or uncompounded unvulcanized rubber and rubber prepd. by the vulcanization of rubber in aq. dispersions thereof. In examples, (1) an unvulcanized aq. dispersion, e. g., latex, is mixed with a vulcanized aq. dispersion, e. g., vulcanized latex, the H<sub>2</sub>O removed and the mixt. masticated, (2) raw rubber is mixed with a vulcanized aq. dispersion and the mixt. subjected to hot mastication until the H<sub>2</sub>O is evapd. and (3) raw rubber is mixed by mastication with the product obtained by removing substantially all the H<sub>2</sub>O from a vulcanized aq. dispersion.

Rubber compositions. The Liverpool Electric Cable Co. Ltd. and Alfred Ellis Hughes. Brit. 433,727, Aug. 20, 1935. A compn., particularly for covering elec. cables, comprises a rubber mixt. and a fire-resistant filler that includes a chlorinated C<sub>12</sub>H<sub>14</sub> and Se. The preferred compn. consists of smoked sheet rubber 35, MgCO<sub>3</sub> 41, Se 1, a chlorinated C<sub>12</sub>H<sub>14</sub> 14.5, C black 5, S 2, antioxidant 1 and accelerator 0.5%. In 433,728, Aug. 20, 1935, similar compns. comprise a rubber mixt. and a fire-resistant filler including a chlorinated pitch of preferably high Cl content, e. g., 30%. Vegetable, animal or mineral pitch may be used and Se is preferably added. A suitable compn. consists of smoked sheet rubber 35, MgCO<sub>3</sub> 41.5, Se 1, C black 4.6, chlorinated elastic cottonseed pitch 12.5, kaolin 2, S 2, antioxidant 1 and accelerator 0.5%.

Rubber compositions. I. G. Farbenindustrie A.-G. Brit. 433,807, Aug. 10, 1935. Rubber mixes are made by using as distributing agent a nonmetallic nonacid product recovered or derived from an oxidation product of a non-aromatic hydrocarbon material of high mol. wt. The oxidation products, e. g., of paraffin, consist of acid constituents, nonacid constituents, e. g., aldehydes, ketones, alcs and esters, and unchanged initial material. The nonacid constituents may be recovered by saponifying the



crude oxidation products with alkalis or alk. earths, extg. the soap soln. with solvents, e. g.,  $\text{C}_2\text{H}_5\text{OH}$ , and evapg. the solvent. Suitable nonacid products may also be obtained from the crude oxidation products of paraffin hydrocarbons by subjecting them to an energetic catalytic hydrogenation with H at elevated temp and pressure so that the carboxylic acids, carboxylic acid esters and lactones are converted into the corresponding aldehydes or alcs., or by esterification with alk., e. g., Me, Et, Pr, iso-Pr, Bu. In an example, a rubber mix, comprising crepe rubber 100, gas C 40,  $\text{ZnO}$  5, S 3, phenylguanidine 1.5 and a condensation product of  $\alpha$ -naphthylamine and aldol 1 parts, is mixed on the rollers with 4 parts of a nonacid product obtained by treating paraffin with air at  $170^\circ$  for 5 hrs., saponig. with alkali, extg. the soap soln. with benzene and evapg. the ext.

Cellular rubber products. Geoffrey W. Trobridge (to Dunlop Rubber Co. Ltd.) U. S. 2,023,296, Dec. 3. An aq. rubber dispersion is mixed with an  $\text{NH}_4$  or alkali metal chloride, sulfate, nitrate or acetate and a metal such as Zn, Mg, Al or Fe which by chem. action produces *in situ* an inflating agent and a coagulating agent, and the resulting frothy mass is permitted to set to a permanent irreversible solid form.

Porous rubber. International Latex Processes Ltd. (Chester E. Linscott and Harold de B. Rice, inventors) Ger. 619,147, Sept. 24, 1935 (Cl. 39a 8 05). See *Fr* 770,501 (C. A. 29, 641<sup>4</sup>).

Coloring rubber. Harry G. Kiernan (to National Aniline & Chemical Co.) U. S. 2,022,887, Dec. 3. A colored unvulcanized rubber which will not deteriorate on standing is prep'd by admixt. of the rubber with a leuco comp'd of a vat dye which has been prep'd by the action of a reducing carbohydrate such as the leuco comp'd of Brilliant Indigo 4B prep'd by the action of corn sugar. Cf. C. A. 29, 1681<sup>1</sup>.

Stable halogen derivatives of rubber. Gerhard Balke and Franz Grom (to I. G. Farbenind. A.-G.) U. S. 2,022,614, Nov. 26. A stabilized product is produced from chlorinated or brominated rubber, etc., by removing the loosely bound halogen by treating the material with a basic substance such as KOH or  $\text{Na}_2\text{CO}_3$  or pyridine at a temp. not exceeding about  $150^\circ$ , in a solvent such as  $\text{CCl}_4$  or solvent naphtha which is resistant to the basic reacting substance.

Rubber products with roughened surfaces. Suerling Latex Products Co. Brit. 433,912, Aug. 22, 1935. See U. S. 1,983,963 (C. A. 29, 959<sup>1</sup>).

Rubber sheeting suitable for making garments for contact with the body. Jacob Stein. U. S. 2,023,231, Dec. 3. A basic ingredient such as borax or  $\text{NaHCO}_3$  is mixed with the rubber used to neutralize acid in body emissions. U. S. 2,023,232 (Jacob Stein and Hugh H. Mosher (to Jacob Stein)) relates to the similar use with rubber of Na benzoate, Na silicate,  $\text{Na}_2\text{HPO}_4$ ,  $\text{Na}_2\text{PO}_4$  or  $\text{NaOAc}$ . U. S. 2,023,233 (Jacob Stein and Hugh H. Mosher) relates to rubber similarly prep'd with a content of basic substance and contg. salicylamide or other specified antipeptic.

Crinkling material such as sheet rubber. James J. Galligan and Wm. J. Robinson (to U. S. Rubber Products Inc.) U. S. 2,022,851-2, Dec. 3. Various details of app and operation.

Compound fabrics. Wingfoot Corp. Brit. 433,777, Aug. 15, 1935. Rubber is bonded to rayon by treating the latter with an aq. protein-latex compn., drying, applying a rubber compn. and vulcanizing. A casein-latex compn. may be made by grinding casein in  $\text{H}_2\text{O}$ , adding dil. alkali, e. g.,  $\text{NH}_4\text{OH}$ , regrounding and mixing with rubber latex. Age-resistors for casein, e. g., hydroquinone, Na chlorophenylphenate, Na *o*-phenylphenate, and insolubling agents may be added. The invention may be used for making automobile tires, rubber hose, fan belting, mats, etc.

Rubber conversion products. I. G. Farbenind. A.-G. (Julius Sell, inventor). Ger. 618,992, Sept. 19, 1935 (Cl. 39a, 3). Addn. to 615,030 (C. A. 29, 6417<sup>1</sup>). Vulcanized rubber is treated with gaseous, liquid or dissolved

1 HF. Horny products similar to those described in Ger. 615,030 are obtained.

Rubber articles. International Latex Processes Ltd. Brit. 433,441, Aug. 14, 1935. Molded articles comprising masticated rubber or like compns., provided with a superficial coating of rubber, etc., deposited from aq. dispersions thereof, are produced by treating the whole or a part of the molding surface of a mold with 1 or more of the aq. dispersions, solidifying the resulting coating, introducing the masticated rubber compn. into the mold, molding the compn. and vulcanizing the whole under pressure in the mold. The process may be applied to the manuf. of floor-covering material, conveyor belts, tire covers, etc., provided on their surface with lettering, etc. The molded articles may be provided with fibrous, fabric or other backings or inserts.

Rubber objects. Metallgesellschaft A.-G. (Hermann Miedel, inventor). Ger. 617,734, Aug. 27, 1935 (Cl. 39b 5). Objects made of or coated with rubber are given a grained or irregular surface by spraying with a concd. aq. rubber dispersion contg. a volatile stabilizing agent. Thus, the dispersion may contain  $\text{ZnO}$ , lithopone, S and tetramethylthiuram disulfide, with  $\text{NH}_3$  as stabilizer.

Apparatus for making rubber strip from aqueous rubber dispersions. International Latex Processes, Ltd. Ger. 620,102, Oct. 14, 1935 (Cl. 39a 8 05). See Brit. 410,000 (C. A. 28, 6346<sup>5</sup>).

Rubber strip material such as surgical drainage tubing. Howard M. Dodge (to B. F. Goodrich Co.) U. S. 2,023,268, Dec. 3. A layer of sponge rubber compn. is applied to an unvulcanized rubber core and the strip is vulcanized in a mold, a layer of rubber is then applied from an aq. dispersion and is vulcanized while the strip is unconfined to provide an impervious cover.

Rubber filaments, tubing, etc. Harvey J. Elwell (to Vultex Corp. of America). U. S. 2,022,462, Nov. 26. A vulcanized latex concentrate, substantially free of protective colloids, proteins and water-sol constituents, is extruded through a die and coagulation of the extruded material is effected by heat alone at a temp. of about  $38^\circ$  or higher.

Rubber printing plates, etc. Theodore C. Browne U. S. 2,022,183, Nov. 26. A sheet formed of a dichromate and gelatin mixt. is exposed to light through a light-controlling screen, and the exposed sheet is placed in an aq. dispersion of rubber, producing a relief-bearing rubber film against the surface of the sheet.

Attaching rubber to metal. Colin Macbeth and Rubber Producers Research Association. Brit. 434,191, Aug. 23, 1935. Rubber-metal units or sections, for use for lining vessels, shutters, conveyors, ball-mills, etc., and for covering cylinders and rolls, are produced by applying a raw rubber to a surface of a metal plate, vulcanizing and shaping the plate so as to relieve the rubber of surface shrinkage and bring it into superficial compression.

Fixing rubber to metal, wood or glass. Soc. anon. des pneumatiques Dunlop. *Fr* 787,072, Sept. 16, 1935. The surface of the rubber is prep'd for fixing or gluing by treating it with a soln. of  $\text{CrO}_3$  in  $\text{H}_2\text{SO}_4$ . The surface is washed, dried and heated to about  $145^\circ$  under pressure for about 10 min. after application of the metal, etc. Cf. C. A. 29, 2393<sup>1</sup>.

Hard rubber coatings on articles such as foot rails. Louis B. Haines (to Western Elec. Co.). U. S. 2,023,582, Dec. 10. A compn. for coating and vulcanization is formed by mixing smoked sheet rubber 500, S 180, diphenylguanidine 2.5 and mercaptobenzothiazole 2.5 parts and dissolving the mixt. in benzene 2300 parts.

Composite articles such as metal and rubber electric plugs. Ferdinand F. Brucker (to B. F. Goodrich Co.). U. S. 2,023,264, Dec. 3. Various operative details of manuf.

Treating reclaimed rubber. Walter C. MacFarlane (to Xylos Rubber Co.). U. S. 2,021,961, Nov. 26. The rubber is strained to remove foreign particles, extruded into strip form, refined by a thin sheet milling operation, the extruded strips being progressively severed to form a plurality of discontinuous particles which are dusted as

formed to prevent them from adhering to each other and conveyed in a continuous stream from the straining and extruding to the refining operation, so that the stream may be diverted to different refining stations. App. is described.

Molding hollow rubber articles having walls of uniform thickness from sheets of unvulcanized stock in molds having cavities. Josef A. Talalay. Bnt. 434,949, Sept. 10, 1935. A stock sheet of nonuniform thickness is used, the parts thereof corresponding to the cavities in the mold being of greater thickness than the parts corresponding to raised portions of the mold, the thickness varying with the varying depth of the cavity.

Apparatus for molding rubber balls. James A. Law. Bnt. 434,577, Sept. 4, 1935.

Apparatus for molding outer covers for pneumatic tires. Leonhard Herbert. Bnt. 434,440, Sept. 2, 1935.

Rubber tubes, etc. John F. Cavanagh (to Wm. W. Potter). U. S. 2,022,230, Nov. 26. App. and various operative details are described.

Inner tire tube. John F. Cavanagh (to Wm. W. Potter). U. S. 2,021,983, Nov. 26. Various details of app. and operation are described.

1 Tire-molding and vulcanizing apparatus. Wm. J. Woodcock and John Bull Rubber Co. Ltd. (formerly The Leicester Rubber Co. Ltd.). Bnt. 434,742, Sept. 9, 1935.

Apparatus for vulcanizing inner tire tubes. Lionel D. Bax. U. S. 2,022,908, Nov. 26. Mech. features.

Apparatus for vulcanizing tires, tubes, etc. Thomas H. Williams (to David Bridge and Co. Ltd.). Bnt. 434,007, Aug. 23, 1935.

2 Apparatus for the continuous vulcanization of rubber goods such as rubber-covered wire. Lewis J. Clayton (to Viceroy Mfg. Co.). U. S. 2,023,665, Dec. 10. Various structural and operative details.

Vulcanizing rubber articles such as tire casings. Geo. P. Bosomworth (to Firestone Tire & Rubber Co.). U. S. 2,023,002, Dec. 3. Various details of app. and operation.

3 Rubber vulcanization. Clyde Coleman (to United States Rubber Co.). U. S. 2,022,953, Dec. 3. An *N*-nitrosophenylmethyl arylmetharyl sulfide such as *N*-nitrosophenylmethylbenzothiazyl sulfide is used as an accelerator.

# CHEMICAL ABSTRACTS

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No. 4

## 1—APPARATUS, PLANT EQUIPMENT AND UNIT OPERATIONS

W. L. RADGER

Devices for automatic regulation of temperature in laboratories. N. A. Udalov. *Zavodskaya Lab.* 4, 805-9 (1935).—Various types of thermometers for thermostat regulation of temps. are illustrated and discussed. C. B.

Application of an objective photoelectric colorimeter in a factory laboratory. A. Zelazny. *Gaz. Charkovskaya* 76, 337-42 (1935), cf. *Werkstätt.* C. A. 29, 3198<sup>1</sup>.—Lange's photoelectric colorimeter giving the extinction coeff. can be standardized in Stammer's degrees, thus it is a valuable tool in tech. lab. J. Wiertel.

Most economical pipe diameters for high-pressure chemical plants. Shun-ichi Uchida. *J. Soc. Chem. Ind. Japan* 38, Suppl. binding 578-81 (1935).—Fundamental equations are derived for fixed cost and for power cost in the case of very large Reynolds' nos., and for values between 2300 and 100,000. Illustrative examples are given. Karl Kammermeyer.

Recent developments in metal sealing into glass. Howard Scott. *J. Franklin Inst.* 220, 733-34 (1935).

E. H.

Porous quartz filters. L. V. Korovnik. *Zavodskaya Lab.* 4, 825-6 (1935).—Methods of manu. of porous quartz filters in Soviet Union are discussed. The products compare well with German articles. Chas. Blanc.

Rapid filtration of silica in volumetric analysis of cast iron and steel. K. A. Yakovleva. *Zavodskaya Lab.* 4, 823-4 (1935).—A tube constructed at the bottom and fitted with a perforated plate is used for filtering. It is charged with filter-paper pulp. Chas. Blanc.

New distillation apparatus. A. Hanal. *Z. Unterzuch. Lehenm.* 70, 394-402 (1935).—Three types are described: (1) steam distn. app., (2) precision distn. app., (3) distillate tester. F. L. Dunlop.

Thermocouple vacuum calorimeter. W. E. Garner and F. J. Veal. *J. Chem. Soc.* 1935, 1436-43.—A thermocouple calorimeter has been developed to measure heats of absorption of gases on oxides. Various discarded types are also described. The device is adaptable to both slow and rapid absorption processes and can be used to measure the differential heats of slow processes. Difficulties are encountered in the attempt to measure differential heats of fast absorptions due to poor distribution of gas throughout the oxide and low-grain cond. The technic is detailed. William E. Vaughan.

Trichromatic colorimeter. R. Donaldson. *Proc. Phys. Soc. (London)* 47, 1068-73 (1935).—The integrating properties of a diffusing sphere are used to mix the 3 colors. S. Bradford Stone.

A chemical hygrometer. Thorne M. Carpenter. *J. Biol. Chem.* 112, 123-33 (1935).—The app. described for the detn. of water vapor by vol. in air is constructed on the principle of a gas analysis app. in which vols. before and after removal of water vapor by an absorbent are read against a const. vol. kept dry by  $P_2O_5$ . A large no. of comparisons with results obtained by weighing the water vapor absorbed from air currents contg. 0.3 to 2.2% show an agreement of under 0.02% on the av. The app. is transportable and easily produced commercially. A. P. Lothrop.

Automatic analysis apparatus of the Ukraine Institute of Applied Physics and Chemistry. N. Ya. Vynnychev and E. E. Korobchanskii. *Zavodskaya Lab.* 4, 812-21 (1935).—Numerous gas analysis app. of original design are illustrated and described. Chas. Blanc.

Sampling apparatus for chemical-climatological and technical air analysis. H. Cauer. *Z. anal. Chem.* 103, 160-88 (1935).—The app. recommended is classified into 2 groups, one uses a motor-driven pump and the other a hand pump. The pump chosen provides for the rapid passage of air and, even when the air passes through a very fine filter, is suitable for making a rapid analysis. The method of sampling and the necessary app. are described and illustrated by photographs of actual practice. Fight references. W. T. H.

Apparatus for aspiration, preservation and analysis of gases soluble in water. M. S. Gershenovich. *Zavodskaya Lab.* 4, 904-7 (1935).—Several app. are illustrated and described. Chas. Blanc.

A new cataphoresis chamber for measuring the velocity of cataphoretic migration of microscopic particles. Robert Havemann. *Biochem. Z.* 281, 402-7 (1935).—Theoretical discussion of cataphoretic methods and description of a simple app. S. Morquais.

Recovery from the gas mixtures which escape during the production of benzyl chloride. E. V. Alekseevich and S. F. Kravayin. *J. Chem. Ind. (Moscow)* 12, 338-40 (1935).—When mixts. of MePh, Cl<sub>2</sub> and HCl are adsorbed on birch C or SiO<sub>2</sub> and desorbed with steam at 120°, mixts. of mono- and dichloro-toluenes are obtained. With Al<sub>2</sub>O<sub>3</sub>, MeC<sub>6</sub>H<sub>4</sub>Cl is obtained. Mixts. of MePh and Cl<sub>2</sub> in air give the same result with SiO<sub>2</sub> as when HCl is present, but with C only monochlorotoluenes are obtained. SiO<sub>2</sub> is a good catalyst for the chlorination of MePh, but C is not. H. M. Leicester.

Constant-flow liquid distributor. A. Bourt. *Documentation scientifique* 4, 43-7 (1935); *Chimie et Industrie* 34, 828.—The principle of the app. is based on the clamping of the liquid inlet by means of a float. A 5-l. flask contg. the liquid to be distributed is mounted above the distributor and a float in the latter is provided with a 1-mm. tube fitting into the outlet tube from the 5-l. flask. The flow is governed by the difference in the level of the liquid in the distributor and that of the outlet of the discharge tube therefrom. A suitably graduated scale can be placed back of the outlet to adjust it for any desired rate of flow. A. Piqueneau-Couture.

Thermal photometers. R. Jouaust. *Rev. gén. Elec.* 38, 419-22 (1935).—Thermal photometers are app. which permit detn. of the energy in a beam of rays after transformation of this energy into heat. Four types are distinguished: (1) app. in which the heating of a mass of air is used; (2) app. utilizing the effects produced in rarefied gases under different temps.; (3) app. in which the increase of elec. resistance due to increase of temp. is utilized; (4) app. with thermocouples. Practical types are described and applications reviewed. Five references. M. Hartenhein.

Höppler viscometer vs. Vogel-Ossag viscometer. J. J. Leendertse. *Chem. Weekblad* 32, 533-5 (1935).—The Höppler viscometer is generally unsuited for work on mineral oils; it lacks theoretical basis. The rapaport viscometer (Vogel-Ossag) is preferred. C. G. Verver. *Ind.* 535-6.—The above results are confirmed. B. J. C. van der Hoeven.

A new micro hydrogen electrode. J. Löbbeck. *Z. anal. Chem.* 103, 180-3 (1935).—It is relatively easy and simple to construct a miniature H<sub>2</sub> electrode with which one can measure the potential against a calomel cell with

less than 0.1 cc. of liquid and with an accuracy of 0.2 mv. in the  $pH$  range 1.8-10.5. The electrode, which is depicted, as well as described, is made of capillary tubing of 1 mm diam and 70-mm height. In the middle, the capillary is blown out somewhat and in the upper third it is connected with another section of capillary tubing which is given a right-angled bend downward and has also a bulb in the middle opposite to the other tube. In the capillary of the first tube is inserted the platinumized electrode through a ground-glass connection. The entire app is placed in 0.1  $N$  KCl when a measurement is to be made and the lower part of the capillary tubing is filled with the soln. to be tested. W T H

An improved hydrogen electrode Witold Hermanowicz *Roczniki Chem* 15, 290-3 (1935).—This electrode is similar in construction to one proposed by Walpole (*Biochem J* 7, 410 (1913), *C A* 8, 2403) and Lasch (*Z ges. exp. Med* 56, 157 (1927)). The electrode is described and a diagram included. It requires 0.2 cc. of liquid for a  $pH$  detn. and can be used in a water thermostat. A const. potential of buffered solns. is attained after 5 min.; that of an unbuffered soln. is attained after 35-40 min.

J F. Matejczyk

A simple apparatus for measuring the heat conductivity of oils E L Lederer *Fettchem Umschau* 42, 152-5 (1935).—The app consists of an arrangement for (1) subjecting to a const., relatively lower temp. the lower end of an otherwise insulated perpendicular cylinder filled to a definite depth with the material being investigated and (2) simultaneously detg., as a function of time, the temp. drop at a predet. point within the material under test. The calcs. are based on Fourier equations (*cf. C A* 22, 1884). The device works well not only with granular or oily bodies but also with solidified substances introduced into the cylindrical part of the app. in a molten condition. J W Perry

Industrial furnace atmospheres C George Segeler. *Am Gas J* 143, No. 6, 9-12, 45 (1935).—A survey of the use of special industrial furnace atm. A table of recommendations and limitations for furnace atm. is given. Twenty-three references. Joseph H Wells

The faults of the heating conditions of the screw conveyor digester R B Gurevich *Kalini (U S S R)* 1935, No. 6, 15-19.—The pressure of steam must be increased from 0.1 to 1.5 atm., to insure the working conditions of the pipes which supply the live steam to the lower part of the digester for mixing and heating purposes. A Pestoff

Radiation from nonluminous flames H C Hottel and V C Smith *Trans Am Soc Mech Engrs* 57, 463-70 (1935), *cf. C A* 29, 7127.—A method is presented for detg. the total radiation from flames, due to  $CO_2$ ,  $H_2O$  and mixts. of these at temps. above 2500°F. The emission of radiation and the corresponding temp. were measured from overventilated flames of  $CO$ ,  $H_2$  and mixts. of these in a Meeker burner of varying length, and the products in the flame were calcd. from the compn. of the burner feed mixt. Results are presented for flames of  $CO$  in mixts. of  $O_2$  and  $N_2$ , in which the burner grid length is varied from 2 to 16 in., the theoretical  $O_2$  from 100 to 300%, and the flame temp. from 2600° to 3800°F. The results establish the validity of the  $CO_2$  radiation chart in its high-temp. range. Less extensive measurements on illuminating gas flames establish the validity of the water-vapor radiation chart at temps. of 2500-2800°F., when the thickness of the gas layer is small. Exptl. data and 22 references are given. Karl Kammermeyer

Evaluation of effective radiant heating surface and application of the Stefan Boltzman law to heat absorption in boiler furnaces W J Mullikin *Trans Am Soc Mech Engrs* 57, 517-29 (1935).—A method is developed for converting well known types of furnace heat-absorbing surface into an equiv. effective radiant heating surface, which is controlled by (1) spacings of wall tubes, (2) kinds of blocks on the water-wall tubes, (3) amts. of slag on the water-wall tubes or blocks, and (4) the emissivity of the water wall surface exposed to the flame. The fraction of the heat supplied to the furnace that is given up

by the gases in the furnace is found to be obtained most simply and practically by use of the Stefan-Boltzman law. Results computed in this manner are compared with results reported in another paper (*cf. following abstr.*), and it is found that for large pulverized coal, and oil-fired and gas-fired furnaces, the assumption of an effective furnace emissivity of unity gives results in good agreement with the exptl. data. For stoker-fired furnaces, in general, the effective furnace emissivity appears to be less than unity. Exptl. data and 6 references are given.

Karl Kammermeyer

An experimental investigation of heat absorption in boiler furnaces W J Wohlenberg, H F. Mullikin, W. H. Armacost and C W Gordon. *Trans Am Soc Mech Engrs* 57, 541-54 (1935).—Test data are reported from 10 boilers in different power plants in the U S A., including stoker fired pulverized fuel, and oil- and gas-fired units with various combinations of refractory and water-cooled furnaces. The data include furnace-exit gas temps., measured by means of the high-velocity thermocouple, which are used to det. the fraction of the heat input to the furnace that was absorbed in its walls. Sixty references.

Karl Kammermeyer

Review of methods of computing heat absorption in boiler furnaces W J Wohlenberg and H F. Mullikin *Trans Am Soc Mech. Engrs* 57, 531-40 (1935).—The heat absorption in boiler furnaces is computed by a no. of methods and the results are compared with the results reported by the A S M. E. Comm. on Absorption of Radiant Heat in Boiler Furnaces (*cf. preceding abstr.*). Particular emphasis is placed on the methods of Broido, Orrock and Wohlenberg. It is shown that the influence of various conditions as found by expt. is in substantial agreement with results expected from a theoretical consideration of the problem and those reached by certain well known methods now in use. Karl Kammermeyer

Fumigation of ships by Clayton gas [buret for estn. of gas concns.] (Gilmour) 13

Chemical apparatus Metallgesellschaft A-G *Fr* 786,450, Sept. 3, 1935. App. is constructed of material insensitive to shocks, e.g., a mixt. of rubber with substances such as cement, plaster, asbestos, leather fibers, vulcanizing agents, this material being coated with a layer of soft rubber or ebonite at the places where the app. is exposed to the attack of chem. agents.

Vacuum distillation apparatus Eric W. Fawcett, John L. McCowen and Imperial Chemical Industries Ltd. *Brit* 434,726, Sept. 9, 1935. In the distn. of liquids in which a no. of distn. units are housed in a common vacuum chamber connected to a high-vacuum pumping system, each unit comprises a disty. surface heated internally by a heater and an adjacent condensing surface. Colormeter. Abraham G. Sheffield *Ch. Eng.* 621,221, Nov. 2, 1935 (*Cl* 42k 18.01). See *Brit.* 405,362 (*C A* 28, 3943).

Potentiometer for measuring  $pH$  values Imperial Chemical Industries Ltd. *Fr* 787,824, Sept. 30, 1935.

Apparatus for electrometric analysis Udo Ehrhardt (to I G Farben) A-G. *U S* 2,024,819, Dec. 17. A calomel, a Ag and at least one Pt electrode are inserted in a titration vessel for carrying out electrometric analyses by measuring the variations of the anodic current of an electron tube operatively connected with the titration vessel and are adapted to be operatively connected to the electron tube. Various details are described.

Precision resistance Paul H. Dike (to Leeds & Northrup Co.) *U S* 2,026,616, Jan. 7. An unsealed precision resistor comprises a resistance conductor (such as a "manganin" wire) having a low temp. coeff. with a covering comprising mixed natural vegetable and animal fibers serving to reduce changes in resistance due to variations in humidity. The covering may be impregnated with a material such as ceresin.

Electrical resistance thermometers Wilhelm Bonwitz *Brit.* 474,635, Sept. 5, 1935.

**Thermometer casings** Herbert C. S. Bullock. Brit. 435,282, Sept. 18, 1935 A thermometer pocket for insertion in a pipe line is provided with means for mixing the stream of fluid under exam. and passing it over the thermometer element.

**Blowpipe suitable for use with oxygen and acetylene.** Samuel R. Oldham (to Oxford Railroad Service Co.) U. S. 2,024,037, Dec 10 Structural details

**Classifying apparatus of the oscillating or reciprocating raking-blade type** Charles K. McArthur (to Dorr Co.) U. S. 2,025,690, Dec 24 Structural, mech. and operative details

**Crystals** Compagnie nationale de matières colorantes et manufactures de produits chimiques du Nord réunies établissements Kuhlmann Fr 786,040, Aug 24, 1935 Absolutely regular crystals of controlled size are obtained in an app. in which starting crystals are dipped into and withdrawn from a saturated supersaturated lye at raised temp. The crystals may be placed in pans dipping into and raised out of 2 vessels by a rocking motion

**Filters** Frank O. L. Chorlton Brit 431,576, Sept 4, 1935 A removable filter element formed by winding wire in a discontinuous helical groove cut in longitudinal ribs or bars on a cage or former has end flanges which fit in sealings of a casing having an inlet and outlet

**Filters** Edward Zahm Brit 434,581, Sept 4, 1935 A filter-press is built up of frames each having an inwardly-projecting flange and a drainage member comprising coarse wire fabric with finer wire fabric on either side, held against a shoulder on the flange. Filter cloth is held against either side of the drainage member by rings within which filter aid is deposited to form a filtering bed

**Filters** Schuchtermann & Kremer-Baum A G für Aufbereitung Ger 618,786, Sept 14, 1935 (ci 12d 13) A device is described for stretching filter cloths on drum filters

**Filters with concentric filtering elements** Établissements A. Guiot Fr 787,482, Sept 23, 1935

**Filtering apparatus (with vibrating filter leaves on brackets)** Robert S. Butler (to Nordberg Mfg Co.) U. S. 2,024,426, Dec 17. Various structural, mech. and operative details

**Rotary-disk filters** Soc. alsacienne de constructions mécaniques. Fr 787,265-266, Sept 19, 1935

**Filters for gas or vapors** Siemens-Mariawerke A-G für Kohlefabrikate (Oswald v. Wartburg, inventor) Ger. 618,505, Sept 11, 1935 (ci 12c 3 02) Rings of gas-adsorbing material such as C are embedded in a plastic container, and hardened. The plastic substance may also be a gas adsorbent

**Rotary-drum filter, with rolls for compressing the filter cake.** Humboldt-Deutemotoren A-G Ger 621,111, Nov. 4, 1935 (ci 12d 15 02)

**Rotary-drum apparatus for filtering and dewatering slimes such as those from coal, wool, ores or sewage** Rene A. Henry. U. S. 2,021,353, Dec 17 Various structural, mech. and operative details

**Filter for liquids.** Frederick G. Seeley. Fr 789,011, Aug 24, 1935.

**Apparatus suitable for filtering liquids through diatomaceous earth.** Edgar Cocke and Lee D. McGown (to Los Angeles Wall Bed Co.) U. S. 2,024,719, Dec 17. Various structural and operative details are described of a filtering app. with vacuum and air supply connections for reverse-flow cleaning of a screen in the filter chamber

**Continuously acting rotary filter for separating liquids** David McCrone Shannon. Ger. 618,906, Sept. 21, 1935 (ci 12d 12 02)

**Continuous-pressure rotary filter** Charles L. Riou. Fr 787,143, Sept. 17, 1935

**Pressure filter and pump, etc., for filtering solvent liquid such as that used in "dry cleaning"** Stanley Malanowski. U. S. 2,024,463, Dec 17 Various structural and operative details

**Filter press** Dario Teatini. U. S. 2,024,055, Dec 17  
**Gas filters** Società italiana Pirelli Brit 434,700, Sept. 6, 1935. Addn. to 433,190 (C. A. 30, 331). In a gas filter a mixt. of fiber and auxiliary granular material is

felted to form the filtering medium. Thus a suspension of wood cellulose in alc. is added to 1 of ebomite dust in alc. and the mixed suspension is filtered, e. g., on a fine sieve. In another example, cotton fibers are wetted by a soln. or suspension, e. g., of colophony in CCl<sub>4</sub>, dried and the mixt. carded and wrapped on a drum or lapped to the desired thickness to effect the filtering

**Apparatus (with a disk of porous stone-like material) suitable for filtering steam, etc.** Emil F. Wemhoener (to Universal Boiler Filter Corp.) U. S. 2,024,122, Dec 10 Various structural and operative details

**Filter comprising loose filtering material enclosed between two concentric rotary drums** Richard Mensing. Ger 605,239, Nov 8, 1935 (ci 12d 15 01) Means for cleaning the filtering material is described

**Cleaning apparatus for bag filters for separating dust from gases** Standard Filterbau Ges m b H Ger. 621,415, Nov 6, 1935 (ci 50e 6)

**Filtering mass for aerosols** Società italiana Pirelli. Fr 786,421, Sept 3, 1935 The mass is composed of fibrous material having been submitted to one of the treatments used in the spinning or weaving industries, e. g., in the form of cloth on which the inert powder is deposited

**Combination funnel and measure** Francis J. Kneer. U. S. 2,024,169, Dec 17 Structural details

**Sieves and strainers** Francis Ward Brit 435,161, Sept 16, 1935 The sieves, etc., are constructed so that a perforated plate or sieve bottom can be inserted at the top or bottom of the frame to provide either a deep or shallow sieve

**Separating dispersed matter from liquids** John O. Samuel and Emily Anthracite Colliery Ltd. Brit 435,126, Sept 16, 1935 The sepn. is promoted by adding a flocculating gel obtained by subjecting a mixt. of amylose material and a neutral metal salt or salts, e. g., ZnCl<sub>2</sub>, the thiocyanates of Li, Ca, Mg, Sr, Mn or Ce, the double thiocyanates of Na and Mn, of Ba and Mn and of Na and Zn, CaCl<sub>2</sub> in admixt. with ZnCl<sub>2</sub>, HgCl<sub>2</sub> or the above thiocyanates or MgCl<sub>2</sub> in admixt. with HgCl<sub>2</sub>, to heat and agitation until the cells of the amylose material burst and form a thick gummy paste. The dispersion to be treated may first be rendered alkal.

**Separating solids from liquids** Westfalia-Dinnendahl-Gröppel A-G and Gewerkschaft Sophus-Jacobsa. Fr 787,522, Sept 21, 1935 See Ger. 609,094 (C. A. 29, 24057)

**Press roll apparatus for separating water from various materials** Karl G. Hüllner and Axel G. Johansson U. S. 2,025,348, Dec 24. Structural details.

**Apparatus for gravity separation of different liquids such as oil and water** Wm R. Mobley U. S. 2,025,883, Dec 31 Various structural and operative details.

**Separating materials (such as coal and stone) of different specific gravities** Gerard J. de Voors U. S. 2,026,343, Dec 31 The materials are introduced into a sepg. liquid such as an aq. suspension of clay and barytes in finely divided condition App. and operative details are described

**Centrifugal separator for gas purification** Charles H. W. Cheltnam and Cyril H. Cheltnam Fr 787,678, Sept 26, 1935

**Centrifugal apparatus for cleaning air and gases.** Willy Neumann Fr 787,141, Sept 23, 1935.

**Centrifugal apparatus for degassing liquids.** Società italiana Pirelli Fr 787,186, Sept 18, 1935

**Apparatus for purifying gases by filtering them through granular material** Jean Journer. Fr 787,190, Sept. 18, 1935

**Apparatus for cleaning gases** Hall & Kay Ltd, Percy Kay and James H. Hall Brit. 433,943, Aug 16, 1935. The app. comprises baffles assembled to form zigzag air or other passages

**Apparatus for analyzing gases.** Claude Bonner. Fr. 787,500, Sept. 23, 1935

**Apparatus for chemical analysis of gases** Pierre L. L. Vallery and Joseph P. P. Rosello. Fr 786,491, Sept. 3, 1935.

Apparatus for automatic gas analysis René Moritz. *Fr* 787,621, Sept. 26, 1935

Electric system, etc., for indicating gas pressures, flows, etc., at a distance. Garrett B. Luederman, Jr. (to Pittsburgh Equitable Meter Co.) U. S. 2,024,378, Dec. 17, 1935. Various structural, elec. and operative details.

Rare gases boiling higher than oxygen. Egeyüllt Izzölampa és Villamosági R. T. *Brit* 434,193, Aug. 28, 1935. Kr and other atm. constituents less volatile than O are obtained by partial liquefaction of air, the Kr being washed out by liquid air or N at a temp. lower than that of liquid O at the pressure employed, the Kr, etc., being thus obtained as a mixt. with O which is worked up to sep. Kr, etc., while the N and O pass off together as gas. *Cl* C 29, 3358<sup>8</sup> 8250<sup>9</sup>

Separating gases by liquefaction. Egeyüllt Izzölampa és Villamosági R. T. *Brit* 434,194, Aug. 23, 1935. Addn. to 434,193 (preceding abstr.). To obtain Kr or hke heavy rare gas from air, atm. air is introduced through a pipe into a packed tower supplied with liquid air from an external source by a pipe and supplied also by a pipe with air liquefied by passage in a compressed state through coils in vessels through which the liquid from the tower flows in succession, the vapors evolved from the vessels passing back into the vessels and escaping by a pipe.

Compressing gases such as those in refrigerating apparatus. Reginald G. Wyld (to Baldwin-Southworth Corp.) U. S. 2,024,323, Dec. 17, 1935. Various details of app. and operation are described.

Apparatus for producing compressed gas from liquefied gas. Akt.-Ges. für Industriegasverwertung. *Ger.* 621,462, Nov. 12, 1935 (*Cl* 17g 5 02)

Two stage system for rectifying liquefied gas mixtures. Oesterreichische Aga-Werke A-G. *Austrian* 143,311, Nov. 11, 1935 (*Cl* 12d)

Removing weak gaseous acids from gases. I. G. Farbenind. A-G. *Fr* 787,782, Sept. 28, 1935. In processes wherein weak acids are removed by washing with org. bases contg. at least 1 amino group, the app. is made wholly or partly of Al and an alloy thereof and the concn. of the org. bases in the solns. or liquids in question is kept at 20% at least. If the solns. contain Al this must be removed.

Apparatus for solidifying gases such as carbon dioxide. Harry W. Cole and Malcolm W. McLaren (to International Carbonic Engineering Co.) U. S. 2,025,608, Dec. 24, 1935. A liquefied gas is supplied to form an accumulation of solidified gas in a chamber in which a definite pressure may be maintained, and the solidified gas is compressed in the chamber.

Means for regulating, indicating or recording varying quantities of liquid flowing out of a pipe. Felix Meyer. *Brit* 434,375, Aug. 30, 1935.

Device for subdividing and distributing liquid in a timed sequence. Leonard B. Harris (to International Oil Gas Corp.) U. S. 2,024,690, Dec. 17, 1935. Various structural, mech., and operative details.

Device for the discharge of liquid from apparatus by gas pressure. Holden & Brooke Ltd. and Daniel G. McNair. *Brit.* 434,688, Sept. 6, 1935.

Apparatus and method for measuring viscosity of liquids. Andrew Gemant and Richard V. Southwell. *Brit* 434,994, Sept. 12, 1935. The viscosity is measured by applying pressure to a column of a liquid in such manner as to produce oscillations and observing the characteristics of the oscillations.

Viscometer with rotating disks suitable for testing liquids. John M. Bell. U. S. 2,026,297, Dec. 31, 1935. Various structural, mech. and operative details.

Electrical liquid level indicator suitable for use with boilers. Fletcher M. Boyd. U. S. 2,026,299, Dec. 31, 1935. Various structural, elec., and operative details.

"Liquid proofing" gaskets such as those of asbestos. Benjamin J. Victor (to Victor Mfg. & Gasket Co.) U. S. 2,025,486, Dec. 24, 1935. The gasket is treated with a resinous residue derived from the partial decomposition of a metallic fatty acid salt such as Al stearate in a solvent such as C<sub>2</sub>H<sub>6</sub> and CCl<sub>4</sub> and the solvent is evaporated.

Mercury-vapor generators. The British Thomson-Houston Co. Ltd. *Brit.* 433,919, Aug. 22, 1935. In a Hg boiler, liquid-vapor sep. means are arranged in the space of the drum between the discharge pipe and the tubes that supply to the drum the mixt. of Hg and vapor.

Mercury-vapor generators. The British Thomson-Houston Co. Ltd. *Brit.* 434,386, Aug. 30, 1935.

Rectification of liquids in a distillation column. Samuel C. Carpey (to Shell Development Co.). U. S. 2,026,019, Dec. 31, 1935. Various structural and operative details for rectifying by decarbon fractions, etc.

Powdered substances. Soc. anon. des pneumatiques Dunlop. *Fr.* 787,363, Sept. 21, 1935. Finely divided powders are obtained by dissolving the primary material (1), emulsifying the soln. in a medium in which this soln. and 1 are practically insol. and removing the solvent without coalescence from 1. Examples are given of the prepn. of powd. chlorinated rubber, nitrocellulose, benzyl-cellulose and a mixt. of chlorinated rubber and chlorinated biphenyl.

Mixing apparatus of the continuous type suitable for use in chemical industries, etc. John E. Chiquone (to Blaw-Knox Co.). U. S. 2,024,509, Dec. 17, 1935. Various structural, mech. and operative details.

Apparatus for mixing aqueous liquids with powdered adsorbents. Carbo-Nort-Union Verwaltungs-Ges. m. b. H. *Ger.* 621,108, Nov. 1, 1935 (*Cl.* 85b.3 03).

Roll apparatus for mixing and grinding. Firma J. M. Lehmann (Kurt Wiemer and Alfred Goll, inventors). *Ger.* 621,095, Nov. 1, 1935 (*Cl.* 50c.13 10).

Rollers composed of a number of washers of asbestos or like material threaded on an axle and compressed longitudinally and intended for displacing articles at high temperatures, particularly glass plates or sheets. Soc. anon. des manufactures des glaces et produits chimiques de St.-Gobain, Chauny & Cirey. *Brit.* 434,315, Aug. 29, 1935.

Evaporating apparatus. Alteselskapet Krystal. *Ger.* 618,727, Sept. 17, 1935 (*Cl.* 12a.2). App. is described for producing large crystals by evapn. *in vacuo*, or by vacuum cooling.

Circulatory tubular evaporating apparatus. Hermann Claassen. *Ger.* 621,110, Nov. 1, 1935 (*Cl.* 12a.2).

Film evaporators. Eric Wm. Fawcett, John L. McCowan and Imperial Chemical Industries Ltd. *Brit.* 435,032, Sept. 9, 1935. In the high-vacuum distn. of materials yielding a solid distillate or residue in app. where the disty. surface and condensing surface are in close proximity, the solid is removed, e. g., from the condensing surface, by a scraper in form of a piston ring traversing the surface by means of a screw and is delivered to a receiver.

Evaporator and crystal separator suitable for treating various salt solutions. Martin J. Kerner (to Buffalo Foundry & Machine Co.). U. S. 2,025,059, Dec. 24, 1935. Various structural details.

Evaporator suitable for refrigerating plants. Raoul Bernat and Henri Bernat. U. S. 2,025,634, Dec. 24, 1935. Structural and operative details of an evaporator suitable for use with liquefied gas.

Drying apparatus. Kurt Goldschmidt. *Austrian* 143,336, Nov. 11, 1935 (*Cl.* 82a). A part only of the moist gas leaving a drying app. is caused to exchange heat with the ingoing gas. The amt. of vapor condensed from the outgoing gas, and the amt. of latent heat recovered, are thus increased.

Film driers. Peter Spence & Sons Ltd., Walter V. Coles, Geo. Scott & Son, London, Ltd. and Geo. W. Raley. *Brit.* 434,883, Sept. 11, 1935. In drying wet clay and like pastes of finely divided material that is substantially refractory at the temp. used, by passing through a heating zone as a thin deposit upon a conveyor, the material is heated mainly by radiant heat from surfaces heated to incandescence.

Pneumatic drier. Büttner-Werke A-G. *Ger.* 618,501, Sept. 9, 1935 (*Cl.* 82a.1 02).

Vacuum drier and cooler. Metallgesellschaft A-G. *Ger.* 618,502, Sept. 10, 1935 (*Cl.* 62a.25 08).

- Chamber furnace.** Hermann Limberg. Ger. 818,857, Sept. 17, 1935 (Cl. 10a. 12 01). Details of construction are given.
- Cupola furnace with a movable bottom for sintering and fusing quartz.** Fritz Winkler (to I. G. Farbenind. A.-G.). U. S. 2,026,370, Dec. 31. Various structural, mech. and operative details.
- Regenerative flame furnace.** Hoersch-Köln Neussen A.-G. für Bergbau und Hüttenbetrieb. Ger. 621,140, Nov. 2, 1935 (Cl. 24c. 6). Addn to 507,211 (C. A. 27, 2351).
- Drum for roasting, distilling and drying cork chippings.** Anton Köhler. Ger. 618,709, Sept. 14, 1935 (Cl. 39a 19 50).
- Continuous regenerative kiln suitable for various heat treatments.** Allen M. Rossmann (to Rossmann Engineering Co.). U. S. 2,026,060, Dec. 31. Various structural and operative details.
- Apparatus (with a vertical reaction chamber) for roasting or calcining various materials.** Eugene C. Saint-Jacques (to Soc. Chantierine d'applications industrielles de brevets). U. S. 2,025,402, Dec. 24. Various structural and operative details.
- Furnace wall with cooling tubes.** James F. Millar (to Babcock & Wilcox Co.). U. S. 2,025,000, Dec. 24. Various details of construction.
- Electrically heated vacuum-flask apparatus for maintaining small quantities of substances at a predetermined temperature.** Rosa Citron. Austrian 143,695, Nov. 25, 1935 (Cl. 42b).
- Heat-exchange apparatus suitable for use with various liquids and gases.** Charles A. Harrison. U. S. 2,024,521, Dec. 17. Various details of manuf. and construction are described.
- Heat-exchange apparatus suitable for cooling hot granular material such as cement clinker on a circular oscillating surface.** Otto Lelupp. U. S. 2,024,934, Dec. 17. Structural, mech. and operative details.
- Heat exchanger for heating oil-saturated steam of a "mallet" type engine by superheated steam from the engine.** Richard B. Salley. U. S. 2,026,438, Dec. 31. Structural details.
- Finned heat-exchange tube.** Alfred J. Berg. U. S. 2,025,036, Dec. 24. Structural details.
- Thermostatically controlled valve device for use in apparatus for exchanging heat between two liquids.** Northern Equipment Co. Brit. 435,150, Sept. 16, 1935.
- Calorimeter and method of use with liquid or gaseous fuels or the like.** Rafael W. Keith. U. S. 2,026,179, Dec. 31. Various structural, elec. and operative details. U. S. 2,026,180 also relates to details of calorimeters "of the multiple type."
- Apparatus for coating articles with viscous compositions by dipping.** N. V. Machinerieën-en Apparaten-Fabrieken. Ger. 621,234, Nov. 4, 1935 (Cl. 76a. 21).
- Apparatus for coating sheet materials.** Raymakers Syndicate Ltd. Ger. 621,331, Nov. 5, 1935 (Cl. 55f 3 01). See Brit. 405,761-2 (C. A. 28, 53627).
- Apparatus for classifying materials such as sands**
- 1 Royal S. Handy.** U. S. 2,025,412, Dec. 24. Various structural, mech. and operative details.
- Core extractor or sampler for removing samples of resin from barrels, etc.** Frederick W. Thomas. U. S. 2,024,076, Dec. 10. A wire coil extending into material such as a resinous gum in a barrel or the like has its outer end secured to a plug adapted to close a hole in the receptacle.
- 2 Apparatus for proportioning the flow of liquids as in refining vegetable or petroleum oils.** Robert J. Short (to Procter & Gamble Co.). U. S. 2,024,478-9-80, Dec. 17. Various structural, mech. and operative details.
- Apparatus for sampling or for visual examination of oils.** Gustave A. Brandlin. U. S. 2,026,267, Dec. 31. Structural and operative details.
- Apparatus for lifting oil (from wells) or other liquids by the action of compressed gases.** Johan Entrop (to Shell Development Co.). U. S. 2,026,226, Dec. 31. Various structural and operative details.
- Exhaust steam condenser, oil separator and feed water heater and purifier.** Charles E. Dillinger. U. S. 2,025,043, Dec. 24. Various structural and operative details.
- Sizing and separating fines from crude materials such as cement, flour or ores.** Meshack T. McWhorter and Jacob R. Hiestand. U. S. 2,026,318, Dec. 31. App. and various operative details are described.
- 4 Clarifying and removing coloring matter from aqueous liquids by use of acid sludge from oil refining.** John C. Bird (to Standard Oil Development Co.). U. S. 2,025,715, Dec. 31. Materials such as sugar solns., etc., are treated with water-sol. sulfonic acids or their salts such as those of from acid sludge capable of forming substantially water-insol. alk. earth sulfonates as a ppt. which adsorbs coloring matter, and the ppt. is removed from the liquid.
- 5 Air-conditioning apparatus.** Lachlan W. Child (to Aeriet Air Conditioner Co.). U. S. 2,025,802, Dec. 31. Various structural, mech. and operative details.
- Air-conditioning apparatus.** Dion K. Dean (to Foster Wheeler Corp.). U. S. 2,025,937, Dec. 31. Various structural and operative details.
- Odor removing apparatus for treating gases such as air.** Arthur B. Ray (to National Carbon Co.). U. S. reissue 19,791, Dec. 17. A reissue of original pat. No. 1,820,030 (C. A. 25, 5599).
- 6 Metal container for holding carbon dioxide, etc., under high pressures.** Daniel Mapes (to Walter Kidde & Co.). U. S. 2,026,133, Dec. 31. Various structural and mg details.
- Controlling sublimation of solid carbon dioxide for refrigerating and carbonating.** Henry C. Williamson (to Beckley-Ralston Co.). U. S. 2,026,335, Dec. 31. App. and various details of operation are described.
- Use of "dry ice" for refrigeration.** Lennie V. Van Ike (to Koldaire Utilities Co.). U. S. 2,024,490, Dec. 17. Various details of an app. are described.
- Still suitable for use with steam-operated absorption refrigerating apparatus.** Glenn F. Zellhoefer. U. S. 2,025,489, Dec. 24. Structural and operative details.

## 2—GENERAL AND PHYSICAL CHEMISTRY

FREDERICK L. BROWNE

- Frank Wigglesworth Clarke.** Charles E. Munroe. *J. Am. Chem. Soc.* 57, Proc. 21-30 (1935).—Biography with portrait.
- Fritz Ephraim.** Ed. Michel. *Helv. Chim. Acta* 18, 1448-64 (1935).—Obituary with list of publications.
- The life and works of Julien Flatau.** K. Hrynakowski. *Bull. soc. chim.* [5], 2, 2033-4 (1935).—Obituary and list of publications.
- H. Fonzes-Diacon.** 1868-1935. A. Astruc. *J. pharm. chim.* 22, 333-5 (1935).—Obituary.
- Ernest Gérard.** 1863-1935. F. Morvillez. *J. pharm. chim.* 22, 487-8 (1935).—Obituary with portrait.
- S. Waldhott**
- Jakob Meisenheimer.** Heinrich Wieland. *Z. Elektrochem.* 41, 817-20 (1935).—Obituary with portrait.
- The life and works of Maurice Vèzes.** Georges Dupont. *Bull. soc. chim.* [5], 2, 2017-32 (1935).—Obituary with portrait and list of publications.
- Letters of a Lausitzer alchemist from the years of 1496-1506.** W. Ganzmüller. *Angew. Chem.* 48, 761-4 (1935).—A historical treatise with 10 references.
- Karl Kammermeyer**
- Eighteenth-century English medicine.** John D. Cornie. *Proc. Roy. Soc. Med.* Oct., 1935; *Chemist and Druggist* 123, 759-60 (1935).—A historical account is given of leading English physicians, notorious quacks, systems of

medicine and scientific advances in chemistry and medicine during the 18th century. S Waldbott  
Nobel Prize winners in chemistry Harrison Hale  
*Ind Eng Chem, News Ed* 13, 462 (1935). E J C.  
Applications of chemistry to archeology Wm Foster.  
*J Chem Education* 12, 577-9 (1935)—Brief analytical findings are given of mortars and cements, beeswax, rock and lead Philip D. Adams

Second year chemistry for high school students Warren J Harman *J Chem Education* 13, 27 (1936).

E. H.  
Demonstrations as a substitute for laboratory practice in general chemistry II What, why and to whom shall we demonstrate? Herschel Hunt *J Chem Education* 13, 29-31 (1936), cf *C A* 29, 1691<sup>4</sup>. E H

Introduction to isomerism and structural theory. Y. L. Goldfarb and L. M. Smorgonsky. *J Chem Education* 13, 22 (1936) E H

Determination of chemical equivalents by the eudiometer method A T Lincoln and H P. Klug *J Chem Education* 12, 589-91 (1935) Philip D Adams

Influence of substituents on organic reactions A quantitative relationship G N Burkhardt *Nature* 136, 684 (1935), cf *C A* 29, 7767<sup>4</sup>. Julius White

Some component errors in determining the volume of a mole of oxygen Charles F. Eckels. *J Chem Education* 13, 39-40 (1936) E H

Advances in physics Henry A Barton *Ind Eng Chem, News Ed* 13, 475-6 (1935) F J C.

Helium content of the stratosphere F A Paneth and E Gluckauf *Nature* 136, 717-18 (1935)—Samples of air taken at altitudes up to 18.5 km had identical He contents A sample taken at 21.0 km contained 8% more He than samples taken below 18.5 km. Evidently winds cease between these heights. G M P

Iron in the sea and in marine plankton L. H. N. Cooper *Proc Roy Soc. (London)* B118, 419-38 (1935)—Fe was detd. colorimetrically by the 2,2'-dipyridyl or preferably 2,2',2''-tripyridyl method. In sea water Fe in true soln (Fe<sup>++</sup> and Fe<sup>+++</sup>) was less than 2 mg per cu m; most of the Fe was present in particulate form with a value 4-25 mg per cu m The plankton present in 1 cu m contained 0.5-2.3 mg Fe The soly of Fe<sup>++</sup> is controlled by that of a sparingly sol basic salt. Ferrihydrite and colloidal Fe compds. are concerned in the Fe metabolism of diatoms Zooplankton has a lower Fe requirement than diatoms When minute sea plants are eaten by herbivores, the excess Fe over requirements is excreted in fecal pellets, and more or less rapidly becomes available for a new growth cycle Forty-four references Joseph S Hepburn

Measurement of density with a float Seibi Torawshi and Ichizo Ouchi *J Chem Soc. Japan* 56, 1323-8 (1935)—The relation between the concn (0.119-0.575%) and the d of H<sub>2</sub>SO<sub>4</sub> was detd by means of a float. The change in d of Au sol prep'd by Bredig's method in an elec. field was followed by a float and the ratio (av.) between charge and the apparent mass of the colloid particle was estd; the value being  $0.7 \times 10^{-4}$  e s u The change in the d of PhNO<sub>2</sub> placed between condenser plates was studied also T. Katsura

The thermodynamic scale below 1°K W. H. Keesom *Physica* 2, 805-6 (1935)—Theoretical

B J C. van der Hoeven  
Effects of high shearing stress combined with high hydrostatic pressure P W. Bridgman *Phys Rev* 48, 825-47 (1935)—A method is described by which thin disks can be subjected simultaneously to hydrostatic pressures up to 50,000 kg/sq cm and shearing stresses up to the point of plastic flow Substances unaffected by these conditions were graphite, mica, NH<sub>4</sub>NO<sub>3</sub>, sugar, rosaniline, CuS, SiO<sub>2</sub>, tremolite, NiF<sub>2</sub>, CuO, MgO, Ag<sub>2</sub>S, Fe<sub>2</sub>O<sub>3</sub>, LiF, AgCl, K<sub>2</sub>CO<sub>3</sub>, NaBrO<sub>3</sub>, Si, Rochelle salt, HgNO<sub>3</sub>, and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> Substances that decomposed with detonation were celluloid, iodoform, PbO<sub>2</sub>, KMnO<sub>4</sub>, Ag<sub>2</sub>O, MnO<sub>2</sub>, (NH<sub>4</sub>)<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, Ag<sub>2</sub>NO<sub>3</sub>, Sr(NO<sub>3</sub>)<sub>2</sub>, Cr<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, K<sub>2</sub>SO<sub>4</sub>, 24H<sub>2</sub>O, Cr<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, K<sub>2</sub>SO<sub>4</sub>, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, and CuCl<sub>2</sub>·2NH<sub>4</sub>Cl·12H<sub>2</sub>O Combinations that reacted with detona-

tion were Cu + S, Fe<sub>2</sub>O<sub>3</sub> + Al, SiO<sub>2</sub> + Mg, K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> + Al, Si + MgO. Rubber, duprene, wood, paper and linen cloth were changed to horn-like substances, in some cases translucent. Bromothymol blue became insol Amorphous Se partially changed to the metallic form, but cryst. S became amorphous. PbO decomposed to the metal without detonation Red HgO and red PbO both changed to the black varieties Besides these qual. observations, curves of max. shearing stress against pressure were detd for substances showing no permanent change, and used to locate polymorphic transitions Breaks in the curves were found for Li, Sr, Ca, Ba, Cd, Zn, V, Mn, Sb, Te, I, La, Ce, Cr, Ti, Bi, Th, Sn, Y and Pr. No breaks were found for Be, B, C as graphite, Na, Mg, Al, Si, P, S, Pd, Cr, Fe, Ni, Co, Cu, Ge, As, Se, Zr, Cb, Mo, Ru, Rh, Pt, Ag, In, Ta, W, Re, Os, Ir, Pt, Au, Pb and U, these substances therefore probably have no transitions within this pressure range. L. S. Kassel

Powdered mercury A. Galatzky. *Bull. soc. chim* [5], 2, 1401-7 (1935)—Hg powder was prep'd. by the following procedure: Ppt. Hg<sub>2</sub>O from a dil. soln of Hg-NO<sub>3</sub> with NaOH Wash by decantation until neutral Clear the Hg<sub>2</sub>O most. Place 3-3.5 g of Hg<sub>2</sub>O in 200 cc H<sub>2</sub>O, and agitate vigorously. Reduce by adding 5% hydrazine hydrate at the rate of about 60 drops per min The color changes from brownish black to black, then to clear gray. Stop adding the reducing agent when the supernatant liquid is clear. Agitate 30 sec. more, decant, wash with 25 cc of EtOH, then with 20 cc of Et<sub>2</sub>O, and dry in thin layers at room temp in air or in a vacuum Remove visible drops of Hg To det. the Hg content dissolve 0.3-0.5 g in 2 cc. HNO<sub>3</sub>, dil to 100 cc, boil off nitrous vapors, add 1-2 drops of sat'd Fe(NH<sub>4</sub>)(SO<sub>4</sub>)<sub>2</sub>, and titrate with 0.1 N NH<sub>4</sub>CNS The Hg may also be detd as HgS To sep metallic Hg from combined Hg boil 0.5-1.0 g in 50 cc H<sub>2</sub>O, which contains not more than 2-3 drops of free HNO<sub>3</sub> The particles of Hg are protected by a film of Hg<sub>2</sub>O, the presence of Hg<sub>2</sub>O is caused by a reduction which is not quite complete. The usual analysis is metallic Hg, 89.4-99.8%, combined Hg, 0.1-0.2%; other elements, 0.1-0.3% Hg powder has those characteristics that would be expected of Hg having a large surface It oxidizes readily at room temp, it appears to be very poisonous, when freshly prep'd it has a strong odor identical with that of boiling Hg Two hundred and thirty-seven mg contg 99.9% Hg was exposed to the air for 12 hrs; 105 mg of Hg evapd and the residue contained 70.1% Hg This method of prep'n is more economical than that proposed by Krauss and Möhlmann (*C A* 24, 1308). G M P.

The electrolytic separation factor of the hydrogen isotopes under various experimental conditions A Eucken and K. Bratzler. *Z. physik Chem* A174, 273-90 (1935)—In order conveniently to make a large no of detns of the conditions affecting the electrolytic sep factor of H isotopes, the D content of the evolved H<sub>2</sub> was detd by measuring the thermal cond The sep factors found lay within wide limits (2.7 and 17), a dependence upon the nature of the cathode was indiscernible There was no parallelism between the characteristic overvoltage of the metal and the sep'n factor. An anodic preliminary treatment of the cathode increases the sep'n factor while the addn of an alkaloid-like material (α-naphthoquinoline) lowers it With increasing amperage the sep'n factor seems to increase when it is large at the beginning. There is no relation between the size of the sep'n factor and the slope of the logarithmic current density-potential curve R. H. Baechler

Experiments on electrical separation of the isotopes of lithium A Eucken and K. Bratzler. *Z. physik Chem* A174, 269-72 (1935)—Li was deposited on Hg from Li<sub>2</sub>SO<sub>4</sub> soln After repeated electrolysis the at wt changed only within the exptl error so that an extremely small change in the isotopic component can be prevent, the upper limit being 1.07% R. H. Baechler

Atomic weight of gallium G E. F. Lundell and James I Hoffman *J Research Natl Bur Standards* 15, 409-20 (1935) (Research Paper No 838), cf *C A* 17, 2207—



Pure metal was converted to the hydroxide, the sulfate and the nitrate, resp. These were then heated until they were changed to  $Ga_2O_3$  which was finally ignited at  $1200^\circ$  to  $1300^\circ$ . By this procedure the at. wt. is related directly to O. Preliminary tests showed that the metal was free from an appreciable film of oxide and did not contain occluded gases. The highly ignited oxide obtained through the hydroxide or sulfate contained no gases and was not appreciably hygroscopic, but the product obtained from the nitrate came to const. wt. slowly because of retained gases. The d. of  $Ga_2O_3$  was detd. to be 5.95 g./cc. The av. of 11 detns. gave 69.74 as the at. wt. of Ga.

P. S. Roller

The advantage of introducing quantum periods in the Mendeleev table. Arrigo Mazzucchelli *Gazz. chim. ital.* 65, 467-73 (1935).—A discussion of the manner in which the Mendeleev table of elements can be subdivided on the basis of the principal quantum nos. of the external electrons. Particular attention is paid to the added significance of the first small period and the octave group.

C. C. Davis

Compounds of the type  $BCl_3 \cdot n(OR)_3$ . III. Preparation of  $BCl_3 \cdot OCH_3$  and  $BCl_3 \cdot OCH_2CH_3$  from boron trichloride and methyl borate. Lgon Wiberg and Herman Smedsrud *Z. anorg. allgem. Chem.* 225, 204-8 (1935), cf. C. A. 26, 1540; 29, 5002.— $BCl_3 \cdot OCH_2CH_3$  was prepd. by reaction of 2 moles of  $BCl_3$  with 1 mole of  $B(OCH_2CH_3)_3$  in a Stock high-vacuum app. A 1:2-mole mixt. of the same reactants gave  $BCl_3 \cdot OCH_2CH_3$ . Electronic formulas are used to explain the formation and decomposition of the intermediate addn products involved.

J. H. Reedy

Dirac's spin theory and nonlinear field equations. W. Wessel. *Z. Physik* 96, 920-33 (1935).—Dirac's theory is extended in such manner that the elec. and magnetic fields satisfy nonlinear equations. The connection with Born's electrodynamics is discussed.

B. Swirles

Analytical expressions for the potential energies of diatomic molecules and their determination from spectroscopic data. I. Fgl. A. Hylleraas *Z. Physik* 96, 643-60 (1935).—The work of Morse (C. A. 23, 5406) and others on the detn. for a mol. with a given electron configuration of the potential-energy function which gives the observed spectroscopic quantities  $D$ ,  $r_{eq}$ ,  $B_v$ , is summarized, and it is shown how the results can be more simply obtained by the phase-integral method instead of direct use of Schrödinger's equation. A form of the potential energy const. 3 addnl consts. is then discussed, the consts. can be adjusted to give  $a_1(B = B_e - a(n + 1/2))$  and 2 higher terms in the energy formula, which is given in a form probably applicable to all homopolar diatomic mols. II. *Ibid.*, 661-8.—The theory of the above paper is applied to the  $\Sigma$  states of CdH and the  $(1^1\Sigma)$  states of  $N_2$ .

B. Swirles

A general method of statistics and its application to chemical energy. Georges Allard *Ann. phys.* [11], 4, 305-76 (1935).—By an extension of the method of Planck, a new statistical method is obtained which allows a finer analysis of the elementary phenomena than the older methods. The results of Bose-Einstein are obtained by assuming that each elementary cell of the phase extension is able to contain any no. of particles and those of Fermi-Dirac by assuming the no. cannot exceed unity. The method was applied to the case of mixts. with or without chem. reaction when the particles are statistically independent. This means that the presence of particles of a certain kind in one cell has no effect on the probability of the presence of particles of another kind in that same cell and that the laws of distribution of each species are the same as if they existed alone. It is also shown that all the classical results of chem. energy are established by this purely statistical method which has led to the study of condensed phases. The coexistence of several phases is interpreted by assuming that the energy of interaction between mols. is, on the av., very different when there is a change from one phase to another. Then it is the principle of max. probability that should govern the distribution of the mols. between the different phases. A general form of the law of mass action is obtained and in

the limited case where the substances reacting are perfect gases, the chem. consts. can be calcd. for monatomic mols., diatomic with quantized rotation, diatomic with internal vibration.

A. A. Vernon

Thermodynamic potentials and affinity. Pierre Van Rysselberghe *Compt. rend.* 201, 1126-7 (1935).—Math. Cf. de Donders, C. A. 19, 3044. C. A. Silberrad.

Coercive force of magnetite powders. V. H. Gottschalk. Progress Repts.—Met. Div. 10. Mineral physics studies. Bur. Mines, Rept. Investigations No. 3268, 83-90 (1935).—Coercive force increases as grain size decreases and is directly proportional to sp. surface (sq. cm. surface area/g.). Data are given for 30 hysteresis curves of magnetite from the Urals Mts. and from Hayden slag.

A. H. E.

Entropy of manganous ammonium sulfate at temperatures close to zero absolute, in relation to the magnetic anisotropy of the salt at room temperature. K. S. Krishnan and S. Banerjee *Proc. Indian Acad. Sci.* 2A, 82-5 (1935).—It is possible to calc.  $\theta_m$ , which det. the deviation of the magnetic entropy of the crystal from its ideal value of  $R \log_e (2J + 1)$  from measurements on the magnetic anisotropy of crystals at room temps. It is possible to predict the entropy-temp. curve close to  $0^\circ$  abs. This curve plays an important part relative to the production of low temps. by adiabatic demagnetization of paramagnetic crystals and in the calcn. of sp. heats in this temp. region.

James H. Hibben

Magnetic study of ferric oxide sols. Augustin Bontaric and Régis Bonneville *Bull. soc. chim.* [5], 2, 1938-2008 (1935).—The magnetic susceptibility of concd colloidal sols. of  $Fe_2O_3$  (contg. up to 75 g./l.  $Fe_2O_3$ ) was detd. to obtain the sp. coeff. of the oxide by the method of mixts. After verifying the applicability of that law to  $Fe_2O_3$  sols it was proved that the sp. coeff. of magnetization of the colloidal oxide is not sensibly altered by the length of dialysis. The coeff. decreases in proportion to the age of the sol, the decrease being the more rapid the higher the temp. at which the sol is kept. A very definite effect of temp. on the magnetic properties of the sol was revealed.

F. L. Browne

Diamagnetism of alkyl acetates. Donald B. Woodbridge *Phys. Rev.* 48, 672-82 (1935).—The magnetic susceptibility was detd. from  $5^\circ$  to  $70^\circ$  for MeOH, MeOAc, EtOAc, PrOAc, BuOAc and AmOAc. The mol. susceptibility of the acetates increases almost linearly with the no. of  $CH_2$  groups added.

L. S. Kassel

Determination of paramagnetic susceptibility of crystals of salts of rare earths by determining paramagnetic rotatory power. Jean Becquerel *Compt. rend.* 201, 1112-15 (1935).—Measurements of the paramagnetic rotatory power along the optic axis of the ethylsulfates of Pr, Nd, Dy and Er from the temp. of liquid He to  $20^\circ$  confirm Van Vleck's conclusion that in such cases the ratio between magnetization and paramagnetic rotation is const. (cf. C. A. 28, 4280).

C. A. Silberrad

Conception of a complex viscosity and its application to dielectrics. A. Cervant *Trans. Faraday Soc.* 31, 1582-90 (1935), cf. C. A. 29, 7143.—The conception of complex viscosity,  $\eta_c$ , is introduced instead of the simple, real viscosity,  $\eta$ , to aid in explaining observed decreases with increasing frequency, of the apparent viscosity in cases concerned with mol. vibrations in amorphous solids such as acoustical waves and dielec. losses. Special application of the theory to dielec. losses is discussed.

C. L. P. Jeffreys

Dielectric anomalies of Seignette salt. Hans Staub *Naturwissenschaften* 23, 728-33 (1935).—A review of the anomalous behavior of Seignette salt which in a general way can be described as the elec. analog of a ferro-magnetic substance.

B. J. C. van der Hoeven

A new seignettelectric substance. G. Busch and P. Scherrer *Naturwissenschaften* 23, 737 (1935).— $KH_2PO_4$  shows the same anomalous relationship between dielec. const. and temp. as Seignette salt. At room temp. the main dielec. const.  $\epsilon_{33}$ , along the  $c$  axis, is 30, it rises from  $-50^\circ$  down to  $-130^\circ$  to a max. of 130 and drops off to a value of 7 near  $-200^\circ$ . The 2 Curie points are  $-130^\circ$  and  $-105^\circ$ . The piezoelec. modulus  $d_{31}$  runs parallel with

$\epsilon_a$  No change in crystal structure was found by the Debye-Scherrer method at the upper Curie point. Crystal platelets from a soln. satd. at the b. p., slowly cooled, of 1 sq. cm. area, 1 mm. thick, were used for the expts. Al foils were glued on as electrodes. Similar phosphates and arsenates are now being studied. B. J. C. v. d. H.

The dielectric constants of hydrogen at pressures up to 1425 atmospheres and at temperatures of 25° and 100°. A. Michels, P. Sanders and A. Schipper. *Physica* **2**, 753-6 (1935).—The dielec. const.  $\epsilon$  of  $H_2$  was detd. by the method previously described (C. A. 27, 2875); the accuracy was  $\pm 0.0005$  or better. Values for the const. at 24.9° vary from 1.00028 to 1.00500 for 1.15 and 1425.4 atm., resp., at 99.9° from 1.00163 at 8.03 to 2.025 at 1425.4 atm. The value of const.  $P = (\epsilon - 1)/(\epsilon + 2)d$  is 2.017, the value of  $\epsilon$  at standard condition is 1.002037.

B. J. C. van der Hoeven  
Transitions in camphor and chemically related compounds. I. Dipole rotation in crystalline solids. W. A. Yager and S. O. Morgan. *J. Am. Chem. Soc.* **57**, 2071-8 (1935).—A study of the dielec. properties of *d*-camphor, *d*-camphor, *d*-camphonic anhydride, borneol, isoborneol and bornyl chloride showed that these materials undergo solid-solid transitions. Above the transition the value of the dielec. const. of the solid is that normally expected of the polar material in the liquid state. Below the transition the dielec. const. has a low value, approx. equal to the square of the optical  $n$ . This dielec. behavior is explained by the rotation of the dipoles in the solid. II. Vibration of atomic groups. Addison H. White and S. O. Morgan. *Ibid.* **57**, 2078-89.—A rise of about 18 cal./degree mole in the sp. heat of *d*-camphor at the transition at  $-30^\circ$  was observed and is attributed to unusually energetic intramolecular vibration at the transition temp. The rise of mol. rotation at the transition which is evidenced by the dielec. data (cf. above) is qualitatively explained in terms of Pauling's theory of mol. rotation in crystals (cf. C. A. 24, 5558) by assuming that the forces opposing this rotation are greatly diminished by a transition which reduces the rigidity of the mol. The behavior of total polarization of *d*-camphor in dil. soln. is explained by assuming that at room temp. dipole moment is smaller and at. polarization much larger than formerly supposed and that the latter polarization begins to decline with temp. below  $-10^\circ$  when the transition to the more rigid mol. begins. Similar transitions are observed in crystals of derivs. of cyclohexane. H. H. Rowley.

Calculation of dipole interaction. R. P. Bell. *Trans. Faraday Soc.* **31**, 1557-60 (1935).—The method of Martin, cf. C. A. 28, 7099, is shown by comparison with statistical treatment to be valid for sufficiently small values of the dipole moment.

C. E. P. Jeffreys  
Dipole induction and the solvent effect in dipole-moment measurements. F. C. Frank. *Proc. Roy. Soc. (London)* **A152**, 171-96 (1935).—The ideas of Weigle and Higashi (C. A. 27, 3371, 28, 2622) are developed into a theory that accounts for the effect of the solvent on the apparent dipole moment. A classification of mol. geometry shows the very different solvent effects in the different classes. The empirical laws previously advanced for the solvent effect apply only to a single class. The theory is in good qual. and approx. quant. agreement with measurements. Bearings of the theory on dipole induction in radicals forming part of the polar mol., the attraction of solvent mols. by dipole forces, the structure of amphoteric ions, and the form of the dispersion curve in polar liquids, are discussed.

L. E. Steiner  
Dipole measurements of isomeric platinum complexes. K. A. Jensen. *Z. anorg. allgem. Chem.* **225**, 97-114 (1935). cf. C. A. 24, 2961.—Dipole moments were detd. for the  $\alpha$ - and  $\beta$ -forms of the thioether derivs. of the type  $PtX_2(SR)_2$  (where  $X = Cl, Br, I, NO, NO_2$ , and  $R = Et, Pr, iso-Pr, iso-Bu, sec-Bu, C_6H_5, CH_3$ ), also those of  $PtCl_2(EtSe)_2$ . The values are approx.  $2.4 \times 10^{-18}$  for the  $\alpha$ -derivs., and  $9 \times 10^{-18}$  for the  $Cl$  and  $Br$  derivs., and  $13 \times 10^{-18}$  for the  $NO_2$  and  $NO$  derivs. of the  $\beta$  type. These results are in line with the assumption that the  $\alpha$ - and  $\beta$  forms are *cis-trans* isomers,

however, the  $\alpha$ -form must have the *trans* configuration, and the  $\beta$ -form the *cis* configuration. Since the dipole moment of the  $\alpha$ -derivs. is not 0, the structure is not of the plane type, but the 4 radicals must be on the same side of the plane. A value of  $110^\circ$  for the valence angle of the  $S$  atom is indicated. Mol.-wt. detns. of the  $\alpha$ - and  $\beta$ -isomers in  $C_6H_6$  show that the compds. are unimol. at ordinary concns., but the  $\beta$ -forms are assoc. in concd. solns. No equil. exists between the  $\alpha$ - and  $\beta$ -forms in  $C_6H_6$  except with the diethyl selenide derivs. J. H. R.

Reactivity and dipole moment. E. ducl. Hertel and Eugen Dumont. *Z. physik. Chem.* **B30**, 139-48 (1935), cf. C. A. 29, 6216.—Dipole moments were detd. for  $PhNMe_2$ ,  $p$ - $BrC_6H_4NMe_2$ ,  $p$ -dimethylaminodine,  $p$ -dimethyltoluidine,  $p$ -dimethylaminobenzonitrile,  $p$ -nitrodimethylaminodine and  $p$ -nitrosodimethylaminodine. The results are discussed in connection with the reactivity of the substituting groups. G. M. Murphy.

Remark on the work of I. Sakurada. "Simultaneous double and triple complexes in dipole association." E. ducl. Hertel and Eugen Dumont. *Z. physik. Chem.* **B30**, 149-51 (1935); cf. C. A. 29, 6117.—Polemic.

G. M. Murphy  
A new addition to the potential-energy expression for diatomic homopolar molecules—applications to  $CdH$  and  $N_2$ . E. L. Egl. A. Hylleberg. *Physik. Z.* **36**, 599-600 (1935).—Explicit expressions are given for the moment of inertia and energy of a diatomic mol. which depend on a phase integral method for evaluation. This method and the expressions are applied to the construction of potential curves for  $CdH$  and the results agree closely with those obtained by Rydberg (C. A. 26, 2375) by a graphic method. The method applied to  $N_2$  leads to a value of the disson. energy close to Herzberg and Spenser's value 7.34 v. (C. A. 28, 5321).

A. B. F. Duncan  
Configuration of mercuric halides. W. J. Curran and H. H. Wenzke. *J. Am. Chem. Soc.* **57**, 2162-3 (1935), cf. C. A. 29, 3572.—The elec. moments of  $HgPh$ ,  $HgCl$ ,  $HgBr$ , and  $HgI$ , were found to be 0.42, 1.29, 1.01 and 0.58  $\times 10^{-18}$ , resp. Dioxane was used as a solvent. The configuration of the halides is not linear.

H. E. Phipps  
Reciprocal solubility and surface tension. II. Surface activity and dipole moment. V. K. Semenchenko and T. N. Ivanova. *J. Gen. Chem. (U. S. S. R.)* **5**, 897-13 (1935); cf. C. A. 28, 7113.—The crit. temp. of soly. was detd. for the pair nicotine- $H_2O$ , in the presence of  $C_6H_5COOH$  (I),  $C_6H_5CONH_2$  (II),  $C_6H_5OH$  (III),  $C_6H_5CN$  (IV),  $C_6H_5CHO$  (V) and  $C_6H_5Cl$  (VI), in concns. up to 0.8 mol. %. The interface surface tension was measured by the drop method for the same pair in the presence of I (0, 0.14, 0.26, 0.39 and 0.44 mol. %), II (0, 0.21, 0.39, 0.61 and 0.80 mol. %), and III (0.19, 0.24, 0.38 and 0.60 mol. %), at 70° and 80°, also in the presence of VI (0.29, 0.33, 0.49 and 0.68 mol. %), IV (0.16, 0.37, 0.57 and 0.73 mol. %) and III (0.29, 0.35, 0.45 and 0.60 mol. %) at 60° and 70°. Parallelism between the effect of admixtures on crit. temp. and on surface tension, found in previous work, was confirmed. Surface tensions of solns. of the 6 org. compds. in concns. of 0, 0.006, 0.01, 0.017 and 0.025 mol. % were detd. at 25°. No definite conclusions could be drawn from the results as to the relation between the dipole moment and surface activity. The surface tensions of solns. of the same org. compds. in nicotine in concns. of 0, 0.10, 0.15, 0.20, 0.25, 0.30, 0.40 and 0.50 mol. % were measured at 25°. All the compds. proved to have a high surface activity, which was directly proportional to the dipole moment for compds. not contg. N and inversely proportional to the dipole for compds. contg. N. The surface tensions of solns. of I, III, VI and IV in  $C_6H_5NO_2$  in concns. of 0, 0.10, 0.20, 0.30, 0.40 and 0.50 mol. % were detd. at 25°. The results were the same in regard to compds. not contg. N. In the interface nicotine- $H_2O$  the activity of the chloride, acid and alc. was inversely proportional to dipole moment, and in the case of nitrile, aldehyde and amide the proportionality was direct.

S. L. Madorsky  
The optical dispersion of hydrochloric acid gas in the

infrared R. Rollefson and A. H. Rollefson *Phys. Rev.* 48, 779-85 (1935).—The  $n$  of HCl gas was detd. for the range 1-10. The results yield a value of  $1.00 \times 10^{-10}$  e. s. u. for the effective charge of the rotator-vibrator and  $1.18 \times 10^{-18}$  e. s. u. for the elec. moment. The former value shows that the contribution of the rotator-vibrator to the  $n$  extrapolated to infinite wave length is far too small to account for the discrepancy between that quantity squared and the temp-independent part of the dielec. const. The elec. moment obtained in this work agrees with that detd. from the dielec. const., and indicates that the low absorption coeffs found for pure rotation lines in the far infrared must be erroneous. L. S. K.

Excitation by collision of intramolecular vibrations in gases and gas mixtures measured by the dispersion of sound. III. Measurements on nitrous oxide. A. Fucken and H. Jaacks *Z. physik. Chem.* B30, 85-112 (1935), cf. C. A. 29, 1206<sup>1</sup>.—The dispersion of sound was measured in  $N_2O$  at frequencies between 288 and 2350 kc, temp between  $-60^\circ$  and  $200^\circ$  and pressures from  $1/2$  to 1 atm. The foreign gases A, He, D, H,  $CO$ ,  $CH_4$ ,  $NH_3$  and  $H_2O$  were also added in various mixts. and a few measurements were made with  $CO_2$  in the presence of  $H_2O$  and  $H_2$ . Double collisions of  $N_2O$  with  $NH_3$  and other  $N_2O$  mols result in deactivation in a similar way to the deactivation of  $CO_2$  by  $H_2$ . Investigation of the effect of temp on the heat of vibration shows that H excites the valence bonds of  $N_2O$ . G. M. Murphy.

Density fluctuations at the critical point. Jacques Yvon. *Compt. rend.* 201, 1099-1102 (1935).—It is shown mathematically that the opalescence observed in a substance at the crit. point is attributed to fluctuations in  $d$  caused by gravity. C. A. Silberrad.

A theoretical study of the liquid-side resistance to gas absorption by a liquid drop. Siroji Hattai and Arimasa Baba. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 544-6 (1935), cf. C. A. 28, 5728<sup>1</sup>.—Three possible formulas are derived for the assumption that (a) no damping of the turbulence (const. turbulence) which is created in the drop formation takes place, (b) gradual damping takes place during the fall of the drop, and (c) perfect damping takes place at the start of the fall, i. e., no turbulence during the fall. An experimental study of the liquid side resistance to gas absorption by a liquid drop. *Ibid.* 546-50.—The absorption of  $CO_2$  in  $H_2O$  was studied with a great excess of  $CO_2$  and with varying height of fall. The absorption during drop formation was very small and the total absorption was considered therefore as absorption during the fall. The exptl. results are presented and the best agreement is with case (c). Most of the absorption took place in the initial stages of the fall and it is therefore more effective to increase the frequency of drop formation, than to increase the height of fall. The absorption formula for case (c) is:  $C/C_0 = (6/\sqrt{\pi}) \sqrt{q} - 3q$ , for  $C/C_0$  less than about 0.015, where  $q = kh/a^2$ ,  $C$  is the mean concn. at time  $t$ ,  $C_0$  is the satn. concn.,  $a$  is the radius of the drop and  $k$  is the diffusion coeff. of the dissolved gas. Karl Kammermeyer.

The determination of the boiling point of small quantities of material. R. Dolque. *Bull. soc. chim.* [5], 2, 1832-47 (1935).—App. and technic for the microdetn. of vapor pressure (and of the b. p.) are described. The app. consists essentially of a vaporization chamber whose vol. is kept const. by a Hg piston. By coupling 2 of these chambers, a fractional distn. can be made, and the purity of the product tested. Temp. and pressure data are given for  $H_2O$ ,  $CaH_2$ ,  $CCl_4$  and for the azeotropic mixts.  $H_2O$ -cyclohexane,  $AcOH$ - $CCl_4$ ,  $H_2O$ - $CCl_4$ ,  $CaH_2$ -cyclohexane and  $EtOH$ - $CCl_4$ . G. M. P.

Parachor as a function of density and molecular volume of compounds of a given class. I. R. Morozov *J. Gen. Chem.* (U. S. S. R.) 5, 1020-3 (1935).—In place of Sugden's parachor  $P = V \gamma^{1/4}$ , where  $P$  is mol. parachor,  $V$  the mol. vol. and  $\gamma$  the surface tension, the following more nearly universal parachor is recommended.  $P = (V + V_1)^{1/4} V_1^{1/4} (2d)^{1/4}$ , where  $V_1$  is the sum of vols. of the elements in the compd.,  $V_1$  is the mol. vol. and  $d$  the density of the

compd. in the liquid or solid state. This equation is used to calc. the parachors for a no. of homologous org. series, such as hydrocarbons, alcs., aldehydes, ethers, acids, etc. If Sugden's equation is combined with the equation suggested by  $M$ , the following equation is obtained  $\gamma = (V + V_1)^{1/4} V_1^{1/4} (2d)^{1/4}$ , thus making it possible to calc.  $\gamma$  as a function of  $V$ ,  $V_1$  and  $d$ .

S. L. Madorsky. Sigmoid curves of internal friction. M. Usanovich. *J. Gen. Chem.* (U. S. S. R.) 5, 996-1001 (1935).—The existence of a new type of viscosity diagram for a binary system, characterized by the fact that the curves do not pass through a max. with lowering of temp., is established. Such a diagram is obtained in those cases where the components react to form a compd. with a lower viscosity than that of one of the components. This happens when one of the components is an assoc. liquid and the product of reaction a normal liquid, e. g.,  $H_2SO_4$ - $CaH_2NO_2$ . As  $CaH_2NO_2$  the inflection points on these types of isotherms shift with rising temp. toward the liquid with the higher viscosity when  $\eta_{AB}$  is less than  $(\eta_A + \eta_B)/2$ , where  $\eta_A$ ,  $\eta_B$  and  $\eta_{AB}$  are viscosity coeffs. of the components A and B and of the product of reaction AB, resp., or toward the liquid with the lower viscosity when  $\eta_{AB}$  is greater than  $(\eta_A + \eta_B)/2$ . S. L. Madorsky.

Viscosity of the system chlorine-sulfur. A. A. Mazel. *J. Gen. Chem.* (U. S. S. R.) 5, 1066-72 (1935).—Viscosity isotherms for the system Cl-S were constructed for  $-15^\circ$ ,  $0^\circ$  and  $25^\circ$ . Compn. of the system varied between 39.96 and 71.21 mols % of Cl. In view of the fact that the system is very complicated through the formation of a no. of compds., such as  $S_2Cl_2$ ,  $S_2Cl_4$ ,  $SCl_2$ ,  $S_2Cl_6$ ,  $S_2Cl_8$ , and  $S_2Cl_{10}$ , the viscosity isotherms are smooth curves, rising with concn. of S and giving no clue as to existence of these compds. S. L. Madorsky.

Viscosity of liquid phosphorus. Alan N. Campbell and Sidney Katz. *J. Am. Chem. Soc.* 57, 2051-5 (1935), cf. C. A. 29, 1304<sup>1</sup>.—The viscosity of liquid P was detd. from  $20^\circ$  to  $140^\circ$  in a modified Ostwald viscometer. Heating the P from  $80^\circ$  to  $120^\circ$  and chilling did not alter its viscosity at  $45^\circ$ . Assoc. was appreciable below  $45^\circ$ . The authors thought it probable that the inner equl. shifts so rapidly on cooling that Smits' theory is not invalid. H. E. Phipps.

Viscous properties of polyisobutylene. John D. Ferry and George S. Parks. *Physica* 6, 356-62 (1935).—The viscosity of polyisobutylene was detd. from  $70^\circ$  to  $175^\circ$  and from  $-53^\circ$  to  $+24^\circ$  by the falling-sphere and concentric-cylinder methods, resp. The viscosity was 19 poises at  $175^\circ$ ,  $10^{10}$  at  $-53^\circ$  and about  $10^{18}$  poises at  $-76^\circ$ .  $-76^\circ$  is the midpoint of the "transition region" for polyisobutylene glass. Macroscopic and microscopic interpretations of viscosity anomalies are discussed. G. M. P.

New liquids for determining refractive indexes by immersion. L. Déverin. *Schweiz. mineral. petrog. Mitt.* 14, 529-30 (1935).—The following mixts are suggested for this purpose: (a) 4,4'-dibromodiphenyl oxide (5 parts), benzyl sulfide (7), isooquinoline (5), n 1.614; (b) isooquinoline (1), 1,4-dibromonaphthalene (1), n 1.630; (c) benzalaniline (1), 9-bromophenanthrene (1), n 1.676; (d) thionaphthene (1), methylendiphenylamine (1), n 1.637; (e) dibenzylamine (5), dibromothionaphthene (5), ethylene trithiocarbonate (2), n 1.672; (f) dibromostyrolene (5), phenyl orthoformate (9), dibromothionaphthene (5), n 1.663; (g) dibromothionaphthene (7), phenyl disulfide (5), n 1.683; (h) dibromothionaphthene (7), dibromonaphthalene (5), n 1.694; (i) dibromonaphthalene (1), phenyl disulfide (2), ethylene trithiocarbonate (2), n 1.714; (j) dibromothionaphthene (1), phenyl orthoformate (1), n 1.681. C. A. S.

Method of determining the structure of sublimates. V. G. Zhurav. *Zashchita Lab. 4*, 821-3 (1935).—The structure and degree of dispersion of sublimates can be detd. by placing an object glass in the path of the gases during sublimation, and then examg. with a microscope. Chas. Blanc.

Vapor pressure of zinc chloride, lead chloride, cadmium

chloride, cuprous chloride and silver chloride D N Tarasenkova and P A Kozhmyakov *J Gen Chem* (U S S R) 5, 830-5(1935) —The vapor pressure of  $ZnCl_2$  in the interval 516-729° is  $7.4 \cdot 10^{-4}$  mm. It can be represented by the equation  $\lg p = 8.7849 - (6952.6/T)$ . The vapor pressure of  $PbCl_2$  in the interval 650-942° is  $4.5 \cdot 10^{-4}$  mm. It can be represented by the equation  $\lg p = 8.3421 - (7832.1/T)$ . The vapor pressure of  $CdCl_2$ ,  $Cu_2Cl_2$  and  $AgCl$  was determined in the intervals 618-932° ( $p = 3$  to  $722.3$  mm), 632-1070° ( $p = 3$  to  $215.7$ ) and 852-1120° ( $p = 3$  to  $50.9$  mm), resp.

S L Madorsky  
Aggregate states L I Allumov *J Gen Chem* (U S S R) 5, 843-7(1935) —The increase of vol of a given substance heated from 0 abs through the solid, liquid and gaseous states is analyzed mathematically on the basis of Pictet's empirical rule. The following empirical rule is suggested for the liquid state: the relative increase of vol of a substance heated from  $m$  to  $p$  does not depend on the nature of the substance, but is a const., i.e.  $\beta_L(T_p - T_m) = C_L$ , where  $\beta_L$  is the coeff of expansion of the liquid,  $T_p$  and  $T_m$  are  $b$  p and  $m$  p, and  $C_L$  is a const. This rule is verified for 6 morg and 23 org compds.

S L Madorsky  
Methods for measuring vapor pressure of metal salts D N Tarasenkova, A N Grigorovich and A V Bogoslovskaya *J Gen Chem* (U S S R) 5, 924-33(1935) —Methods for detg the vapor pressure of metal salts, developed by Volmer (C A 24, 538) and Jellinek and Rosner (C A 24, 276), were compared experimentally with the method of  $b$ -p detn at reduced pressures, in the case of  $PbCl_2$  at 435-945°. Johnston's statistical method (C A 2, 2033) was tested in the case of pressure of dissociation of  $CuCl_2$  at 430-550°. Volmer's method was found difficult because of the corrosive effect of metal salts on the quartz container, it also gives inaccurate results. Jellinek and Rosner's method gave more accurate results. The method of  $b$ -p detn at reduced pressures is suitable for measuring higher vapor pressures of substances that do not decompose at high temps. Johnston's statistical method gives accurate results for high vapor pressures and is suitable in cases where the compd dissociates, giving off a gas.

S L Madorsky  
The growth of metal crystals in metal vapors IV M Strassman *Z physik Chem* B30, 132-8(1935), cf C A 28, 6041<sup>1</sup> —Te crystals were grown by sublimation in an atm of H at pressures between 0.01 and 7.0 mm and temps below and above the  $m$  p. The crystals obtained at low pressures were measured, the results disagreeing with the theory of Strassman and Kashaev (cf C A 29, 5324<sup>1</sup>). Similar discrepancies are also to be expected for Se.

G M Murphy  
The arbitrarily caused crystallization of melts C Weygand *Z anorg allgem Chem* 224, 265-72(1935) —Specific centers of crystal in melts of org compds are classified as crystals of identical kind, 1) nucleating desmotropes and of foreign kind, II (such as those of homologs, derivs, isomers and polymers). For I crystal starts instantaneously, with a definite crystal form in a certain temp interval. Some seed crystals may produce several crystal modifications in a melt, depending on temp, but only one at any definite temp. For II, crystal may depend on whether complete crystals or fragments are used, whether the seed crystals were obtained from a melt or from some particular solvent, and on the age of the crystals. Although crystal caused by II often starts more slowly than for I, the kind of crystal started is just as specific. Any seed crystals of kind II, prep'd by any definite method, which starts crystal in a melt, always starts a specific kind at any given temp and pressure.

L E Steiner  
The electrical resistance of hydrogen saturated wires of palladium alloyed with silver and with gold A Sieverts and H Hagen *Z physik Chem* A174, 247-61(1935) —The pressure-conc isotherms of alloys with 5 and 10% Ag are similar to those of pure Pd, at 15% they show hysteresis, at 200° an approaching region in which increase in pressure produces no change. All other isotherms are

parabolic. Previous observations on the effect of Ag content on solvent power of the alloys for H were substantiated and extended. The pressure resistance isotherms up to 30 atm % Ag resemble those of Pd. The change in resistance decreases with increase in Ag content. In alloys of 30 and 40% Ag the resistance is decreased by the absorption of H. The Pd Au alloys behave similarly to the Pd Ag alloys. R H Baerlein

The electron theory in metals; superconductivity. L Brillouin *Rev gen Electric* 38, 491-501(1935) —Work on the cond of metals and especially the phenomenon of supercond is reviewed and the principles of the electron theory which suffices for the cond of metals are explained, for the elucidation of supercond, however, this theory is not yet sufficient. The latest theories of Gorter, London, Meissner, et al have not succeeded in explaining the problem entirely.

M Hartenstein  
Superconductivity and other low-temperature phenomena J C McLennan *Proc Roy Soc (London)* A152, 1-46(1935) —A symposium. Among the subjects discussed are lambda phenomena in liquid He, the magnetic effects occurring on transition to the superconducting state, interpretations of supercond, and the sp. heat of Fe NiAl alloy below 1°K and preliminary detn of the thermodynamic temp scale.

L E Steiner  
An expression of deliquescence and of efflorescence A Damens *Bull soc. chim* [5], 2, 1893-1901(1935) —It is not generally recognized that deliquescence and efflorescence depend upon the relative humidity of the air. These terms, as commonly used, are incorrect. It is proposed to substitute for these terms the crit hygroscopic state (Hc). This is the relative humidity at which the compd (or its satd aq soln) is in equl with the atm. The temp. should also be specified. The max Hc is the limit of deliquescence; the min Hc is the limit of efflorescence. Also in *IX Congr intern chim pura applicada, Madrid* 3, 535-46(1934).

G M P  
The determination of the critical hygroscopic state M. Driesms *Bull soc. chim* [5], 2, 1901-7(1935); cf preceding abstr. —App and technic for detg the vapor pressure of salts and of their solns are described. This was detd for 75 satd aq solns at 20° and at 25°, and for some of the solids at these temps. Upon dividing this pressure by the vapor pressure of  $H_2O$ , the crit hygroscopic state (Hc) is detd. Hc max is detd with the satd soln, Hc min is detd with the salt.

G M P  
Effect of surface perturbations upon the interior of real crystal systems D Balarev and Vera Christoforova *Z physik Chem* B30, 152-6(1935), cf C A 29, 2814<sup>1</sup> —Vapor pressure detn of the system  $BaCl_2 \cdot 2H_2O \rightleftharpoons BaCl_2 + H_2O + H_2O$  with pure substances and when mixed with other salts confirms the theory previously proposed.

G M Murphy  
The Rayleigh scattering in crystals F Matossi *Z Physik* 96, 695(1935), cf C A 29, 2042<sup>1</sup> —Correction of an error in the case of quartz. The agreement with observation is improved.

B Swales  
The crystal structure and electron configuration of the transition and alkali metals U. Dehlinger *Z Physik* 96, 620-33(1935) —The empirical data show that the crystal structures of the transition metals be symmetrically about the Cr, Mo, W column. It is shown how the structures are detd by the gradual building of the d-shell. Zero crystal momentum of the d-electrons leads to a body-centered cubic lattice, otherwise the structures are cubic or hexagonal of closest packing. The stability of a body-centered cubic lattice for Fe is deduced from its ferromagnetic properties.

B Swales  
Explosive antimony II Its structure, electrical conductivity and rate of crystallization C. C. Coffin *Proc Roy Soc (London)* A152, 47-63(1935), cf C A 29, 974<sup>1</sup> —The polished and etched surfaces of an explosive, electrolytic deposit of Sb shows a heterogeneous gel like structure in which one phase is oriented parallel to the lines of the depositing current. This oriented phase is regarded as an  $SbCl_3$ -Sb complex, and the other phase as probably amorphous Sb. For fresh deposits, the sp elec. resistance,  $\rho$  is about  $10^4$  times that of ordinary Sb.



detd from 120° to 450°K. and found to be 0.31 at the higher temp and rising steadily below 350°K. to 0.55 at 120°K. This rise with decreasing temp is compared to similar results obtained with Pt and W surfaces and discussed on the basis of 2 distinct types of adsorbed H<sub>2</sub>. Comparison of the  $\alpha$  obtained by numerous investigators on different surfaces led to the conclusion that the exchange of energy between H<sub>2</sub> gas moles and an adsorbed layer of H<sub>2</sub> is measured in each case. An attempt to remove this adsorbed layer lowered  $\alpha$  from 0.31 to 0.18 at 350°K. H. H. R.

**Exchange of energy between diatomic gas molecules and a solid surface** J. M. Jackson and A. Howarth *Proc Roy Soc (London)* A152, 515-29(1935)—The theory of accommodation coeff. given by Jackson and Mott, cf. C. A. 26, 5489, is extended to a gas composed of diatomic molecules. Two limiting cases are discussed. The gas mol is treated as an oscillator, and as a plane rotator. In the first case the exchange of energy between rotations of the gas moles and vibrations of the solid is negligible. In the second, the effect of the rotation is very small for H<sub>2</sub> and somewhat larger for O<sub>2</sub>, taking the same repulsive field as the solid in each case. Under these conditions the energy exchange is larger the larger the moment of inertia of the gas mol. C. B. P. Jeffreys

**Absorption of some organic solvents by Yamagata absorbing clay** M. Uchida *J Soc Chem Ind, Japan* 38, Suppl. binding 513-14(1935)—The clay absorbed much H<sub>2</sub>O selectively, but only a little of each of 20 common org solvents. The swelled vol seemed to have no systematic relation with either dipole moment or dielectric const of the solvent. The relative viscosity of the system clay sol-H<sub>2</sub>O-Et<sub>2</sub>O became infinitely great near the max point of the H<sub>2</sub>O-alc. system. A kind of base-exchange reaction was observed between clay and HCl, NaOH and NaCl solns., and the reaction velocity increased rapidly with increasing concn of the electrolyte soln. Karl Kammermeyer

**Absorption of hydrogen sulfide and sulfur dioxide from an air current** V. A. P'yankov *J Gen Chem (U S S R)* 5, 1112-18(1935)—I-treated activated charcoal has an absorptive power for H<sub>2</sub>S and SO<sub>2</sub> at 20°, 3 and 18 times as great, resp., as pure activated charcoal when the concn of either gas is 0.05%, and 2.2 and 1.2 times as great when the concn is 0.02%. At 40° the ratios are higher for H<sub>2</sub>S absorption and about the same for SO<sub>2</sub> absorption. S. L. Madorsky

**The effect of a foreign solid phase on the freezing point of water and dilute aqueous solutions** I. System quartz sand water A. V. Rakovskii, D. N. Tarasenkova and A. V. Komandin *J Gen Chem (U S S R)* 5, 1273-8(1935)—In a mixt of H<sub>2</sub>O and sand, the  $f$  of H<sub>2</sub>O, for any given ratio of the two components, is inversely proportional to the total surface of the sand. The same was found true in the case of sand and dil. solns. It is concluded, on the basis of these results, that mol forces can act at a distance of 10,000 mol diams. S. L. Madorsky

**Surface potentials and force-area relations of unimolecular films** II.  $d$ -Pimanic acid and tetrahydro- $d$ -pimanic acid William D. Harkins, Herman E. Ries, Jr., and Everett F. Carman *J Am Chem Soc* 57, 2224-7(1935), cf. C. A. 28, 854—Surface potentials and force-area curves are given for  $d$ -pimanic and tetrahydro- $d$ -pimanic acids, corresponding unsatd and satd compds, resp. A technique for obtaining more accurate clean surface potentials is described. The max thickness of the mono layer is about 12 Å for both compds. The coeffs of compressibility are caled. The structures of these acids are compared with that of cholesterol, and their orientations in surface films are related to the different positions in which the polar groups are located. The expl data compare favorably with results predicted from models. The surface potentials are 200 and 100 mv for the satd and unsatd acids, resp. The unsatd acid films had an unprecedented decrease in surface potential on compression. These facts may be related to an increasing intramolecular dipole compensation on compression. G. M. P.

**Constitution of the capillary layer in solutions of**

malachite green M. T. Salazar *Compt rend* 201, 1120-3(1935)—The surface tension,  $\sigma$ , of aq solns of malachite green diminishes with increasing concn,  $c$ , thereof, becoming const for  $c$  greater than 8.5 g/100 cc. By means of Gibbs' equation,  $U = (c/RT)(d\sigma/dc)$  ( $U$  = no of moles per sq cm adsorbed in the surface), if the moles are spherical, it is shown that for this concn the surface layer is unimol. (D of malachite green is 139 Å). C. A. Silberrad

**Rapid method of determining stability of emulsions** V. Bockko *Masloboino Zhirovno Delo* 11, 489-90(1935)—A glass cylinder 25 mm in diam with a bulbous extension at the bottom is fitted with a side cock and rests in a wooden support. The cylinder is charged with 100 cc of the emulsion and placed in a drying oven to settle at a definite temp and for definite time. After 20 cc of the liquid has been removed and discarded, 10 cc is withdrawn into a tared Al beaker and weighed before and after evapn in a drying oven, giving the moisture content. If the emulsion contains  $B\%$  of dispersed phase, and  $a$  is the percentage of the dry substances of the phase and  $b$  the wt of the evapd moisture, then the content of non-fatty part in the emulsion is  $b/(100 + a)/a$ , and the stability of the emulsion is  $b/(100 + a)/B$ . Chas. Blanc

**Stability of emulsions** Joseph B. Parke, Quart J. Pharm Pharmacol 8, 484-9(1935)—To insure stability, an emulsion should contain (1) a suitable emulsifier and (2) a suitable phase ratio having regard to its type. If the emulsion is homogenized in an efficient machine, (2) is not so important as (1) because there is very little creaming. As a general rule the homogenization of an emulsion contg more than approx 74% by vol of disperse phase leads to a partial breakdown. If, on the other hand, the emulsion is prepd by agitation alone, then the percentage of disperse phase should be at least 74 by vol, otherwise it is very probable that creaming will occur. Very stable, noncreaming emulsions are possible if the continuous phase can be induced to set to a jelly-like mass by the addn of a substance such as gelatin. Since the latter is subject to decompn by bacteria, its value for the above purpose is greatly lowered. The ideal noncreaming emulsion would contain just sufficient disperse phase for the globules to be in closest packing after homogenization. Various examples of emulsions are illustrated. W. O. E.

**Properties of detergent solutions** I. Influence of hydrogen ion concentration on the surface tension of soap solutions J. Powney *Trans Faraday Soc* 31, 1510-21(1935)—The surface tensions of Na and K salts of oleic, lauric and myristic acids were detd at 20° in the presence of atm CO<sub>2</sub> by means of a torsion balance of the du Nouy type with Pt rings. Small changes in  $pH$  produced very large changes in surface tension, especially in the case of laurates. The effect of CO<sub>2</sub> on surface tension of soap solns can be correlated with absorption of CO<sub>2</sub> with consequent change in  $pH$ . C. E. P. Jeffreys

**The investigation of differing and of isomeric undissociated organic compounds by dialysis** H. Brintzinger and H. G. Beier *Z anorg allgem Chem* 224, 325-8(1935)—Dialysis experiments on a no of org compds show that the dialysis coeff of an undissoc org compd depends on the constitution of the compd as well as on its mol wt. The coeffs differ for aliphatic and aromatic compds and for  $o$ -,  $m$ - and  $p$  isomers. If the method is to be used for detg the mol wt of a compd in H<sub>2</sub>O soln a reference substance of the same structure and nearly the same wt should be chosen. L. E. Steiner

**The formation of zirconate hydrosols and their disintegration by certain neutral salts** Arthur W. Thomas and Harry S. Owens *J Am Chem Soc* 57, 2131-5(1935), cf. C. A. 29, 7754—Neutral salts may displace coordinatively bound OH ions decreasing the acidity of the dispersion medium. They may displace Cl<sup>-</sup> ion increasing the Cl<sup>-</sup> ion activity of the dispersion medium. The following anions also displace aquo groups and change the nuelles from cationic basic Zr chloride nuelles to anionic zirconate nuelles: citrate, glycolate, lactate, malate, mucate, pyruvate, tartrate, dihydrogen phosphate,

pyrophosphate. Sixteen other anions failed to reverse the charge on the micelle. The conditions necessary for reversing the charge are: (1) ability of the anion to displace the aquo group, (2) the formation of a sol anionic complex, and (3) the equal const. of the anionic complex must be near to, or less than, the soly. product of  $Zr(OH)_2$ . A large part of the added anion is available to maintain equilibrium. Boiling decreases the excess required. K citrate, tartrate and glycolate react with basic Zr chloride hydrosols and the resultants diffuse through a nitrocellulose bag. When  $ZrO_2$  is peptized by tartaric acid (or other acids with powerful coordinating anions) positively and negatively charged micelles appear simultaneously. The polyolation and oxolation theory of the structure of "metallic oxide" hydrosol micelles would predict and explain all of these behaviors.

**F E Brown**  
Variation of viscosity during the coagulation of colloidal aluminum hydroxide by potassium chloride solutions. S. Shridhar Sarvottam Joshi and K P N Pannikar. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 5, 41-5 (1935).—The progress of coagulation at lowest concn. of electrolyte is discontinuous at the beginning. An initial diminution of viscosity of about 1% takes place and the time  $T_{90}$  corresponding to the occurrence of viscosity  $\eta$  is unaltered even when the concn. of coagulator is increased from 0.025 N to 0.1 N. This indicates that  $\eta_{90}$  might not denote a definite stage of coagulation since the corresponding time is in general susceptible to changes in electrolyte concn. When 0.5 N KCl is used as coagulator a net rise in viscosity takes place.

**C E P Jeffreys**  
Coagulation of colloidal gold. K. Jablczynski and W. Koschany. *Rozprawy Chem* 15, 283-8 (1935).—The kinetics of colloidal coagulation were studied with a König-Martens spectrophotometer (*Rozprawy Chem.* 4, 185, 251 (1924)). Colloidal Au was prepd. by reduction in the presence of  $K_2CO_3$ . Each cc. of the prepn. contained 0.0001227 g. Au,  $1.6 \times 10^{13}$  particles,  $16.3 \mu$  in diam. The absorption cell, 30 mm internal width, cont. 20 cc. of prepn. was kept in a water thermostat at  $15^\circ$ . After measurement of the angle of the hydrosol ( $\alpha_{90}$ ), 5.5 - 10 cc. of KCl was added and the angle was remeasured ( $\alpha_0$ ) after 20 sec. All measurements were made in red light.  $\alpha_{90}$  remains const. but  $\alpha_0$  varies, being affected by improper mixing and by the rate of KCl addn. The value of  $k = (1/\alpha_0)(\lg \alpha_{90} - \lg \alpha_0)$  calcd. by the Jablczynski formula (loc. cit.) decreases with time, whereas it is const. for Fe(OH)<sub>3</sub> and the sulfides of As, Sb and Cd. The Au particles transmit at first only red light but as coagulation sets in violet and blue light are transmitted. The spectrophotometer method for colloidal gold is of qual. value only.

**J F Matejczyk**  
The variation of the flocculation threshold in starch sols. H. Kober and F. Dittmar. *Kolloid-Z.* 73, 219-26 (1935).—Sols of nine starches characterized by soly in cold water and viscosity of aq. sols at  $20^\circ$  and  $70^\circ$  were studied in the presence and absence of  $MgSO_4$  and a capillary-active substance (Prästabitol, Isepon T). Either type of addn. reagent alone has little effect on the turbidity of the sols, but, if added together, they cause an instantaneous increase in turbidity (sometimes even coagulation). The flocculation is then a function of  $MgSO_4$  as well as starch concn.

**Oscar T. Quimby**  
The coagulation of organosols of nitrocellulose by electrolytes. Serg. Papkov and M. Chvileva. *Kolloid-Z.* 73, 202-3 (1935); cf. *C. A.* 30, 147.—The stability of nitrocellulose dispersed in acetone is not affected by charge but depends largely on solvation. Thus aq. 0.3 M  $AlCl_3$  had exactly the same effect as pure water on the organosol.

**Oscar T. Quimby**  
The effect of alcohols on organosols (in particular polystyrene, rubber and acetylcellulose). H. Erbring and K. Sakurada. *Kolloid-Z.* 73, 191-201 (1935); cf. Yamaguchi, *C. A.* 29, 7755f.—E. and S. studied the pptg. effect of homologous aliphatic alcs. (up to  $C_9$ ) on sols of (1) polystyrene in EtOAc, butylacetone,  $CCl_4$ ,  $CHCl_3$ , Me Et ketone,  $C_4H_8$ , xylene, toluene and monostyrene, (2) acetylcellulose in acetone, (3) paraffin in

$C_4H_8$  and (4) rubber in  $C_4H_8$ ,  $CCl_4$  and  $CHCl_3$ . In most of the cases studied the relative pptg. powers  $\alpha$  ( $MeOH = 1$ ) of the alc. increased with its mol. dielec. polarization; however, with systems 3 and 4, the reverse effect was observed while, with polystyrene in Me Et ketone all alcs. had the same pptg. power. In systems 1 and 2  $\alpha$  was proportional to sol concn. For a given concn. of a solute in various dispersing media, a rough parallel was found between viscosity of the original sol and the pptg. concn. of alc. (in mol.). Addn. of alcs. to polystyrene in  $C_4H_8$  produced 2 liquid layers. Sols of polystyrene in binary mixts. of  $C_4H_8$  and EtOH did not behave in a simple additive way.

**Silicic acid gels. V. Determination of the hydrogen-ion concentration of the gel mixtures.** Charles B. Hurd and Robert L. Griffith. *J. Phys. Chem.* 39, 1155-9 (1935). cf. *C. A.* 28, 4061f.—Electrolyte-free gels were made by mixing Na silicate (1 brand, Phila. Quartz Co.) and  $H_2O$ , breaking up and washing the gel with distilled water agitated by a high speed elec. motor. The  $pH$ , measured by a Leeds and Northrup quinhydrone indicator No. 7654, varied from 4.02 to 9.92. The gel does not appear to affect the Pt electrode.

**States of aggregation of colloids and calculation of molecular weights.** F. F. Nord and F. L. M. Lange. *Naturwissenschaften* 23, 722 (1935).—Exposure of sols of lyophilic (albumin, gum, etc.) or lyophobic (myosin) colloids to temps. below the ice point in concns. of  $10^{-4}$  up to  $5\%$  results in aggregation or disaggregation depending on the concn. This was brought out by diffusion expts. (Schott cell) on the thawed colloids. It proves that different particle sizes exist in these sols.

**B. J. C. van der Hoeven**  
Cryolysis, diffusion and particle size. III. Studies on gum arabic and polyacrylic acid. G. Endö, F. E. M. Lange and F. F. Nord. *Ber.* 68B, 2004-11 (1935).—Lyophilic and lyophobic biocolloids undergo on freezing disaggregation or aggregation of particles depending upon their concn. The particle size is calcd. from diffusion measurements. The diffusion studies show further that the particle size of certain colloids hitherto considered const. is actually detd. by previous treatment.

**S. Morgulis**  
The relation between the concentration and viscosity of solutions of highly polymerized substances. Serg. Papkov. *Kunststoffe* 25, 233-5 (1935).—The viscosity-concn. relations of various colloidal sols can be graphically represented as a straight line with the aid of the equation,  $\log \eta = Kc^z$ , where  $z$  and  $c$  represent viscosity and concn., resp., and  $K$  and  $z$  are constants. Max. deviations amount to  $\pm 5-10\%$ . The applicability of the equation is demonstrated with data from the following sources: Fikentscher, *C. A.* 26, 5414 (acetylcellulose in 91%  $Me_2CO$ ) and Bincer, *C. A.* 26, 3424 (various kinds of gelatin in water). A similar equation developed by Abernethy (*C. A.* 20, 124) for rubber sols is shown to be a special case of P's equation.

**J. W. Perry**  
Fiber diagram of polyvinyl alcohol. Friedrich Halle and Wilhelm Hofmann. *Naturwissenschaften* 23, 770 (1935).—Threads of polyvinyl alc. are doubly refractive; on stretching beyond the elastic limit the transparent threads (all-o films) become milky and behave like an optical monoaxial substance of pos. double refraction. X-ray diagrams of the original thread have 4 or 5 interference rings, on stretching the crystallites are better orientated especially if the stretching is done (up to 7000%) in an air bath at  $90-100^\circ$  and a fiber diagram is obtained. At room temp. the max. elongation is 300-400%. The fiber diagram has an identity period of  $2.57 \pm 0.02$  Å, which is only slightly more than the length of a side of the C tetrahedron ( $2.52$  Å). The symmetry is taken to be monoclinic.

**B. J. C. van der Hoeven**  
Sperier lens and colloidal structure. Wm. Seifritz. *Ind. Eng. Chem.* 28, 136-40 (1936).—"Pictures of colloidal matter produced by the Sperier dark-field lens represent a true structure when suitable material is used and the optical system is properly handled. The major types of structure observed have been seen by several other

methods. However the Spierer picture is regarded, it can exist only in virtue of a definite and corresponding type of structure within the material observed. The stratified structure seen in cellulose represents a fine and naturally occurring grating. The Spierer lens reveals several distinct types of structure in colloidal matter. Proper handling of the lens produces sharp pictures, devoid of diffraction lines, of so small an object as a colon bacillus. Improper handling or unsuitable material produces artifacts. Recent x-ray work on cellulose gives evidence of a structural unit comparable in size to the supermicelles shown by the Spierer lens." **I. L. B.**

**The solution of nitrocellulose. I. III. T. Tomonari. J. Soc. Chem. Ind., Japan 38, Suppl. binding 517-28 (1935), cf. C. A. 29, 6422<sup>2</sup>.**—The behavior of various types of nitrated micelles was investigated. If an inert substance is present at the micelle exterior, it acts to prevent dispersion in the inner substance in the process of soln. This holds for nitrofibers  $(\text{Tr})_{\text{cell}}$  and  $(\text{Tr})_{\text{Dx}}$  in acetone, and  $(\text{D})_{\text{Tr}}$  in MeOH. If a sol substance is formed at the micelle exterior, the outer micelles go into soln only after the amt. of dissolved substance becomes sufficiently great, so that micelles may be dissolved that show a considerable degree of nitration at the micelle surface, while the micelle interior is unchanged cellulose. In the ideal case of equal micelle size and even progress of nitration for all micelles within the fiber, the soln. of the whole fiber mass takes place spontaneously at a certain degree of nitration. This case is approached in the soln. of nitrofibers  $(\text{Cell})_{\text{Tr}}$  in acetone and of  $(\text{Tr})_{\text{D}}$  in MeOH. In the soln. of  $(\text{Cell})_{\text{D}}$  in acetone the phenomenon is hidden by the great effect of the fiber heterogeneity of the prepns. In the soln. of nitrocelluloses prep. with  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{O}$  mixts. an unknown substance X plays an important role in preventing the soln. of dinitro- and trinitro-cellulose. Therefore the effect of the geometric arrangement of the nitroesters in the micelles upon the soln. processes of the prepns does not prevail. Four references.

**Ideal and real egg white solutions. G. Ettisch and R. Havemann. Z. Physik. Chem. A174, 199-221 (1935).**—If quartz suspensions are allowed to sediment in egg-white solns the max sediment vol occurs in isoelec solns. The simple sediment vol method for detg the isoelec pt. is refined by the introduction of the sediment vol difference ( $\Delta S V$ ). If the  $\Delta S V$  is measured in ovalbumin- and serum albumin-quartz suspension in relation to the protein concn, above a certain concn a relation between the  $\Delta S V$  max and the protein concn appears. From the course of the  $p_H$  of egg white soln a relation is obtained between concn and isoelec. pt. above this boundary concn. From this detn. of the isoelec. pt. as well as the change in cathaphoretic migration velocity of quartz suspension it is found that a reciprocal action takes place between the isoelec. protein mols as soon as a definite concn is passed. The nature of this reciprocal action may be explained through a reciprocal action potential based on the electrolytic theory of egg-white solns. Many other phenomena may be thus explained. Thus egg white solns can be divided into those in which the powers of reciprocal action are present (real solns) and those in which they are not (ideal solns). **R. H. Baechler.**

**The combination of bivalent manganese with certain proteins, amino acids and related compounds. Raynond K. Main and Carl L. A. Schmidt. J. Gen. Physiol. 19, 127-47 (1935).**—The criteria of complex formation among these substances were: (a) equal between the test substance and  $\text{Mn}^{++}$  dissolved in aq. buffered soln. and isonitrosacetophenone in  $\text{CHCl}_3$ ; (b) the electrophoretic migration of Mn in the presence of the test substance at varying  $p_H$  values; (c) anomalous titration. Classes of substances that possess the necessary groupings to form Mn complexes are: hydroxy monocarboxylic acids (lactic, gluconic), dicarboxylic acids (oxalic, malonic), hydroxy di- and tricarboxylic acids (citric, tartaric), dicarboxylic amino acids (aspartic, glutamic), certain inorg. acids ( $\text{H}_3\text{PO}_4$ ,  $\text{H}_2\text{SO}_4$ ), certain phosphoric acid-contg. compds. (nucleic, glycerophosphoric), certain

aromatic enol sulfonic acids (phenolsulfonic, catecholsulfonic), and certain proteins (casein, edestin, gelatin). A relation exists between the amt. of Mn bound by several proteins and the no. of free  $\text{COOH}$  and phosphonic acid groups. The enol groups furnish, in the compds that contain them, the addn. attraction necessary for the formation of complexes with  $\text{Mn}^{++}$ . Formulas, based upon the residual charges of certain atoms, are given in explanation of the union of Mn with the compounds studied.

**C. H. Richardson.**  
**Addition to the article. Quinone gelatin gels. Janos Buchholz. Kolloid Z. 73, 237 (1935), cf. C. A. 29, 4238<sup>2</sup>.**—Toluquinone but not naphthoquinone nor anthraquinone forms a gel of the type previously described.

**Oscar T. Quimby.**  
**Supplementary remarks on our article. The effect of various metal compounds on gelatin. E. Elöd and Th. Schachowsky. Kolloid Z. 73, 236 (1935); cf. C. A. 29, 3892<sup>1</sup>.**—Polemic with Kuntzel (cf. C. A. 30, 157<sup>2</sup>).

**Oscar T. Quimby.**  
**The diffusion of colored substances in gelatin and the morphology of their reactions with electrolytes. Suzanne Veil. Bull. soc. chim. [5], 2, 1830-2 (1935), cf. C. A. 29, 2424<sup>1</sup>.**

**F. L. Browne.**  
**Diffusion in gelatin and rhythmic precipitation of magnesium hydroxide. G. Ammon and R. Ammon. Kolloid Z. 73, 204-19 (1935).**—Quant studies were made at room temp. on the rhythmic pptn. of  $\text{Mg}(\text{OH})_2$  resulting from diffusion of  $\text{NH}_4\text{OH}$  into gelatin gels contg. bromothymol blue indicator and  $\text{MgCl}_2$ . Various concns of gelatin ( $C = 0$  to  $14$  g./100 cc.  $\text{H}_2\text{O}$ ) and  $\text{MgCl}_2$  ( $c = 0$  to  $16$  g./100 cc.  $\text{H}_2\text{O}$ ) were used with a fixed concn of  $\text{NH}_4\text{OH}$  (25% aq.  $\text{NH}_3$ ). The quantities measured were (1) the distance  $Y$  the  $\text{NH}_4\text{OH}$  boundary (indicator) had traveled, (2) the distance  $y$  of each  $\text{Mg}(\text{OH})_2$  band measured to center from zero position and (3) the time  $t$  at which each band formed. The law of Morse and Pierce (*Z. physik. Chem.* 45, 589 (1903)),  $Y/\sqrt{t} = K$  a const., was verified for  $\text{NH}_4\text{OH}$  diffusion in absence of  $\text{MgCl}_2$ , in the presence of  $\text{MgCl}_2$   $K$  decreased for the first 6 hrs., but became practically const. thereafter. Likewise  $y/\sqrt{t} = k$  a const. was approx. satisfied after 3-4 hrs. Both  $K$  and  $k$  were larger if  $\text{NH}_4\text{OH}$  diffused upward instead of downward. The Schleussner relation,  $y_0/y_{n-1} = q$  a const. (C. A. 18, 2991), applied from the beginning. As required by these two laws, it was found that  $t_n/t_{n-1} = q^2$  where  $t_n$  and  $t_{n-1}$  are the times required for the  $n$ th and the preceding band to form. From the above laws there follows the relation of Mikhailov, Nikiforov and Shemyakin (C. A. 28, 2594<sup>1</sup>), namely,  $(\Delta y/\Delta t)\Delta y = k(g-1)/(g+1) = \text{a const.}$ , which was also verified within the region of  $k$  constancy. The implication of Christiansen and Wulff (C. A. 28, 6047<sup>1</sup>) that  $(\Delta y/\Delta t)\Delta y$  is a const. dependent on  $\text{MgCl}_2$  concn was not supported.

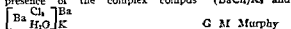
**Oscar T. Quimby.**  
**Emission-wave theory of periodic reactions. VII. F. M. Shemyakin. J. Gen. Chem. (U. S. S. R.) 5, 943-9 (1935), cf. C. A. 29, 3901<sup>2</sup>.**—A discussion of the application of wave equations of de Broglie and Schrödinger to the mechanism of formation of Liesegang rings, as worked out by Shemyakin and co-workers (cf. Christiansen and Wulff, C. A. 28, 6047<sup>1</sup>).

**S. L. Madorsky.**  
**Theory of physicochemical periodic processes. A. A. Vitt and F. M. Shemyakin. J. Gen. Chem. (U. S. S. R.) 5, 814-17 (1935), cf. C. A. 29, 6487<sup>2</sup>.**—It is shown mathematically that periodic changes of concn (Liesegang rings, periodic salting out, chemotaxis) take place, with time, in systems  $A + B \rightleftharpoons AB$ ,  $A + AB \rightleftharpoons A_2B$ ,  $A_2B + B \rightleftharpoons 2AB$ , where  $A$  is the external component,  $i$  e., the diffusate,  $B$  is the internal component,  $i$  e., the diffused substance uniformly distributed through the medium  $A$ ,  $AB$  the product of reaction between  $A$  and  $B$ , and  $A_2B$  a reaction component of a complex or adsorptive nature.

**S. L. Madorsky.**  
**A study of periodic reactions by the method of physicochemical analysis. VI. F. M. Shemyakin, E. A. Fokina and P. F. Mikhailov. J. Gen. Chem. (U. S. S. R.) 5,**



Refractometric investigation of aqueous solutions of salt mixtures. The system  $\text{BaCl}_2 + \text{KCl}$ . G. Spacu and B. Popper. *Z. physik. Chem.* B30, 113-16 (1935); cf. C. A. 28, 5314. Measurements were made of  $n$  and  $d$  for aq. solns. of a  $\text{BaCl}_2$  and  $\text{KCl}$  mixt. at  $21 \pm 0.03^\circ$ . The change of sp. refraction with concn. indicates the presence of the complex compds.  $(\text{BaCl})_2\text{K}$  and



G. M. Murphy

Viscosity of solutions of salts in methanol. Grinnell Jones and Holmes. *J. Fornwall. J. Am. Chem. Soc.* 57, 2041-5 (1935), cf. C. A. 27, 1804; 28, 7108. The abs.  $d$  (expressed in terms of the Root equation for aq. soln.) and relative viscosities of varying concns. of the K halides and  $\text{NH}_4\text{Cl}$  were detd. in  $\text{MeOH}$ . The Jones and Dole equation held only to 0.01  $M$ . The Onsager and Fuoss equation was better for intermediate concns. All the solns. showed the "Grüneisen effect" and all have a viscosity greater than that of pure  $\text{MeOH}$ . H. E. P.

Viscosity of aqueous solutions of electrolytes. II. A comparison of the specific ionic viscosity with other ionic properties of homologous elements. Hans Tollert. *Z. physik. Chem.* A174, 239-46 (1935), cf. C. A. 29, 3217. The sp. ionic viscosity of the alkali metals and of the halogens was compared graphically and mathematically with newly detd. ionic properties. The trend of ionic viscosity in 0.1  $N$  concn. agrees with the trend of the lattice energy of the alkali chlorides, the ionic radiations in solid lattice, the hydration and heat of hydration with exception of the Na value, and in the alkali series the energy of ionization. From this it may be deduced that the hydrated ions and free water are in equl. which is a function of the sp. no.

R. H. Baechler

Some physicochemical studies of organometallic and furan compounds. Willard E. Catlin. *Iowa State Coll. J. Sci.* 10, 63-7 (1935).—A. Relative reactivities of halides and super-aromatic properties of furan.—If the activity of  $\text{BuCl}$  is one, the activities are: 2-furylmethyl chloride 3184, 5-nitro-2-furylmethyl chloride 12,703, 2-furylmethyl chloride 93,230, 2-tetrahydrofurylmethyl chloride 0.025,  $\gamma$ -(2-furyl)propyl chloride 2.51 and  $\gamma$ -(2-tetrahydrofuryl)propyl chloride 1.33. Halogens attached to the furan ring are inert. B. Parachors of some furans.—The values of the parachors were: furan 160.4, 2-methylfuran 199.8, 2,5-dimethylfuran 240.6, 2-nitro-furan 220.8, 3-bromofuran 212.6, 2-furfuryl alc. ( $\text{H}_2\text{O}$ -sol) 218.9, 2-furfuryl alc. ( $\text{H}_2\text{O}$ -insol) 216.2, 2-furfuryl methyl ether 260.8, 2-tetrahydrofuryl methyl ether 321.3, 2-furfural, 212.5, ethyl 2-furoate 309.6 and 2-furfuryl 2-furoate 398.9. This supports the dolefin formula or a related structure. Probably, there is a dynamic equl. of several forms. C. Ionization constants of some acids of the furan series and super-aromatic properties of furan.—Ionization constants ( $\times 10^4$ ) were calcd. from  $pK$  measurements on half-neutralized solns. of the acids as follows: 3-chloro-2-furoic 204.1, 5-chloro-2-furoic 147.4, 5-bromo-2-furoic 144.3, 5-iodo-2-furoic 116.0, 3,4-dichloro-2-furoic 400.3, 3,5-dichloro-2-furoic 377.4, 4,5-dichloro-2-furoic 248.6, 3,5-dibromo-2-furoic 326.8, 4-nitro-2-furoic 870, 5-methyl-2-furoic 38.12, 2-methyl-3-furoic 2.94, 2,4-dimethyl-3-furoic 2.79, 2,5-dimethyl-3-furoic 2.290, muco-bromic 5.26, furylacrylic 3.83, 2-furoic 75.2, 3-furoic 11.3, and thiophene-2-carboxylic 34.26. D. Relative reactivities of some organometallic compounds.—Reactivities of organometallic compds. of elements of groups 2, 3, 4 and 5 of the periodic table can be measured by adding to an acid of suitable strength and soly. an excess of the organometallic compd. and following the reaction by extg. the unchanged acid by  $\text{H}_2\text{O}$ . The relative reactivities are:  $\text{PbEt}_2$ , 6,  $\text{PbPh}_2$ , 56,  $\text{HgPh}_2$ , 57,  $\text{BiPh}_3$ , 40,  $\text{PbPh}_2\text{Et}$ , 2000, when  $\text{CCl}_3\text{CO}_2\text{H}$  was used at  $25^\circ$ ; with  $\text{HCl}$  at  $25^\circ$ :  $\text{SnEt}_2$ , 0.9, at  $10^\circ$   $\text{PbEt}_2$ , 410,  $\text{SnPh}_3$ , 75,  $\text{HgEt}_2$ , 30. As catalysts diatomaceous earth and oxygen (on oxidation products) greatly increased the rates of reaction.

F. E. Brown

The dissociation constants and rotations of some  $\alpha$ -substituted ethylamines. J. Marvin Burch. *Iowa State Coll. J. Sci.* 10, 55-7 (1935).—In this study secondary butylamine,  $\alpha$ -benzylethylamine,  $\alpha$ - $p$ -tolylethylamine,  $\alpha$ -phenylethylamine, and  $\alpha$ - $p$ -diphenylethylamine were prepd. and resolved according to published directions, and  $\alpha$ - $\alpha$ -chlorobenzylethylamine was prepd. and resolved for the first time. Rotations of the pure amines, of the  $\text{MeOH}$ ,  $\text{EtOH}$  and  $\text{C}_2\text{H}_5$  solns. of the amines, and of the amine hydrochlorides in  $\text{MeOH}$  were measured with a Na arc as the light source. The mol. rotations of the amine hydrochlorides in  $\text{MeOH}$  were lower than that of the amines. The mol. rotation of  $\alpha$ -phenylethylamine in  $\text{MeOH}$  was observed while it was being neutralized in steps by gaseous  $\text{HCl}$ . When mol. rotation was plotted against  $pH$ , the curve resembled an electrometric titration curve except that the greatest slope occurred at half neutralization of the amine. The disocn. const. at half neutralization of the  $\alpha$ -substituted ethylamines (A), mentioned above, were detd. (cf. C. A. 28, 4378) in  $\text{MeOH}$ . When the radical substituted in A is varied the mol. rotation varies. The change from the phenyl group to cyclohexyl group produced a change of mol. rotation from +49.4 to -4.06 at  $15^\circ$ . The other substituents produced changes of less magnitude. The magnitude of the change was a function of elec. properties of the substituent. When disocn. const. of A in  $\text{MeOH}$ , of the primary amines in  $\text{H}_2\text{O}$  and the dipole moments of the chlorides in  $\text{CaH}_2$  were compared with the mol. rotations of A, with but few exceptions, a decrease in the disocn. const. of the amines, or an increase in disocn. const. of the acid, or a decrease in the dipole moment of the chloride corresponded to an increase in the mol. rotation of the active As.

F. E. Brown

The relative strengths of acids in butyl alcohol. Leland A. Wooten and Louis P. Hammett. *J. Am. Chem. Soc.* 57, 2289-96 (1935).—A method of measuring relative strengths of acids in  $\text{BuOH}$  is developed by which 33 representative carboxylic and phenolic acids are measured. The effect of a substituent upon the variation in relative strength with changing medium is discussed.

H. W. Leahy

Statistical treatment of strong electrolytes. S. Levine. *Proc. Roy. Soc. (London)* A152, 829-59 (1935).—The method of Kramers (C. A. 21, 1746) was used in a statistical mech. treatment of strong electrolytes. The deviations from the inverse square law due to satn. and to polarization effects on the  $\text{H}_2\text{O}$  dipoles, and to polarization, van der Waals, and exchange forces between typical  $\text{H}_2\text{O}$  ions, and  $\gamma$  is accounted for by a correction term  $\gamma_{\text{cor}}$  in the expression for energy of interaction. The ad.  $\epsilon$  of this term is equl. to a modification of the dielectric const.  $D$  to  $D - \delta$ , where  $\delta$  depends upon  $E_i$  and is a function of concn. and temp. When the proper form for  $\delta$  is known the method proposed should describe the properties of solns. of strong electrolytes. C. L. P. Jeffreys

The diffusion of strong electrolytes. Seiji Kaneko. *J. Chem. Soc. Japan* 56, 1322-3 (1935), cf. *J. Ind. 1265* (1935).

T. Katsurai

Ionic migration as a molecular kinetic problem. A. Magnus. *Z. physik. Chem.* A174, 262-8 (1935).—The const. migration velocity of ions in an elec. field is explained by the assumption that the ions are accelerated in the direction of the elec. field between 2 collisions with mols. of the solvent and are again retarded by the collision. The mean free path can be calcd. from observations of ionic mobility which, especially in the case of small and bivalent ions, is much smaller than the mol. kinetically calcd. values. These deviations can be explained on the basis of the dipolar structure of the solvent. R. H. B.

Concentrated solutions. I. Electric conductivity, viscosity and density of molten  $\text{NH}_4\text{Ag}(\text{NO}_3)$ , and its concentrated solutions. M. S. Skanavi-Gingor'eva and E. B. Shternin. *J. Gen. Chem. (U.S.S.R.)* 5, 799-806 (1935).—The equl. concn. of molten  $\text{NH}_4\text{Ag}(\text{NO}_3)$  at  $110^\circ$ ,  $112^\circ$ ,  $113^\circ$ ,  $115^\circ$ ,  $118^\circ$ ,  $120^\circ$  and  $123^\circ$  was found to be 0.134, 0.142, 0.150, 0.156, 0.168, 0.174 and 0.187 reciprocal ohms, resp. Its viscosity,  $1/\eta$ , in  $\text{cm}^2$ , at  $110^\circ$ ,  $120^\circ$  and  $124^\circ$ , was 12.03, 10.62 and 9.95, resp., where  $t$  is time of flow of fused salt and  $t_{\text{H}_2\text{O}}$  is time of flow of  $\text{H}_2\text{O}$  at  $100^\circ$ , through the

Ostwald viscometer. Elec. cond., viscosity and  $d$  of 1  
sols of this salt were also studied in concns. from 41.80  
to 100% at temps. of 25–112°. Conc'd. sols. of  $\text{NH}_4\text{Ag-}$   
 $(\text{NO}_3)_2$  represent very complicated systems. No explana-  
tion is given of the nature of curves representing varia-  
tions of elec. cond., viscosity and  $d$  with concn or temp

S. L. Madorsky

Conductance of some acids and other solutes in pyri-  
dine. Mansel M. Davies *Trans. Faraday Soc.* 31,  
1561–7 (1935).—From cond. measurements in pyridine  
the following dissoxn. consts were det'd for  $\text{HClO}_4$ ,  
 $\text{HNO}_3$  and  $\text{HI}$ ,  $7.55 \times 10^{-4}$ ,  $4.96 \times 10^{-4}$  and  $5.9 \times 10^{-4}$ ,  
resp. Picryl chloride gave very divergent values for cond  
I or  $\text{BzCl}$  a value of  $K = 1.30 = 0.02 \times 10^{-4}$  was obtained  
 $m\text{-C}_6\text{H}_4(\text{NO}_2)_2$  had only a light cond.  $\text{C}_6\text{H}_5(\text{NO}_2)_2$   
reacted with pyridine, developed a red color and gave a  
progressively increasing cond. A mean value of  $1.6 \times$   
 $10^{-19}$  for  $\text{BzOH}$  was obtained from very low cond. values

C. E. P. Jeffreys

Conductivity of strong electrolyte. Supplement Seiji  
Kaneko *J. Chem. Soc. Japan* 56, 1320–2 (1935),  
C. A. 29, 6819†—Math. T. Katsuragi

Conductance measurements of dilute solutions. M.  
Hilasko and A. Salūtina *Roczniki Chem.* 15, 273–82  
(1935).—The method used and previously described by  
the authors (C. A. 29, 6127†) permits measurement of the  
conductance of very dil. sols ( $10^{-4} N$ ). The limiting  
conductance of  $\text{Ca}(\text{OH})_2$ ,  $\text{Sr}(\text{OH})_2$ ,  $\text{Ba}(\text{OH})_2$  and  $\text{TiOH}$   
is det'd directly. The mol. conductance of  $\text{Mg}(\text{OH})_2$   
and  $\text{AgOH}$  agrees closely with the limiting conductance.  
The latter is attained in sols  $\sim 2 \times 10^{-4} N$   $\text{Ca}(\text{OH})_2$ ,  
 $5 \times 10^{-4} N$   $\text{Sr}(\text{OH})_2$ ,  $1 \times 10^{-4} N$   $\text{Ba}(\text{OH})_2$ ,  $2 \times 10^{-4} N$   
 $\text{TiOH}$ ,  $3 \times 10^{-4} N$   $\text{AgOH}$ , within an explt. error of 0.2%.  
These agree within 0.2% of the calc'd. values by the Kohl-  
rausch formula. If 200.2 is the velocity of  $\text{OH}^-$   $\text{Mg}^{++}$ ,  
 $\text{Sr}^{++}$ ,  $\text{Ca}^{++}$ ,  $\text{Ba}^{++}$ ,  $\text{Ag}^+$  and  $\text{Ti}^+$  have values of 50, 60, 1,  
60.3, 65.05, 63.3 and 78.7. The conductance coeffs.  
( $\mu = \lambda \cdot \kappa \cdot 100$ ) differ by 0.5–1% from those calc'd. by the  
M. Onsager formula, and the order of magnitude is the  
following:  $\text{NH}_4\text{OH} < \text{HgOH} < \text{Mg}(\text{OH})_2 < \text{Ca}(\text{OH})_2$ ,  
 $< \text{Sr}(\text{OH})_2 < \text{Ba}(\text{OH})_2 < \text{TiOH} < \text{LiOH} < \text{NaOH} < \text{KOH}$   
 $< \text{RbOH} < \text{CsOH}$ .

J. F. Matejczyk

Effect of temperature on borax solutions in the presence  
of polyhydric substances and organic acids. S. M.  
Mehta. *Proc. Acad. Sci. United Provinces Agra Oudh*,  
*India* 5, 46–9 (1935).—The effect of many polyhydric  
alcs. and org. acids on the change in  $p_n$  of borax sols  
with temp. was investigated. The behavior of the mixt  
and simple borax sols was due simply to a change in  
the degree of hydrolysis of Na borate with temp

C. E. P. Jeffreys

Methods for determining  $p_n$ . H. Brückner *Z. anal.*  
*Chem.* 103, 285–92 (1935).—A review. W. T. H.

The contact layer between solutions. V. Čupr *Chem.*  
*Listy* 29, 253–7 (in French 257); *Collection Czechoslov.*  
*Chem. Communications* 7, 445–50 (1935).—A graph is  
presented in which the sum of 3 diffusion potentials  
 $\pi + \pi_1 + \pi_2 = R_1/R_2 + R_2/R_3 + R_3/R_1$  is plotted against  
time. Measurements were made in capillary tubes of an  
app. designed for the purpose, in which  $R$  was 1.0 N  
 $\text{HCl}$ ,  $R_2$  was 0.01 N  $\text{KCl}$ , and  $R_3$  was 0.5 N  $\text{HCl}$  contg.  
a 0.005 N concn. of  $\text{KCl}$ . During the first 15–30 min. of  
contact between the sols., irregularities in the sum of  
potentials continued to exist because of changes in compn.  
in the layers of contact; after these irregularities sub-  
sided, the sum of the 3 potentials  $\pi + \pi_1 + \pi_2$  became zero  
for as long as 40 hrs.; between the 40th and 50th hr.,  
the sum of the potentials rose to  $-1.50 = 0.4$  mv. Since  
the Planck equation gives a sum of potentials equal to  
zero only in special cases the Henderson equation, which  
depends upon a mixed layer at the zone of contact, shows  
why  $\pi + \pi_1 + \pi_2 = 0$  only when the  $R_2$  (inserted between  
 $R_1$  and  $R_3$ ) is a mixt. of  $R_1$  and  $R_3$ . The results confirm  
the hypothesis stated by Planck that after the sols. are  
in contact a mixed layer exists long enough to compute  
the potentials by the Henderson equation; later, the mixed  
layer becomes a diffusion layer. This transformation of  
layers occurred after the 4th hr. of contact. The diffusion

potential computed from Planck's equation was  $-1.46$ ;  
the explt. values after the 40th hr. were  $-1.50$  mv., but  
since the electrolytes were diffusing at this time into wide  
portions of capillary tubes a closer approach to the com-  
puted value was not possible. The results also agree with  
those of Shu-Tsu-Chang

Frank Mareš

The electric activation of passive iron wires in nitric  
acid. Ralph S. Lillie *J. Gen. Physiol.* 19, 109–26  
(1935).—The characteristic intensity-duration curves for  
passive Fe wire in  $\text{HNO}_3$  soln. resemble those for irritable  
living tissues with moderate speeds of response to stimula-  
tion. The intensity of the current required for activation,  
and its minimal effective duration for a given intensity,  
increase rapidly with increase of  $\text{HNO}_3$  concn. To brief  
currents, the responsiveness of the wire is low immediately  
after activation, responsiveness then returns progressively  
to the original level, following a time curve which resembles  
that for living tissues during the relative refractory  
period. The responsiveness to brief currents is decreased  
reversibly by surface-active compds., the degree depend-  
ing upon the concn. of the compds. Under somewhat  
special conditions, Fe wire, like irritable tissue, is activated  
by the interruption of a flowing concn. current.

C. H. Richardson

The proof of the law of mass action in fused solutions  
by potential measurements. L. Holub, F. Neubert and  
F. Sauerwald *Z. physik. Chem.* A174, 161–98 (1935).—  
Through the potentiometric detn. of the equil. const. and  
activity ratios in the reaction between metals and their  
molten salts assumed mol. wts. can be verified as well as  
const. and ratios det'd analytically. The Lorenz-  
van Laar law ( $MAL$ ) does not hold. The smallest devia-  
tion of the mean value of the const. compared to the  
electrolytically detd. const. is 25%. When the const. of the  
ideal  $MAL$  are correct it is not necessary for the partici-  
pants in the reaction to be in the state of ideal soln.  
If the  $MAL$  const. are calc'd. from the equil. with pre-  
ponderantly noble metal a better agreement with the elec-  
trochemically detd. const. is obtained. The theoretically  
indicated approach to agreement between ideal and  
electrochemically detd. const. obtained by detn. of the  
soln. is present in 2 cases ( $\text{Pb-Sn-Cl}$  with 0.45% Pb and  
Pb-Sn-Cl). Comparison of const. detd. electrochemi-  
cally from Daniell chains with those obtained by combining  
individual activities show no agreement. Evidently  
individual activities cannot be detd. electrochemically  
because of irreversibility of the electrodes. Comparison  
of values obtained by varied assumptions of mol. size  
shows that those giving the most nearly const. mass-action  
quotients do not always show the most probable reaction.  
Simple and assoc. mol. as well as ions are present to-  
gether

R. H. Baechler

Application of the law of mass action in the synthesis  
of  $\beta$ -glucosides. I. Vintilescu, C. N. Ionescu and A.  
Cizy *Bul. soc. chim. Romania* 17, 137–50 (1935);  
C. A. 29, 4390†.—Correct application of the mass law  
is accomplished only by consideration (besides the usual  
factors) of (1) the displacement of the equil.  $\beta$ -glucose  $\rightleftharpoons$   
 $\alpha$ -glucose in the presence of alc., and (2) the specificity  
of  $\beta$ -glucosidase.

W. J. Peterson

Law of equilibrium displacement. Paul Renaud and  
Ernest Baumgardt. *Compt. rend.* 201, 1129–31 (1935).—  
Arguments are adduced showing the validity of Le  
Chatelier's principle, and the invalidity of Braun's criti-  
cisms thereof (*Z. physik. Chem.* 1, 259 (1887)). Cf.  
Planck, C. A. 28, 3620†

C. A. Silberrad

Formation of amorphous sulfur. Wawrzyniec Jacke.  
*Roczniki Chem.* 15, 258–72 (1935).—A theoretical con-  
sideration of the velocity of formation of amorphous S  
from  $\text{Na}_2\text{S}_2\text{O}_3$  and  $\text{HCl}$ .

J. F. Matejczyk

Steric factor of bimolecular association reactions. C. E.  
H. Bawn. *Trans. Faraday Soc.* 31, 1536–47 (1935).—  
The steric factor is related to the probability of the transi-  
tion state, and it is concluded that the small steric factor  
of bimol. assocn. reactions between complex mol. com-  
pared with those of atoms or simple mol. arises from the  
decreased probability of the transition state in these

cases. Application of these views to rupture of long-chain mols is discussed C. E. P. Jeffreys

The reactivity of halogen compounds III The velocities of reaction, energies of activation, and probability factors for the reaction between 2,4-dinitrobenzene and some aromatic primary amines Attar Singh and D. H. Peacock *J. Chem. Soc.* 1935, 1410-11 — Exptl data for the reaction between 2,4 (NO<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>Br and PhNH<sub>2</sub> and *m*- and *p*-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> in EtOH soln. were obtained which indicate that the difference in reaction velocity is partially, or wholly, due to a difference in the probability factor IV The effect of the addition of inactive substances on the rate of reaction, energy of activation and probability factor *Ibid.* 1411-12. — Addn of C<sub>6</sub>H<sub>6</sub> had no effect on the velocity const. while C<sub>6</sub>H<sub>5</sub>N (C<sub>6</sub>H<sub>5</sub>)<sub>3</sub> had the greatest effect All effective compds. contained the C<sub>6</sub>H<sub>5</sub> nucleus Their dipole moments did not seem related to effect H. W. Leahy

Mechanism of, and constitutional factors controlling, the hydrolysis of carboxylic esters VII. Cyclopentane-dicarboxylic esters Attempted check on the calculation of molecular dimensions Christopher K. Ingold and H. G. G. Mohrhehn *J. Chem. Soc.* 1935, 1482-6, cf. C. A. 29, 7763 — The hydrolysis of the Me esters of *cis*- and *trans*-cyclopentane-1,2 and 1,3-dicarboxylic acids by NaOH was studied The exptl data confirm the validity of the correction for local elec. effects, but deviations remain H. W. Leahy

A reaction-mechanism study—action of fused sodium amide on mono-, di- and trimethylamines II Shipley I. Fry and Wilbur V. Culp *Rec. trav. chim.* 54, 838-46 (1935) — If NaNH<sub>2</sub> and NaOH have a chem. relation similar to the relation between CH<sub>3</sub>NH<sub>2</sub> and CH<sub>3</sub>OH, the type equation for the reaction between NaNH<sub>2</sub> and amines should be analogous to the type of reaction between NaOH and alcs This equation for the former case is thus R(H)<sub>3</sub> + nNaNH<sub>2</sub> → R(NHNa)<sub>n</sub> + nH<sub>2</sub> For the first 3 amines, then, the reaction equation is CH<sub>3</sub>NH<sub>2</sub> + NaNH<sub>2</sub> → NaCN + NH<sub>3</sub> + 2H<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>NH + 2NaNH<sub>2</sub> → 2NaCN + NH<sub>3</sub> + 4H<sub>2</sub>, and (CH<sub>3</sub>)<sub>3</sub>N + 3NaNH<sub>2</sub> → 3NaCN + NH<sub>3</sub> + 6H<sub>2</sub> CH<sub>3</sub>NH<sub>2</sub> and (CH<sub>3</sub>)<sub>2</sub>NH reacted in accordance with the equations to the extent of 95% of the theoretical (CH<sub>3</sub>)<sub>3</sub>N did not react This can be explained by assuming the formation of the NH<sub>2</sub> compd. as the first step The disocn. const., which measures the basic property, of (CH<sub>3</sub>)<sub>3</sub>N is too small to allow the formation of the NH<sub>2</sub> compd. Arthur A. Vernon

Mechanism of hydrolysis in solutions of salts of heavy metals Marguerite Quintin *Compt. rend.* 201, 1176-8 (1935) — The law of the proportionality of the activity of the metallic cation to that of H<sup>+</sup> established empirically in the case of MSO<sub>4</sub> and M(NO<sub>3</sub>)<sub>3</sub>, M = Cu, Cd or Zn, (cf. C. A. 22, 2097) applies to more strongly hydrolyzed solns, e. g., of CdCl<sub>2</sub>, and is attributed to formation of a complex, e. g., CdCl<sub>2</sub>(OH)<sub>2</sub> or M(SO<sub>4</sub>)(OH)(H<sub>2</sub>O)<sub>2</sub> C. A. Sühner

The diffusion of gases through metals II Diffusion of hydrogen through aluminum C. J. Smithell and C. F. Ransley *Proc. Roy. Soc. (London)* A152, 706-13 (1935); cf. C. A. 29, 4951 — H diffuses through Al at measurable rates at temps. above 400° The rate depends on the state of the surface The highest rate, obtained with surfaces freshly scraped in H<sub>2</sub>, falls off rapidly, and after some hrs. reaches a steady value (about 1/10 the original) which corresponds to the low rate of an anodically oxidized surface The effects of temp. and pressure are represented by Richardson's equation L. E. S.

Kinetics of the displacement of silver from silver nitrate solutions by metallic lead S. Krzyżanek *Z. anorg. allgem. Chem.* 225, 151-62 (1935) — Vertical surfaces of Pb were exposed to uncurrent solns. of AgNO<sub>3</sub>, and the progress of the reaction was followed by detg. at 10-min. intervals the Ag<sup>+</sup> left in the soln. The pptd. Ag adheres well, and comparable diffusion effects are maintained. The av. speed of reaction increases with the concn. of the AgNO<sub>3</sub>, reaching a max. at approx. 1 N, but decreases sharply beyond that point K for the reaction does not follow Boguski's log law (*Ber.* 9, 1616),

but increases as the reaction proceeds. The amt. of reaction per unit area of Pb varies directly as the concn. of the Ag<sup>+</sup>, indicating a reaction of the 0 order Temp. coeffs. were found to be independent of the initial concn. J. H. Reedy

Temperature coefficients in the acid hydration of sodium pyrophosphate Samuel J. Kiehl and Edward Clausen, Jr. *J. Am. Chem. Soc.* 57, 2284-9 (1935). — At the temps. 30°, 45°, 60°, 75° and 90°, the velocities of hydration for 0.125 M solns. of Na<sub>2</sub>P<sub>2</sub>O<sub>7</sub> 10H<sub>2</sub>O in the presence of 0.500 M, 0.425 M and 0.350 M HCl were detd. The pyrophosphate was detd. by pptn. of the Zn salt in carefully regulated acid concns. The data are fairly well represented by the equation,  $kt = -102.860 \log X - 68.143 + 109.590X - 42.598X^2$ , where X is the fraction of the pyrophosphate unchanged Temp. coeffs. calcd. for 15° intervals range from 3.4 for interval 90-75° in 0.500 M HCl to 7.0 for the interval 45-30° in 0.350 M HCl. When the velocity consts. are plotted against 1/T they are connected by a straight line. If the values of Q at 15° intervals are calcd. they are within the usual limits of constancy. P. E. Brown

Nonadiabatic reactions The decomposition of N<sub>2</sub>O Allen E. Stearn and Henry Eyring. *J. Chem. Physics* 3, 778-85 (1935) — Exptl. and theoretical abs. rates for the nonadiabatic decompn. N<sub>2</sub>O → N<sub>2</sub> + O agree well. The amt. of chem. inertia present in other reactions involving the singlet-triplet transition of O is considered. A convenient method is given of constructing potential functions for polyat. mols. which fit the spectroscopic data, and which reduce in the proper way for the various disocn. processes; it is applied to the N<sub>2</sub>O mol. G. M. P.

Effect of hydrogen ion concentration on oxidation-reduction reactions A. K. Babko. *Z. anal. Chem.* 103, 98-103 (1935). W. T. H.

Quantum mechanics and kinetics of organic chemical reactions C. N. Hinshelwood. *Bull. soc. chim.* [5], 2, 1786-90 (1935) — A lecture G. M. P.

The primary process of photochemical and thermal decomposition of azomethane I. Patat *Naturwissenschaften* 23, 891 (1935) — Results of Forbes, et al. (C. A. 29, 7810) were confirmed, decompn. of azomethane between 20° and 226° is not a chain reaction. By use of 365 mμ light, 10-600 mm. Hg pressure, a zero temp. coeff. of the photoreaction was found The primary reaction takes place as Me<sub>2</sub>N<sub>2</sub> → 2Me + N<sub>2</sub>; this was proved by the decompn. of Me<sub>2</sub>O (150-600 mm.) in the presence of irradiated MeN<sub>2</sub> (10-30 mm.) at 230-265°. Free Me radicals appear but do not form chains; hence the activation energy for Me + Me<sub>2</sub>N<sub>2</sub> is more than 20 kcal. Likewise a stable MeN<sub>2</sub> radical does not exist, therefore the thermal decompn. follows the same pattern (Allen and Suckman, C. A. 29, 7245) B. J. C. van der Hoeven

The velocity of decomposition of diazo compounds in water. XVIII. E. Yamamoto *J. Soc. Chem. Ind. Japan* 38, Suppl. binding 528-32 (1935); cf. C. A. 29, 6492 — Exptl. results are reported on the decompn. velocities, at 0-100°, for *p,p'*-tetrazodiphenyl chloride, *p,p'*-tetrazo-*o,o'*-diethyl chloride, *o,o'*-dimethoxy-*p,p'*-tetrazodiphenyl chloride and *o,o'*-disulfo-*p,p'*-tetrazodiphenyl chloride. The velocity consts.  $K \times 10^{-4}$  at 0° were 1.00, 7.94, 0.158 and 2.34, resp. K. K.

The aging and stability to light of ferrous oxide hydrates in the presence and absence of alkali nitrates Oskar Baudisch *Ber.* 68B, 2046-9 (1935); cf. Wolf and Baudisch, C. A. 28, 7102 — Fe(OH)<sub>2</sub> and FeCO<sub>3</sub> can be kept in contact with aq. solns. of KNO<sub>3</sub> in sealed containers for many months without reduction of nitrate or oxidation of Fe provided that they are kept in the dark and the system is absolutely free of O<sub>2</sub>. After such aging, exposure to sunlight for 3 weeks develops color due to Fe<sub>2</sub>O<sub>3</sub>. The light energy splits off atomic O from the nitrate which oxidizes the Fe. The resulting nitrite reacts at once with excess Fe<sup>2+</sup> and further reduction of the O-N compds. takes place governed by the *p<sub>H</sub>* of the medium. F. L. Browne

Sorption phenomena and chemical processes. VII

Theory of permutoid reactions. I. Putilova. *J. Gen. Chem.* (U. S. S. R.) 5, 834-7 (1935); cf. Lipatov and Sokolova, *C. A.* 25, 4120—It was previously shown that solid nitroaluminum (I) reacts with  $\text{Cu}(\text{OAc})_2$  solns. to form the lake and  $\text{AcOH}$  (cf. *C. A.* 24, 766). The lake is a definite compd., the equiv. wt. of I being 135. Further expts. show that the reaction const. changes with temp. according to van't Hoff's equation. The equation, when integrated, reads  $\ln(K_1/K_2) = U \times (T_2 - T_1)/R \times (T_1 \times T_2)$ ,  $K_1$  and  $K_2$  being the equl const. at the temps  $T_1$  and  $T_2$ , resp.,  $U$  being heat of reaction and  $R$  the gas const.  $K$  was detd. at 20° and 40° from the equl amts of Cu adsorbed by I from  $\text{Cu}(\text{OAc})_2$  solns. of varying concn. The corresponding value of  $U$  was 1600 cal. Direct measurements of  $U$  were made by rubbing I with water and mixing the resulting paste with  $\text{Cu}(\text{OAc})_2$  soln. in a Schottke calorimeter. The av. result (corrected for the heat of diln. of the  $\text{Cu}(\text{OAc})_2$  soln.) was 1576 cal. Permutoid reactions, therefore, obey the same laws (mass-action law, le Chatelier principle) that govern the equl in homogeneous media. B. Soyenko

Dissociation of nickel sulfide. D. N. Tarasenko and A. V. Bogoslovskaya. *J. Gen. Chem.* (U. S. S. R.) 5, 836-8 (1935)— $\text{NiS}$  was prepd. by heating a mixt. of powd. Ni and S, in the ratio of 5/3, for 13 hrs. at 800-50°. The product was mixed with 10% by wt. of S and heated again for 6 hrs. The final product contained 64.12% Ni. Jellinek's method was used for detg. dissociation of  $\text{NiS}$  at 750°, 850° and 900°.  $\text{NiS}$  sulfide is not a definite chem. compd. but rather a solid soln. The existence of  $\text{NiS}$  or higher sulfides as definite compds. could be detd. only by a melting diagram of  $\text{Ni-S}$  (where  $S > 40\%$ ), i. e. by an extension of Bornemann's diagram, *C. A.* 2, 1686.

S. L. Madorsky  
A critical discussion of some experimental work on physicochemical analysis of binary systems. V. F. Ust-Kachintzev. *J. Gen. Chem.* (U. S. S. R.) 5, 892-8 (1935)—A discussion of the exptl. work of Kendall, Howell and Houdford, Usanovich and of Terpuvov, diagrams of compn. properties, at various temps., of binary systems of the rational and irrational types. It is concluded that electrically conducting systems may not necessarily show a deviation from additivity in the measurement of other properties. In irrational systems there is displacement of max. and min. of various properties and deviation from additivity. Inflection points on curves of temp. coeff. vs. properties of irrational systems cannot serve as guides for detn. of compn. of compds. formed in a binary system. S. L. Madorsky

Internal friction of binary systems in critical zones (of layer formation). R. V. Mertzlin. *J. Gen. Chem.* (U. S. S. R.) 5, 899-903 (1935)—A crit. discussion of Tsakalotos' theory (*C. A.* 4, 402) in regard to the relation between the geometric form of viscosity isotherms and the nature of the zone of layer formation of binary systems near the crit. temp. of layer formation. It is concluded that this theory is wrong in assuming that the nature of viscosity isotherms is independent of the zone of layer formation. S. L. Madorsky

Form of curves of critical temperatures of binary mixtures. R. V. Mertzlin. *J. Gen. Chem.* (U. S. S. R.) 5, 1073-6 (1935); cf. *C. A.* 29, 5002<sup>a</sup>—It is shown geometrically, on the basis of Stakhovskii's formula (cf. *C. A.* 22, 2302),  $\sigma = \sigma_1 \sigma_2 / [\sigma_1(1-x) + \sigma_2 x]$ , where  $\sigma_1$  and  $\sigma_2$  are surface tension of the mixt. and the 1st and 2nd components of the binary mixt., resp., and  $x$  is the mol. fraction of the 1st component, that the polytherms of crit. temps. could be either concave or convex to the compn. axis, the form depending on the relation of  $\sigma_1$  and  $\sigma_2$  and the temp. coeffs. of the two components. The linear formula of Pavlevskii and Straus does not give a general soln. and is true only for special cases. S. L. Madorsky

The melting curve of mixtures of heavy and ordinary water. The solubility equilibrium in the system. Mladen Deželić. *Z. anorg. allgem. Chem.* 225, 173-4 (1935)—Thermal analysis of the system  $\text{D}_2\text{O-H}_2\text{O}$  indicates a continuous series of solid solns. The graph is a straight

line, indicating impossibility of sepg. heavy water from ordinary water by fractional freezing. J. H. Reedy

Determination of the melting points of calcium, strontium and barium. F. Hoffmann and A. Schulze. *Z. Metallkunde* 27, 155-8 (1935).—See *C. A.* 29, 7165<sup>a</sup>.

G. Dege  
Allotropy of phosphorous pentoxide. A. N. Campbell and A. J. R. Campbell. *Trans. Faraday Soc.* 31, 1567-74 (1935).—The d, soln. tensions, and soly of amorphous, vitreous, and one cryst. form of  $\text{P}_2\text{O}_5$ , were investigated. The only homogeneous form, vitreous, has the lowest soly and highest d., and is most stable. The vitreous forms from amorphous and forms with it a true soln. of vitreous in amorphous. It is prepd. by heating amorphous for 3 weeks in a sealed tube at 450°. The cryst. is formed with vitreous on heating amorphous at 350-600° for not too long a period. It is not considered possible to prep. pure cryst. because it arises from the heating of amorphous and then passes into the more stable vitreous form. C. E. P. Jeffreys

The melting curve of oxygen. J. H. C. Lissman and W. H. Keesom. *Physica* 2, 839 (1935)—For pressures from 48.2 to 167.7 kg. per sq. cm.  $0^\circ\text{M}$  54.90-56.25° abs. B. J. C. van der Hoeven

Solubility of cobalt nitrate in aqueous solutions of nitric acid and transition points of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  into  $\text{Co}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ . A. Valdmann and L. L. Klyachko-Gurvich. *J. Gen. Chem.* (U. S. S. R.) 5, 791-4 (1935)—The soly of  $\text{Co}(\text{NO}_3)_2$  in aq. solns. of  $\text{HNO}_3$  was studied at 25° and 80°. In the case of the 25° isotherm, concns. of  $\text{HNO}_3$  and  $\text{Co}(\text{NO}_3)_2$  varied from 0 to 39.59% and from 50.57 to 21.68%, resp., and the solid phase consisted of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ . In the case of the 80° isotherm, concns. of  $\text{HNO}_3$  and  $\text{Co}(\text{NO}_3)_2$  varied from 0 to 10.27% and from 67.86 to 63.68%, resp., and the solid phase consisted of  $\text{Co}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ . Points of transformation  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} \rightarrow \text{Co}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  were studied also by the dilatometric and thermometric methods, also the effect of  $\text{HNO}_3$  concn. on the displacement of these points, by the thermometric method, in the interval 37-56°. In a neutral soln. the transition point is 54-5°, in acid solns. the points are 52°, 50°, 48°, 43° and 41° for  $\text{HNO}_3$  concns. of 1, 3, 10, 16.05, 26.75 and 35.87%, resp.  $\text{HNO}_3$  affects considerably the ratio of  $\text{Co}(\text{NO}_3)_2$  and  $\text{H}_2\text{O}$  in solns., particularly at 25° and has a salting-out effect on the hydrate. On satg. the soln. at 80° and then cooling to 25°, the entire soln. becomes cryst., and contains 39.5%  $\text{Co}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and 60.5%  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ . On satg. the soln. at 80° and then cooling to a little above 56°, 39.5% of the  $\text{Co}(\text{NO}_3)_2$  crystallizes as a trihydrate. To obtain crystn. of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  free from  $\text{Co}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ , a soln. conrg. 62.88%  $\text{Co}(\text{NO}_3)_2$  is prepd. at 80° and on cooling the entire mass crystallizes as  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ . Another way is to sat. the soln. with  $\text{Co}(\text{NO}_3)_2$  at 56° (m. p. of the hexahydrate) and then cool the soln. S. L. M.

The iron-oxygen diagram. D. P. Bogatskii. *Metallurg* 10, No. 4, 64-75 (1935)—The differences in the results of various investigators, particularly in regard to soly of Fe in FeO and of  $\text{O}_2$  in Fe, are discussed. H. W. Rathmann

Thermal analysis of the system  $\text{POCl}_3\text{-SO}_2\text{Cl}_2$ . G. P. Luchinskii and A. I. Likhacheva. *Z. anorg. allgem. Chem.* 225, 175-6 (1935)—F-p measurements on mixts. of  $\text{POCl}_3$  and  $\text{SO}_2\text{Cl}_2$  show a eutectic conglomerate at -73.8°, consisting of  $\text{POCl}_3 + 3\text{SO}_2\text{Cl}_2$ . No definite compds. are indicated. J. H. Reedy

Equilibrium in the system water-magnesium bromide. Frederick H. Getman. *Rec. trav. chim.* 54, 886-72 (1935)—The soly. of  $\text{MgBr}_2$  in  $\text{H}_2\text{O}$  was detd. from -42.7° to 100° and the m. p. of  $\text{MgBr}_2 \cdot 6\text{H}_2\text{O}$  found to be 172.4°. The temp. of equl. between  $\text{MgBr}_2 \cdot 10\text{H}_2\text{O}$ , ice, soln., and vapor is -42.7°.  $\text{MgBr}_2 \cdot 10\text{H}_2\text{O}$  and  $\text{MgBr}_2 \cdot 6\text{H}_2\text{O}$  are the only hydrates that exist and the transition temp. is 0.83°. The  $\text{MgBr}_2\text{-H}_2\text{O}$  equl. diagram is plotted. Arthur A. Vernon

Physicochemical properties of some amine-water systems capable of separation. R. V. Mertzlin. *J. Gen. Chem.* (U. S. S. R.) 5, 888-91 (1935); cf. Ust-Kachintzev, *C. A.* 29, 7759<sup>a</sup>—The existence of chem.

comps and their thermal decompn in the systems of  $\beta$ -collidine-water and  $\text{Et}_3\text{N}$ -water was disclosed by the method of surface tension. The polytherms of surface tension of some amine-water systems capable of sepn show properties analogous to those of tautomeric comps. This may be explained by the rapid thermal decompn of the compd and the conversion of the pseudoternary system into the binary. The 2 systems are typically anomalous, because the isotherms show a sharply displaced active branch and 2 faintly developed maxima shifted with increase of temp toward the amine. C. B.

Homogenization with amines of some amine-water systems with higher critical temperature of separation into layers R. V. Merzhan and V. F. Ust-Kachkintzev. *J. Gen. Chem. (U. S. S. R.)* 5, 904-19(1935).—The homogenizing effect of the amines capable of giving inseparable mixts with  $\text{H}_2\text{O}$  on amine-water systems was studied with  $\text{PhNH}_2$ - $\text{H}_2\text{O}$  of the crit. temp.  $167^\circ\text{C}$  and  $\text{PhNHNH}_2$ - $\text{H}_2\text{O}$  of crit. temp.  $55^\circ\text{C}$  at a temp. interval of  $0$ - $50^\circ\text{C}$  in the following ternary systems:  $\text{H}_2\text{O}$ - $\text{PhNH}_2$ - $\text{C}_6\text{H}_5\text{N}$ ,  $\text{H}_2\text{O}$ - $\text{PhNH}_2$ - $\text{C}_6\text{H}_5\text{N}$ ,  $\text{H}_2\text{O}$ - $\text{PhNH}_2$ - $\text{PhCH}_2\text{NH}_2$ ,  $\text{H}_2\text{O}$ - $\text{PhNHNH}_2$ - $\text{C}_6\text{H}_5\text{N}$ ,  $\text{H}_2\text{O}$ - $\text{PhNHNH}_2$ - $\text{C}_6\text{H}_5\text{N}$ ,  $\text{H}_2\text{O}$ - $\text{PhNHNH}_2$ - $\text{PhCH}_2\text{NH}_2$ , and  $\text{H}_2\text{O}$ - $\text{PhNHNH}_2$ - $\text{PhNH}_2$ . The homogenizing ability of the amines decreases in the order  $\text{C}_6\text{H}_5\text{N}$ ,  $\text{C}_6\text{H}_5\text{N}$ ,  $\text{PhCH}_2\text{NH}_2$ ,  $\text{PhNHNH}_2$ . With increasing temp. ( $40$ - $50^\circ\text{C}$ ) the homogenizing ability of  $\text{C}_6\text{H}_5\text{N}$  and  $\text{PhCH}_2\text{NH}_2$  increases, while that of  $\text{C}_6\text{H}_5\text{N}$  decreases. No direct connection exists between the homogenizing action of compds at an arbitrarily selected temp and their phys. consts. On the basis of the results of homogenization of the mixt.  $\text{PhNH}_2$ - $\text{H}_2\text{O}$ , the existence of a lower crit. temp. in the system is assumed. The existence of considerable retrograde soly and the arrangement of crit. points on the binodal curves accord well with the previous supposition of the existence of highly placed false lower crit. temps. for a series of nonstratifying amine-water systems. Chas. Blanc.

System water-potassium thiocyanate Andre Chretien and Oscar Hoffer. *Compt. rend.* 201, 1131-3(1935).—A satd. soln of KCNS has no p., the curve rising steadily to the m. p. of the anhyd. salt ( $177.2^\circ\text{C}$ ). There are 2 hydrates:  $\text{KCNS} \cdot 0.5\text{H}_2\text{O}$ , stable  $-29.5^\circ$  to  $6.8^\circ$ , and  $\text{KCNS} \cdot 0.8\text{H}_2\text{O}$ , metastable,  $-30.1^\circ$  to  $-17.75^\circ$ ; and 3 eutectic points at  $-20.2^\circ$  (51.2%), stable, and at  $-33.2^\circ$  (54.4%) and  $-30.05^\circ$  (51.8%) metastable. C. A. Silberrad.

Tertiary system potassium nitrate-nitric acid-water V. I. Nikolaev, S. K. Chirkov, and A. G. Kogan. *Kali (U. S. S. R.)* 1935, No 7, 23-7.—The field of crystal of  $\text{KNO}_3$  was studied and the melting curve of the binary system  $\text{KNO}_3$ - $\text{HNO}_3$  is given. The fields of crystal of ice,  $\text{KNO}_3 \cdot 2\text{HNO}_3$  (potassium trinitrate (I)),  $\text{HNO}_3$ , and hydrates of  $\text{HNO}_3$  were studied. The tertiary diagram is given. I is very stable, in the presence of excess  $\text{HNO}_3$ , to the dissociation action of  $\text{H}_2\text{O}$ , it can be transported long distances in sealed containers at a temp. nearing  $0^\circ$ . I can be used to obtain pure  $\text{KNO}_3$  and highly concd.  $\text{HNO}_3$  (by heating to  $100$ - $120^\circ$ ).  $\text{HNO}_3$  of 99.8% was obtained. A. Pestoff.

The comparative action of mixed catalysts when used for the simultaneous dehydration of ethyl alcohol and ammonia J. N. I. Shufkin, A. A. Balandin, and Z. I. Plotkin. *J. Phys. Chem.* 39, 1197-1205(1935).—See C. A. 29, 3584. II Catalytic preparation of monethylamine N. I. Shufkin, A. A. Balandin, and F. T. Dumov. *Ibid.* 1207-16.—See C. A. 29, 3585. G. G.

The oxidation of carbon monoxide catalyzed by nitrogen dioxide R. H. Crist and O. C. Roehling. *J. Am. Chem. Soc.* 57, 2196-2203(1935); cf. *Science* 80, 338 (1934).—The oxidation of CO catalyzed by  $\text{NO}_2$  in a Pyrex vessel was measured at  $527^\circ$ . The rate-dep. reaction at low concn. of the catalyst appeared to be a chain mechanism, and at higher concn. to be the trimol oxidation of NO. The rate is first-order with respect to  $\text{O}_2$  at high concn. of  $\text{NO}_2$  and at low concn. it is proportional to the  $\text{O}_2$  concn. and also to the second power of the CO concn. The initial rate is approx. proportional to the square of the H pressure and is infinite with the H pressure

greater than  $0.53$  mm. The effect of  $\text{H}_2\text{O}$  on the rate was measured. H. H. Rowley.

Decomposition of chloral catalyzed by nitric oxide. F. H. Verhoek. *Trans. Faraday Soc.* 31, 1521-6(1935).—The decompn. of chloral by NO is first-order with respect to chloral, and the rate increases in direct proportion to the concn. of NO.  $\text{N}_2\text{O}$  and  $\text{O}_2$  have no catalytic effect. A heat of activation of  $37,100$  cal. was calcd., the use of 2 squared terms seems to account for the catalyzed reaction.  $\text{CHCl}_3$  decompn. is nearly first-order with respect to initial pressure at  $512^\circ$  above  $50$  mm. The decompn. is catalyzed by NO and the products. C. I. P. Jeffreys.

Decomposition of acetaldehyde catalyzed by nitric oxide. F. H. Verhoek. *Trans. Faraday Soc.* 31, 1533-6 (1935).—Decomposition of  $\text{AcHl}$  is catalyzed by NO homogeneously, but the reaction is complicated by the apparent superposition of two effects: (1) an induced catalysis initiated by an oxidation and (2) a simple collisional catalysis. At  $480^\circ$  the reaction is approx. of the  $3/2$  order with respect to  $\text{AcHl}$ , and the rate becomes proportional to NO concn. when NO is in excess. A value of  $37,300$  cal./g. mol. was calcd. as the energy of activation. C. E. P. Jeffreys.

Decomposition of acetaldehyde catalyzed by nitrous oxide I. H. Verhoek. *Trans. Faraday Soc.* 31, 1527-33 (1935).—The decompn. of  $\text{AcHl}$  is catalyzed by  $\text{N}_2\text{O}$ , but in clean silica vessels the decompn. is complicated by a surface reaction. At  $480^\circ$  it appears first-order with respect to  $\text{AcHl}$  and rises to a max. with increasing pressure of  $\text{N}_2\text{O}$ . With the vessel walls covered with C the reaction is of the  $3/2$  order for a single expt., and the rate is proportional to  $[\text{N}_2\text{O}]^{1/2}$ . An activation energy of  $39,800$  cal./g. mol. was calcd. The  $\text{N}_2\text{O}$  is assumed to form an intermediate which acts as a catalyst or chain initiator.  $\text{AcHl}$  decompn. catalyzed by  $\text{O}_2$  is first-order with respect to  $\text{O}_2$  concn. The amt. of  $\text{AcHl}$  decomposed in the presence of a given amt. of  $\text{O}_2$  is greater the greater the excess of  $\text{AcHl}$ . C. L. P. Jeffreys.

The thermal decomposition of sugar and its catalytic acceleration E. H. Ruesenfeld and F. Muller. *Ber.* 68B, 2052-4(1935).—The thermal decompn. of sucrose during 3 hrs. heating at  $150^\circ$  was detd. by dissolving the product in water and observing the intensity of color of the soln. (caramelization) and the amt. of carbonaceous residue. Pure sugar showed little caramelization and no residue. When 5% NaCl was mixed with the sugar caramelization increased and there was a moderate amt. of residue. Addn. of  $\text{FeCl}_3$  along with the NaCl failed to increase the decompn. until it amounted to 0.005% of the mixt. (sugar + NaCl +  $\text{FeCl}_3$ ). Greater quantities of Fe decreased caramelization but greatly increased the carbonaceous residue. In the well-known expt. of making a lump of sugar burn by spreading cigar ashes over it, the Fe in the ash probably is less important as a catalyst than the alk. salts in the ash. F. L. Browne.

Catalytic properties of rhenum and of some of its compounds S. B. Anusimov, V. M. Krasheinnikova, and M. S. Platonov. *J. Gen. Chem. (U. S. S. R.)* 5, 1059-65(1935).—With metallic Re as a catalyst, expts. were performed on hydrogenation of maleic acid in aq. soln. and cyclohexane in EtOH soln. at  $15$ - $20^\circ$ , of cyclohexane at  $150^\circ$  and  $250^\circ$ , of  $\text{C}_6\text{H}_6$  at  $100^\circ$ ,  $150^\circ$  and  $200^\circ$ , of  $\text{PhNO}_2$  at  $250^\circ$  and  $300^\circ$  and of NO at  $200^\circ$ ,  $250^\circ$ ,  $300^\circ$ ,  $350^\circ$  and  $400^\circ$ . Expts. were performed also on dehydrogenation of EtOH at  $200$ - $600^\circ$ , and on oxidation of  $\text{NH}_3$  at  $500^\circ$ , in the presence of metallic Re, and of  $\text{CH}_4$  at  $300$ - $400^\circ$  in the presence of Re and K perchlorate. Re is a weak catalyst for hydrogenation, but active for dehydrogenation of EtOH. Catalytic action of Re on oxidation at high temps. is hindered by the formation of highly volatile Re oxides. S. L. Madorsky.

Catalysis with organic fibers H. G. Bredig, I. Gersinger, and H. Lang. *Biochem. Z.* 282, 88-98(1935), cf. C. A. 26, 5110.—A catalyst such as  $\text{Et}_3\text{NHCl}$ , which contains no asym. atom, produces only optically inactive nitriles, but coupled with an optically active cellulose fiber it acts stereochemically in a relatively sp. manner on the substrate. Fibers of diethylamino-cellulose con-

dense not only benzaldehyde with HCN to optically active hydroxy nitrile, but also various other aldehydes. 1 ether catalysts were made from cellulose and different bases and the results obtained with them are discussed.

S Morgulis

Catalytic decomposition of hydrogen peroxide on different metals Joseph Weiss. *Trans. Faraday Soc.* 31, 1547-57 (1935)—The initial process in the catalytic decompn of  $H_2O_2$  by metals involves a transfer of 1 electron from the metal to adsorbed  $H_2O_2$  forming the radicals  $OH$  and  $HO_2$ . The 2 principal reactions are  $H_2O_2 + e_{metal} = OH^\cdot + OH_2^\cdot$  and  $HO_2^\cdot = HO_2 + e_{metal}$ . This mechanism is favored by the fact that the decompn is definitely increased through cathodic polarization, which decreases the work function of the metal.

The catalytic decompn on Au, Pt, Pd, Ag and Zn was investigated and discussed from this point of view. Poisoning by KCN, Na<sub>2</sub>S and CO is explained on the assumption that coordination surface compds are formed with electron shells of the inert-gas type, which practically prevent electron transfer from Pt or Pd. C E P. J

Catalytic action of Japanese acid clay I Condensation of benzyl chloride and benzene. Tsutomu Kuwata *J Soc Chem Ind, Japan* 38, Suppl binding 503-b (1935)—Japanese acid clay is an effective catalyst in the condensation reaction  $PhCH_2Cl$  and  $p$ -dibenzylbenzene were found in the condensation products, and the presence of  $o$ - and  $m$ -compds was suspected. The activity of the clay was decreased when  $Fe_2O_3$  and  $Al_2O_3$  were removed from it with HCl. Prep.  $Fe_2O_3$  also was an effective catalyst. Five references. Karl Kammermeyer

Exchange of heavy hydrogen atoms between hydrogen and ammonia K Wirtz *Naturwissenschaften* 23, 721-2 (1935)—Exchange of D from  $HD$  to  $NH_3$  is catalyzed by a Pt wire at 300°. The results are detd from the D content of the  $H_2$  after the  $NH_3$  has been frozen out (Parkas method, C A 28, 3657). The const.  $K = [NH_2D][H_2]/[NH_4][HD] = 1.9$  at 300°. From spectroscopic data 1.82 was estd. B J C v d. H

Measurement of specific heat of iron at high temperatures. K. Mehss *Arch Eisenhüttenw* 9, 209-12 (1935)—The methods of detn used at present are discussed as to accuracy and the sp heat is detd for an Fe contg 0.06% C with an accuracy of 0.05%. It is recommended to make sp-heat measurements relatively instead of directly, and to refer sp heat to a standard material, preferably sinter-corrundum, which is particularly suitable for this purpose because of its mech properties.

M Hartenhem

The specific heat of  $Ag_2HgI_4$  in connection with its crystal structure J. A. A. Ketelaar *Z physik. Chem* B30, 53-60 (1935), cf C A 28, 7098<sup>4</sup>—The true sp heat of  $Ag_2HgI_4$  was measured between 20° and 100° by a modified Nernst method. The curve begins to show anomalies at 37° with a transition point at 50.7° = 0.2°, where the sp. heat is three times normal. The heat of transition is 0.29, kg.-cal./mol. for the homogeneous transition and 1.04 kg.-cal./mol. for the heterogeneous part. The "averaged structure" previously described is the best explanation for both sp heat and cond. changes.

G M. Murphy

Second virial coefficient and specific heats of oxygen J. A. van Lameren. *Physica* 2, 833-9 (1935)—Previous detns. were extended (cf. C A 26, 889) at liquid- $C_{2}H_2$  temps. from sound-velocity measurements. At 86.27° and 83.80° abs., resp. the values of the  $S$  function are  $5 \times 10^3 = -7.613$  and  $-8.214$ ,  $c_p/c_v$  for  $p = 0$  is 1.406 and 1.404, resp. An empirical equation for  $B$ , the second virial coeff., is given. The calcd. values for sp heat of  $O_2$  at 90.00°, 80.27° and 83.80° are at zero pressure  $c_p = 6.86$ , 6.87 and 6.90 cal. per mole. B. J. C. v d. H

Entropy of heavy hydrogen Klaus Clusius and Ernst Bartholomé. *Z. physik. Chem.* B30, 258-64 (1935); cf. C A 29, 7169<sup>4</sup>—The entropy of D at 298.15°K., obtained from thermal data is 33.91, cal/mol./degree. Calcn of this quantity from statistical mechanics and spectroscopic data gives 38.08. The difference of 5.06,

can be explained by assuming that para-D can rotate freely in the solid state up to 10°K. with  $J = 1$ , where  $J$  is the rotational quantum no. and in addn, the nuclear spins of the ortho and para mols. are preserved. The entropy term is  $R \ln 9 + 1/2 R \ln 3 = 5.09$ , which agrees well with the thermal calcn. For practical calcs., the abs. value of 34.63 entropy units is used, this being corrected by the term  $R \ln 9$  for the entropy of nuclear spin. At low temp, the chem const for ortho-D,  $s'_1 = -0.68$  and for normal D,  $s'_1 = -0.622$ . The vapor pressure const,  $s'_p = -0.67$ , for normal D. At high temp,  $s'_1 = -2.01$  and  $s'_p = -2.76$ . G. M. Murphy

Caloric and thermal properties of condensed heavy hydrogen Klaus Clusius and Ernst Bartholomé. *Z. physik. Chem* B30, 237 57 (1935)—App. is described for making certain thermal measurements on D. The following results were obtained, the second figure in each case being the corresponding value for H, which was not measured in this investigation but is given for comparison. Heat of vaporization at 194.6 mm Hg, 302.3 = 0.6 cal, 210.7, heat of fusion, 47.0 = 0.4 cal, 28.0, heat of sublimation at 0°K, 274.4 cal, 183.4,  $\theta$  value of Debye,  $(C_p) 89^\circ, 91^\circ$ , triple point, 18.65°K, 13.95, slope of the fusion curve at the triple point, 40.5 kg./sq. cm., 30.3; mol. vol of liquid at triple point, 23.14 cc, 20.15; mol. vol of solid at triple point, 20.48 cc, 23.31; zero-point energy of lattice, approx 215 cal, 305. The differences can all be explained on the basis of the difference of the zero-point energies and anharmonicity const in the lattice vibrations, by assuming the same intermol forces for each. G. M. Murphy

The critical data of light and heavy water and the density-temperature diagrams E H Riesenfeld and T L Chang *Z physik. Chem* B30, 61-8 (1935); cf C A 29, 4648<sup>2</sup>—The crit temp of  $H_2O$ - $D_2O$  mixt. is an almost linear function of the const for the pure liquids and is given by  $t_c = 374.2 - 2.7n$ , where  $n$  is the mole fraction of  $D_2O$ . For the latter,  $t_c = 371.5^\circ$ ,  $p_c = 218$ , atm,  $d_k = 0.363$  g./cc. The  $d_s$  of  $H_2O$  and  $D_2O$  are equal at 49.5°, where the satd vapors have equal  $d_s$ , and at 370.0°, where the liquids have the same  $d$ .

G. M. Murphy

Phase equilibria in hydrocarbon systems IX. Specific heats of butane and propane. B. H. Sage and W. N. Lacey. *Ind. Eng. Chem.* 27, 1484-8 (1935); cf. C A 29, 4650<sup>2</sup>—The sp. heat at atm. pressure and at 500 lb./sq. in. was detd. for the range 60° to 220°F. The value  $C_p$ , defined as the heat required to raise the temp. of the satd. liquid by 1°F., was detd. for the range 50° to 200°F. X. Thermodynamic behavior of liquid mixtures of butane and crystal oil *Ibid.* 28, 106-11 (1936)—The data cover the pressure range from below atm. to 3000 lb./sq. in. and the temp range 70° to 220°F. Data are reported for the relation of sp. vol. to pressure and temp., compressibility and thermal expansion of the liquid as functions of compn, effect of temp. and compn. on bubble-point pressure, sp. heat at const. pressure for bubble-point liquid as functions of temp. and compn, isothermal change in entropy and in heat content with pressure at 220°F., temp.-heat content diagram for liquid contg. 40.0% butane and partial sp. heat and sp. vol. for each component of bubble-point liquid as functions of compn.

F. L. Browne

The heats of formation and solution of potassium dithionate. H. Zeumer and W. A. Roth. *Z. anorg. allgem. Chem.* 224, 253-6 (1935)—For the decompn.,  $K_2S_2O_8(s) \rightarrow K_2SO_4(s) + SO_2(g)$  the heat absorbed is 5 kg.-cal./mole. The heat of formation of  $K_2S_2O_8$  from the elements is calcd., from this heat and the known heats of formation of  $K_2SO_4$  and  $SO_2$ , to be 118.9 kg.-cal. At 20° the heats of soln. (in kg.-cal. absorbed) of 1 mol. of  $K_2S_2O_8$  in 400, 800 and 1600 mols.  $H_2O$  are, resp., 12.9, 13.1, and 13.1.

L. E. Steiner

The heat of formation of zinc sulfide. Critical remarks on Mixer's sodium peroxide method. H. Zeumer and W. A. Roth. *Z. anorg. allgem. Chem.* 224, 257-64 (1935); cf. C A 29, 7779<sup>2</sup>—The heat of formation of regular 7n blende ( $d_2^2 = -4.088$ ), detd. by Mixer's  $Na_2O_2$  method,

is 41.5 ± 0.9 kg.-cal. The sources of error of the method are pointed out. All heats of formation of oxides, determined by this method are uncertain, owing to variation in composition of the various melts obtained as end products.

L. E. Steiner

Solubility relationships of water solns. saturated with respect to sucrose and dextrose in relation to the storage

of sherbet and water ice (Leighton, Leviton) 12. Absorption law for slow neutrons (Rasetti, *et al.*) 3. Absorption spectra of the vapors of the monoxides of Cu, Fe, Ni and Co and the determination of their heats of sublimation (Trivedi) 3. Absorption spectra of the vapors of the monosulfides of Fe, Ni, Co and Cu and the determination of their heats of sublimation (Trivedi) 3. Velocity of solution of Fe in molten Zn (Grubitsch) 9.

### 3—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

W. ALBERT NOYES, JR.

The natural philosophical foundations of quantum mechanics Grete Hermann *Naturwissenschaften* 23, 718-21(1935)

Problems of the new quantum theory of the electron Viktor Weisskopf *Naturwissenschaften* 23, 631-7, 647-53, 669-74(1935)—A review discussing Schrödinger's equations, relativity, spin, Dirac's theory, positron theory, etc.

The neutrino theory of light H. R. de L. Kronig *Physica* 2, 854-60(1935)—Continuation of a previously developed theory (C. A. 29, 7175) B. J. C. van der Hoeven

The Born theory of the electron Arthur Bramley *Science* 82, 438-9(1935)—The Born theory of the electron (Born, *Proc. Roy. Soc. A* 143, 410(1934), Born and Infeld, *Proc. Roy. Soc. A* 144, 425(1934)) is discussed.

Exchange forces and the structure of the nucleus James H. Bartlett, Jr. *Phys. Rev.* 49, 102(1935)

A quantum mechanical discussion of orientation of substituents in aromatic molecules G. W. Wheland and Linus Pauling *J. Am. Chem. Soc.* 57, 2086-95(1935), cf. C. A. 29, 3599—By use of the method of molecular orbitals, a quantum discussion of the charge distribution in aromatic molecules undergoing substitution reactions is carried out, with consideration of the inductive effect, the resonance effect, and the polarizing effect of the attacking group. It is shown that, with reasonable values for the parameters involved, the calculated charge distributions for pyridine, toluene, phenol, the halobenzenes, etc., are in qualitative agreement with the experimental results regarding position and rate of substitution, the auxiliary hypothesis is made that the rate of substitution of the group R' for H on the *i*th C atom increases with increase in the negative charge on the *i*th C atom when R' is in position to react.

Ground state of (H<sub>2</sub>), the molecule ion (H<sub>2</sub><sup>+</sup>) and wave mechanics O. W. Richardson *Proc. Roy. Soc. (London)* A152, 503-14(1935)—The agreement between values of the fundamental constants such as dissociation energy of the ground state of the H<sub>2</sub><sup>+</sup> as determined by observation and as calculated by wave mechanics is discussed critically. The properties of H<sub>2</sub><sup>+</sup> predicted empirically from a comparative study of various excited states of H<sub>2</sub> are compared with those predicted by wave mechanics.

The three center problem I. G. S. Goudas Z. *Physik* 96, 542-5(1935)—A quantum-mechanical treatment of the H<sub>2</sub><sup>+</sup> ion, one electron in the field of 3 fixed protons, is given. A variation method is used, in which the allowable proper functions are linear functions of 3 H-like proper functions. In particular 2 models are considered, (1) with the 3 protons in a line (2) with the 3 protons forming an equilateral triangle. The numerical calculations will be published later.

Calculation of the self-consistent field with exchange for lithium V. A. Fok and M. I. Petrashen *Compt. rend. acad. U. R. S. S. (N. S.)* 3, 295-6(1935), cf. C. A. 29, 13—The wave functions for the normal and 4 excited states of neutral Li were constructed by the generalized self-consistent field method (electron exchange was taken into account) and the corresponding term values and the transition probabilities for the principal series of Li were calculated. A comparison of the observed term values of Li and Na with those calculated with and without exchange,

shows that in some cases 90% of the discrepancies of the latter are due to the neglect of the quantum exchange. The transition probabilities calculated with the functions including exchange gave a satisfactory reproduction of the observed intensities of the Li principal series.

Morris Muskat  
Absorption in mercury high-pressure discharges W. Elenbaas *Physica* 2, 763-8(1935)—Recent results of Fabrikant and Pulver (C. A. 29, 3231) on absorption of Hg light in the Hg arc were checked. Absorption of 5461, 5770, 5790 and 5791 Å, in a Hg tube was measured. The absorption increases with increased *c* in the absorbing tube and with decreasing *c* in the emitting tube. A different interpretation is given to the incomplete results of F. and P. The filling up of 2P levels is not constant, but increases with the *c* in agreement with temperature requirements.

Temperature and gradient of the mercury arc W. Elenbaas *Physica* 2, 757-62(1935)—The energy used, in a high-pressure Hg arc is expressed by  $L = A + Bm$ , where *m* = the amt of Hg per cm of tube length (C. A. 29, 6930), *A* represents the conduction loss, *Bm* the radiation loss. By assuming *A* to be independent of *L* and *m* and using a mean excitation level of *V<sub>m</sub>* volts the relations between *L* and *T* are evaluated. For *V<sub>m</sub>* a practical value of 7.8 v. is obtained and the gradient *G* is found proportional to  $L^{1/2} m^{1/2} d^{-1/2} (L - A)^{-1/2}$  with *d* the tube diam. This leads to a minimum of *G* depending on *L* for  $L - A = 28 \text{ w per cm}$ . The *G* values at higher *L* agree well with experimental data. In different form the derived temperature relation is  $T = 40,400 / [k - \log(L - A)/m]$  with *k* = 7.5. This relation is, however, not in agreement with the similarity law and an empirical equation  $T = 6025 [L/(8.5 + 5.75 m)]^{1/2}$  follows the data somewhat better.

The gradient of the mercury high-pressure discharge as a function of pressure, diameter and current density W. Elenbaas *Physica* 2, 787-92(1935), cf. C. A. 29, 7683

Total radiation of super high-pressure mercury discharge W. Elenbaas and W. de Groot *Physica* 2, 807-10(1935)

Magnification of photocurrents by emission of secondary electrons F. M. Penning and A. A. Kruthof *Physica* 2, 793-804(1935).

The ionization of a gas at different pressures under the influence of photons and corpuscular radiation J. Clay *Physica* 2, 811-16(1935)—In a gas filled vessel the no. of ions *I* produced by corpuscles depends on *p*, the pressure required to limit the path of the corpuscles to the vessel dimension and on *p<sub>0</sub>* required to limit the path of the wall particles to the vessel. For pressure *p* smaller than *p<sub>0</sub>* and *p<sub>0</sub>*' the value of  $I = (Ap/p_0) + (Bp/p_0')$ , for  $p_0 < p < p_0'$ ,  $I = Ap + (Bp/p_0')$ ; for  $p > p_0$  and  $p > p_0'$ ,  $I = Ap + B$ . These equations hold for photons. For corpuscular radiation a term proportional to *p* is to be added. Experimental values on saturation current in several gases by γ-rays or cosmic rays at different pressures confirm the theory (graphs).

Ionization measurements in air at high pressures J. Clay and M. A. van Tijn *Physica* 2, 825-32(1935)—The ionization current of air under pressure in a steel vessel was measured. At 20° and 30° with 95 atm. air pressure no temperature effect was found. The pressure rela-

tions are completely expressed by sepn. of space and wall radiation combined with the theory of column ionization. The satn. value obtained for fields higher than 800 v. per cm. agrees with the theory (cf. preceding abstr.). At satn., difference between neg. and pos. charging disappears. Even at 500 v. per cm. the satn. is incomplete for 1 atm. pressure; the field required for satn. increases with pressure. B. J. C. van der Hoeven.

A short method for determination of saturation current by the Jaffe theory of column ionization. H. Zanstra. *Physica* 2, 817-24 (1935).—A rapid graphical method is described to find from the measurement of ionization current for finite fields values for the satn. current. The method is based on the Jaffe theory (C. A. 8, 1541).

B. J. C. van der Hoeven.

The effective cross section of helium and the charged area of the helium ion. Antonio Rostagni. *Ann. Physik* 24, 543-4 (1935).—The discrepancy found by Wolf, cf. C. A. 29, 6832<sup>7</sup>, 7787<sup>8</sup>, is due to a high value for the area of the charge. Measurements of the cross section agree. R. E. DeRight.

Effect of light on diamagnetic susceptibilities. H. C. Bhuyan. *Nature* 136, 872 (1935).—The effect of light on paramagnetic susceptibilities has been reported by Bose and Raha, *Phil. Mag.* 20, 145-66 (1935). When Cl, Br and I vapors were exposed to light, a slight increase in the diamagnetic susceptibility was obtained. This is evanescent at the red end and remains very small until the violet end is reached. The shift of equilibrium is temporary, and the dark deflection is reproducible. The susceptibility of an air tube was unaffected by light. G. M. P.

Temperature dependence of free electron susceptibility. Edmund C. Stoner. *Proc. Roy. Soc. (London)* A152, 672-92 (1935).—The various formulas required in the application of the Fermi-Dirac statistics to calcn. of magnetization are given, and calcns. made of magnetization,  $M$ , as a function of  $H$  and  $T$  for the spin and diamagnetic effects considered separately and for the resultant effect both at low and at high temp. C. E. P. J.

Determination of the saturation ionization current from high-speed electrons in air. Lauriston S. Taylor. *Phys. Rev.* 48, 970 (1935).—G. Jaffe's theory of columnar ionization (C. A. 8, 1541) was successfully applied to expl. data on the ionization of air at normal pressure by a beam of electrons with a max. energy of 150 e. kv. G. M. P.

"Extra" rings in electron diffraction patterns. G. I. Finch and A. G. Quarrell. *Nature* 136, 720 (1935); cf. C. A. 29, 2443<sup>7</sup>.—"Extra" rings in electron diffraction patterns from metal films may be due either to grease or to amalgamation. The detection of "extra" rings due to amalgamation is difficult. Photographs and measurements are given for 6 sets of "extra" rings. G. M. P.

Characteristics of the adsorption of indium and thallium atoms on tungsten oxide. C. F. Powell and R. L. Mercer. *Trans. Roy. Soc. (London)* A235, 101-24 (1935).—An app. is described for measuring the atoms that are adsorbed on the surface of a metallic oxide, such as is formed on W when it is heated in air. If the W is in the shape of a strip or filament the oxide may be heated uniformly to various temps. with an elec. current through it. From elec. furnaces contg. in or Ti streams of atoms are emitted and of those that reach the filament a const. fraction is adsorbed, and this fraction (probably equal to 1) is independent of the temp. and the total no. of atoms adsorbed over a wide range. The filament when heated will drive off a current of pos. ions which are collected by electrodes in the app. A state of dynamic equl. ensues in which the no. of atoms arriving at the hot filament is balanced by the emission of neutral atoms and pos. ions. From the ratio of the pos. ion emission to that of neutral atoms can be detd. the electronic work function of the surface when free from adsorbed atoms. This is 6.13 v. for In and 6.25 v. for Ti. The ratio of the emission of neutral atoms to that of pos. ions increased with the surface concn. of adsorbed atoms in a manner corresponding to a linear decrease of the electronic work function. The characteristics of an oxide necessary for its use as a base for

the thermionic emission of pos. ions are discussed, and these are described for several oxides. Those of Ni, Fe, U and Mo. C. C. Kiess.

The structure and physical properties of thin films of metal on solid surfaces. E. N. da C. Andrade and J. G. Martindale. *Trans. Roy. Soc. (London)* A235, 69-100 (1935).—Thin films of Ag and Au, of from 20 to 100 atoms thick, were deposited on glass and other solid surfaces by the method of cathodic sputtering for the purpose of studying their color and other optical properties. The variations in these properties reported by others under apparently identical conditions of prepn. are due to uncontrolled heating. Uniform and reproducible films are obtained if the sputtering is done slowly and the base is kept cool. Such films are apparently amorphous when microscopically examd. with the highest powers. When films of the order of 50 atoms in thickness are maintained at high temps. which are, however, well under the m. p. (230° for Ag, 400° for Au) a flow of the atoms in the upper layers of the film occurs, with condensation into aggregates. On prolonged heating at somewhat higher temp. the aggregates develop into crystals with the cubic lattice structure, the (111) face being parallel to the surface of the supporting base. The flow of atoms into the crystals leaves the film blotched with large irregular patches only a few atoms thick. These areas can also begin to form crystals with a further increase in temp. until at about 600° perfect cubic crystals are formed on a background of very thin film. Heated films on glass and quartz, but not on the cleavage faces of mica, exhibited characteristic alignments of the crystals, the patterns of which were repeated after several cleanings and sputterings. These linear arrangements do not follow the course of minute polishing scratches and are, therefore, attributed to submicroscopic surface cracks. C. C. Kiess.

The diffraction of electrons by metals and organic polymers. J. J. Trillat and H. Motz. *Ann. phys.* [11], 4, 273-304 (1935).—Expts. on electron diffraction of nitrocellulose and cellulose acetate, synthetic resins and rubber indicate a striking analogy which is attributed to a very thin film of heavy substance such as satd. acid, paraffin or wax. The first diffraction diagram alters and is replaced by an amorphous type after about 10 min. and if the material is washed with ether or benzene a new ring diagram appears which is very different from the first. The same phenomenon is evident with metallic films of Au, Pt, Ag, Al and Ni. Thin deposited films of  $C_{18}H_{38}$ , stearic acid, tristearin, ceryl alc., oleic acid and beeswax gave no supplementary pattern, hence it is concluded that the layers formed naturally on metal and org. polymers are due to the crystn. of long-chain mols. of C atoms. The mols. are oriented perpendicularly to the surface and the method of electron diffraction can be used to study the structure of such thin layers. The formation of such layers is very common and many diagrams previously published are the result of a ring superimposed on another diagram. Some previous interpretations in terms of amorphous layers due to polishing and adsorption of gases may be wrong. In working with org. polymers the best method is to det. the pattern after the electron stream has been directed at a certain point for 15 or 30 min. By examg. the patterns at the beginning and later, it is possible to det. whether the layer was present or absent. If present it will be destroyed by the bombardment of the electrons. The theoretical interpretation is applied to perpendicular and oblique incidence and can be used to study the structure of org. compds. of long chain. Several diffraction patterns are shown. Arthur A. Vernon.

The photoeffect in thin adsorbed layers of alkali metals. V. Gei and I. Truten. *Physik Z. Sammelunion* 8, 342-51 (1935).—Explanations (cf. Fleischer, C. A. 21, 1223) of selective max. in curves of the photoelec. yield at metal surfaces were investigated. Measurements were made with ad. layers of alkali metal, free from oxides and hydroxides, deposited on well-dried  $SiO_2$  gel. The prepn. of the  $SiO_2$  gel, deposition of the metal, detn. of its purity and thickness, and technic of measurement of the elec. cond. and photocurrent are described in detail.



The gel is colored blue by the adsorbed layer of alkali metal. The color is attained rapidly with Cs, more slowly with Rb, and most slowly with K. The rate depends on the temp. of the gel, its distance from the metal, and the width of the tube connecting the metal with the gel. A yellow deposit is formed above 60° which possesses neither an elec. cond. nor a photoeffect. The elec. cond. of the alkali metal layers follows Ohm's law up to potentials of 4 to 5 v., beyond which reproducible results are not obtained. Since saturation of the photocurrent occurs at high potential differences, all measurements were made at 300 v. The red limit of the photoeffect for such layers appears at 78.0 and 95.0 Å. for K and Cs, resp. The relative photocurrent curves are normal and have no selective max. No temp. dependence, characteristic of composite photocathodes, could be established. These results indicate that the metal layers were essentially free from oxides and hydrides. The large displacement of the long wave limit of the photoeffect in the alkali metal layers is ascribed to the absorptive power. The normal course of the photocurrent curves indicates that the absorption power alone cannot lead to the appearance of selective max. as de Boer and Teves (C. A. 26, 915) assumed. It is probable that the appearance of selective max. is determined by the presence of oxides, hydrides and other substances in an intermediate layer.

Alvin S. Smith

A magnetic electron lens without rotation of the image. G. Stuebenow. *Z. Physik* 96, 634-42 (1935).—An arrangement of two magnetic lenses can be made, by a suitable choice of currents, to give an inverted image in the same way as an optical lens.

B. Swirles

Interaction of light nuclei. II. Binding energies of the nuclei of  ${}^2\text{H}$  and  ${}^3\text{He}$ . H. S. W. Massey and C. B. O. Mohr. *Proc. Roy. Soc. (London)* A152, 693-705 (1935), cf. C. A. 29, 2537.—The accuracy of the variation method in dealing with short-range interactions was studied by comparison with exact solns. for  ${}^2\text{H}$ . Trial functions in great variety were used in calcn. of the binding energy of  ${}^2\text{H}$  on the assumption of zero interaction between neutrons. No function was found which gave a binding energy greater than  $3.4 \times 10^6$  e. v. (observed value  $8.1 \times 10^6$ ). When an interaction is introduced between neutrons, the observed binding energy can be obtained if this attraction is even  $1/3$  of that between neutron and proton, on the assumption that the ranges of interaction in the 2 cases are comparable. This precludes the possibility of existence of a stable nucleus  ${}^4\text{He}$ . Similar calcs. for  ${}^3\text{He}$  show that an anomalous attraction between 2 protons at small distances of the same order as that between 2 neutrons will give the observed binding energy, and consequently the nucleus  ${}^3\text{He}$  must be unstable. C. E. P. J.

Photographs of cosmic rays with a Wilson Blackett chamber under special conditions. Pierre Auger and Paul Ehrenfest. *J. phys. radium* 6, 255-6 (1935).—Results of these experiments agree with A's theory, i. e., the existence of 2 kinds of corpuscular radiations ( $M$  and  $D$ ) of nearly the same energy, but having different penetrating powers (especially in the elements of high atomic no.). The group having the least penetrating power is the source of most of the numerous rays observed in the Jungfrau station and at sea level. The other group, having greater penetrating power, produces very rarely secondary radiation of great energy. But to those rays are perhaps due the feeble photons and delta rays that are observed along certain penetrating trajectories.

Antibos

Analysis of corpuscular cosmic radiation under twenty-eight meters of soil. Pierre Auger and Albert Rosenberg. *Compt. rend. 201*, 1, 17-20 (1935); cf. C. A. 29, 2537.—Measurements were made of corpuscular radiation that had passed through 28 m. of soil (sand or calcareous, d. 2.2) by means of 3 counters vertically above each other and which were 10 and 10 cm. Pb. Under these conditions the  $M$  group does not exceed 37%. The coeff. of absorption of the corpuscles attaining this depth is about  $0.4 \times 10^{-12}$  cm<sup>-1</sup>/g.

C. A. Silberrad

Absorption in matter of particles of great energy. Jacques Solovine. *Compt. rend. 201*, 1119-12 (1935).—The correction to be introduced into the ordinary quantum-mechanics formulas for the loss of energy of an electron by radiation or of a photon by production of pairs when the energy of the colliding particle approaches  $137mc^2$  is calculated. For Pb and electrons of energy  $20mc^2$  or  $40mc^2$  it is of the order of 5% (cf. Bethe and Heitler, C. A. 28, 7145).

C. A. Silberrad

Sign and nature of ultrapermeating particles of cosmic radiation. Louis Leprince-Ringuet. *Compt. rend. 201*, 1184-6 (1935).—Corpuscular cosmic radiation was passed first through a Wilson chamber  $55 \times 15$  cm. in which it was subjected to a magnetic field of 13,000 gauss, then through 2 counters, 7 cm. of Pb and finally through a third counter. About  $1/3$  of the 60 odd trajectories recorded in the third counter were practically straight, indicating particles of very great energy; in the rest (with energies of  $7 \times 10^6$  e. v.) there were approx. 2 pos. to 1 neg. These results, while not excluding other explanations, are consistent with existing views (cf. Auger, C. A. 29, 5345).

C. A. Silberrad

The theory of internal pair production. Hideo Yukawa and Shochi Sakata. *Proc. Phys.-Math. Soc. Japan* 17, 397-407 (1935).—The probability of internal pair production induced by radiationless  $S$ - $S$  transitions of a radioactive nucleus was calculated and its ratio to the probability of  $K$  electron emission by the same nuclear transition was compared with expn. in the case of  $\text{RaC}'$ . The comparison shows that this process for internal pair production will provide only a very small part of the experimentally observed pair productions.

Morris Misra

The deuteron theory. D. I. Blokhintsev. *Physik. Z. Sowjetunion* 8, 270-4 (1935).—Math. The present conception concerning the binding forces between the proton and neutron requires an application of the relativistic theory of the reciprocal effect of ponderable particles in order correctly to calc. the motions of particles in the deuteron.

Allen S. Smith

The theory of atomic nuclei. W. Heisenberg. *Z. Physik* 96, 473-84 (1935).—The mass defects of light nuclei are calculated with the assumption of an exchange force between proton and neutron of the form  $J(r) = ae^{-\mu r}$ , and the use of a simplified Hartree approximation, instead of the Thomas-Fermi method used by Majorana (C. A. 27, 5099). The agreement of the relation between  $a$  and  $\mu$  with that calculated by Wigner (C. A. 27, 2576) is much better than Majorana's; the causes of the residual discrepancy are discussed.

B. Swirles

A system of masses of light atoms deduced from nuclear reactions alone. L. Isakov. *Compt. rend. Acad. Sci. U. P. S. S. (N. S.)*, 3, 391-4 (1935).—A least-square soln. has been obtained for the masses of the light elements, including  $\text{B}^{10}$ , from the 19 best-established equations for their nuclear reactions. With these masses the resulting residuals in the reaction equations do not exceed 3 units in the 4th decimal place (in mass units), and the probable errors of the masses are within the 5th decimal place. The values obtained for  $\text{H}^1$ ,  $\text{H}^2$ ,  $\text{He}^3$ ,  $\text{Li}^6$  and  $\text{Li}^7$  are in good agreement with the mass-spectroscopic values if the latter are increased by 1.8 parts in  $10^4$  for the effect of the error in the ratio  $\text{He}^3/\text{H}^1$ . The differences from Bethe's values are within the probable errors of the latter.

Morris Misra

The theory of nuclear masses. C. F. v. Weizsäcker. *Z. Physik* 96, 431-58 (1935).—According to Majorana's theory of the nucleus (C. A. 27, 5099), the mass defect per particle is constant for all nuclei. This is not in agreement with observation for the lighter elements and the discrepancy is traced to a "surface tension" effect. It is shown how this effect can be derived by a modification of the Thomas-Fermi method. A method of interpolation is given for deriving the mass defects of nuclei with an odd number of particles from those of nuclei with an even number. A half-empirical classification of the mass defects of the nuclei is made.

B. Swirles

The structure of the light atomic nuclei. S. Flügge. *Z. Physik* 96, 459-72 (1935).—v. Weizsäcker's treatment (preceding abstract) is modified to a form suitable for very light nuclei, on the assumption of a law of exchange force between proton and neutron of the form,  $ae^{-\mu r}$ .

The values of the constants  $a$  and  $b$  calculated from different nuclei other than He show good agreement with each other, but not with Wigner's values; it appears that this agreement cannot be reached by using the Thomas-Fermi approximation. The constants are also made for a law of force  $a'e^{-b/r}$ . A modification of the  $\sqrt{Z}$  law for the nuclear radius is derived for light elements and the influence of Coulomb forces is discussed. B. Swirles

**Energies of nuclear reactions.** H. A. Wilson. *Proc. Roy. Soc. (London)* A152, 497-502 (1935), cf. C A 29, 5012. The energies of 17 nuclear reactions are discussed, and some of the values revised, making the 17 reaction equations consistent. The energies are nearly equal multiples of  $q = 0.000115$  in at. wt. units. The at. wt. of 13 light elements are calculated from the reaction energies and values obtained nearly equal to those of Bethe and of Oliphant, Kempton and Rutherford (cf. C A 29, 4618). C. P. P. Jefferys

**Emission of positrons from radioactive sources.** A. I. Alkhanov, A. I. Alkhanov and M. S. Kozlov. *Nature* 136, 719-720 (1935), cf. C A 29, 7790. A spectrum of positrons from Rn is given, and is compared with one from a Th active deposit. The spectrum is continuous up to about 1700 e. kv., plus discontinuities at 770 and 1160 e. kv., due to the internal conversion of 2 strong Ra  $\gamma$ -rays with energies of 1700 and 2200 e. kv. The ratio of positrons to Ra C  $\beta$  particles is  $2.3 \times 10^{-4}$ . An approx. equal no. of positrons was produced in Pb by Ra C  $\gamma$ -rays from the source. When Pb and Al were irradiated by both  $\gamma$ - and  $\beta$ -rays, most of the pair production was due to Ra C  $\gamma$ -rays. G. M. P.

**Creation of electron pairs by fast charged particles.** H. J. Rihl. *Proc. Roy. Soc. (London)* A152, 550-561 (1935).—The creation of electron pairs in the collision of particles moving with relative velocity near that of light is calculated. The effect of screening and the variation of probability of pair creation as a function of impact parameters are considered. C. P. P. Jefferys

**Scattering of neutrons by protons.** M. Bronstein. *Compt. rend. acad. sci. U. R. S. S.* 3, 75-8 (1935).—Math. An extension of the derivations of Wigner (C A 28, 4019). The conclusions of Peierls (cf. C A 29, 3909) have been verified. W. J. Peterson

**Radioactivity of some rare earths induced by neutron bombardment.** J. C. McLennan and W. H. Kann. *Nature* 136, 821-2 (1935).—These elements of the rare earth group for which the results of other workers disagree were reinvestigated. The compounds were bombarded with slow neutrons from Ra + Be. The half-lives obtained were:  $\text{Nd}_2\text{O}_3$ , 35 = 6 min.;  $\text{Ce}_2(\text{C}_2\text{O}_4)_3$ , 64 = 0.3 hr.;  $\text{Dys}_2(\text{C}_2\text{O}_4)_3$ , 2.5 = 0.1 hr.;  $\text{Er}_2\text{O}_3$ , 5.8 = 0.2 min.; 2.7 = 0.2 hr.;  $\text{Ho}_2\text{O}_3$ , 2.6 hr.;  $\text{Lu}_2(\text{C}_2\text{O}_4)_3$ , 1.6 = 0.4 hr. The quantities used and initial intensities are also given. G. M. P.

**The velocities of slow neutrons.** G. A. Fink, J. R. Dunning, G. B. Pegram and D. P. Mitchell. *Phys. Rev.* 49, 107 (1935); cf. Dunning, et al., C A 30, 375. A frequency-velocity curves are given for slow neutrons from a Ra-Be source at 90°K. The small deviations from theory are discussed. G. M. P.

**The absorption law for slow neutrons.** G. B. Pegram, F. Segre, G. A. Fink, J. R. Dunning and G. B. Pegram. *Phys. Rev.* 49, 104 (1935).—The absorption of slow neutrons by Ag follows closely the  $1/v$  law ( $v$  = velocity of the neutron); that of Cd does not. G. M. P.

**Excitation of nuclei by neutrons.** W. L. Hurens. *Nature* 136, 870 (1935).—The activation observed by Danysz, Rothblat, Wretenstein and Zyw (C A 29, 1692) was observed, with a Ag cylinder, with Si,  $-23 = 4\%$ , Al,  $-19 = 3\%$ , Ag,  $+40 = 5\%$ , I,  $+19 = 17\%$ , Pb, 0 =  $\pm 2\%$ . The decrease of activation of Al and Si gives directly the cross section for the total absorption of fast neutrons:  $3 \times 10^{-24}$  sq. cm. Collisions of neutrons with Ag nuclei can lead either to an excitation without capture or to capture with the formation of a radioactive element. The activity induced in Ag and in Rh is very differently affected by the absorber although these substances show

almost the same increase when the neutrons are slowed down by paraffin. G. M. P.

**The disintegration of deuterium by deuterons.** K. D. Alexopoulos. *Naturwissenschaften* 23, 817 (1935).—Expts with coincidental counters indicated that in the process of destruction of D nuclei by rapid deuterons, (A)  $\text{D}^2 + \text{D}^2 = \text{H}^2 + \text{H}^2$ , or (B)  $\text{D}^2 + \text{D}^2 = \text{He}^4 + \text{e}^-$  no rapid  $\gamma$  rays are emitted, i. e., less than 1  $\gamma$  quantum per 1000 neutrons made. For a deuteron current of 2  $\mu$  amps,  $140 \text{ e} \times 1.7 \times 10^7$  neutrons are produced. A He-filled Wilson chamber gave the same order of magnitude in results. The yields of reactions A and B (A yields from Oliphant, et al., C A 28, 4670) coincided perfectly at different values of deuteron energy (0-130 e. v.). B. J. C. van der Hoeven

**A particle yield from lithium under proton bombardment.** N. P. Heydenburg, C. T. Zahn and L. D. P. King. *Phys. Rev.* 49, 100-1 (1935).—The yield of  $\alpha$ -particles (from Li) per proton is plotted against voltage for the range 42-225 kv. The data agree with those of Herb, Parkinson and Kerst (C A 29, 7178) and extend to lower voltages. G. M. P.

**The occurrence of radium in north and central German waters.** Otto Hahn and Hans-Joachim Born. *Naturwissenschaften* 23, 729-30 (1935). On the basis of the He content ofylvinites (C A 28, 3654) it was expected that the water in contact with these salts would have a high Ra content. The Ra content was determined in several subterranean water sources, particularly from oil well drillings. As compared with max. known Ra contents of 7 and  $1.9 \times 10^{-12}$  g. Ra per cc. (Kruznach and Heidelberg) many samples from salt and oil wells in Hannover and Thuringen gave from 0.7 to  $5.5 \times 10^{-12}$  g. per cc. The He theory is thereby supported. The waters high in Ra also contain Li, the Ra content varies up and down with the Ca content (0.5 to 2.0%). B. J. C. van der Hoeven

**The  $\gamma$  radiation emitted in the disintegration of  $\text{Be}^8$  by  $\alpha$  rays.** Simone Franchetti. *Nuovo cimento* 12, 516-21 (1935).—Increasing the range of the bombarding  $\alpha$ -rays from 1.4 to 3.9 cm. air produces no measurable change in the absorption coefficient of the  $\gamma$  rays emitted. Conclusion. The  $\gamma$ -rays are emitted by  $\text{C}^{12}$  formed in the reaction  $\text{Be}^8 + \text{He}^4 = \text{C}^{12} + \text{e}^-$ . J. B. A.

**$\text{K}^{44}$  and the radioactivity of potassium.** H. J. Walke. *Nature* 136, 755 (1935).—Bittie has suggested that the radioactivity of K is due to  $\text{K}^{44}$  (C A 29, 7705). Isotopes containing the most neutrons are the least stable, and have the shortest half-lives. The period of  $\text{K}^{44}$  is 16 hrs., that of  $\text{K}^{46}$  would be shorter. Evidently this isotope cannot be the source of the radioactivity of K. The isotope  $\text{K}^{44}$ , proposed as the source of the radioactivity of K by Newman and Walke (C A 29, 2437, 5734) and by Klempner (C A 29, 2484) has been detected by Nier (C A 29, 6501) and by Brewer (C A 29, 7797) in "normal" K. Nier stated that  $\text{K}^{44}$  and  $\text{K}^{46}$ , if present at all, had an abundance less than 1 in 10,000. This evidence confirms W.'s selection of  $\text{K}^{44}$  as the source of the radioactivity of K. G. M. P.

**Radioactivity of samarium.** H. J. Taylor. *Nature* 136, 719 (1935), cf. C A 29, 5731.  $\alpha$ -Particles from Sm were found to have a range of  $1.13 \pm 0.02$  cm. in air. About 1% of the tracks observed had longer ranges, up to 7.5 cm. These long-range particles are not  $\alpha$ -particles, but are probably protons. G. M. P.

**Induced radioactivity by bombarding magnesium with  $\alpha$ -particles.** C. D. Ellis and W. J. Henderson. *Nature* 136, 755 (1935). When Mg is bombarded by  $\alpha$ -particles, the chief product has a period of 137 sec.,  $^{24}\text{Mg}^{24} + \text{He}^4 = ^{24}\text{Al}^{24} + \text{H}^1$ ,  $^{24}\text{Mg}^{24} + \text{He}^4 = ^{24}\text{Si}^{24} + \text{e}^-$ . Two other reactions occur to small extents,  $^{24}\text{Mg}^{24} + \text{He}^4 = ^{24}\text{Al}^{24} + \text{H}^1$ ,  $^{24}\text{Mg}^{24} + \text{He}^4 = ^{24}\text{Si}^{24} + \text{e}^-$ , with a period of 11 min.;  $^{24}\text{Mg}^{24} + \text{He}^4 = ^{24}\text{Al}^{24} + \text{e}^-$ ,  $^{24}\text{Mg}^{24} + \text{He}^4 = ^{24}\text{Si}^{24} + \text{e}^-$ , with a period of 6.7 min.  $^{24}\text{Mg}^{24}$  has a strong resonance level for  $\alpha$ -particles of less than  $5.4 \times 10^6$  v., and either  $\text{Mg}^{24}$  or  $\text{Mg}^{26}$ , or both, have a resonance level for  $\alpha$ -particles of  $5.4$  to  $6.1 \times 10^6$  v. By using  $\alpha$ -particles of energies up to  $6.6 \times 10^6$  v. to bombard a thick layer of Mg, the cross

sections for proton emission from  $Mg^{24}$ ,  $Mg^{23}$  and  $Mg^{26}$  have the ratio 1 300 10

**Energy of disintegration of radiophosphorus** C D Ellis and W J Henderson *Proc Roy Soc (London)* A152, 714-23(1935)—The max energy of positrons emitted from radio P( $P^{30}$ ) was detd by measuring the end point of the absorption curve in Cu. The value obtained  $2.9 \pm 0.1 \times 10^6$  agrees well with previous detns. No  $\gamma$ -rays accompanied the disintegration other than annihilation radiation of the positrons. The energy value  $2.9 \times 10^6$  v detns the difference in energy of the ground states of  $P^{30}$  and  $Si^{31}$ . This value combined with other data shows difference in mass to be the same between  $Al^{27}$  and  $Si^{28}$  when calcd from  $Al^{27} + {}^1_1H^1 \rightarrow {}^{28}_{12}Si + {}^1_0e^+$  or  $Al^{27} + {}^1_1H^1 \rightarrow {}^{28}_{13}Al + {}^1_0e^+$  and  $Al^{27} + {}^1_1H^1 \rightarrow {}^{28}_{14}Si + {}^1_0e^+$

**Isotope abundance in platinum** B Venkatesachar and L Sibaiya *Proc Indian Acad Sci* 2A, 101 3(1935), cf C A 29, 6129—The relative abundance of the Pt isotopes is estd from the structure of the pattern of the Pt band at 3408 13 Å. The values for mass nos 196, 195, 194 and 192 are 16, 13, 10 and 2, resp. This assumes a pos shift and is not consistent with the suggestion of a neg shift. The computed at wt is approx 195

**The escape of radon from cells which contain radium** J M A Hoflake and A E Korvecze *Rec trav chim.* 54, 768-78(1935)—Ra, surrounded by Ag gauze and by a thin layer of paraffin ( $15 \times 8 \times 2$  mm), is used in prep. an aq soln of Rn, which is used medicinally. The escape of Rn from this cell was found to be irregular, and to depend on the temp

**The effect of alteration on the lead-uranium ratio and the calculated age of Wilberforce, Ontario, uraninite** Chester M Alter and Egbert M Kipp *Science* 82, 464-5 (1935)—Sep detns of Pb, U and Th are reported for the outer and middle layers and the core of a crystal of uraninite which was altered to some extent. In the alteration of uraninite, Pb is lost less rapidly than U and Th. To calc accurately the age of a mineral by the Pb method, the analysis must be made on fresh, unaltered specimens. Alteration causes the calcd age to be too high. The Th/U ratio may vary in different zones of an unaltered single crystal of uraninite, owing to a change in the concn of these elements in the surrounding medium, during the growth of the crystal. Disregarding the outer layer, an age of  $1.42 \times 10^8$  years was found for the specimen. A method is suggested for calcd the relative Pb producing power of U and Th from the analytical data on sections of single crystals of minerals in which the Th/U ratio varies, owing to some other process than alteration

**Radioactivity of ferromanganese formations in seas and lakes of the U S S R** L M Kurbatov *Nature* 136, 871(1935)—Ferromanganese concretions from the bottoms of 9 lakes and seas were found to contain  $1.2-31.8 \times 10^{-10}$  Ra. Th is absent. The activity is more concd in the surface of the concretions

**Radioactive minerals** H Buttgenbach *Rev universelle mines* 11, 449-58(1935)—Radioactive minerals contain Th or U, which disintegrate spontaneously into a series of products of which Ms-Th and Ra are the most important industrially. Th is produced mainly from monazite sands, U from pitchblende, phosphates from Portugal and vanadates from Colorado, and lately especially from ores from the Congo. Deposits, modes of treatment and measurement of radioactivity are described

**Emanation content of soil air and tectonics of subsoil** H Israel Kohler and F Becker *Naturwissenschaften* 23, 818(1935)—The emanation content of the soil is derived from a special form of the Graham-Fick diffusion equation. Three cases are distinguished: homogeneous soil, active layers and active cracks. From measurements at Naumburg an approx figure of 0.65 sq cm per sec is derived for the diffusion coeff. of Rn in soil air

**The radiative collision between fast charged particles**

C. Møller *Proc Roy Soc (London)* A152, 481-96 (1935)—A relativistically invariant correspondence method generalizing a previous method for treating non-radiative collisions is applied to the problem of the radiative collision between two particles. In the limiting case, where one of the particles becomes infinitely heavy, the formula for the differential cross section goes over into that of Bethe and Heitler. The same results are derived by using the quantum electrodynamics of Heisenberg, Pauli and Dirac

**The collisions of very fast heavy particles** Werner Braunkopf *Z Physik* 96, 600-6(1935)—In view of the suggestion that the primary penetrating radiation may consist of heavy particles of high energy, an investigation is made of the collisions of heavy particles with free electrons at rest, by an approx. method appropriate to particles of infinite mass and applicable to protons up to  $10^{11}$  v. The cross section for collisions is obtained from the Rutherford scattering formula by a Lorentz transformation

**Intensity of dispersive radiation in x-ray irradiation** Otto Vaupel *Arch Eisenhüttenw.* 9, 213-14(1935)—In order to be able to employ and lay out satisfactory protective measures against x-ray radiation the magnitude of the dispersion of rays in the arrangement used must be known exactly, especially in the now frequent use in industrial operations. Expts are described which were made to det. the required dose of x-ray radiation and the dispersion in a definite arrangement, and a formula was developed which permits a quick calcd. of the dispersive radiation at any place surrounding the source of radiation

**Occurrence of the reversed absorption edges of the long wave lengths of x-rays** M Bačkovsky and V Dolejšek *Nature* 136, 836-7(1935)

**Excitation potential of the x-ray satellites in the L series** D Coster, H H Kuipers and W J. Huizinga *Physica* 2, 870-83(1935)—The doubly ionized state  $L_{II} M_{IV}$ , responsible for the weak satellites on the short-wave side of the  $L_{\alpha 1}$  line is due to radiationless transitions  $L_{II} L_{III}$  with ejection of an M electron (Auger process). The x-ray spectrum of Cb (41),  $L_{\alpha 1}$  line, was studied. The excitation function of the satellites was different from that of the parent line. A value of 2700 v, the excitation level of  $L_{II}$  is required to produce the satellites. Below 2700 v traces of satellites are left which may be due to a direct double ionization process ( $L_{II} M_V$ ).

**The absorption of monochromatic x-ray beams, of wave length in the region 50 to 20 X units, in lead, tin, copper and iron** John Read *Proc Roy Soc (London)* A152, 402-17(1935), cf Read and Lauritsen, C A 28, 3299—Filtered x radiation, reflected from a NaCl crystal and limited by slits to a beam width of 5 X U, was passed through two ionization chambers between which the absorber could be inserted. A graphic relation of the absorption coeff.,  $\mu$ , to the wave length was obtained for Pb accurate to 2%. The calcd photoelec coeffs.,  $\tau$ , are 7-20% higher than those obtained from Gray's law (cf C A 25, 2049), they may be related to Allen's value at 102 X U (cf C A 20, 1942) by  $\tau (\text{cm}^{-1}) = 0.01377 \lambda + 0.0003326 \lambda^2 + 0.00003474 \lambda^3 (\lambda \text{ in X U})$ . The relation  $\tau \propto Z^4$  holds closely for Pb, Cu and Fe, but for Sn it is more nearly  $\propto Z^{3.8}$ . The observed  $\mu$ 's agree closely with those calcd. from the curves of Hulme, McDougall, Buckingham and Fowler (cf C A 29, 3905), except for Pb for  $\lambda < 20$  X U, for which the observed are high

**Effect of pressure and of other gases on absorption of chromyl chloride** Michael Kantzer *Compt. rend.* 201, 1020-1(1935); cf C A 28, 3303—Passing the light exd. through the same amt. of  $\text{CrO}_2\text{Cl}_2$  but at different pressures the lines observed fall into the same 3 series, A, B and C, as pressure increases absorption increases in series A, but decreases in B and C, more rapidly in C. Addn. of  $\text{H}_2$  increases the absorption, by about 50% for addn. of an equal no. of mols. Other gases, e.g.,  $\text{N}_2$ ,  $\text{CO}_2$ ,  $\text{SO}_2$  and  $\text{SF}_6$ , show similar effects, decreasing

as the density increases until with  $\text{CO}_2$  it is very slight. SF<sub>6</sub> has almost the same effect as increasing the density of  $\text{CrO}_2\text{Cl}_2$ . C. A. Silberrad

"Extra" electron diffraction rings L. H. Germer *Nature* 136, 832 (1935) — Extra electron diffraction rings are due to surface contamination, not to diffraction from two-dimensional gratings made up of the atoms in the crystallographic faces through which electrons might leave the foil ("exit faces"). Cf. Litch and Quarrell, *C. A.* 29, 2445; Litch, Quarrell and Wilman, *C. A.* 30, 17.

"Extra" electron diffraction rings G. I. Litch *Nature* 136, 832 (1935) — Cf. preceding abstract. — The exptl. data, the ring radii attributed to scattering from a (110) exit face, and the values calculated by Litch *et al.* and by Germer, are compared. G. M. P.

Absorption coefficients and hydrogen line intensities Donald H. Menzel and Chaim L. Pekeris *Monthly Notices Roy. Astron. Soc.* 96, 77 (1935) — Theoretical C. C. Kiess

Fine structure of  $D_\alpha$  with increased resolution Robt. C. Williams and R. C. Gibbs *Phys. Rev.* 48, 971 (1935) — The first member of the Balmer series of  $D_\alpha$  ( $D_\alpha$ ) is a doublet with an interval of 0.134  $\text{cm}^{-1}$  between centers. This deviates considerably from the theoretical interval of 0.108  $\text{cm}^{-1}$ . The Shane and Spedding value of 0.142 (C. A. 29, 2367) is discussed. G. M. P.

The fine structure of the metastable levels of nitrogen R. Stejneger *Physik. Z. Supplement* 8, 352 (1935) — Spectrograms of the CO emission with a small admixt. of N<sub>2</sub> obtained for other purposes, led to the disclosure and measurement of the sept. of the lines 1742.740 and 1745.240 Å. The values 2.16 or 2.13  $\text{cm}^{-1}$  were obtained for the magnitude of the sept. The sept. of the metastable levels of N predicted by Compton and Boyce (C. A. 23, 2654) is considered to be proved. A term diagram is given. Allen S. Smith

The nuclear spin of iodine II Fine structure in the  $\alpha$  spectrum and a fine structure perturbation effect S. Tolansky *Proc. Roy. Soc. (London)* A152, 663 (1935); *C. A.* 29, 5912a — An analysis of the fine structure of 13 of the class of 1 Å lines ( $\lambda\lambda$  6300–4740), emitted by a water-cooled hollow-cathode discharge and measured with a silvered Fabry-Perot interferometer, confirm the nuclear spin value (5/2) previously found from the first spark spectrum. The  $6s^2P_{1/2}$  term is found to be perturbed by a term with  $J = 3/2$ , causing a breakdown in the interval rule, the interval factor for this term is 2.17 times that of  $6s^2P_{3/2}$ , but is exceeded by those of some of the  $5p^2P$  terms. This shows that the  $p$  electrons make important contributions to the fine structure coupling factors even in the presence of an  $s$  electron. M. M.

Hyperfine structure and the gross structure analysis of the spectrum of doubly ionized antimony J. S. Radami *Nature* 136, 826 (1935) — The gross structure analysis of Sb III (C. A. 27, 1423) is modified, in order to obtain satisfactory agreement with Coulomb's formula for nuclear magnetic moments (C. A. 27, 3139). G. M. P.

A determination of the profile of the calcium line  $\lambda$  4227 in the solar spectrum, using a prism spectrograph and monochromator, R. O. Redman *Monthly Notices Roy. Astron. Soc.* 95, 742 (1935) — It was found from measurements on high-dispersion prism spectrograms of sunlight that the central intensity of the Ca I line at 4227 Å. is 26% of the adjacent continuous spectrum. C. C. Kiess

Spark spectra of cadmium, Raymond Kernal and Antoine Saunier, *Compt. rend.* 201, 1115–16 (1935) — By means of the electrodeless discharge through Cd vapor at about 250°–300° or revised lengths of old lines in the Cd III and IV have been measured between  $\lambda\lambda$  4951 and 3468, with an accuracy of  $\pm 0.05$  Å. for lines of  $\lambda$  less than 4500 Å., and  $\pm 0.1$  for those above. C. A. S.

Relative  $f$ -values for lines of Fe I from electric furnace absorption spectra Robert R. King and Arthur S. King, *Astrophys. J.* 82, 877–95 (1935) — The total absorptions of lines of Fe I in the region 3400 to 4400 Å. were measured

microphotometrically on spectrograms for which the electric furnace at 2100° was the source. From these measurements the relative  $f$ -values of the lines, i. e., their oscillator strengths, were then derived from the curve of growth of the lines which expresses the functional relation between the total absorption (equivalently width) of a line and the no. of atoms active in absorbing it. C. C. Kiess

The variable spectrum of  $\gamma$  Cassiopeiae Wm. J. S. Lockyer *Monthly Notices Roy. Astron. Soc.* 96, 2–4 (1935) — The spectrum of  $\gamma$  Cass. already known as variable, has recently displayed the sudden appearance of a strong absorption line nearly coincident in position with H $\delta$ . It is probably due to the He I line at 5885.6 Å. C. C. Kiess

The energy distribution in the continuous spectrum of the sun G. I. W. Mulders *Astrophys. J.* 132, 44 (1935) — The relation between Rowland's intensities of lines in the solar spectrum and their equivalent breadths (C. A. 29, 7849) has been used to estimate the total energy that has disappeared from the sun's continuous spectrum as a function of the wave length. The new results are in good agreement with those found by Abbot with the bolometer and are found to differ notably from the black-body radiation. From the energy distribution the coeff. of absorption is determined as a function of the wave length. C. C. Kiess

Bolometric measurement of line contours in the infrared solar spectrum A. Dahme *Z. Astrophys.* 11, 93–7 (1935) — A quantitative evaluation of the total absorption and the no. of atoms over 1 sq. cm. of the photosphere has been carried out for the H lines 5, 6, 7, 8 of the Paschen series, and the Ca II lines at 8498, 8542, 8642 Å., as observed bolometrically in the solar spectrum by Abbot (C. A. 27, 18) and Green (C. A. 23, 1057). The results are in good accord with those obtained from photographic observations by others and, therefore, justify the use of bolometric observations for this problem in those spectral regions not accessible photographically. C. C. Kiess

Fluorescence phenomena and central intensities in Fraunhofer lines A. Pannekoek *Monthly Notices Roy. Astron. Soc.* 95, 725 (1935) — It is shown theoretically that the fluorescence light, superposed on a Fraunhofer line, has the character of an emission line. But owing to lack of resolving power in most spectrographic app. this light is diffused into a general moderate brightening of the dark central parts of the absorption lines so that their central intensities will be greater than zero. C. C. K.

Observations of intensity with a spectrohelioscope R. v. d. Wooley and H. W. Newton *Monthly Notices Roy. Astron. Soc.* 96, 5 (1935) — An auxiliary app. attached to the spectrohelioscope at Greenwich is described with which it is possible to measure the light intensity of the Pöschel and references of the sun with reference to the intensity of the solar disk outside the lines  $H_\alpha$  and  $H_\beta$ . In particular the profile of  $H_\alpha$  has been determined without photographic plotting, the result being in close agreement with that obtained photographically by Fläckner (C. A. 29, 3239). C. C. Kiess

The spectrum of Nova Herculis  $\lambda\lambda$  5150 6550 Å. Paul W. Merrill *Astrophys. J.* 82, 413 (1935) — In the visual region of the spectrum of Nova Herculis absorption lines of Na, Ba II, O I, Si II, Fe II, Ti II, Sc II, Cr II, Y II and He I were measured, and the emission lines of Fe II, O I, Si II, Na I and H. These lines may be divided into several distinct groups according to the displacements from their normal positions. C. C. Kiess

Ionic dispersion in the extreme infrared, C. Hawley Cartwright *Phys. Rev.* 49, 101 (1935) G. M. P.

Dissociation of diatomic molecules in the stars abundant in hydrogen Yoshino Umu *Japan. J. Astron. Geophys.* 13, 21–42 (1935) — Theoretical C. C. Kiess

The spectra of van der Waals molecules, W. Finkelburg *Z. Physik* 96, 199–713 (1935) — A theoretical discussion is given of the properties of "van der Waals" or "polarization" molecules and their spectra, special emphasis being laid on the differences between their behavior and that of valence-bound molecules. In considering transition-

probabilities for van der Waals molecules the Franck-Condon principle must be applied in its quantum-mech form. Line resonance-fluorescence and the question of excited van der Waals states in liquids are discussed. B. S.

The spectrum of the cadmium van der Waals molecule, Cd<sub>2</sub>. W. Finkelburg. *Z. Physik* 96, 714-19 (1935).—A theoretical interpretation of the exptl data for the Cd mol spectrum. It is based on the results of the preceding paper. B. Swirles.

Aspects of gross intensities in electronic bands with special reference to C<sub>2</sub> (Swan) and N<sub>2</sub> (second positive) systems. N. R. Tawde. *Proc. Indian Acad. Sci.* 2A, 67-81 (1935).—The development of the subject of spectral intensity in relation to gross intensity of bands has been reviewed. The transition probabilities, the temps, and the centers of intensity have been discussed in relation to each other. An attempt has been made to explain unusual intensity features of bands excited by different sources particularly in A. The Swan bands of C<sub>2</sub> and N<sub>2</sub> have been treated with reference to conditions of excitation. James H. Hibben.

The rotational structure of the band system (b') of the nitrogen molecule in the Schumann region. V. M. Chulanovskii. *Compt. rend. acad. sci. U. R. S. S.* [N. S.], 3, 155-6 (1935) (in German).—Analysis of  $\nu'$  progression (from  $\nu' = 0$ ) shows only P and R branches and shows that the upper state is not <sup>11</sup> but <sup>12</sup>. B' is calcd to be 1.144. Extrapolation of B' shows that the lower level of the system (b') is the normal state of N<sub>2</sub>. Four bands of a  $\nu' = 1, \nu'$  progression ( $\nu' = 18, 19, 20, 21$ ) were observed. B' is calcd to be 1.141. Conclusion.—The band systems of Watson and Koontz (C. A. 28, 5757<sup>2</sup>) are  $\nu'$  progressions with  $\nu' = 2$  and 3.  $\omega_e = 754.42 \text{ cm}^{-1}$  and  $x_e \omega_e = 4.16 \text{ cm}^{-1}$ . A. B. F. D.

Absorption bands of gaseous HI. D. E. Kirkpatrick. *Phys. Rev.* 49, 104 (1936).—The centers of the  $1 \leftarrow 0$  and  $2 \leftarrow 0$  absorption bands of HI lie at  $2260 \pm 30$  and  $4416 \pm 75 \text{ cm}^{-1}$ , resp. Cf. Czerny (C. A. 21, 3553), Salant and Sandow (C. A. 25, 3568), and Nielsen and Nielsen (C. A. 29, 4672<sup>2</sup>). G. M. P.

Absorption spectra of the vapors of the monoxides of copper, iron, nickel and cobalt, and the determination of their heats of sublimation. Hrishikesh Trivedi. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 5, 27-33 (1935).—FeO and CuO show 1 region of continuous absorption, while NiO and CoO show 2. The wave lengths of the beginnings of absorption are: FeO 2500, NiO 3270, 2380, CoO 2750, 2100, and CuO 2410 Å. The differences between the 2 regions of absorption for NiO and CoO were approx 32 kg.-cal, which may be due to disson. of the oxides into the metal atom and either O(<sup>1</sup>P) or O(<sup>1</sup>D). The heats of vaporization were calcd to be: FeO 97.5, CoO 101.5, NiO 111.5 kg.-cal. C. E. P. Jeffreys.

Interference spectroscopic examination of the gold hydride band spectrum in search of the isotope effect due to the suspected gold isotopes. Sunao Imamishi. *Sci. Papers Inst. Phys. Chem. Research (Tokyo)* 28, 129-34 (1935); cf. C. A. 29, 7793<sup>2</sup>.—The high resolving power afforded by a reflection echelon was applied to the rotational lines of several bands belonging to the <sup>12</sup> → <sup>12</sup> system of AuH in an effort to find the fainter lines due to the theoretical isotope Au<sup>199</sup>, the existence of which is inferred from the at. wt. 197.21 of Au. No fainter lines were found, although the precision of the expt. was sufficient to detect them if their intensities conformed to the abundance ratio 1.8 derived for the isotopes from the adopted at. wt. The neg. results of the expts. confirm other exptl. evidence on the singleness of the Au atom and cast doubt on the reality of the decimal part of the at. wt. C. C. Kiess.

Rotational analysis of the S<sub>2</sub> bands. E. W. van Dyk and A. J. Lamers. *Physica* 2, 785-6 (1935).—New spectrograms of the 4th order of bands of S<sub>2</sub> (0.6 Å per mm dispersion) indicate a sym. wave function like that of O<sub>2</sub>. The B values obtained agree better with Morse's rule (C. A. 24, 1020) than with Badger's (C. A. 29, 2996<sup>2</sup>). B. J. C. van der Hoeven.

Emission spectrum of tellurium oxide. Choong Shim-

Piaw. *Compt. rend.* 201, 1181-3 (1935); cf. C. A. 30, 380<sup>2</sup>.—The spectrum of TeO between 3190 and 3820 Å (that for greater λ is masked by that of Te) consists of 30 bands, and closely resembles those of SO and SeO. Of the bands 27 have simple and 3 double heads. The simple bands and the component of smaller λ of the double-headed are given by  $\nu = 29499.0 + [372.9 (\nu' + 1/2) - 5.36 (\nu' + 1/2)^2] - [796.1 (\nu'' + 1/2) - 3.40 (\nu'' + 1/2)^2]$ , whence the fundamental frequencies of the TeO mol in the normal and excited states are deduced as 796.1 and 372.9, resp., and the energy of disson. in the normal state as 5.70 v. C. A. Silberrad.

The absorption spectra of the vapors of the monosulfides of iron, nickel, cobalt and copper, and the determination of their heats of sublimation. Hrishikesh Trivedi. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 5, 34-40 (1935).—The absorption spectra of FeS, NiS, CoS and CuS show a continuous region, a region of retransmission, and a second continuum. The wave lengths of the onset of the continua are: FeS 3100, 2325, CoS 3190, 2400, NiS 2810, 2170; CuS 3400, 2400. The long wave continuum is ascribed to the process  $MS + h\nu_1 = M + S(^1P)$  and the second to  $MS + h\nu_2 = M + S(^1D)$ . The heats of vaporization were calcd to be: FeS 79.3; NiS 69.3; CoS 81.7; CuS 70.25 kg.-cal. The difference  $h\nu_1 - h\nu_2 = 1.31 \text{ v.}$  is attributed to the difference <sup>1</sup>P - <sup>1</sup>D of S. No bands characteristic of the transition group were obtained, but it is possible this failure was due to the obscuring of the weak bands by the strong continuous radiation coming from the heated furnace. C. E. P. Jeffreys.

Interpretation of the vibrational spectrum of organic molecules by means of the isotope effect. E. Bartholome and H. Sachse. *Z. physik. Chem.* B30, 40-52 (1935).—Infrared absorption spectra were obtained for MeOH and MeOD. Comparison of all available data for MeOH shows that the results are discordant. Qual. considerations of the isotopic spectrum help in detg. the fundamental frequencies. Some of the vibrational frequencies are displaced only slightly from the corresponding MeOH frequencies and in these cases the D atom takes no part. Where the D atom also vibrates the differences are much greater. In this way, the fundamental frequencies of MeOH are established. Similar considerations on the isotopic spectra of C<sub>2</sub>H<sub>4</sub> clear up some disputed points for the bands at 827 and 1400 cm<sup>-1</sup>. G. M. M.

Study of atmospheric ozone by visual spectroscopy. J. Gauzit. *Ann. Phys.* [11], 4, 450-532 (1935).—The construction and standardization of a high-precision visual spectrophotometer are described. The app. is used to det. the atm. O<sub>3</sub> and more generally the spectrophotometry of the sun, sky and moon. Two methods are employed, viz., direct vision of the sun and direct vision of the blue sky. A table is given showing 156 detns. between Jan., 1933 and March, 1934. A great variation in the amt. of O<sub>3</sub> was observed having a max. during March and April and a min. during Aug. and Sept. Five night detns. are given obtained by spectroscopy of the moon. Direct observation of the sun at the horizon shows the inequality of distribution of O<sub>3</sub> in the same horizontal layer. The av. altitude of the "thin layer" of O<sub>3</sub> was found to be 33 km. The values varied from 20 to 50 km, which indicates that the altitude increases with its thickness. The spectroscopy of the sun shows clearly the insufficiency of the preceding hypothesis and gives an approx. value for the quantity of O<sub>3</sub> in the troposphere. Sixty-six references. W. George Parks.

The rotation vibration spectrum of acetylene in the photographic infrared. Costa W. Funke and Gerhard Herzberg. *Phys. Rev.* 49, 100 (1936).—Nine new absorption bands are reported for C<sub>2</sub>H<sub>2</sub>. The bands are interpreted according to the plan of Herzberg and Sparks (C. A. 29, 1010<sup>2</sup>). G. M. P.

Near ultraviolet absorption bands of SO<sub>2</sub>. R. K. Asundi and R. Samuel. *Proc. Indian Acad. Sci.* 2A, 30-45 (1935).—The absorption bands of SO<sub>2</sub> have been measured and analyzed for the near ultraviolet. Preference is given to an arrangement of SO<sub>2</sub> bands by which the

sym. valence vibration of excited  $\text{SO}_2$  gains almost exactly the same values as that of excited  $\text{SO}$ . J. H. H.

Temperature dependence of the methyl iodide absorption spectrum in the quartz ultraviolet. A. Henrici and H. Griener. *Z. physik. Chem.* B30, 1-59 (1935).—The ultraviolet absorption spectrum of MeI was photographed between pressures of 240 and  $10^{-4}$  mm Hg and a temp. between  $400^\circ$  and room temp. Flowing gas was used in an absorption cell 20 cm long. The quartz spectrograph had a dispersion of 4.4 Å. per mm at 1945 Å. A large no. of new bands were found and analyzed. Fundamental vibrational frequencies are 525, 580 and 1237  $\text{cm}^{-1}$  with corresponding frequencies of 508, 780, 1090 and 1230  $\text{cm}^{-1}$  in the excited electronic state. The most intense bands are considerably broadened by increasing pressure but the apparent broadening of some other bands is due to unresolved rotational structure. An addnl set of bands has fundamental frequencies of 495, 805 and 985  $\text{cm}^{-1}$  and they probably belong to a new electronic band system since they are very sensitive to temp.

G. M. Murphy  
The infrared absorption spectra of water and alcohols in nonpolar solvents. E. L. Kansey and J. W. Ellis. *Phys. Rev.* 49, 105 (1936).—Absorption spectra are given for  $\text{H}_2\text{O}$ ,  $\text{H}_2\text{O} + 300 \text{ CS}_2$ , atm  $\text{H}_2\text{O}$  vapor, MeOH vapor, MeOH liquid and 1MeOH + 7.9  $\text{CCl}_4$  in the region 0.8-2.4  $\mu$ .

G. M. P.  
The absorption spectra of certain trivalent halides dissolved in ether and their dissociation products. Sechi Katō and Fujiko Someno. *Sci. Papers Inst. Phys. Chem. Research* (Tokyo) 28, 95-111 (1935).—The absorption spectra of solns. in ether of the trivalent halides of As, Sb, Bi, Fe and Ti were observed in the region from 2000 Å in the ultraviolet to 7000 Å in the red. Each of the salts was also volatilized in a vacuum quartz tube and its absorption observed to det. the effect of the solvent. Another set of observations was made on the Raman spectra of ether solns. of  $\text{AsCl}_3$ ,  $\text{AsBr}_3$  and  $\text{SbCl}_3$ . From the wave lengths of the max. of the absorption bands and the beginnings of their long-wave edges the energies available for the photochem. dissociation of the mols. were derived. It was found that for the Fe salts only 1 halogen atom is sep'd. from the mol. by absorption of radiant energy, whereas for the Ti, As, Sb and Bi salts, also 2 and 3 halogen atoms may be sep'd. from the comp'd. The effect of the solvent, indicated by a shift of the absorption toward shorter wave lengths, is to deform the mols. without, however, exciting them from the deepest energy state of the normal mol.

C. C. Kiess  
Remarks on the investigation of Bullroth—"Absorption of substituted benzenes VI." Erwin Steurer. *Z. physik. Chem.* B30, 157-8 (1935), cf. C. A. 29, 6142.  
Absorption spectra were measured for *p*-xylene, 1,2,4- and 1,3,5-trimethylbenzene and the results compared with those of B.

G. M. Murphy  
The ultraviolet absorption spectra of some complex aromatic hydrocarbons. I. W. V. Mayneord and E. M. F. Roe. *Proc. Roy. Soc. (London)* A152, 299-324 (1935).—The ultraviolet absorption spectra in the region 2000-4000 Å. are given for the alc. solns. of benzene (I), neoergosta-triene (II), -tetraene (III), and -pentaene (IV), naphthalene (V), 4,5-benzobicyclopentadiene (VI), dimeride of 1-isopropynaphthalene, *cis*- and *trans*-9,10-dimethyl-9,10-dihydro-1,2,5,6-dibenzanthracene (VII, VIII), anthracene, 1,2-benzanthracene and its 6-Me-, 7-Me-, 6,7-di-Me-, 6,7-cyclopentene-, 5,6-cyclopentene- and 10-isopropyl (IX) derivs, methylcholanthrene (X), phenanthrene (XI), 1,2-cyclopentene-phenanthrene (XII), 2,1'-naphtha-1,2'-fluorene, 5-methyl-8-isopropyl-2,1'-naphtha-1,2'-fluorene, chrysene, 1,2,5,6-dibenzanthracene and its 9,10-di-Me, and 9,10-dihydro (XIII) derivs, 1,2,5,6-dibenz-9,10-anthraquinone, pyrene (XIV), 2-methylpyrene (XV) and 1,2-benzopyrene and for picene in  $\text{CHCl}_3$ . Some of the comp'ds. are structurally related to the carcinogenic hydrocarbons. The curves of II, III and IV are similar to that of I but with suppressed detail. The effects of addn of various groups and the importance of the structure of the mol. as a whole in

detg. the form of the spectra (e. g., the curve of XIV is similar to that of XV but displaced toward the red) are noted. VII and VIII with the central ring sat'd. behave as though consisting of 2 fairly sep. portions and the curves are similar to those of V and VI, but XIII, although the central ring is also sat'd., is entirely different. The curves of IX and X are very similar, although X is strongly carcinogenic and IX inactive. The facts that the frequency differences between neighboring bands of XI alternate between 650 and 750  $\text{cm}^{-1}$  and that addn of a 5-membered ring (XII) emphasizes or depresses alternate bands suggests that the long-wave system of XI is double.

Janet E. Austin  
Origin of the wing accompanying the Rayleigh line in liquids. S. C. Sirkar. *Nature* 136, 759-60 (1935).—Cross and Vuks (C. A. 29, 2448<sup>5</sup>, 3913<sup>4</sup>, 5744<sup>4</sup>) have suggested that the "wings" which accompany the Rayleigh line in the Raman spectrum of certain org. liquids are due to lattice oscillations of quasicryst. groups in the liquid, and that only the portion closest to the Rayleigh line which increases in intensity with temp. is connected with mol. rotation. On dissolving benzene in cyclohexane (which gives only a feeble "wing"), the intensity of the portion of the wing close to the Rayleigh line diminishes; that at about 50  $\text{cm}^{-1}$  from the center of the line increases slightly, the total width of the "wing" is practically const. In benzene, the intensity of the portion of the "wing" nearest the Rayleigh line diminishes with rising temp. (C. A. 29, 6143<sup>5</sup>). These data contradict the Gross-Vuks hypothesis. The lines are not due to lattice oscillations, but may be due to oscillations of very small groups of mols. Groups of polar mols. would be more stable than groups of nonpolar mols.

G. M. P.  
The Raman spectra of light and heavy phosphine. Marc de Hemptinne and Jean-Marie Delfosse. *Bull. sci. acad. roy. Belg.* 21, 793-9 (1935).—The lines of  $\text{PH}_3$  and  $\text{PD}_3$  were photographed in the liquid and gaseous states. The observed frequencies of liquid  $\text{PD}_3$  are 1664, 807, 740  $\text{cm}^{-1}$ . The frequencies in the gas are about 20  $\text{cm}^{-1}$  greater. The frequencies of  $\text{PH}_3$  and  $\text{PD}_3$  are corrected for anharmonicity by a valence force calcn. Some of the force consts. of  $\text{PH}_3$  are calc'd.

A. B. F. D.  
Raman spectrum and benzene symmetry. K. W. F. Kohlrausch. *Naturwissenschaften* 23, 624-5 (1935).—A previous conclusion that the frequency of 1000 in the mono-, *m*-di- and sym. tri-substituted derivs. corresponds to the 992 frequency of  $\text{C}_6\text{H}_6$  and indicates trigonal symmetry of the latter is incorrect. Further work on the Raman spectrum of 250 derivs. indicates that the  $\text{C}_6$  ring has hexagonal symmetry  $D_{6h}$ , the mono-, *o*- and *m*-derivs.  $C_{2v}$ , the *p* derivs.  $C_{3v}$ . An investigation of the flat vibration possibilities of the  $\text{C}_6$  ring from the Trenkler mech. model (C. A. 29, 3562<sup>7</sup>) indicates 3 possible deformational vibrations, 6 valency vibrations. Two of them have trigonal symmetry; one corresponds to  $\omega = 1000$ . From the models the expected frequencies of the vibrations are calc'd. by the method of van den Bossche and Masnebeck (C. A. 29, 2409<sup>8</sup>) and found in fair agreement with observed values.

B. J. C. van der Hoeven  
Raman effect and organic chemistry. Raman spectra of ethylenic compounds of the general formula  $\text{CH}_2\text{:CHR}$ . Maurice Bourguet and Léon Piaux. *Bull. soc. chim.* [5], 2, 1958-69 (1935); cf. C. A. 26, 4232, 27, 26; 28, 93; 29, 5805<sup>9</sup>, 4507<sup>9</sup>.—The Raman spectra of propene (I), 1-butene, 1-heptene, 1-octene, 1-nonene, 3-phenyl-1-propene, vinyl bromide (II), allyl bromide, allyl alc., acrolein (III) and acrylic acid are given and compared with the spectra of comp'ds. of the same general formula already published (cf. C. A. 27, 3192<sup>9</sup>, 29, 2499<sup>9</sup>, 7936<sup>9</sup>, 7937<sup>9</sup>). There are 5 bands characteristic of the group  $\text{CH}_2\text{:CH}$  1295, 1417, 1642, 3004, 3083 (for 1-butene). The line 1642 corresponds to the double bond and is the same for all hydrocarbons of the form  $\text{CH}_2\text{:CHCH}_2\text{R}$  but varies from 1647 for I to 1598 for II. In general for  $\text{CH}_2\text{:CHR}$  the radicals Me, OH, COOR, etc. increase the frequency (strengthen the bond) but neg. radicals and atoms (COOH, Ph, CHO, Cl, Br) lower the frequency. The radicals  $\text{CH}_2\text{Br}$ ,  $\text{CH}_2\text{Cl}$  and  $\text{CH}_2\text{Ph}$  all lower the fre-

quency slightly. Conjugation of C-C or C=O with the double bond of CH<sub>2</sub>CHR also lowers the frequency. J. E. A.

The effect of imprisonment of resonance radiation in the decomposition of ammonia and deuterioammonia. H. W. Melville. *Proc Roy Soc (London)* A152, 325-41 (1935).—A reexam of the photosensitized decompn of NH<sub>3</sub>, ND<sub>3</sub>, PH<sub>3</sub>, and PD<sub>3</sub> under conditions where the reabsorption of the resonance radiation is negligible, has resulted in smaller velocity coeffs than previously reported. The disson is interpreted as being due to collisions between NH<sub>3</sub> (ND<sub>3</sub>) and metastable atoms, derived in turn from quenching collisions between <sup>3</sup>P<sub>1</sub> atoms and NH<sub>3</sub> (ND<sub>3</sub>). The former collisions are almost equally efficient for NH<sub>3</sub> and ND<sub>3</sub>. This mechanism also explains the abnormally high inhibitory effect of H<sub>2</sub>, which is 20-40 times as great as that computed from the relative radii of NH<sub>3</sub> and H<sub>2</sub> and ND<sub>3</sub> and D<sub>2</sub>. The more rapid decompn of NH<sub>3</sub> than of ND<sub>3</sub> is attributed to secondary reactions unconnected with Hg atom processes. This is confirmed by the fact that in the direct photodecompn NH<sub>3</sub> also reacts more quickly. Morris Muskat.

Free radicals in the photodissociation of gaseous metal alkyls. N. Prilezhayeva and A. Terenin. *Trans Faraday Soc* 31, 1483-7 (1935).—The method of detecting free radicals based on the disappearance of metal layers under their action was improved by detn of the thickness of the film by measurement of light transmitted through it with a photocell. The method was applied to the detection of alkyl radicals produced by illumination of Me<sub>2</sub>Co, Me<sub>2</sub>Hg and Et<sub>2</sub>Pb with ultraviolet light from a spark. By use of various electrodes and filters the range of most effective radiation for Me<sub>2</sub>Hg was found to be 2000-2100 Å. This is exactly the region of a diffuse band absorption (Terenin and Prilezhayeva, *cf. C. A.* 28, 5758). The assumption that the diffuse spectrum is due to a predissoc process liberating free Me radicals is confirmed. C. E. P. Jeffreys.

Asymmetric photochemical decomposition of complex salts. Ryutaro Tsuchida, Akira Nakamura and Masahisa Kobayashi. *J Chem Soc Japan* 56, 1335-8 (1935).—The optical rotatory power of K<sub>2</sub>[Co(ox)<sub>2</sub>] is -5725° (λ = 0.97 mμ) (ox = oxalate radical). On illumination by dextro circularly polarized light of 589 mμ, the aq soln of racemic K<sub>2</sub>[Co(ox)<sub>2</sub>] becomes levorotatory. The power increases with time, reaches a max and then decreases. T. Katsurai.

Fluorescence spectrochemistry of the chlorophyll pigments. I. C. Dhéré and Anne Raffy. *Bull soc chim biol* 17, 1385-7 (1935).—App and methods are described. Visible fluorescence spectra of α- and β-chlorophyll in solution. *Ibid* 1388-96.—The fluorescence spectra of α- and β-chlorophyll in various solvents were examd and the results compared with those of other workers. Infrared fluorescence of chlorophyll in living plants.

*Ibid* 1397-1408.—See *C. A.* 29, 4408<sup>2</sup>, 6273<sup>4</sup>. Fluorescence spectra of pheophorbides. *Ibid* 1409-13.—See *C. A.* 29, 4474<sup>1</sup>. L. E. Gilson.

The decomposition of ozone by alpha particles. P. C. Capron and R. Cloetens. *Bull soc chim Belg* 44, 441-66 (1935).—The decompn and formation of ozone at 25° have been investigated in the absence of Hg and org matter. In the range of intensity of radon of 5-35 millicuries, the decompn was proportional to the concn of ozone. The ratio of mols O<sub>3</sub> formed to pairs of ions produced is of the same order as that found by Lewis (*cf. C. A.* 27, 5249), but is const for a given intensity of radiation. This yield increases with the intensity of radiation, the reverse of which has been found in the radiochem decompn of H<sub>2</sub>S and NH<sub>3</sub>. R. E. DeLight.

The possibility of igniting chlorine detonating gas by high-velocity electrons. A. E. Mal'novskii and K. A. Skrumnikov. *Fizisk Zh Sovetskijunon* 8, 289-93 (1935).—The soln of this question was sought in expts with a Cl and H<sub>2</sub> mixt. which requires less energy for reaction than do the gas mixts previously used (*C. A.* 29, 3916<sup>1</sup>), the strength and concn of the electron current were not increased. The explosion mixt consisted of equal parts of Cl and H<sub>2</sub>; its pressure was varied from 7.5 to 750 mm. The app was that previously used, potentials in the Coolidge tube varied from 35 to 45 kv and the current intensities from 0.6 to 1.0 ma. In all cases without exception no explosion was observed. The electron stream in the explosion chamber attained a d of 5.83 × 10<sup>-1</sup> amp/sq mm. With certain assumptions the electron concn was calcd to be 0.62 × 10<sup>16</sup>/cc corresponding to an ion concn of about 10<sup>16</sup>. The ion concn in a flame with linear velocity of about 1 cm/sec is of the order of 10<sup>14</sup>, this indicates that the possibility of ignition can be detd by increasing the ion concn 10<sup>4</sup> times with the use of a c. d. of about 6 × 10<sup>-4</sup> amp/sq mm. A. S. S.

The stability of Lenard light centers in zinc sulfide. N. Richl. *Ann. Physik* 24, 536-42 (1935).—The sensitivity to α rays shows a definite loss with time, the percentage loss being greatest for strongest radiations. These centers are thermodynamically unstable. R. E. DeRight.

The ultraviolet radiation of chemical reactions. Otakar Viktorin. *Chem Listy* 29, 245-9 (1935).—Ultraviolet radiations accompany the oxidation of pyrogallol in air, pyrogallol by H<sub>2</sub>O<sub>2</sub>, EtOH by Cr<sub>2</sub>O<sub>3</sub>, glucose by KMnO<sub>4</sub>, Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> in an alk medium, the action of Br upon K oxalate, the formation of LiBr, and the neutralization of HNO<sub>3</sub> by NaOH. In a 10% NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> soln the anodic oxidation of Al yielded strong and almost quantitative ultraviolet radiations. Frank Marsh.

Bequerel cells (Athanasu). 4. Primary process of photochem and thermal decompn of azomethane (Patat). 2.

#### 4-ELECTROCHEMISTRY

COLIN G. WINK

Production of ferroalloys directly from minerals in the electric furnace. Alfred Salmony Karsten. *Quim e ind* 12, 231-5 (1935).—Some large com installations for the production of ferro-Si, ferro-Mn, ferro-Cr, ferro-Mo and ferro-W are briefly described. L. E. Gilson.

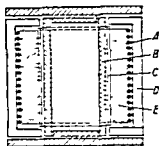
Heat balance in electric furnaces during production of aluminate slags. P. B. Golubkov. *Legkie Metali* 4, No. 2, 16-22 (1935).—Stones contg Ti are formed in the elec furnaces at the Dnieper Aluminum Plant (*cf. C. A.* 29, 4267<sup>7</sup>), because the charge, consisting of a CaO-Al<sub>2</sub>O<sub>3</sub> agglomerate, melts at the same or lower temp than the Al slags. This prevents superheating the slag sufficiently to dissolve the Ti compds. G suggests using raw Al<sub>2</sub>O<sub>3</sub> and CaO as charge and making slag of eutectic compn to prevent this stone formation. H. W. Rathmann.

The use of cast aluminum shields in large electric arc furnaces. A. v. Zierleider. *Aluminium* 17, 529-30

(1935).—The concentric shields of nonmagnetic material in the Miguet furnace, usually made of cast bronze, were successfully replaced by cast Al plates 2.5 m high and 4.6 m in diam., they carry up to 80,000 amp and weigh 5300 kg against 15,000 kg for the bronze shields. The Al shield has been in steady use since 1930. M. H.

Electric furnaces installed in the Pans Mint. Anon. *Electrician* 115, 9 (1935).—For the annealing of the com blanks 3 horizontal rotary furnaces have been installed. Each furnace absorbs 50 kw and is fed with three phase current. The temp of the furnaces is self regulated. Heating is by induction. The system is shown in the accompanying diagram. A is a solenoid coil fed by a c. In the center of this solenoid is a muffle B of magnetic iron. Near the muffle there is an envelope of nickel steel (C) and around the outside of the coil are a no. of sheet-iron cores (D), which complete the magnetic circuit.

E is lagging. The current flowing through the solenoid sets up an induced current in the envelope, which forms, in fact, a winding of one turn per cent. The envelope, therefore, heats up and transmits its heat to the



muffle until this latter reaches the point at which it loses its magnetism. The flux in the magnetic core is then reduced to the point at which the heat produced is exactly equal to that lost by radiation. C. G. F.

Comparison of methods of operating an aluminum electrolytic cell. Ya. A. Bernshon and G. I. Khviltzikh. *Lehne Metal* 4, No. 4, 19-23 (1935).—The output and energy consumption of an Al fused-cryolite cell operating under conditions of const. current, voltage, or power input ( $\rho$ ), resp., were compared. The output per cell per day and the elec. energy consumed per kg. of Al were. For  $I = \text{const.}$  149 kg., 19.4 kw-hr.; for  $V = \text{const.}$  118 kg., 17.5 kw-hr.; and for  $\rho = \text{const.}$  138 kg., 18.8 kw-hr. H. W. Rathmann.

The preparation of oxygen by electrolysis of baryta. Eric M. Stoddart. *Proc. Roy. Soc. (London)* A152, 271-7 (1935).—The electrolysis of baryta with Ni electrodes does not always give pure O at the anode. After a time H also appears at the anode, probably being due to the reaction  $2\text{OH} = \text{H}_2 + \text{O}$ . The liberation of H can be prevented by coating the electrode with a layer of the black oxide of Ni. Morris Muskat.

Investigating the solubility of solvents and the electro-deposition of iron from these solutions. R. Müller and L. Harant. *Berg und Hüttenmännisches Jahrbuch* 23, 93-102 (1935).—The practical possibilities of direct electrolytic production of Fe from the ore are discussed. Two principal cases are distinguished: (1) refining of raw Fe, whereby Fe is usually dissolved anodically and cathodically deposited in very pure form; (2) an electrolyte is obtained by direct action of solvents on the ore, and the pure Fe is deposited from this soln. by electrolysis with an insol. anode. Work of other authors is reviewed.

Cathode polarization during deposition of copper from sulfate solutions. O. Evin and A. Levin. *J. Gen. Chem. (U. S. S. R.)* 5, 1262-15 (1935).—Measurements were made of deposition potentials of Cu at 25° from solns. contg. various concns. of  $\text{CuSO}_4$  (0.3, 0.4, 0.5, 0.6, 0.7 and 1.0 N) in 2 concns. of  $\text{H}_2\text{SO}_4$ , 0.1 and 0.2 N, with c.d.s. of 1 ma. per sq. cm. and up. Gordon's equation  $E_p = E_0 + \frac{b}{a} \log(1 - ED)$  (C. A. 19, 3432), where  $E_p$  is polarization,  $E_0$ ,  $b$  and  $K$  are const. and  $D$  is c.d., is in fairly good agreement with exptl. results down to the point where  $E_p = E_0$ .  $E_0$  should not be considered here as expressing the low limit of polarization, but merely as an indicator of the low limit of applicability of this equation. In the interval  $D = 1$  to 10 ma. per sq. cm. polarization behaves irregularly, showing an increase in electrodepos. direction with rise in c.d., then again rising regularly, above 10 ma. in electrodepos. direction. The so-called "chemical" polarization  $E_c$  in Cu deposition follows more correctly Ertley-Gritz-Volmer's equation  $E_c = 1/(a + k_1 \log D)$  (C. A. 25, 1197), where  $a$  and  $k_1$  are const. and  $D$  is c.d. S. L. Madorsky.

Nickel and chromium plating, progress and present situation. M. Bailly. *Unne* 44, No. 46, 27; *Res. met.* 32, 494-500 (1935).—A brief review of present practices in different countries. M. Hartenbeum.

Alkaline plating baths containing ethanalamines. II. A study of baths containing triethanolamine for the direct nickel-plating of zinc. C. J. Brockman and J. P. Nowlen. *Trans. Electrochem. Soc.* 69, 11 pp. (preprint) (1935).—The following solns. examd. contained either just enough triethanolamine to give a clear soln. or they contained an excess of triethanolamine: (1) Ni NH<sub>4</sub> sulfate soln.; (2) Ni NH<sub>4</sub> sulfate-NiSO<sub>4</sub> soln.; (3) Ni NH<sub>4</sub> sulfate-boric acid soln.; (4) NiSO<sub>4</sub> soln.; (5) NiCl<sub>2</sub> soln.; (6) Ni NH<sub>4</sub> sulfate-NiCl<sub>2</sub> soln. The plating conditions of the 6 solns. were detd. at different temps., concns. and c.d.s. C.d. vs. potential measurements were made during the plating processes. Good deposits of bright Ni were obtained from the 3 sulfate baths. III. Cobalt plating from triethanolamine solutions. From solns. of CoSO<sub>4</sub> contg. triethanolamine it is possible to plate Co successfully on Cu, steel, Ni and Zn at c.d.s. ranging from 0.5 to 25 amp. sq. dm. at room temp. or slightly above. Cathode efficiencies were as high as 98%. The plates are slightly darker than the corresponding Ni plates. The bath was electrolyzed for 1 hr. at a c.d. of 2 amp./sq. dm. after which satisfactory deposits were obtained. The temp. rose from 21° to 24° during electrolysis. C. G. F.

Control of the quality of tin electroplating. Ya. L. Vertman. *Zashchita Lab* 4, 737-9 (1935).—The test is made with 5 cc. of HNO<sub>3</sub> (d. 1.4) in 15 cc. H<sub>2</sub>O by the procedure previously described for Zn electroplating (C. A. 29, 2453). Chas. Blanc.

Electrode dispersion of noble metals. Andr. Voet. *Trans. Faraday Soc.* 31, 1488-91 (1935). d. C. A. 29, 2978.—On electrolyzing with noble-metal electrodes at a high c.d. and a high voltage in alk., acid, and neutral solns. a disintegration of the anode is observed which results in, generally unstable, suspensions. Every electrode dispersion is accompanied by frequent pulsations of current, which were measured with an oscillograph. A min. concn. of electrolyte is necessary to obtain dispersion for every potential difference. The Wehnelt effect is not the cause of the dispersion but it may be explained as the result of the repulsion of a periodically formed oxide layer on the surface of the anode. C. E. P. Jeffreys.

The dependence of the electrochemical properties of an element on its state of aggregation. I. Research on the potential of gallium and gallium amalgam in gallium salt solutions. Otto Stelling. *Z. Elektrochem.* 41, 712-21 (1935).—Amalgam electrodes were prepd. by passing liquid Ga through a capillary tube into Hg in an atm. of H<sub>2</sub>. A slight coat of oxide which appeared in 10-20 hrs. did not affect the results. All the work was done in an atm. of H<sub>2</sub>. The potential of the Ga amalgam in contact with GaCl<sub>3</sub> came to equil. slowly at -0.887-0.888 v. In a soln. of 0.1 M Na<sub>2</sub>Co<sub>3</sub>, which was also 0.36 N with respect to NaOH, the amalgam electrode showed a potential of -1.512 and its potential changed markedly for any change in  $\rho$ . Addn. of 35.5% EtOH to a Ga amalgam electrode in 0.0077 M GaCl<sub>3</sub> and 0.54 N HCl raised the potential from -0.976 to -0.867. Addn. of NaCl to a similar system had little effect. Ga metal for electrodes was freed from oxide by passing it through a capillary tube, the Ga metal electrodes were kept in an atm. of H<sub>2</sub>. The potential of the Ga electrode in contact with GaCl<sub>3</sub> and HCl came to equil. quickly. However, 2 electrodes in the same soln. gave potentials which sometimes differed by as much as 10-20 mv. The potential of the Ga electrode in Ga salt soln. was near that of the Ga amalgam in Ga salt soln. The liquid Ga electrode showed a passivity effect. In contact with an alk. Na<sub>2</sub>Co<sub>3</sub> soln., a supercooled liquid Ga electrode showed a potential of about -1.4 v. vs. a potential of about 1.5 for the solid electrode. In acid soln. a similar though smaller effect was noticed.

F. H. Moser.

Electrolysis of zinc, nickel and cobalt bromides and iodides in aqueous-alcoholic solutions. Claude Charmentant. *Compt. rend.* 201, 1174-6 (1935), cf. C. A. 29, 5749\*.—With a similar arrangement and 0.5 M solns. of ZnBr<sub>2</sub>, Br is liberated at the anode and diffusing to the cathode forms HBr and acetaldehyde in amt. increasing with concn. of EtOH. With ZnI<sub>2</sub>, I is similarly



set free, but does not react further. The Zn deposited is adherent with low concn of EtOH, but is at least partially not adherent for concns of EtOH exceeding 450 g/l. Yield (max 97%) decreases with time and increased concn of LiOH. NiBr<sub>2</sub> and NiI<sub>2</sub> behave similarly, but with NiBr<sub>2</sub> less HBr and acetaldehyde are formed. As with NiCl<sub>2</sub>, NiO is deposited at the cathode with disengagement of H<sub>2</sub> when soln is basic, but if the soln is acid (0.01-0.025 N in HBr or HI) Ni is deposited. The yield increases with concn of EtOH, and is always greater with NiBr<sub>2</sub> than with NiI<sub>2</sub> (max 93 and 90%, resp.). With CoBr<sub>2</sub> and CoI<sub>2</sub> results are very similar save that no oxide is deposited and yield is slightly better (max. 99.5%).

C. A. Silberrad  
Relations among fundamental electrochemical quantities III. Itsuro Uehara. *J. Chem. Soc. Japan* 56, 1364-71 (1935). cf. *C. A.* 29, 6145<sup>7</sup>.—By consideration of a reversible cycle between nonmetallic substances such as F<sub>2</sub>, Cl<sub>2</sub>, Br<sub>2</sub>, I<sub>2</sub>, O and their ionic solns, the relative values of the standard electrode potential of the nonmetallic substance have been calculated.

T. Katsurai  
A contribution to the study of electroosmosis with various ceramic diaphragms. J. Věštek and A. Vašíček. *Chem. Listy* 29, 250-3, Collection Czechoslov. Chem. Communications 7, No. 10, 451-6 (1935), cf. *C. A.* 29, 2050<sup>4</sup>.

F. W. Marech  
Electric storage batteries in recent patents. L. Juma. *Revue électrique* 38, 335-48, 367-82 (1935).—Recent improvements in storage batteries to facilitate the chem. reactions, assembling and dismantling are reviewed and many improvements are described.

M. Hartenhein  
An electron tube and instrument for use with the glass electrode and a description of a rugged type of glass electrode. Lyle D. Goodhue. *Iowa State Coll. J. Sci.* 10, No. 1, 7-15 (1935).—The instrument is a simple resistance-coupled direct amplifier using 2 vacuum tubes. A condenser is used in the grid circuit of the first tube, which is charged and discharged by a switch of special design. The substitution of a milliammeter for a galvanometer prevents drift, secures equal in a few sec and permits a precision of 0.5 mV with resistances up to 500 megohms. Drawings of the app. and detailed directions for assembly and operation are included. The glass electrode consists of an elongated bulb on the end of a 10-mm tube of Corning glass No. 015, filled with 0.2 N HCl, into which extends a AgCl electrode. With this set the walls need not be thin, for large resistances do not interfere with accurate readings.

F. E. B.  
Photoelectric colorimeter with logarithmic response. Ralph H. Muller and Gilbert F. Kinney. *J. Optical Soc. Am.* 25, 342-6 (1935).—Since the light transmitted by a colored soln is a log function of the concn according to the Lambert-Beer law, a device which possesses log response is sought. This instrument consists of a vacuum photoelec. cell feeding into a vacuum tube voltmeter. This has a type 6D6 tube, which with properly chosen resistance gives a plate current proportional to the log of the input voltage. The output meter can thus be calibrated directly in concn. units. Details of the circuit and optical arrangements are shown, and some measurements obtained with the instrument are given.

Helen S. Hopfield  
Photoelectric cells with barrier film artificially deposited. W. Ch. van Geel and J. H. de Boer. *Physica* 2, 892-900 (1935).—The photoelec. qualities of artificial barrier cells with Se as semiconductor and cresol-formaldehyde resin, CaF<sub>2</sub>, SiO<sub>2</sub> or MgO as barrier film were studied. The Se film (10 μ) was sublimed onto an Fe or a brass plate, baked at 200° to a grayish film, then a 10<sup>-4</sup>-cm film of barrier material applied either as soln or as vapor. A thin transparent metal film (e.g., Sb) is then volatilized onto the barrier film as electrode. For a cell of 4 sq cm, MgO barrier, the rectifying ratio was 25; the photocurrent for Sb negative 65 × 10<sup>-8</sup> amp. At 24 v with Se negative a 75 × 10<sup>-8</sup>-amp photocurrent resulted from radiation of 3000 luxes, for 4.5 v, Sb positive, current 10<sup>-8</sup> amp; without potential the current was 0.5 × 10<sup>-8</sup> amp. The current light relation is  $i = AL^{1/2}$  for all types of barrier

material used. The internal photoeffect of the Se used followed  $i = BL^{1/2}$  independently of the potential used (10-100 v). The photoelectrons are liberated in the Se semiconductor; the barrier material need not have any internal photoeffect.

B. J. C. van der Hoeven  
Bequerel cells. G. Athanasu. *Ann. phys.* [11], 4, 377-449 (1935); cf. *C. A.* 20, 1005.—The effect of the H<sup>+</sup> and OH<sup>-</sup> concns on the magnitude and direction of the photovoltaic effect was carefully investigated for the following electrodes: Ag-AgI, Hg-Hg<sub>2</sub>I<sub>2</sub>, Ag-AgBr, Cu-CuI, Cu-CuBr, Cu-CuO, Hg-HgS, Ag-AgS and Cu-Cu<sub>2</sub>O. The effect was greatest with Ag-AgI and Hg-Hg<sub>2</sub>I<sub>2</sub>, where the pos. photovoltaic effect is increased by H<sup>+</sup> and may be reduced to zero or even reversed by OH<sup>-</sup>. The results may be explained as due to the electrode functioning as an "acceptor" of the OH<sup>-</sup> of the electrolyte, i.e., the I<sup>-</sup> liberated by the light on the electrode may temporarily form IOH, which interferes with recombination with the Ag or the Hg. The photovoltaic  $e.m.f.$  decreases with rise in temp. in all cases. This is explained on the basis of two opposing reactions, one photochemical and the other purely thermal. Sensitivity curves in the spectrum and the thresholds of sensitivity were detd. With Ag-AgI maxima were found at λ 0.4225 μ and λ 0.30 μ, which correspond with the max. adsorption of AgI. The max. at λ 0.4225 μ coincides with the max. photoconductance. With Hg-Hg<sub>2</sub>I<sub>2</sub>, the max. was found at λ 0.400 μ and the threshold toward the red at λ 0.530-0.540 μ. With Cu-CuO a max. was found at λ 0.400 μ and the sensitivity extended as far as 1 μ in the infrared. Electrodes of Cu covered with a layer of the suboxide obtained by the wet and dry methods gave a max. photovoltaic effect which coincided with the max. photoconductance. The max. sensitivity was at λ 0.480 μ. It is impossible to explain the phenomena of Bequerel on the basis of the photoelectric effect alone. The app. for investigating these cells is described in detail.

W. George Parks  
Heterogeneous chemical reactions under the silent electric discharge. XV. Preparation of colloidal solutions. 4. Susumu Miyamoto. *J. Chem. Soc. Japan* 56, 1359-64 (1935), cf. *C. A.* 29, 6846<sup>7</sup>.—By using active H<sub>2</sub> prepd. in the silent elec. discharge, (a) the hydrosol and the alcohol of Cu<sub>2</sub>O from CuSO<sub>4</sub>, (b) the hydrosol and the alcohol of CuS from CuS, (c) the hydrosol of Hg from Hg<sub>2</sub>NO, (d) the hydrosol of HgS from HgS and from Hg thiocyanate were prepd. and their stability was studied.

T. Katsurai  
Chemical reaction in the electric discharge. I. The chemical effects of impulse discharges. E. J. B. Willey. *Proc. Roy. Soc. (London)* A152, 158-71 (1935).—Wrede's results (*C. A.* 23, 2886) for the disson of N<sub>2</sub> in the controlled impulse discharge of a condenser are confirmed by chem. means. Concns of active N up to 40%, with NO as reagent, and to 13% with org. vapors (calcd. from the yield of HCN) were obtained. The controlled impulse discharge is superior to a -c. and d -c. discharges in both absolute and power yield. Under fixed conditions of gas flow, pressure, etc., the yield of active N depends only on the current, being independent of the frequency, length of discharge, and magnitude of the individual current impulse. The glow intensity and elec. cond. of the gas are no guide to the amts. of active N present.

L. E. Steiner  
Purifying argon for filling incandescent lamps. Robert P. Moran. *Industrial Gas* 14, No. 6, 13, 20-5 (1935).—Commercial cylinder argon is purified by passing it in turn through soda lime, over P<sub>2</sub>O<sub>5</sub>, through a tube contg. Cu chips heated to 500°, a CuO tube at 500°, and finally again through soda lime and over P<sub>2</sub>O<sub>5</sub>. The steel tubes for Cu and CuO are in a gas-heated furnace, consisting of a vertical steel cylinder, 2 ft. in diam. and 3 1/4 ft. high, lined with 4 in. of firebrick. This plant supplies N<sub>2</sub> and Ar for 15,000 lamps in 8 hrs., using 1100 cu. ft. of 625 B. t. u. fuel gas per 24 hrs.

R. W. Ryan  
Pb metallurgy (Hayward) 9. Elec. furnace for fusing glass (Fr. pat. 786,168) 19. Ni-Cr-Ca alloys [for lamp filaments] (U. S. pat. 2,026,213) 9

Batteries. Soc. anon. Le Carbone. Brit. 435,141, Sept. 16, 1935. See Fr. 778,742 (C. A. 29, 4689P).

Storage battery. Harvey D. Geyer (to General Motors Corp.). U. S. 2,024,637, Dec. 17. Structural features.

Storage battery. Karl W. Gasche (to Firestone Battery Co.). U. S. 2,024,988, Dec. 17. Structural details.

Storage battery. Paul E. Barkhausen and Carl H. King (to Globe-Union Mfg. Co.). U. S. 2,025,149, Dec. 24. Structural details.

Storage battery. Karl W. J. Hjelmblad (to Accumulator Aktiebolaget Leif Nobel). U. S. 2,025,452, Dec. 24. Structural details.

Storage battery. Milton E. Taylor. U. S. 2,025,482, Dec. 24. Structural details.

Storage batteries. Henri G. André. Fr. 786,276, Aug. 20, 1935. In alk. batteries having a sol neg. electrode Cellophane is used to immobilize the electrolyte, favor the liberation of gas, avoid penetration by conductive substances and render the deposition of the neg. metal homogeneous and reversible.

Storage batteries. Soc. anon. des accumulateurs Monoplaque (Paul Pauton, inventor). Fr. 787,259, Sept. 10, 1935. An electrolyte for Ni and Zn batteries contains a substance capable of causing the formation of O during the working of the battery (a mixt. of oxides of Fe and Ti, to which Co oxide may be added as a catalyst, or a mixt. of  $K_2CrO_4$  and oxide of Co or Hg) to prevent the formation of secondary salts of Zn, such as the hydrates or other basic salts and to allow the correct deposit of Zn on the neg. electrode.

Storage batteries. Compagnie générale d'électricité. Fr. 787,330, Sept. 20, 1935. See Brit. 430,897 (C. A. 29, 7831P).

Storage-battery separator with a wood diaphragm and hard rubber dowels. Charles D. Galloway. U. S. 2,026,030, Dec. 31. Structural details.

Storage-battery plates. Edward W. Smith (to Electric Storage Battery Co.). U. S. 2,026,193, Dec. 31. Pencils of active material surround rods of conductive material and are in turn surrounded by slabs of microporous, elec.-insulating, acid-resisting material. Various structural details are described.

Storage-battery terminal. Charles E. Hoover. U. S. 2,024,248, Dec. 17. Structural details.

Storage-battery cases. Joseph Lucas Ltd. and John Merrick. Brit. 434,427, Sept. 2, 1935. To cover cracks that may form in the partition in a cell molded from plastic material, the partition is provided with a covering of impervious and slightly extensible material, e. g., ebonite, on 1 or both sides.

Dry cell. Clemens T. Fleckenstein and Thomas H. Byrne (to Bond Flac Corp.). U. S. 2,025,028, Dec. 24. Structural details of a battery having a disk of material such as paper or felt impregnated with an asphaltic compn placed beneath a metal cover closing the upper end of the battery container.

Dry cell. Frank A. Adamski (to National Carbon Co.). U. S. 2,025,631, Dec. 24. Structural features.

Dry cell. Sergius Apostoloff. U. S. 2,026,203, Dec. 31. Numerous structural details.

Dry cell. Peter Corighano and Louis Platzman (to Bright Star Battery Co.). U. S. 2,026,615, Jan. 7. Structural details.

Selenium cells. Constantin Chidlovsky. Ger. 621,472, Nov. 7, 1935 (Cl. 21g 29). This corresponds to Fr. 747,791 (C. A. 27, 4963).

Fluid cells. Soc. anon. Le Carbone. Fr. 787,232, Sept. 10, 1935. The formation of climbing salt in batteries having an alk. electrolyte is avoided by forming, above the electrolyte, a chamber the atm. of which cannot be renewed.

Galvanic baths. Schering-Kahlbaum A. G. Fr. 787,506, Sept. 21, 1935. Baths, e. g., of Ni or Cu are restored to their original efficacy by adding a small amt. (1-3 g. per l.) of a substance having a surface action,

e. g., Al silicate, fullers' earth or pulverized gelatinous  $SiO_2$ .

Electrolytic cells. Ferdinand Krauss. Brit. 431,165, Aug. 20, 1935. Divided on 431,488 (C. A. 30, 6879). A cell for producing per-salts without the use of a diaphragm comprises a container constituting 1 electrode and an electrode nested within and uniformly spaced from the outer 1. 1 of the electrodes comprises a set of conductive patches sepd. by insulating material.

Electrolytic cells. Maschinenfabrik Oberkron. Brit. 434,348, Aug. 20, 1935. Distd.  $H_2O$  is supplied periodically to the cells of a  $H_2O$  decomposer through branch pipes connected to a main pipe connected to a feed tank situated several meters above the cells.

Electrolytic cells. Hugh B. Franklin. Brit. 434,542, Sept. 4, 1935. Upward circulation of electrolyte through a series of superimposed cells is provided by means of narrow tubes extending from the liquid level of 1 cell into the gas space of the cell above.

Electrolytic cells. N. V. Philips' Gloeilampenfabrieken. Brit. 435,112, Sept. 16, 1935. A cell comprises a tubular electrode support of hard insulating material directly pressed into a bore of the wall of the vessel and secured in a bore of the electrode body, both in fluid-tight manner, said support permitting the leading through, not in fluid-tight manner, of an electrode connection through the wall of the vessel.

Electrolytic cell for production of anhydrous magnesium chloride. Wilhelm Moschel (to Magnesium Development Corp.). U. S. 2,024,242, Dec. 17. Various structural and operative details.

Electrolytic cell (with self renewing anodes) for aluminum manufacture. "Montecatini" Soc. generale per l'industria mineraria ed agricola. Austrian 143,310, Nov. 11, 1935 (Cl. 40c).

Electrolytic rectifiers and condensers. Ralph D. Mershon. U. S. 2,024,240, Dec. 17. Electrodes are made of Al or an Al alloy to which has been added (suitably 0.4-3.3%) of an alkali metal or alk. earth metal silicate such as Na silicate or glass wool which serves to give improved working properties.

Electrolytic condenser. Elektrizitäts-A.-G. Hydraul. Fr. 787,790, Sept. 28, 1935.

Plated metal for jewelry. Ermand H. Davignon (to General Plate Co.). U. S. 2,024,150, Dec. 17. A thin layer of Ni is electrodeposited on a cleaned surface of stainless steel and a layer of precious metal is united to the Ni by an intervening layer of soldering metal, and the material is rolled to produce a finely attenuated stock suitable for jewelry manufacture.

Chromium-coating articles such as those of glass or china. Ernst P. Schreiber. U. S. 2,025,528, Dec. 24.

A nonmetallic rigid article is immersed in an aq. soln. of Sn sulfate of about 5° Bé strength and superficially washed, and there is then poured over the article simultaneously a soln. of 1 oz.  $AgNO_3$  and 1 oz. of a 28%  $NH_3$  soln. in a qt. of water together with a soln. of  $Cl_2O$ , the article is rinsed in water, then submerged in a  $CuSO_4$  soln. of about 25° Bé strength to which an elec. current of 5-15 amp. is applied, then rinsed with water, immersed in a Ni sulfate soln. of about 25° Bé strength, washed, dried, polished, washed in alkali soln., immersed in a chromic acid soln. of 25-40° Bé strength for 3-10 min. at 100-200 amp. and finally rinsed and dried. Coating of leather, paper, celluloid and rubber is also described.

Electroplating aluminum and its alloys with silver. Vereinigte Aluminiumwerke A.-G. (Hans Ginsberg, inventor). Ger. 621,489, Nov. 7, 1935 (Cl. 48a 601). The cleaned metal or alloy is dipped in a strongly acid bath contg.  $HCl$ ,  $HNO_3$  and  $HF$ . A suitable bath contains  $HCl$  (36.3%), 3,  $HNO_3$  (65%), 1,  $HF$  (40%) 1 and water 15 parts by vol. The material is then washed and dipped in an alk. bath, e. g., a 10%  $NaOH$  soln. satd. with  $NaCl$ . Electrodeposition of Ag is then effected in the usual way. Dense coherent coatings are obtained.

Electrolytic cleaning and plating apparatus. Stanley R. Brewer and John F. Hunsley. Brit. 434,116, Aug. 23, 1935. Metal wire or strip or articles connected in a chain.

are descaled by passage through a narrow tank between electrodes mounted in recesses and similarly arranged electrodes of opposite polarity at the other end of the tank, the work forming an intermediate electrode

**Preparing copper wire for enameling** Pierre Fuster (one half to Soc anon J Boucure & Cie) U S 2,023,998, Dec 10 For producing uniform enameled wire for elec purposes, the wire, between drawing steps, is passed, as a sol anode, through an electrolytic soln of  $\text{CuSO}_4$  capable of directly attacking the substance of the wire itself under the action of an elec current, to remove surface inequalities and protuberances of Cu on the wire, and, after further drawing, the wire is enameled

**Ferrophosphorus** Metallgesellschaft A-G (Conway Freiherr von Gurselwald, Hans Weidmann and Gerhard Roesner, inventors) Ger 618,958, Sept 19, 1935 (Cl 185 20) Fe-P with a high P content is obtained in two stages. First, Fe-P contg Si is obtained by reducing a fused mass contg phosphate, Fe,  $\text{CaO}$ ,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ . The amt. of  $\text{Al}_2\text{O}_3$  is adjusted so that a Fe-P-Si alloy and a practically  $\text{SiO}_2$  free aluminate slag results. In the second stage, the Fe-P-Si alloy is treated with further phosphate to remove the Si and increase the P content. In the example, bauxite, Fe shot and coke are heated in an elec furnace to give a Fe-P-Si alloy and a Ca aluminate slag. The slag is converted to  $\text{CaCO}_3$  and  $\text{NaAlO}_2$  by treatment with  $\text{Na}_2\text{CO}_3$  soln. The Fe-P-Si alloy is heated again in an elec furnace with phosphate and Fe shot to give Fe-P. Cf C 29, 5005<sup>1</sup>

**Beryllium** Deutsche Gold- und Silber-Scheideanstalt vorm Roessler and G Jaeger Brit 431,338, Aug 29, 1935 See Fr 785,072 (C A 30,391<sup>1</sup>)

**Coloring aluminum alloys** Siemens & Halske A-G (Nikolai Budiloff, inventor) Ger 618,830, Sept. 17, 1935 (Cl 48a 16) A dark-colored oxide layer is produced on Cu-Al alloys by first producing a light-colored oxide layer by anodic electrolysis in a d c or a c., and then subjecting the coated Cu-Al to cathodic electrolysis. Both operations may be carried out in the same soln., which may be  $(\text{CO}_3\text{H})_2$  soln. Cf C A 30,427<sup>1</sup>

**Electrolyte for filming metal** such as aluminum, tantalum, etc. Philip E Fiedman (to Robert T Mack, as trustee) U S 2,024,210, Dec 17.  $\text{NH}_4$  benzoate is used as a film forming chemical (suitably in a condenser, rectifier or lightning arrester). Various other benzoic or salicylic compds. also may be used.

**Electrolytic manufacture of aluminum** Compagnie de produits chim et electrometallurgiques Alais, Froges et Camargue Ger 621,153, Nov 2, 1935 (Cl 40c 6 04) See Fr 706,998 (C A 29,58<sup>1</sup>)

**Electrolytic system for aluminum production** Jean Grolee (to Compagnie de produits chimiques et electrometallurgiques Alais, Froges et Camargue) U S 2,026,466, Dec 31 Various elec and operative details

**Electrolytic furnace for obtaining aluminum** Siemens-Pfannkuche A G fur Kohlefabrikate (Edwin Leuchner, inventor) Ger 618,601, Sept 11, 1935 (Cl 40c 6 04)

**Electric resistance heated furnace** suitable for use in galvanizing James C Woodson (to Westinghouse Elec & Mfg Co) U S 2,024,714, Dec 17 Various structural details

**Multitubular furnace** suitably heated by electric resistances Emilio Ficorò U S 2,021,516, Dec 17. Structural and operative details

**Rotatable air-tight furnace** with an electrical resistance heater suitable for melting magnesium, etc. Henri George (to Soc anon des manufactures des glaces & produits chimiques de Saint Gobain, Chauny & Crey) U S 2,021,854, Dec 17 Various structural, mech and operative details

**Electric oven** Rosalia C Phelps U S 2,024,386, Dec 17. Structural details

**Electric oven** Hubert Meredith-Jones U S 2,025,515, Dec 24 Various structural features

**Electrically heated annealing furnace** for wire, bands, etc. Siemens-Schuckertwerke A-G (Johann Schnepf, inventor) Ger 618,504, Sept 9, 1935 (Cl 18c 6 60).

**Portable electrically heated crucible** Voigt & Haefner A-G (Robert Trambauer, inventor) Ger 621,302, Nov. 5, 1935 (Cl 21k 15 50).

**Electric-resistance temperature indicator for use with liquid-immersed electrical transformers, etc.** Guglielmo Camilli (to General Elec Co) U S 2,026,375, Dec 31. Structural and elec details

**Glowing cathode** Radio-Röhren-Laboratorium Nickel G m b H Ger 618,917, Sept 18, 1935 (Cl 21g 13 01).

In making glowing cathodes indirectly heated by an insulating mass, the latter is mixed with a strong reducing material such as Al powder.

**Electrical-precipitation apparatus for cleaning gases** such as blast-furnace gas for use as a fuel John P Irwin and George B Garrett U S 2,024,226, Dec 17. Various structural and operative details

**Recovering dust from gases** Siemens-Lurgi-Cottrell Elektrofilter-Ges m b H fur Forschung und Patentverwertung Fr. 786,000, Aug 26, 1935 In chem or metallurgical operations producing dust which is sepd from the waste gases by electrostatic filtration, the temp or other factors of the operation are so chosen that the gases contain hygroscopic dusts. Thus,  $\text{NaCl}$ ,  $\text{MgCl}_2$ , etc., are added to the primary material. Retrograde ionization is thereby reduced.

**Catalytic reactions** Minami Manshū Tetsudō K K Fr 786,008, Aug 24, 1935 Reactions such as hydrogenation are effected in a continuous manner by using the catalyst in the form of a suspension, passing a high-tension a c during the reaction and passing a d c when the reaction is complete so that the catalyst is caused to accumulate in the reaction vessel while the reaction products are being withdrawn and fresh materials introduced. App is described.

**Electron discharge device** Arthur L Samuel (to Bell Telephone Laboratories, Inc.) U S 2,025,075, Dec 24. Structural details

**X-ray diffraction apparatus** Chu-Phay Yap U S 2,025,488 Dec 24 Various structural details

**Photoelectric apparatus** Radio Corp. of America Fr. 787,661, Sept 26, 1935

**Photoelectric cell** N V Philips' Gloeilampenfabrieken Fr 786,412, Sept 3, 1935.

**Photoelectric cells** Compagnie française pour l'exploitation des procédés Thomson-Houston Fr 787,563, Sept 25, 1935 The cell is composed of a metal disk covered with a layer of Se and another layer of an alk earth metal

**Photoelectric tubes** Heinrich Geffcken, Hans Richter and Karl Möller (to Radio Corp of America) U S 2,024,762, Dec 17 For adjusting the emission current during the manu of photoelec tubes, a light-sensitive electrode is subjected to the influence of short-wave length radiation of the lower visible and ultraviolet range of definite intensity and for a definite period

**Mercury-vapor lamp** Alvin J. Thompson U S 2,024,311, Dec 17. Structural details

**Exhausting and gas-filling apparatus** suitable for use with electric lamp bulbs John F Donovan and Geo Hingworth (to General Elec Co) U S 2,025,579, Dec 24. Structural, mech and operative details

**Operating vapor electric lamps** Gordon R Fonda and Andrew H Young (to General Elec Co) U S 2,025,585, Dec 24 For operating an elec discharge through a confined luminosity-producing gas comprising an ingredient such as Na (as when used with Ne) condensable at ordinary temps., the current through the gas is maintained at the value at which the luminous output is substantially const. for materially unlike ambient temps.

**Ultraviolet lamp** with outer and inner bulbs and containing mercury George Sperti (to Sperti Lamp Corp) U S 2,025,182, Dec 24 Various structural and elec details

**Incandescent lamp** with a glower of hafnium carbide Fritz Blau (to General Elec Co) U S 2,025,565, Dec 24

**Fluorescent lamps** Jean-Baptiste J M Abadie Fr 786,500, Sept. 3, 1935 The lamps have a double wall,

the exterior being composed of ordinary thick glass or a hard glass such as Sabor or Pyrex, and the interior being thin and composed of a mixt. of phosphorescent and fluorescent substances such as sulfides, which are mixed with a salt or salts in the vitrified state to form the interior layer.

## 5—PHOTOGRAPHY

R. R. BULLOCK

**Photography in the far ultraviolet** G. Rathenau *Physica* 2, 840-2 (1935)—All sensitizing oils of the 3 types mentioned by Harrison and Leighton (*J. Opt. Soc. Am.* 20, 313 (1930)) are partly transparent in thin layers for light of more than 2100 Å, thereby disturbing the light distribution. A better arrangement for photography of the far ultraviolet is as follows. The photographic plate is impregnated with an absorbing dye which absorbs selectively light of fairly long wave length, then covered with a sensitizer oil film which acts on short wave lengths unabsorbed by the dye. Dyes used are nitrosodimethyl-aniline, methyl orange, fuchsin and sensitizer Cenco pump oil 11021B. R. J. C. van der Hoeven

**Influence of atmospheric oxygen in the photographic process of bleaching-out of the latent image** J. Marietta Blau, *Phot. Korrr* 71, Suppl., 21 8 (1935), cf. *C. A.* 29, 4685<sup>1</sup>—Expts with gaslight and AgBr papers, and with transparency and neg. emulsion plates, show that a diminution of the atm. pressure to about 6 mm. has the effect of diminishing or preventing the Herschel effect and of partially restoring the original sensitivity of the materials after they have been desensitized with pink/ryptol yellow. Capri blue, which ordinarily desensitizes, can sensitize transparency plates, and this sensitization is then found to be greater under the diminished, than under normal, atm. pressure. It thus appears that a reaction in which O is involved is an essential process, alike in the Herschel effect and in desensitization by means of dyes. With ordinary com. color-sensitized materials, under the same conditions, even with addition of KBr, no influence of atm. O is to be observed. E. R. Bullock

**Agfa's new pan-kine type II negative film** Andreas Schilling, *J. Motion Picture Soc. India* 1, No. 8, 15-21 (1935)—In comparison with the Type G, the Agfa Type II film is found to be approx. twice as fast, to be equally fine-grained and to give better shadow-tone reproduction. G. P. Matthews

**Voigtlander-Gevaert Bessapan film** R. Weisacker *Atelier Phot.* 42, 161-2 (1935)—This is a panchromatic film has a speed of 10/10<sup>2</sup> DIN, good gradation, wide latitude, protection against halation, exceptionally good color rendition, freedom from fog and reasonably low graininess. H. Parker

**Color films by the "silver-dye-bleaching" process** Gerd Heymer, *Veroffentl. wiss. Zentral-Lab. phot. Abt. Agfa* 4, 177-80 (1935)—Under this title are classed those processes which effect bleaching of dyes through the agency of a Ag image. Included is Schweitzer's process, in which a Ag image is converted with toning solns. into one of PbCrO<sub>4</sub>, which is then decompd with an acid to liberate CrO<sub>3</sub>, which, in turn, destroys the dye. Also included is Christensen's process, in which the Ag image appears to catalyze the destruction of the dye by reducing agents which otherwise act upon the dye very slowly or only at elevated temps. References are made to publications of Luther and Von Holcklen, Crabtree, Lippocramer, and Gaspard, and to work done in the Agfa research lab. The essential characteristics of the "Ag-dye-bleaching" process are said to be: "1. Only reduced dyes, a Ag image, the unused remainder of which is removed by any Ag solvent, a Ag solvent in acid medium as a bleaching soln., and in case the bleaching soln. has a tendency to liquefy the gelatin, a hardening agent for the gelatin, which is applied before the entrance of the sample into the bleaching soln." The most suitable dyes are said to be found among the substantive azo dyes. The diffusion of the dyes from 1 layer of a multilayer film to another is said to be diminished by the use of heavy-metal salts, basic dyes, or chemically similar colorless substances as

2 pptg. agents. A difficulty in the use of dyed emulsion layers sensitized for the colors which they absorb, as proposed by Christensen, is said to lie in the tendency of the other colors to penetrate the depth of the emulsion and produce clear highlights at lower exposure values than the absorbed colors, even though the absorbed colors give higher threshold speeds. Consequently, layers sensitized for transmitted colors are employed in some practical applications of the "Ag dye bleaching" process. This arrangement prevents making direct photographs in natural colors or direct prints in natural colors from similar originals. A method is described for making original records on lenticular biprism and printing these on a film bearing, on the 1 side, yellow and magenta layers, and, on the other, a blue green layer, differently colored filters being used in taking and printing. M. W. Seymour

**Herschel effect in red- and infrared sensitive photographic plates** J. Narbutz *Wiss. Phot.* 33, 48-51 (1934), cf. *C. A.* 28, 7180<sup>1</sup>—Expts. demonstrating the effect are described. B. C. A.

**Use of Sheppard sensitizers and cystine like substances.** A. Steigmann *Phot. Ind.* 33, 602-3 (1935)—A completely inert bone gelatin (contg. no inhibiting substances) was used for testing the effect of the addition of Sheppard's sensitizers and it was found to produce a gelatin unsuitable for photographic emulsions. However, by combining inhibiting agents like cystine and formal cystine, with (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and gelatins predominantly high in albumin, a good emulsion gelatin was obtained. H. R. B.

**Reactions of gelatin sensitizers** A. Steigmann, *Phot. Korrr* 71, 92-4 (1935)—After a discussion of the methods in use for the detection of gelatin sensitizers, S. describes tests on the wick waters from a gelatin that had been rendered completely inert and from a Nelson gelatin which was exceptionally rich in sensitizers. The results are in harmony with the view that the natural sensitizers in a gelatin consist of thiosulfates. E. R. Bullock

**Properties of photographic gelatin** A. Steigmann *Phot. Ind.* 32, 1172-3 (1934)—The character of the sensitizing and inhibiting substances in gelatin was investigated. Albumin has inhibiting properties but there seems to be no correlation between the amt. of albumin present, as detd. by heat coagulation, and the inhibiting property of the gelatin. The substances present in a good photographic gelatin from bone are thiosulfates, which easily oxidize to polythionates, and cystine-like substances but not pure cystine, since it is a strong desensitizing agent. H. R. Brigham

**Modification of silver sulfide images** E. Asloglou, *Photo-Rev.* 47, 299-31 (1935)—The tone of sulfidized prints can be modified by supplementary toning, for which formulas and directions are given. The procedures described include Se, Au, Fe, Cu and Hg toning, and mordanting for dye toning. P. W. Vittum

**Fogging of the silver bromide plate by dyes** Lippocramer *Z. wiss. Phot.* 34, 150-5 (1935)—Ordinary, noncolor sensitized plates, after being bathed for 2 1/2 min. in a 0.02% soln. of pinilavol or one of 5 dyes related to pinilavol and dried, and then developed in hydroquinone for 6 min., showed fogs of 2.0 with pinilavol and 0.3 to 0.4 with the related sensitizers. Pinicynol, benzoclenazole, benzothiazole and benzoxazole, similarly tested, gave fogs of 0.5, 0.15, 0.35 and 0.1, resp. This is also the order of decreasing toxicity of these dyes. When followed, without drying, by development in ferrous oxalate, 0.01% soln. of phosphine, isooquinoline red, acridine yellow, pink/ryptol yellow and phenosulfaniline gave fogs ranging from 1.6 to 0.15, while no fog was obtained with similar solns. of toluasulfaniline, brilliant rhodu-

line red, fuchsin, erythrosin or rhodamine B. L-C discusses the ease of fogging by a dye in the presence of a heavy metal salt, and in connection with Weber's explanation of this effect (C. A. 27, 3888) suggests that the dyeing of the Ag halide grain diminishes the protective action of the gelatin and thus favors the acceleration by the heavy-metal salt of the reducing action of the developer. E. R. Bullock

**Intermediate soda bath** E. Weyde *Phot. Chronik* 32, 270-1 (1935) —For decreasing the washing time by facilitating the removal of  $\text{Na}_2\text{S}_2\text{O}_3$ , the use, after fixing, of  $\text{Na}_2\text{CO}_3$  (1%) for 1 min is suggested. Greater permanence of the prints is claimed. L. E. Muehler

**Chromium intensification** R. Nannus *Progresso fot.* 41, 294-7 (1934) —In a review of the practice of Cr intensification, N suggests the following modification of his previous bleach soln. (*Progresso fot.* 40, 35-7 (1933)). KBr 50 g.,  $\text{CrO}_3$  5 g.,  $\text{H}_2\text{O}$  to 1 l. Increase of the  $\text{CrO}_3$  above 10 g. per l. decreases the degree of intensification. The gelatin of films bleached for Cr intensification is claimed to be greatly affected by the alteration occurring in the change from the acid bleach to an alk. developer. To avoid this, the 2nd developer used for the Baby Pathé reversal process is suggested. This consists of a soln. contg.  $\text{NaHSO}_3$  1% and  $\text{Na}_2\text{S}_2\text{O}_3$  1%. L. E. M.

**Printing papers with silver salts** P. Weigleb *Schweiz. Phot. Ztg.* 37, 163-71, 175-82 (1935) —A general account of the structure and characteristics of Ag salt printing-out papers and of AgCl, Ag chlorobromide and AgBr development papers. E. R. Bullock

**Ways and means of reducing** R. Weiszacker *Phot. Chronik* 42, 149-50 (1935) —In a list of well-known reducers, the following soln. given by Valenta, employing ammoniacal  $\text{CuO}$  with  $\text{Na}_2\text{S}_2\text{O}_3$ , is suggested for use with gaslight papers.  $\text{NH}_3$  (conc'd) 200 cc. and  $\text{CuSO}_4$  5H $_2\text{O}$  25 g. To use, add 3 to 5 cc. of this soln. to 100 cc. of 10% hypo soln. L. E. Muehler

**The function of alkali in a developer** II. Physical developing G. P. Faerman and N. N. Shishkina. *J. Phys. Chem.* (U. S. R. S.) 5, 456-63 (1934), cf. C. A. 29, 1852\* —With const.  $\text{Ag}^+$  concn the rate of phys. development and the rate of reduction of the  $\text{Ag}^+$  in soln. decreases or increases with decreasing or increasing  $\text{pH}$ . For each  $\text{Ag}^+$  concn in the soln. there is an optimum  $\text{pH}$  for the developer. If the systems  $\text{Ag}^+/\text{Ag}$  and oxidation/reduction have the same  $\text{pH}$ , the velocity of development is independent of the  $\text{pH}$ . Only on longer drawn-out development do factors other than  $\text{pH}$ , such as concn of  $\text{Ag}^+$  ions, of developer, of acid anions, etc., have any effect. The expts. were carried out mainly on "Agfa" films with metol-citric acid developers in a  $\text{pH}$  from 1.6 to 3.0. III. An iron developer *Ibid.* 404-5 —In a ferro-feric oxalate developer the developing influence remains const. over the same interval of  $\text{pH}$  as does the oxidation/reduction potential, i. e., from  $\text{pH}$  = 4-7. F. H. Rathmann

**A comparison of reflection densities measured photoelectrically and visually** R. E. Owen and E. R. Davies *Phot. J.* 74, 463-70 (1934); *Kodak Research Labs. Monthly Abstr. Bull.* 20, 433-4, cf. C. A. 28, 7182\* —The possibility of measuring the reflection  $\rho$  of photographic papers with an instrument embodying a photoelec. cell and an optical wedge is discussed with particular reference to the influence of the spectral sensitivity of the photoelec. cell and the spectral absorption of the wedge. Since neither a photoelec. cell which corresponds exactly with the eye in spectral sensitivity nor an optical wedge which is neutral over the wave-length range of interest is available, it is theoretically impossible to obtain exact correspondence between measurements made with such an app. and those made visually, if the photographic papers show any selective reflection. It is shown, however, that by suitably choosing the conditions of measurement, satisfactory agreement can be obtained for all photographic papers in general use, including sepia toned prints. Since the visual measurements were found to depend to some extent upon the color quality of the light used,

this was standardized as daylight and approximated to by screening a W lamp with a Wratten No. 78 filter. Authors

**Plastic material [for making cinematographic films]** (Fr. pat. 787,142) 13 Photometric app. for measuring the hiding power of photographic emulsions (Brit. pat. 434,136) 26

**Color photography** Béla Gáspár. Brit. 434,875, Sept. 5, 1935 Multicolor photographic images are produced on colored and differently sensitized multilayer or multicolor screen material, the same filters being used for recording and printing purposes, by using filters each having 2 light-transmission ranges, viz., 1 in the light, visible portion of the spectrum for recording and 1 in the dark, invisible portion for printing purposes. An example describes a screen contg. blue filter elements, transmitting at 400-500 m $\mu$  and also at 600-690 m $\mu$ , formed by a mixt. of eosulin, methylene blue, rhodamine and Berlin or Prussian blue; green filter elements, transmitting at 500-600 m $\mu$  and also at 700-750 m $\mu$ , formed by a mixt. of Patent blue, tartrazine and 2,2'-dimethyl-3,4,3',4'-dibenzothiotricarbocyanine chloride, and red filter elements, transmitting at 590 m $\mu$  and also above 750 m $\mu$ , formed by a mixt. of indolemine carbocyanine, thioflavine T and 1,3,3,1',3',3'-hexamethylindotricarbocyanine iodide. The red filter element may also be selected to have a transmission range of 350-390 m $\mu$  by coloring with rhodamine, Prussian blue and nitrosodimethylamine-HCl.

**Color photography.** I. G. Farben und -G. Fr. 787,388, Sept. 21, 1935 In the production of mono- and polychrome images by synthesis of azo dyes, a material is used in which the photographic layers serving for the constitution of the image are allied with components of azo dyes which are substantive with respect to the layers. Thus, azo or diazo components in the dissolved state, which behave substantively toward gelatin are added to a soln. of gelatin and the gelatin is submitted to a careful washing and treated in the usual manner.

**Colored photographic materials** Béla Gáspár. Brit. 434,303, Aug. 29, 1935 See Fr. 777,098 (C. A. 29, 3617\*).

**Photographic sensitive materials** Humphrey D. Murray, Harry Bames, Ralph A. S. Grist and Dufaycolor Ltd. Brit. 435,484, Sept. 23, 1935 In sensitive material using a monochrome or multicolor screen, the color screen is sepd. from the emulsion by a varnish layer which comprises a compn. contg. a "Glyptal" resin, e. g., "Paralac," which also contains a drying oil, e. g., linseed or tung. Inert substances, e. g., cellulose esters, chlorinated rubber compds. or metallic driers, may also be added to assist in the drying of the layer. Plasticizers may be added to increase the flexibility of the layer.

**Light-sensitive layers** Oskar Süs (to Kalle & Co. A.-G.). U. S. 2,025,675, Dec. 24.  $\text{AgNO}_3$ , ferric  $\text{NH}_4$  citrate and free malic acid, or like ingredients, are used together.

**Intensifying photographic images** Kenneth C. D. Hickman and Walter J. Weyerts (to Eastman Kodak Co.) U. S. 2,024,644, Dec. 17. For intensifying a photographic Ag image, it is bleached in a soln. contg. an oxidizing agent such as  $\text{KMnO}_4$  and relatively poor in Ag precipitant, the bleached image is darkened in a soln. contg. Na sulfide and a solvent for Ag bromide such as Na thiosulfate and the resulting Ag sulfide image is then intensified in a soln. contg.  $\text{NaHSO}_3$ ,  $\text{Na}_2\text{SO}_3$  and a Ag salt such as Ag sublim. Various examples are given.

**Positive reflex copies on diazotype layers** Lodewijk P. F. van der Grinten (to N.-V. Chemische Fabriek L. van der Grinten). U. S. 2,020,292, Dec. 31. A sensitive sheet for prep. pos. reflex copies comprises a layer contg. diazo compds., presenting small areas substantially free from diazo compds. sepd. by areas contg. diazo compds. Cf. C. A. 30, 402\*.

**Cinematographic films in natural colors** Georges A. Ragum (to "Société Lumière"). U. S. 2,025,671, Dec. 24. A transparent support is first coated with a glumk transparent varnish along a longitudinal portion corre-

sponding to the succession of images of the finished film but not along the longitudinal portion corresponding to the sound track; transparent multicolored particles are spread on the support, those particles which do not adhere are removed, and the whole width of the support is coated with a light-sensitive emulsion.

**Cinematographic color sound films** Béla Gáspár U. S. 2,025,639, Dec. 24. In a pre-dyed photographic light-sensitive Ag halide emulsion layer there is formed a developed and fixed Ag image constituting a sound record and a Ag image constituting a picture record, and the records are treated with an agent which locally destroys the dye to convert each of the records into a color image, the metallic Ag in the visual and sound records is converted into a Ag salt, and the sound record only is treated with a reducing substance to reconvert the Ag salt into metallic Ag, and the Ag salt is thereafter dissolved from the visual record portion by treating the film with a fixing bath.

**Sound and like records** N. V. Philips' Gloeilampenfabrieken Brit. 434,020, Sept. 11, 1935. A carrier, preferably a film, on which sound, etc., may be recorded mechanically for optical reproduction and which comprises 2 adjoining layers, 1 of which is formed of a material capable of holding a dye or the like, e. g., of tanned gelatin, while an adjacent, preferably the covering, layer is formed of a material that repels dyes, e. g., of untanned gelatin, has its covering layer formed by or contg. a material that is opaque to red or infrared rays, whereas the other layer or layers is or are transparent to such rays.  $\text{CuSO}_4$  may be used as the substance opaque to red and infrared rays.

**Apparatus for drying photographic prints** Charles Jellinek (to Kodak Ltd.) Brit. 431,431, Sept. 2, 1935.

**Photographic printing by x-rays** Siemens & Halske A.-G. Brit. 435,181, Sept. 12, 1935. Addn to 407,830 (C. A. 28, 5358). Photographic positives are produced by first using the sensitive Al or Al alloy supports prep. as described in 407,830 to produce negatives and then printing positives from the negatives by means of x-rays. Before use in the printing process, a Ag negative may be

converted by any known toning process into a negative of a heavier metal, e. g., Au or Pt or of a salt of Pb or other heavy metal.

**Tanned prints** Kalle & Co. A.-G. Brit. 431,073, Aug. 26, 1935. This corresponds to Ger. 615,958 (C. A. 29, 7842).

**Pictures in relief from plane photographs** Ernest L. Zeve (to Scul-Pho, Inc.) U. S. 2,020,202, Dec. 31.

Numerous operative details are described.

**Motion-picture reverse mat.** Frank D. Williams U. S. 2,021,081, Dec. 10. The image of an object reflecting light predominantly red before a background reflecting predominantly blue light of greater intensity than the light from the object is photographed upon a film comprising a panchromatic emulsion stratum on the front of a single base and an orthochromatic emulsion stratum superposed over the panchromatic emulsion stratum and facing the object, and the film is developed to produce a normal detail negative image corresponding to the object and a substantially nonlight-transmissive background.

**Photographic plates** Gaston Maillet U. S. 2,025,696, Dec. 31. A coating is used comprising a colloid mixed with a chromate salt of an org. base of the pyridine group such as pyridine or quinoline chromate or dichromate.

**Printing plate** Ernest S. Ballard (to Standard Process Corp.) U. S. 2,021,080, Dec. 10. A screen positive is made in which tones and shadows are represented by distinct dots in photographic gradation, a negative of the screen positive is produced on a sensitive plate, the negative is superposed on a smooth sensitized metal plate and the latter is exposed to light transmitted through the dot formation; the plate is developed and fixed so that its coating becomes nonconducting, and the fixed coating is electroplated to produce a printing plate having distinct openings of graduated sizes and the plate is stripped from the coating. U. S. 2,024,087 also relates to details of printing-plate production.

**Enamels for use in photoengraving** John J. Murray. Fr. 787,489, Sept. 23, 1935. See U. S. 2,000,453 (C. A. 29, 3928).

## 6—INORGANIC CHEMISTRY

A. R. MIDDLETON

**Coordinate valency rings** III. Some inner complex salts of iron and manganese Tokuchi Tsumaki J. Chem. Soc. Japan 56, 1329-31 (1935), cf. C. A. 29, 3930. By means of the reaction between (a) hot soln. and 150 cc. of alic of salicylaldehyde, 10 g. of 25%  $\text{Ni}(\text{OH})_2$  and 150 cc. of alic, and (b) 80 cc. of 5.6%  $\text{Fe}(\text{NH}_4)_2$  alum, the Fe deriv. of trisalicylaldehydeimine,  $\text{C}_{21}\text{H}_{18}\text{O}_6\text{N}_3\text{Fe}$ , was prep. By similar processes the Mn derivs. of trisalicylaldehydeimine,  $\text{C}_{21}\text{H}_{18}\text{O}_6\text{N}_3\text{Mn}$ , and of salicylaldehydebenzylimine,  $\text{C}_{21}\text{H}_{18}\text{O}_6\text{N}_3\text{Mn}$ , and the hydroxy-manganese deriv. of salicylaldehydebenzylimine,  $\text{C}_{21}\text{H}_{18}\text{O}_7\text{N}_3\text{Mn}$ , were obtained. T. Katsurai.

**Compounds of aluminum bromide with bromides of lithium, copper and silver.** V. A. Plotnikov and L. Ya. Gorenbeln J. Gen. Chem. (U. S. S. R.) 5, 1108-11 (1935), cf. C. A. 28, 1501. Crystals of complex compds.  $\text{LiBr} \cdot \text{AlBr}_3$ ,  $\text{CuBr} \cdot \text{AlBr}_3$  and  $(\text{AgBr})_2 \cdot (\text{AlBr}_3)_2$  crystallize from an  $\text{EtBr}$  soln. of  $\text{AlBr}_3$  mixed with the bromides of Li, Cu and Ag, resp. Melting points of the 3 complexes, in the order given above, are: 192, 241 and 180-185°.

S. L. Madorsky

**The formation of barium aluminates from barium carbonate and alumina in solid state** G. Grube and G. Heintz Z. Elektrochem. 41, 797-804 (1935).—II pulverized mixts. of  $\text{BaCO}_3$  and  $\text{Al}_2\text{O}_3$  are heated in a  $\text{CO}_2$  atm. in the temp. range between 900° and 1050°, monobarium aluminate is formed as the reaction product whether the starting mixt. contains  $\text{BaCO}_3$  or  $\text{Al}_2\text{O}_3$  in excess. Aluminates in which more than 1 mol.  $\text{Al}_2\text{O}_3$  is united to 1 mol.  $\text{BaO}$  do not form under these conditions. At temps. from 1000° upward, reaction products

are formed in which the  $\text{Al}_2\text{O}_3$  is bound entirely as aluminate and the  $\text{BaCO}_3$  is practically entirely transformed into  $\text{BaO}$ . Extensive measurements were made of the equal pressures of the reactions to det. whether di- or tri-barium aluminates are formed.

**Chloroarsenate of calcium.** C. M. Smith, J. Wash. Acad. Sci. 25, 435-6 (1935).—To 50 g. anhyd.  $\text{CaCl}_2$  in 50 cc.  $\text{H}_2\text{O}$  was added 5 cc. concd.  $\text{HCl}$ .  $\text{Ca}(\text{H}_2\text{AsO}_4)_2 \cdot \text{H}_2\text{O}$  was then added to satn. and the mixt. put on the steam bath. In the course of 11 days crystals sep'd. A large vol. of  $\text{EtOH}$  was added and the crystals were filtered off. The empirical formula assigned to this new compd. was  $(\text{CaCl})_2 \cdot \text{H}_2\text{AsO}_4 \cdot 2\text{H}_2\text{O}$ .

Louise Kelley

**Induced oxidation of potassium iodide by ozone** Genevieve Guéron, Jules Guéron and Marcel Prettre Compt. rend. 201, 1376-8 (1935), cf. C. A. 29, 6372.  $\text{CaO}$  from a reservoir of  $\text{O}_3$  contg. 2-7 mg.  $\text{O}_3/\text{l.}$  is passed at a measured rate (a) directly into aq. KI, and (b) into a similar soln. after diln. to a known extent by air, and the results are compared, the total oxidation and that due to formation of  $\text{IO}_3^-$  are separately det'd. As ozone becomes more dil. the total oxidation increases, but that due to  $\text{IO}_3^-$  decreases, the variations being almost linear. C. A. S.

**Structure of nitrogen peroxide as deduced from action of potassium iodide** Maurice Dodé. Compt. rend. 201, 1378-80 (1935).—When  $\text{N}_2\text{O}$  reacts in a vacuum with aq. KI of varying concn part is hydrolyzed to  $\text{HNO}_3$  and  $\text{HNO}_2$ ; this proportion increases from 14% with a 60% soln. to 85% with a 0.5% soln., the balance reacting according to  $\text{N}_2\text{O} + 2\text{KI} = 2\text{KNO} + \text{I}_2$ , which implies either  $\text{ON-O-O-NO}$  or  $\text{O}_2\text{N-N-O}$  as the structure of  $\text{N}_2\text{O}$ .

With solid KI the reaction is  $2\text{N}_2\text{O}_5 + 2\text{KI} = 2\text{KNO}_3 + 2\text{NO} + \text{I}_2$ , which is best explained by the formula  $\text{O}_2\text{N}-\text{O}-\text{NO}$ . With moist KI (0.5%  $\text{H}_2\text{O}$ ) both reactions occur simultaneously. C. A. Silberrad

Phosphorus pernitride,  $\text{P}_4\text{N}_8$ , Henri Mureau and Georges Wetroff *Compt rend* 201, 1391-3 (1935). —When  $\text{P}_4(\text{NH})_6$ , obtained by heating the product of the action of liquid  $\text{NH}_3$  on  $\text{PCl}_5$ , is heated at  $500-600^\circ$  there is not, as thought by Renaud (C. A. 29, 5033), formed  $(\text{PN})_2$ , but  $\text{H}_2$  and  $\text{P}_4\text{N}_8$  are also evolved and the pernitride  $\text{P}_4\text{N}_8$  results.  $2\text{P}_4(\text{NH})_6 = \text{P}_4\text{N}_8 + 3\text{H}_2$ , followed to some extent by  $\text{P}_4\text{N}_8 + 3\text{H}_2 = 6\text{NH}_3 + (2x - 6)\text{PH}_3 + (10 - 2x)\text{P}$ .  $\text{P}_4\text{N}_8$  is white, insoluble, nonvolatile and spontaneously inflammable in the air. Heated for 12 hrs with  $\text{H}_2\text{O}$  at  $215^\circ$  it hydrolyzes:  $\text{P}_4\text{N}_8 + 15\text{H}_2\text{O} = 2(\text{NH}_4)_2\text{HPO}_4 + \text{NH}_4\text{H}_2\text{PO}_4 + \text{NH}_4\text{HPO}_4$ . The phosphate is, however, partially converted into phosphate with evolution of  $\text{H}_2$ . Treated similarly,  $\text{P}_4\text{N}_8$  hydrolyzes according to  $\text{P}_4\text{N}_8 + 12\text{H}_2\text{O} = 2(\text{NH}_4)_2\text{HPO}_4 + \text{NH}_4\text{H}_2\text{PO}_4$ . Heated in a vacuum above  $750^\circ$ ,  $\text{P}_4\text{N}_8$  evolves  $\text{N}_2$ , forming  $\text{PN}$  identical with that previously obtained (C. A. 28, 3997).  $\text{PN}$  dissociates above  $840^\circ$  into  $\text{P}$  and  $\text{N}_2$ , and only hydrolyzes on heating with  $\text{H}_2\text{O}$  at  $215^\circ$  for 3 days, with formation of  $\text{NH}_4\text{H}_2\text{PO}_4$ ,  $\text{NH}_4\text{HPO}_4$ , and  $\text{H}_2$ . C. A. Silberrad

Hydration of chromic chloride in heavy water. Georges Champetier *Compt rend* 201, 1118-20 (1935). —The changes in constitution of solns of  $\text{CrCl}_3$  in  $\text{H}_2\text{O}$  represented by  $[\text{Cr}(\text{H}_2\text{O})_6]\text{Cl}_3$  (I)  $\rightleftharpoons [\text{Cr}(\text{H}_2\text{O})_5\text{Cl}]\text{Cl}_2$  (II)  $\rightleftharpoons [\text{Cr}(\text{H}_2\text{O})_4\text{Cl}_2]\text{Cl}$  (III) is shown by cond. dens. to occur similarly in 98.5%  $\text{D}_2\text{O}$ , the only differences being that the cond. in  $\text{D}_2\text{O}$  is less owing to the smaller mobility of the ions therein (cf. Lewis and Doody, C. A. 27, 4730), and that the changes proceed more slowly, that of II to III being at about  $1/3$  of the rate in  $\text{H}_2\text{O}$ . C. A. Silberrad

A method for the preparation of pure carbon monoxide. G. Meyer, R. A. Henkes and A. Slooff *Rec trav chim* 54, 797-9 (1935). —Pure  $\text{Ni}$  was made by the reduction of a mixt. of the hydrides of  $\text{Ni}$  at  $350^\circ$ . The  $\text{Ni}$  was allowed to react with  $\text{CO}$  in a bomb under a pressure of 60 atm. and the  $\text{Ni}(\text{CO})_4$  was decompd. by passing through a hot tube at  $200^\circ$ . The  $\text{CO}$  produced, which was purified by passing through a liquid-air trap and collected over  $\text{H}_2$ , analyzed from 99.7 to 100.3%. A. A. V.

The nitroprusside reagent and its changes by the action of alkali sulfide. E. Justin Mueller, *J. pharm. chem.* 22, 496-503 (1935). —The finally adopted formula for  $\text{Na}$  nitroprusside (A) is  $\text{Na}(\text{CN})_5\text{Fe}(\text{NO})$  (NO) (CN) (Na) Fe (CN) (Na). One mol. A reacting with  $2\text{Na}_2\text{S}$  forms  $4\text{NaCN}$  plus an unstable purple-red compd. (B).  $\text{Na}(\text{CN})_5\text{Fe}(\text{SNO})\text{Na}$  (SNO)Na Fe (CN) (Na). With  $\text{Na}_2\text{O}$ , 2B rapidly loses the (SNO)Na, complex which in the nascent state reacts with 1 of the 2 remaining  $\text{Na}_5(\text{CN})_5\text{Fe}$  groups, forming  $4\text{HCN}$ ,  $2\text{NaCN}$ ,  $2\text{NaNO}$ , and the ferrous nitrosulfide,  $\text{FeS}_2(\text{NO})\text{Na}$  (or  $\text{FeS}_2(\text{NO})\text{Na}_2$ , Naquet, in Naquet's Dictionary, 1, 1106). The other  $\text{Na}_5(\text{CN})_5\text{Fe}$  in the presence of  $2\text{NaOH}$  forms  $\text{Na}_2\text{Fe}(\text{CN})_6 + \text{Fe}(\text{OH})_2$ , the latter gradually oxidizes, and upon acidulation, Prussian blue is formed. The various phases of these reactions can be made visible by the touch method on wool. Ten references.

S. Waldbott  
Chemical investigation of silicates. III. Behavior of talc toward  $\text{MgO}$  and chlorides of magnesium, cobalt and manganese on heating. Erich Thilo *Z. anorg. allgem. Chem.* 225, 40-63 (1933); cf. C. A. 27, 4185. —On heating mixts. of talc with chlorides of  $\text{Mg}$ ,  $\text{Co}$  and  $\text{Mn}$  at about  $800^\circ$ , such products as  $\text{MgSiO}_3$ ,  $\text{SiO}_2$  and orthosilicate and such mixed crystals as  $(\text{Mg}, \text{Mn})\text{SiO}_3$  are formed. The definite compd.  $\text{Mg}_2\text{Si}_2\text{O}_7$  was not recognized. This behavior is different from that of pyrophyllite, which reacts with  $\text{CoO}$  and  $\text{MgO}$  but not  $\text{MnO}$  and does not form orthosilicate. It is concluded that the crystal structures of talc and pyrophyllite, while very similar, are not identical. Pyrophyllite reactions depend upon octahedral voids, which in the structure of talc are occupied by  $\text{Mg}$ . For talc to react the lattice must be completely altered. For the dehydration of talc, therefore, a greater quantity of

energy is required, accounting for the occurrence of orthosilicate in the reaction products. H. Stoertz

Formation of "persulfate." Hugo Ditz, *Z. anorg. allgem. Chem.* 225, 90-2 (1935). —Comments upon an article by Krauss and Oettner (C. A. 29, 5763). D. claims that their statement that he was unable to obtain persulfate from  $\text{H}_2\text{O}_2$  and  $\text{Na}_2\text{SiO}_3$  is incorrect. H. S.

The existence of orthosilicic acid,  $\text{H}_2\text{SiO}_4$ . E. Zintl and W. Hauke *Z. physik. Chem.* A174, 312-16 (1935). —With the help of a K hl camera powder pictures of  $\text{HNO}_3$  -  $\text{H}_2\text{O}$  were produced and compared with diagrams of the 5 low pressure modifications of  $\text{NH}_4\text{NO}_3$  and cryst.  $\text{H}_3\text{PO}_4$ . The authors agree with W. Beltz, *Nachr. Ges. Wiss. G ttingen Ges. Mitt. Math.-physik. Klasse* 111 1, 95 (1935), that the monohydrate of  $\text{HNO}_3$  has the structure  $\text{H}_2\text{NO}_4$ . R. H. Baechler

Preparation of selenic acid. L. I. Culbertson and C. B. King *J. Am. Chem. Soc.* 58, 180 (1936). —Oxidation of 150 g.  $\text{SeO}_2$  with 500 g. of 30%  $\text{H}_2\text{O}_2$  for 24 hrs at room temp., followed by refluxing for 12 hrs while  $\text{O}_2$  is bubbled through the soln. (more  $\text{H}_2\text{O}_2$  may have to be added) and removal of the  $\text{H}_2\text{O}$  on a steam bath and finally at  $150-60^\circ$  and 4-6 mm., gives 192-5 g.  $\text{H}_2\text{SeO}_4$ , m.  $57-6^\circ$ . C. J. West

The mixed polyhalides of titanium. G. P. Luchinski and A. I. Likhacheva. *Z. anorg. allgem. Chem.* 224, 420-6 (1935). — $\text{TiCl}_3$  mixes with  $\text{Br}$  in all proportions, forming mixts. of intense red color. The melting curve of the system  $\text{TiCl}_3\text{Br}$  has 3 eutectic and 2 singular points. Two mol. compds. are formed,  $\text{TiBrCl}_2$  and  $\text{TiBr}_2\text{Cl}$ . Under the influence of heat and of  $\text{H}_2\text{O}$  the entire  $\text{Br}$  splits off from both compds. The  $\text{Ti}$  can be detd. colorimetrically in the presence of the  $\text{Br}$  compds. and after removal of the  $\text{Br}$  by chloroform extn. M. M.

Spontaneous decomposition of polythionate solutions. A. K rtenacker, A. M schin and F. Stastny. *Z. anorg. allgem. Chem.* 224, 399-419 (1935). —Investigation of the influence of  $\text{pH}$  on the decompn. of K salts of polythionates showed: (1) Hexathionates decompose in the region between  $\text{pH} = 8.9$  and  $\text{pH} = 0.3$ , smoothly and without the formation of by-products, into  $\text{S}$  and pentathionate. (2) Pentathionate in weakly acid soln. ( $\text{pH} = 4.6$  and  $\text{pH} = 3.8$ ) decomposes without formation of secondary products into  $\text{S}$  and tetrathionate. In neutral and weakly alk. soln. ( $\text{pH} = 7$  and  $\text{pH} = 8.9$ ) small amts. of trithionate, thiosulfate and sulfate appear probably on account of the decompn. of the tetrathionate. (3) The tetrathionate decompn. is more complicated. With  $\text{pH}$  not more than 8.9 the known decompn. into tri- and pentathionate occurs. With  $\text{pH} = 11.5$   $4\text{S}_2\text{O}_8^{2-} + 6\text{OH}^- = 2\text{S}_2\text{O}_4^{2-} + 5\text{S}_2\text{O}_3^{2-} + 3\text{H}_2\text{O}$ . (4) The trithionate decomps. thus: (1)  $\text{S}_3\text{O}_6^{2-} + \text{H}_2\text{O} = \text{S}_2\text{O}_3^{2-} + \text{SO}_4^{2-} + 2\text{H}^+$  at  $\text{pH}$  12 or less, quantitatively at  $\text{pH}$  8.9-5.6, (2)  $2\text{S}_3\text{O}_6^{2-} + 6\text{OH}^- = \text{S}_2\text{O}_4^{2-} + 4\text{SO}_3^{2-} + 3\text{H}_2\text{O}$  only at high  $\text{OH}^-$  concn. M. McMahon

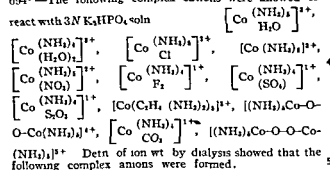
The systems alkali oxides- $\text{CaO}$   $\text{H}_2\text{O}$ ,  $\text{SiO}_2$ - $\text{CO}_2$ . V. Stable equilibrium in the system  $\text{Na}_2\text{O}$ - $\text{SiO}_2$ - $\text{CO}_2$  and the  $\text{CO}_2$  pressure in the reaction of  $\text{K}_2\text{CO}_3$  and  $\text{K}_2\text{SiO}_3$ . Carl Kr ger and Ernst Pingas *Z. anorg. allgem. Chem.* 225, 1-23 (1935), cf. C. A. 30, 693. —Equal and  $\text{CO}_2$  pressures were detd. in the reaction between cryst.  $\text{Na}_2\text{SiO}_3$  and  $\text{Na}_2\text{CO}_3$  at various temps. between  $368^\circ$  and  $1015^\circ$ .  $\text{CO}_2$  pressure curves are shown and results indicate the equl. is bivalent. The action of  $\text{Na}_2\text{SiO}_3$  upon  $\text{Na}_2\text{SiO}_3$  was studied between  $480^\circ$  and  $790^\circ$ . A cryst. form of  $\text{Na}_2\text{SiO}_3$  was not obtained but only glass-like material, and the reaction with  $\text{Na}_2\text{CO}_3$  shows a univariant equl. curve. In studying absorption of  $\text{CO}_2$ , it was found that neither  $\text{Na}_2\text{SiO}_3$  nor  $\text{Na}_2\text{SiO}_3$ - $\text{Na}_2\text{SiO}_3$  mixts. would take up  $\text{CO}_2$ , but it is taken up by  $\text{Na}_2\text{SiO}_3$  even at temps. of  $200-300^\circ$  to form  $\text{Na}_2\text{SiO}_3$  and  $\text{Na}_2\text{CO}_3$ . In the reaction of  $\text{Na}_2\text{CO}_3$  upon the pyrosulfate  $\text{Na}_2\text{S}_2\text{O}_7$  the equl. varies between tri- and bivalent. The  $\text{p}$ - $\text{H}$  diagram and stable equl. were detd. for the system  $\text{Na}_2\text{O}$ - $\text{SiO}_2$ - $\text{CO}_2$ . In the action of  $\text{K}_2\text{CO}_3$  upon  $\text{K}_2\text{SiO}_3$  it was shown that in the system  $\text{K}_2\text{O}$ - $\text{SiO}_2$ - $\text{CO}_2$  as in the system  $\text{Na}_2\text{O}$ - $\text{SiO}_2$ - $\text{CO}_2$  there is considerable difference in the behavior of glass-like and cryst. basic bodies.

p-4 diagrams were detd. for the system  $K_2O-SiO_2-CO_2$  <sup>1</sup>

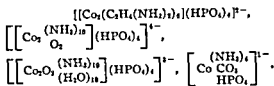
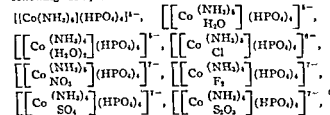
H. Stoertz  
Cuprtetra-chlorides and -bromides of some secondary and tertiary amines and alkaloids. Jean Amiel *Compt. rend.* 201, 1383-5(1935); cf. C. A. 30, 406<sup>1</sup>.—By similar methods  $[CuX_4](BH)$ , have been prepd., where X = Cl and Br, B = NHEt<sub>3</sub>, NHPMe, NHPPhEt, NPBMe, NPhEt<sub>3</sub>, and also  $[CuCl_4](NEt_3H)$ . Except for  $[CuCl_4](NHEt_3H)$ , which is green, non-deliquescent and very sol in COMe<sub>2</sub>, all closely resemble the compds previously prepd. Similar compds were obtained with some alkaloids:  $[CuX_4](B'H_2)$ , where X = Cl and Br, B' = nicotine or quinine, with strychnine  $[CuX_4](B'H)$ , with nicotine also  $[CuCl_4](B'H_2)$  H<sub>2</sub>O, and with brucine  $[CuCl_4](B'H)$ . C. A. Suberrad

A new group of complex compounds—complex compounds whose central ion is a complex cation. III. Complex phosphato compounds with complex cobalt cations as central ions H. Bruntzinger and H. Osswald. *Z. anorg. allgem. Chem.* 225, 33 7(1935), cf. C. A. 30, 694<sup>1</sup>.—The following complex cations were allowed to

react with 3N  $K_3HPO_4$  soln



$(NH_3)_6]^{3+}$  Detn. of ion wt by dialysis showed that the following complex anions were formed.



H. Stoertz  
Complexes of boric acid with one and two molecules of polyhydric alcohols in aqueous solution J. Böseken, N. Vermaas, W. H. Zaayer and J. L. Leefers *Rec. trav. chim.* 54, 853-60(1935).—Measurements of the rotation of polarized light by solns. of mannitol in H<sub>2</sub>O with NaBO<sub>2</sub> show the existence of complex ions of the formula BD<sup>-</sup> and BD<sub>2</sub><sup>-</sup>, where D represents a mol. of a polyhydric alc. Measurements on a soln. contg. sorbitol and NaBO<sub>2</sub> also showed the presence of the ions BD<sub>2</sub><sup>-</sup> and BD<sup>-</sup>.

Arthur A. Vernon  
Composition and properties of potassium phosphotungstate E. A. Nikitina *J. Gen. Chem.* (U. S. S. R.) 5, 1133-8(1935).—Analyses show that the compn. of H<sub>2</sub>O-free K phosphotungstate is 3K<sub>2</sub>O·P<sub>2</sub>O<sub>5</sub>·24WO<sub>3</sub>; the H<sub>2</sub>O-contg. mol. is K<sub>2</sub>H<sub>4</sub>[P(W<sub>2</sub>O<sub>7</sub>)<sub>4</sub>]. Its properties were studied with a view to utilizing the formation of this compd. as a method of quant. analysis for K. The ppt. formed in the reaction of a K salt soln. with H<sub>2</sub>[P(W<sub>2</sub>O<sub>7</sub>)<sub>4</sub>] is microcryst. It is difficult to filter and it settles very slowly. The salt is sol. in H<sub>2</sub>O but difficultly sol. in HCl soln. Its soly. in 18% HCl is 0, 0, 0, 0, 19, 0, 44, 0, 67 and 0, 80 g. per 100 g. soln. at 0, 20, 43, 53, 70, 80 and 90°, resp. H<sub>2</sub>[P(W<sub>2</sub>O<sub>7</sub>)<sub>4</sub>] can be used for the detn. of K in the absence of Na or for detection of K in the presence of Na. S. L. Madorsky

Stability of sodium aluminate solutions V. D. D'yachkov and O. S. Kozhukhova *J. Gen. Chem.* (U. S. S. R.) 5, 1139-43(1935).—The stabilizing effect of an excess of alkali on Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub> solns. increased sharply at Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> = 1.4-1.5. The range of lowest stability for any given ratio Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> is about 120-180 g. of Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub> per l. Agar-agar is a good stabilizer for Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub> solns. S. L. Madorsky

Asymmetric adsorption of complex salts on quartz (Tsuchida, *et al.*) 2 Asymmetric photochem. decomposition of complex salts (Tsuchida, *et al.*) 3.

## 7—ANALYTICAL CHEMISTRY

W. T. HALL

Theory of electrocapillary method of qualitative analysis III. S. I. D'yachkovskii. *J. Gen. Chem.* (U. S. S. R.) 5, 728-30(1935); cf. C. A. 28, 1625<sup>1</sup>.—The electrocapillary method of qual. analysis is explained on the basis of the movement of superimposed layers of anions and cations which arrange themselves with respect to the moist filter paper placed between the electrodes. In case of simple dipoles (e. g., NaCl), only electrostatic forces act between the layers. In case of complex heteropolar mols. (e. g., NH<sub>4</sub>Cl), chem. forces act in addn. to electrostatic forces. S. L. Madorsky

Application of organic reduction-oxidation systems in qualitative analysis. I. Theory of the application in oxidation processes Leonid M. Kul'berg *J. Gen. Chem.* (U. S. S. R.) 5, 1085-90(1935); cf. C. A. 29, 4280<sup>1</sup>. Application of organic reduction-oxidation systems in quantitative analysis II. Theory of application in colorimetry. Oxidation processes *Ibid.* 1240-5.—A general discussion, with math. treatment, of the use of leuco dyes in the detection and colorimetric detn. of various inorg. compds. based on the exact coordination of their resp. oxidation-reduction elec. potentials. In the preliminary communications various factors, such as temp., concn.,  $\mu_n$ , capable of affecting the reactions, and the literature are discussed. Twenty references. C. B.

Methods of quantitative Röntgen analysis. N. D. Borisov and P. I. Kisel'gof. *Zavodskaya Lab.* 4, 800-4 (1935), cf. C. A. 29, 1355<sup>1</sup>.—A review with about 30 references. Chas. Blanc

Microchemical spectral analysis in high-frequency sparks A. Schleicher and N. Brecht-Bergen. *Z. anal. Chem.* 103, 198(1935), cf. C. A. 29, 5765<sup>1</sup>, Gerlach and Gerlach, C. A. 27, 2909.—Further details are given. W. T. H.

Exact measurement of volume and exact titration R. Gol'tz *J. Gen. Chem.* (U. S. S. R.) 5, 779-82(1935).—A discussion of errors involved in volumetric titration and suggestions for reducing these errors to a min. S. L. Madorsky

Vacuum titration T. Thunberg *Skand. Arch. Physiol.* 72, 291-4(1935).—Description of procedure. S. Morgulis

Volumetric determinations in strongly alkaline solutions. III. Titration of thallium and cerium with hypobromite O. Tomiček and M. Jašek *J. Am. Chem. Soc.* 57, 2499-11(1935), cf. C. A. 29, 5376<sup>1</sup>.—Thallous soln. in an alk. soln. are oxidized smoothly to insol. Ti(OH)<sub>3</sub> by means of 0.05 M NaBrO soln., the end point of the reaction can be detd. potentiometrically. This provides a suitable method for standardizing NaBrO solns. with Ti<sub>2</sub>CO<sub>3</sub> or Ti<sub>2</sub>SO<sub>4</sub> as standard. Cerous salts can be ti-



trated similarly in 20-30%  $K_2CO_3$  soln but it is necessary to work in an indifferent atm. W. T. H.

A new method for the determination of the water contents of liquids and solids. A contribution to the treatise of Karl Fischer Richter *Angew Chem* 48, 776(1935); cf. C. A. 29, 6532. It was found that Fischer's I method can be applied to foodstuffs with only a few exceptions. Results are reported on wheat flour, powd cocoa, margarine, soy oil, marmalade, malt extract and cheese. Good agreement is shown with detns by other methods, except for cheese which gave a low result with the I method, as it was not possible to subdivide the substance sufficiently.

Karl Kammermeyer

Use of resorcinol in chemical analysis. II. Alan H. Ware *Chemist and Druggist* 123, 282(1935), cf. C. A. 28, 7195. Trease and Tincey, C. A. 20, 3777. (1). *Org. halogen deriv.*, 1 e,  $CHCl_3$ ,  $CHBr_3$ ,  $CHI_3$ ,  $CCl_4$ ,  $CH(OH)_3$ ,  $CCl_3CHOH$ ,  $CH(OH)COH$ ,  $CCl_3CO_2H$ ,  $CCl_3$ ,  $C_6H_5CO_2H$ , give a deep pink, carmine or brownish carmine color when 0.05 g. solid or 1 cc. liquid substance is heated for 1 min with 0.05 g. resorcinol (I), 10 cc. of 5% KOH and a little Cu as catalyst. Cool, and if necessary, boil again for 5 min. "Chlorbutol" gives a neg. result. (2) If Cu is omitted,  $CCl_3$  gives no characteristic color, with  $CHI_3$  the test is then indistinct. (3) With the above *Cl derivs.* contg. a CO group, also with  $MeCHClCCl_3$ ,  $CH(OH)_3$ , if 5% KOH is replaced by 2.5% aq.  $NH_4OH$ , a brownish carmine color is produced, when diluted with  $H_2O$  until pink or yellow in transmitted light, a sp. intense green fluorescence is seen. (4) To test for  $C_6H_5$ ,  $CCl_3$ , warm it with A in a dry test tube, dissolve in aq. alkali and dil. with  $H_2O$ , a strong green fluorescence is obtained. If heated with KOH from the start, the test is neg. (5) To test *nitriles*, mix 2 cc. aq. soln with 0.05 g. A and allow 3 cc. N-free  $H_2SO_4$  to run down the side of the tube, then shake lightly. Let stand 2 min., then add 5 cc.  $H_2O$ . Add 5 cc. of the mixt. to 5 cc. coned. cooled aq.  $NH_4OH$ , with  $NH_4$  always in excess. A rich scarlet fluorescence is seen whose intensity by comparison with solns. of known strength is a measure of the nitrite content. Dln. of  $1 \times 10^{-4}$  shows a wine-purple color,  $2 \times 10^{-4}$  purple violet,  $5 \times 10^{-4}$  blue violet. This test permits ready detection of nitrite in coned.  $H_2SO_4$ .

S. Waldbott

The Mohler-Denigès reagent for tartaric acid. C. H. Liberali *Rev. chim. e. farmacia* 1, 12-15(1935).—Mohler-Denigès reagent (to 2 or 3 cc. coned.  $H_2SO_4$ , add 1-2 drops of aq. soln of 2% resorcinol and 1-2 drops of soln to be assayed, mix, heat to 108-110°, red-violet color) for tartaric acid, although not specific, is to be recommended. In the presence of chlorates, nitrates, nitrites, bromates and iodates, the color is changed. Gluconic, lactic and pyruvic acids are the cause of error, as they give the same color as tartrates. F. S. G. B.

The use of 5-methyl-8-hydroxyquinoline in analysis. Carlos E. Gietz and Adolfo S. *Anales asoc. quim. Argentina* 23, 45-57(1935).—Addn. of 5-methyl-8-hydroxyquinoline to a 5% NaOAc-10% AcOH soln will show, by various color reactions, the presence of 1,000,000  $Fe^{+++}$ , 1,000,000  $Pd^{++}$ , 1,300,000  $Cu^{++}$ , 1,300,000  $Ti^{+++}$ , 1,000,000  $Mo$ , 1,300,000  $W$  and in a 0.2 N NaOH 10% said by Na tartrate soln, 1,250,000  $Cu$  or  $Fe^{++}$  and 1,500,000  $Pd^{++}$ . The sensitivity of other ions is less. E. M. Symmes

Diphenylpicarbazide. C. F. Miller. *Chemist Analyst* 25, No. 10, 11(1936).—A method of prep. the reagent is described, a procedure is given for detg. Cr colorimetrically, and tests for Cd and Hg are given. The more or less fugitive color reactions with Cu, Ag, Pb, Mg, Zn, Fe, Ni, Co and Mn are given. W. T. H.

Colorimetric method of determining small quantities of arsenic in iron and steel. V. Ya. Tartakovsky. *Zarodskaya Lab* 4, 750-4(1935).—The method is based on that of Denigès and depends on the formation in soln of  $H[As(MoO_4)_2]$  which with  $SnCl_2$  in dil. HCl gives a blue compd.  $H[As(MoO_4)_2]ONaO_2$ . C. B.

Determination of trivalent and quaquevalent arsenic in the presence of each other. I. M. Dubrovyn. *Zarod-*

*skaya Lab* 4, 888-91(1935).—Dissolve a sample in 50% HCl, dil. and titrate  $As^{+++}$  at 50-60° with 0.1 N  $KBrO_3$  in the presence of methyl orange as indicator. Make the titrated soln strongly acid with HCl, introduce pyridine sulfate, distil, titrate as above and calc.  $As^{+++}$  by difference. Chas. Blanc

Application of the logarithmic sector in the spectrum analysis of barium and strontium solutions. N. V. Luzinov. *Zarodskaya Lab* 4, 901-9(1935).—The exper. were carried out with the aid of the sparing vessel of Twyman and Hitchen (C. A. 25, 5864). Accurate results were obtained in  $HNO_3$  solns. at concns. of 0.05% BaO and SrO and lower. The procedure, with math treatment, is described in detail. Chas. Blanc

New (color) test for bismuth. M. V. Gapchenko and O. G. Sheintsis. *Zarodskaya Lab* 4, 835(1935).—A mixt. of 1 g. quinine in 100 cc. alc. with 20 cc. of 25% KI gives a highly intense red-orange with Bi with sensitiveness of 0.02 mg. Bi in 1 (50,000). By the spot method 1.6  $\gamma$  Bi in 1 drop can be detected. This reagent gives with  $Pb^{++}$  yellow,  $Hg^{+}$  green,  $Hg^{++}$  white, Ag<sup>+</sup> very faint yellow,  $Sb^{+++}$  yellow, and with  $Cu^{++}$  and  $Fe^{+++}$  brownish yellow (liberation of I). All these cations, except the last 2, do not affect the detn. In the presence of  $Cu^{++}$  and  $Fe^{+++}$ , place 1 drop of 10%  $NaHSO_4$  on the test paper said with the reagent and follow with 1 drop of the acidified soln to be tested. Chas. Blanc

Possibility of determining bismuth as basic carbonate. I. Friedrich Hecht and Richard Reissner. *Z. anal. Chem.* 103, 180-9(1935), cf. C. A. 30, 693<sup>a</sup>, 696<sup>a</sup>.—If a soln of  $Bi(NO_3)_3$  contg. not over 60 mg. Bi in 100 ml. is treated with an excess of a satd. soln of  $(NH_4)_2CO_3$ , added in small portions, and is then boiled a few min., filtered, washed with hot water and dried in the app. of Dworzak and Reich-Rohrig (C. A. 25, 5465) in a current of air for 2-3 hrs., the ppt. can be weighed as  $(BiO)_2CO_3$ . The results obtained in 5 analyses were good, the greatest error was 0.5 mg. with a sample weighing 0.22 g.  $Bi_2O_3$ . The procedure is satisfactory for analyzing as little as 1 mg.  $Bi_2O_3$ . W. T. H.

Fundamentals and applications of absolute colorimetry. XII. Determination of iron. A. Thiel and O. Peter. *Z. anal. Chem.* 103, 161-6(1935); cf. C. A. 29, 71<sup>a</sup>.—The method of Lapin and Kill, C. A. 26, 789, which depends upon the formation of a red-colored soln by the action of sulfosalicylic acid upon  $Fe^{+++}$  in a soln. slightly acid with HCl or upon the formation of a yellow-colored soln by both  $Fe^{++}$  and  $Fe^{+++}$  in the presence of  $NH_4$ , is suitable for the detn. of both total Fe and  $Fe^{+++}$  by abs. colorimetry. From 0.24 to 8.0 mg. per l. of Fe can be detd. with remarkable accuracy. W. T. H.

Rapid analysis of iron, ferrous and ferric oxides in a mixture. S. P. Leiba and Yu. I. Dolgina. *Zarodskaya Lab* 4, 743-7(1935).—The modification of the Wilner and Merck method for detg. Fe, FeO and  $Fe_2O_3$  in a mixt. by oxidation of Fe with  $HgCl_2$  is based on the observation that a part of the  $FeCl_3$  is reduced by Hg and  $HgCl$  formed in the process of solvation of the mixt. by boiling with HCl. Det. metallic Fe in the mixt. by the method of Wilner and Merck. For the detn. of FeO, introduce 5 g. of powd. sample into a dry flask (100 cc.) contg.  $CO_2$ , 5 g.  $HgCl_2$  and 30 cc.  $H_2O$  (preferably charged with  $CO_2$ ). Boil the mixt. in a strong current of  $CO_2$  for 1 min. Allow to cool and add 30 cc. of coned. HCl with shaking. Dil. the mixt. to 100 cc. Remove 15-20 cc. of the clear supernatant soln, dil. to 200 cc., add 15-20 cc. of the Renhardt soln and titrate with  $KMnO_4$ . Calc. FeO from the total value for FeO and Fe. Det. the total Fe in the mixt. by the Zimmermann-Renhardt method and calc.  $Fe_2O_3$ . The method is accurate to 0.04% for Fe and FeO and 0.2% for  $Fe_2O_3$ . Chas. Blanc

Determination of small quantities of iron in phosphates with sulfosalicylic acid. V. M. Peshkova and A. D. Egorov. *Zarodskaya Lab* 4, 835-7(1935), cf. Lapin and Kill, C. A. 26, 789.—Good results were obtained by colorimetric detn. of  $Fe^{++}$  and  $Fe^{+++}$  in  $NH_4OH$  soln with sulfosalicylic acid as reagent. Chas. Blanc

Determination of lead in alloys. M. V. Gapchenko and

O. G. Shelutsky, *Zarodskaya Lab.* 4, 868-70 (1935).—(1) Treat the filtrate from  $\text{SnO}_2$  and  $\text{SnO}_3$  with an excess of  $\text{NH}_4\text{OH}$  (to dissolve any  $\text{Cu}(\text{NO}_3)_2$ ), add  $\text{cryst. AcO-NH}_4$  to dissolve  $\text{Pb}(\text{OH})_2$  and  $10\%$   $\text{NaHSO}_4$ , filter off  $\text{PbSO}_4$  in a Gooch crucible, wash with cold  $\text{H}_2\text{O}$ , dry at  $105^\circ$  and weigh. (2) Filter off  $\text{H}_2\text{SO}_4$ , wash it free from  $\text{SO}_3^{2-}$  (test with I and starch soln.), dissolve  $\text{PbSO}_4$  in the filtrate with 10 cc. of 2 N  $\text{NaOH}$  and wash the filter to a neutral reaction (litmus). Introduce into the filtrate a standard soln. of I and after 10 min.  $25\%$   $\text{H}_2\text{SO}_4$  and titrate the excess of I with  $\text{Na}_2\text{S}_2\text{O}_4$ . Chas. Blanc.

Rapid method of analysis of red lead Stanley Kettle *Chemist Analyst* 25, No. 1, 6-8 (1936).—Treat 5-10 g. of sample with 20-40 ml. of  $50\%$   $\text{HNO}_3$  and measure the vol. of  $1.2\%$   $\text{H}_2\text{O}_2$  required to dissolve the  $\text{Pb}_2\text{O}_3$ .

Determination of nickel by the combined Chugaev-Bruck and Moore methods I. I. Pomerantsev *Zarodskaya Lab.* 4, 960-7 (1935).—The Ni is pptd. with dimethyl glyoxime, the ppt. is removed, dissolved in  $\text{HCl}$  and the Ni titrated with  $\text{KCN}$ . Chas. Blanc.

A new microchemical reaction for potassium A. Kniga *Akhil (U. S. S. R.)* 1935, No. 7, 22-3.—The finely ground sample was fused on a Pt wire with  $\text{Na}_2\text{CO}_3$ , treated with  $\text{HCl}$ , put on a microscope glass, heated to remove excess  $\text{HCl}$  and treated with 1 drop  $\text{NaMnO}_4$ . After 1-2 min. the formation of black-violet crystals of  $\text{KMnO}_4$  was noticed through the microscope. Na, Mg, Ca, Sr, Ba, Zn, Al, Cd, Ni, Bi and Cu did not interfere with the reaction. Mn, Co, Cr, iodine, evanide, ferrocyanide and other reducing anions and org. matters did interfere. The same reaction was obtained with salts of Ni, (removed on ignition), Rb and Cs (both are rare). The sensitivity of this reaction is 0.04 mg. of K. A. P.

The refractometer method of determining potassium without separating the sodium M. Grindel *Khimiya i Selskoye Zemel'noye (Moscow)* No. 3, 101-6 (1935).—A discussion of the method and some results are presented. J. S. Joffe.

Rapid colorimetric method of determining silicon in cast iron and steel P. P. Fedorov *Zarodskaya Lab.* 4, 747-50 (1935); cf. Gutman and Froshutin *Khim.* C. A. 29, 7218.

Analytical determination of the form of silicon in aluminum castings Yu. A. Khachko *J. Gen. Chem. (U. S. S. R.)* 5, 1180-2 (1935). cf. C. A. 29, 1373.—An analytical method designed to differentiate between free and combined Si in Al castings is based on the fact that combined Si reacts with mineral acids (except  $\text{HNO}_3$ ) to give  $\text{SiH}_4$ , which in turn reacts with O of the air to form  $\text{SiO}_2$ . However, if a stream of  $\text{H}_2$  is passed through the soln., the  $\text{SiH}_4$  is protected from oxidation and is removed by the  $\text{H}_2$  stream. The method then consists in dissolving one sample of the Al casting in  $\text{HCl}$  in air. This gives total Si. Another sample is dissolved in  $\text{HCl}$  while a stream of  $\text{H}_2$  is passed through the acid soln. This gives free Si, since the combined Si escapes as  $\text{SiH}_4$ . The difference between the two dectns. is combined Si. S. L. M.

Determination of the fineness of silver bullion M. R. Bercovitsh *Chemist Analyst* 25, No. 1, 11-14 (1936).—It is recommended to ppt. the greater part of the  $\text{Ag}^+$  with a standard soln. of  $\text{NaCl}$ , filter, evaporate the filtrate somewhat, and titrate the remaining  $\text{Ag}^+$  with  $\text{KCN}$  soln., with ferric alum as indicator. W. T. H.

Determination of titanium in stainless steels and zirconium in ferrozirconium with the aid of 8-hydroxyquinoline. S. L. Trimbberg *Zarodskaya Lab.* 4, 783-5 (1935).—The method of detn. of Ti in Cr-Ni-Ti steels is based on their soln. in  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$ . To a soln. of 1 g. sample in 50 cc. of  $50\%$   $\text{HCl}$  or  $20\%$   $\text{H}_2\text{SO}_4$ , add 120-80 cc., add dropwise  $5\%$  cupferron until the yellow ppt. begins to turn red (pptn. of Fe). Allow to stand 40-5 min., and filter through a filter cone, filter paper pulp and wash the ppt. with  $5\%$   $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{O}$ . Ignite the ppt. with the filter and then with the addition of 10 parts of  $\text{Na}_2\text{CO}_3$  for 15-2 hrs. Decompose the melt with 50 cc. of  $50\%$   $\text{HCl}$ . Add to the soln. 1 g. of tartaric acid, neutralize with  $\text{NH}_4\text{OH}$  to methyl red, add 2.5 cc. of strong  $\text{AcOH}$

and ppt. Fe with 10 cc. of  $1\%$  "oxine" at  $70-80^\circ$  (cf. C. A. 29, 709). Ppt. Ti in the filtrate with  $5\%$  cupferron as above and ignite to  $\text{TiO}_2$ . The detn. of Zr in Zr-Fe is based on the soln. of Zr oxine in dil.  $\text{AcOH}$ . Decompose 1 g. of powder sample in 20 cc.  $\text{HNO}_3$  and 15 cc.  $\text{HF}$ . Evaporate the soln. with 5 cc.  $\text{H}_2\text{SO}_4$  to fuming. Dissolve the residue in hot  $\text{HCl}$  and add to 1 l. To 100 cc. of the soln. add 1 g. tartaric acid,  $\text{NH}_4\text{OH}$  to a neutral reaction, 3 cc. of concd.  $\text{AcOH}$  and 10 cc. of  $1\%$  "oxine". Filter off and wash the Fe ppt. and det. as usual. Det. Zr in the filtrate either by direct addn. of  $5\%$  cupferron, or by first neutralizing the filtrate with  $\text{NH}_4\text{OH}$  and acidifying with 10 cc. of  $50\%$   $\text{H}_2\text{SO}_4$  and then pptg. with cupferron and igniting the ppt. to  $\text{ZrO}_2$ . Chas. Blanc.

Colorimetric determination of titanium in the presence of bromine compounds G. P. Lushinskii and Antonina Ivanovna Likhacheva *Z. anal. Chem.* 103, 190-8; *Zarodskaya Lab.* 4, 831-5 (1935).—The colorimetric detn. of Ti by the Weller test ( $E_{\text{Fe}}^{1\%} 1\text{cm} = 15, 250\%$  (1922)) with  $\text{H}_2\text{O}_2$  gives satisfactory results in the absence of  $\text{Br}_2$  whose color in dil. solns. resembles that of the colored Ti compd. This error can be avoided, however, by taking advantage of the fact that  $\text{Br}_2$  is sol. in  $\text{CHCl}_3$ , whereas the colored Ti compd. is not. The soln. to be tested colorimetrically should not contain more than  $0.2\%$   $\text{Ti}$  and  $1\%$   $\text{Br}_2$ ; it should contain at least  $10\%$   $\text{H}_2\text{SO}_4$ . To 25 cc. of Ti soln. add 25 cc. of  $\text{H}_2\text{SO}_4$  contg.  $5\%$   $\text{H}_2\text{O}_2$ . It is important that the reagent and the soln. to be analyzed both have equal  $\text{H}_2\text{SO}_4$  concns. before mixing. After allowing the color to develop for 0.5 hr., transfer to a separatory funnel and shake 0.5 hr. with 50 cc. of  $\text{CHCl}_3$ . Draw off the aq. soln. and subject to the colorimetric comparison. W. T. H.

Determination of uranium, vanadium and iron in the presence of each other E. A. Ostroumov *Zarodskaya Lab.* 4, 754-7 (1935).—The volumetric detn. of U, V and Fe in a mixt. is made in 3 aliquot parts of the soln. V is detd. by the Hammer method of reducing V with  $\text{FeSO}_4$ , oxidizing the excess of  $\text{FeSO}_4$  with  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  and titrating  $\text{V}_2\text{O}_5$  soln. with  $\text{KMnO}_4$ . V and Fe are detd. by the Zimmermann-Reinhardt method by reducing  $\text{V}_2\text{O}_5$  to  $\text{V}^{4+}$  with  $\text{SnCl}_2$  and titrating with  $\text{KMnO}_4$ . U, V and Fe are detd. by Semeva's method (C. A. 19, 224) as modified by Dumov and Volodina (C. A. 28, 71979). By substituting  $\text{H}_2\text{SO}_4$  for  $\text{HCl}$  in the last procedure, the results are improved. Chas. Blanc.

Rapid determination of zinc in granulated slags. M. I. Shubin *Zarodskaya Lab.* 4, 85-7 (1935).—The method of detg. Zn in slags, obtained in smelting of waste Cu-Zn, depends on electrolytic deposition of Zn in alk. or  $\text{NH}_4\text{OH}$  solns. in the presence of the hydroxides of Ca, Al, Fe, Pb, Sb and Sn. After sepn. of  $\text{SO}_2$  by filtration and Cu by electrolysis, the  $\text{H}_2\text{SO}_4$  soln. is heated to  $40-50^\circ$  and treated with 5 g.  $(\text{NH}_4)_2\text{SO}_4$ , 7-8 drops of  $10\%$   $\text{H}_2\text{O}_2$  and an excess of  $2\%$   $\text{NH}_4\text{OH}$ . The mixt. is directly electrolyzed at  $20^\circ$  with a Pt electrode, with stirring, for 20-30 min. The electrode is washed with running water and alc. and dried at  $70-80^\circ$ . The detn. is accurate to  $\pm 0.1\%$ . C. B.

Separation of zinc from cobalt based on a new method for reducing post precipitation John R. Caldwell and Harvey V. Moyer *J. Am. Chem. Soc.* 57, 2275-7 (1935).—Adjust the pH of the  $\text{Cl}^-$ -free soln. contg. about 0.25 g. of Zn so that the hydroxides just remain in soln., and 6-8 g. of  $(\text{NH}_4)_2\text{SO}_4$ , d. l. to 230-300 ml. and add 0.2 ml. of acrolein. Introduce a rapid stream of  $\text{H}_2\text{S}$  for 20 min., add 5-10 ml. of  $0.02\%$  relative soln., filter after standing 15 min. and wash with cold water. Acrolein reacts with  $\text{H}_2\text{S}$  at the surface of the ZnS particles and thereby reduces the tendency of  $\text{CoS}$  to ppt. with the  $\text{ZnS}$ . W. T. H.

Microdetermination of carbon Wilhelm Littens and Walter Christian *Fis. Ann. Z.* 281, 310-13 (1935).—The org. material is oxidized in a combustion tube prepd. according to Pregl. The  $\text{CO}_2$  is absorbed in 2 tubes with 0.1 N  $\text{NaOH}$ , which is measured out from a microburet. To aid the absorption the tubes are filled with 0.5 g. Ag filings. The absorbed  $\text{CO}_2$  is liberated and measured in a Warburg app. The method for calg. the results is

discussed The C is detd in 0.2-0.6 g of substance

S Morgulis  
Determination of pale red phosphorus in commercial red phosphorus A A Korinfski Zavodskaya Lab. 4, 762-4(1935) —The detn depends on the pptn of 2 mols of Cu from  $\text{CuSO}_4$  by 1 mol of pale red P (inflammable), and the inability of the red modification of P to give this reaction

Chas. Blanc  
Determination of sulfur in refined copper M R. Beronvitch *Chemist Analyst* 25, No 1, 4-5(1936) —Heat 200 g of the sample to redness in  $\text{H}_2$ , absorb the  $\text{H}_2\text{S}$  in ammoniacal  $\text{CdSO}_4$ , and finish the analysis as in detg S in steel

W T H  
Rapid determination of sulfur in ores and other products L P Elenovitch and V G Makarova *Zavodskaya Lab* 4, 839-61(1935) —The sample is ignited with a mixt of ZnO and  $\text{Na}_2\text{CO}_3$  and the melt extd with  $\text{H}_2\text{O}$  After pptn of  $\text{SO}_4^{2-}$  in the filtrate with  $\text{BaCl}_2$ , the soln is neutralized with  $\text{NH}_4\text{OH}$  to methyl orange and treated with an excess of 0.2 N  $\text{K}_2\text{Cr}_2\text{O}_7$ . The filtrate is dil to a definite vol, and an aliquot part, after the addn of an excess of  $\text{HCl}$ , 10% KI and starch soln, is titrated with  $\text{Na}_2\text{S}_2\text{O}_3$

Chas Blanc  
Rapid determination of sulfur in ores, concentrates and flotation tailings L M Tol'son and E I Dyadicheva *Zavodskaya Lab*, 4, 873 9(1935), cf *C A* 29, 7865-7

The combustion is carried out in an elec furnace at  $900^\circ$  for 10 min in a current of air conducted at an exact rate The combustion gases are absorbed in neutral 1.5%  $\text{H}_2\text{O}_2$ , and the  $\text{SO}_2$  is titrated with 0.1 N NaOH in the presence of neutral red as indicator The analysis is completed in 20 min with uniformly accurate results by following exactly the procedure which is described in detail Cf Khlopin, *C A* 29, 787, Gerke, *C A* 29, 787, C B

Organic flocculating agents in the quantitative precipitation of zinc sulfide John R. Caldwell and Harvey V. Moyer *J Am Chem Soc* 57, 2372-4(1935) —If 0.5-2 mg of gelatin or 2-5 mg of agar agar is added to a Zn soln of appropriate pH before or after treatment with  $\text{H}_2\text{S}$ , complete flocculation of 0.3 g ZnS takes place instantly Pptn can take place at room temp and the soln can be filtered in 15 min after the pptn Satisfactory seps were obtained from all other ions of the third group except Co, and the ZnS can be washed with cold water

W T H  
Amalgamation as an aid in metal analysis Wolfgang Boehm *Metall u Erz* 32, 543-4(1935) —Large quantities of amalgam-forming heavy metals can be sepd from small quantities of alkali metals with the aid of Hg

H S  
Rapid analysis of basic Martin slags V P Remin *Zavodskaya Lab* 4, 965-6(1935) —By quick immersion of hot slags in  $\text{H}_2\text{O}$  for a few sec they become sol in acids After a short drying to remove retained  $\text{H}_2\text{O}$ , the slags are powd and metallic Fe is extd with a magnet  $\text{SiO}_2$  is detd by evapg a sample first with concd  $\text{HCl}$  and then with  $\text{HCl}$  with a few drops of  $\text{HNO}_3$ , and proceeding as usual Ca is detd in the filtrate by pptn as oxalate and titration with  $\text{KMnO}_4$  in  $\text{H}_2\text{SO}_4$  soln Fe is detd by dissolving a sample in 20%  $\text{H}_2\text{SO}_4$  and, after reduction with  $\text{SnCl}_2$  and addn of  $\text{H}_2\text{Cl}_2$  and Knopp mixt, titrating with  $\text{K}_2\text{Cr}_2\text{O}_7$  Mn is detd by titrating a  $\text{H}_2\text{SO}_4$  soln with  $\text{Na}_2\text{AsO}_4$  by the persulfate method

Chas Blanc  
Analysis of Martin slags obtained in smelting of special steels F K Gerke and V P Zvereva *Zavodskaya Lab* 4, 738-43(1935) —A systematic analysis of slags, obtained in the production of Cr-Ti-V-Ni steels, is based on known methods

Chas Blanc  
Determination of the zirconium-hafnium ratio. Grant Wernimont and Thomas De Vries *J Am Chem Soc* 57, 2386-7(1935) —The optical rotation of polarized light by basic tartrate solns contg varying quantities of Zr was measured, and as a result a procedure is given which will serve for the analysis of Zr-Hf mixts with the polar scope

W T H  
Accuracy of the titration of thioacynat with mercuric mercury I M Kolthoff and J J Lingane *J Am Chem Soc* 57, 2377-9(1935) —KCNS can be recommended as a standard in mercurimetry when a precision

1 not greater than 0.5 part in 1000 is satisfactory. The effective strength of KCNS in titrating  $\text{Hg}(\text{NO}_3)_2$  is 99.95% when the  $\text{Hg}(\text{NO}_3)_2$  is added to KCNS soln and 100.03% when the reagents are mixed in the reverse order.

W T H  
Rapid method for determining silica in clays R W Ellison, *Chemist Analyst* 25, No 1, 6 8(1936) —Instead of fusing with considerable soda, it is recommended merely to sinter 0.5 g of sample with 0.6 g  $\text{Na}_2\text{CO}_3$  at a temp to be detd empirically, so as to furnish a friable mass which is treated in the usual manner

W T H  
Semimicroanalysis of minerals I General. T. Milobedzki *Roczniki Chem* 15, 294-7(1935) —A discussion of general methods II Brass Walerja Janczak *Ibid* 298-303 —A sample of brass was analyzed by the macro- and semimicro-method The results were identical There is an economy of material 90% and of time 50% by the semimicro-method III. Dolomite *Ibid* 304-9 —The macro- and semimicro-methods gave identical results There is a saving of 90% on material and 40% on time by the latter method

J F Matejczyk  
Analysis of stellites Z. S. Mukhina and K. A. Sukhenko *Zavodskaya Lab* 4, 870-4(1935), cf *C A* 29, 5381\* —Methods of analysis of various types of Ni and Co stellites with the aid of org pptg. reagents are reviewed

Chas Blanc  
Methods for determining lead oxides in lead Ya Gamzulov and V. Bedova *Zavodskaya Lab* 4, 867-8(1935) —Heat 2 g of partly oxidized Pb with 5%  $\text{AcOH}$  for 1-1.5 hrs, add to the filtrate 5 cc of concd  $\text{H}_2\text{SO}_4$  and det. as  $\text{PbSO}_4$ , or dissolve the  $\text{PbSO}_4$  in  $\text{AcONH}_4$  and titrate with  $\text{NH}_4$  molybdate Another method depends on heating a sample in a H current at  $700-800^\circ$  in an elec furnace and collecting the  $\text{H}_2\text{O}$  in  $\text{P}_2\text{O}_5$  H is purified by conducting it through 10%  $\text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{SO}_4$ , KOH and over a Pt spiral at  $700^\circ$  and absorbing the  $\text{H}_2\text{O}$ .

Chas Blanc  
Analysis of products of chlorination of oxides and sulfides of copper Sh Sh Margolina *Zavodskaya Lab* 4, 861-3(1935) —A systematic analysis of the products of chlorination of Cu bearing ores in the extn of Cu was studied with chlorinated pure  $\text{CuO}$  and  $\text{Cu}_2\text{S}$  In each case the detns were made with 3 1-g samples

Chlorinated  
6  $\text{CuO}$  —To det the total Cu and Cl, dissolve the sample in 5 cc of 33%  $\text{HNO}_3$ , dil to a definite vol and det in aliquot parts Cu iodometrically and Cl by the Volhard method To det the total Cu<sup>+</sup>, present as  $\text{Cu}_2\text{O}$ ,  $\text{Cu}_2\text{Cl}_2$  and  $\text{Cu}_2\text{SO}_4$ , oxidize the sample with  $\text{KFe}(\text{SO}_4)_2$  and titrate  $\text{Fe}^{3+}$  with  $\text{KMnO}_4$  in  $\text{CO}_2$  atm The detn of  $\text{CuCl}_2$  depends on the soly of  $\text{CuCl}_2$  and insoly of other Cu compds in abs alc Lix the sample with abs alc in an atm of dry  $\text{CO}_2$  and filter from the insol residue (1) Evap the soln to dryness, dissolve the residue in  $\text{HNO}_3$  and det Cu iodometrically Wash the residue (1) with alc and with boiled and cooled 15%  $\text{NaCl}$  and  $\text{H}_2\text{O}$ , and filter from undissolved residue (2) Dil the soln (possibly contg  $\text{Cu}_2\text{Cl}_2$ ,  $\text{Cu}_2\text{SO}_4$ ,  $\text{Na}_2\text{SO}_4$  and  $\text{NaCl}$ ) to a definite vol in a  $\text{CO}_2$  atm, det in an aliquot part  $\text{Cu}^{+}$  as above and calc  $\text{Cu}_2\text{Cl}_2$  In a 2nd aliquot part det total Cu by pptg with  $\text{H}_2\text{S}$ , dissolving the ppt in concd  $\text{HNO}_3$ , evap the soln nearly to dryness and detg Cu iodometrically Calc  $\text{Cu}_2\text{SO}_4$  on the basis of the values obtained for total Cu and  $\text{Cu}^{+}$  Det  $\text{SO}_4^{2-}$  with  $\text{BaCl}_2$  and calc  $\text{Na}_2\text{SO}_4$  Dissolve the residue (2) (contg  $\text{CuO}$  and  $\text{Cu}_2\text{O}$ ), in concd  $\text{HNO}_3$ , evap, det in the soln Cu iodometrically and calc  $\text{Cu}_2\text{O}$  and from this  $\text{CuO}$  Chlorinated  $\text{Cu}_2\text{S}$  —Det total Cu, Cl and S as above, and  $\text{CuS}$  in another sample by the Schulte method For the detn of  $\text{Cu}_2\text{Cl}_2$ ,  $\text{Cu}_2\text{Cl}_2$ ,  $\text{Cu}_2\text{O}$ ,  $\text{Cu}_2\text{SO}_4$  and  $\text{Cu}_2\text{S}$ , ext  $\text{CuCl}_2$  with abs alc and det Cu iodometrically as above Wash the insol residue in 15%  $\text{NaCl}$  and  $\text{H}_2\text{O}$ , and proceed with the detn of  $\text{Cu}_2\text{Cl}_2$ ,  $\text{Cu}_2\text{SO}_4$ ,  $\text{Na}_2\text{SO}_4$  and  $\text{NaCl}$  as described above For detn of  $\text{Cu}_2\text{O}$ ,  $\text{Cu}_2\text{S}$  and  $\text{CuS}$  in the residue from the washing with 15%  $\text{NaCl}$ , treat the residue with 5%  $\text{NH}_4\text{OH}$  in a H atm for 2-3 hrs, until  $\text{Cu}_2\text{O}$  and  $\text{CuO}$  are dissolved and filter Dil the filtrate to a definite vol, and det in aliquot parts  $\text{Cu}_2\text{O}$  and  $\text{Cu}_2\text{S} + \text{CuO}$  as above Dissolve the insol residue ( $\text{Cu}_2\text{S}$  and  $\text{CuS}$ ) in

$\text{HNO}_3$ , det. Cu iodometrically and calc.  $\text{Cu}_2\text{S}$  and  $\text{CuS}$ . 1 while the lower limit of response is less than 0.001%  
The complete analysis is accurate to 97.5–102.5%.  
G. Tonnesen

Chas Blanc  
Solubility of precipitates in acids A K Babko Z anal Chem. 103, 190–6 (1935) —A general formula is worked out for expressing the soly of ppts such as  $\text{CaC}_2\text{O}_4$  and the formula was tested with  $\text{CaC}_2\text{O}_4$  and  $\text{BaC}_2\text{O}_4$  ppts. The results obtained in varying the concn of  $\text{H}^+$  and of  $\text{C}_2\text{O}_4^{--}$  agreed well with the formula, but the effects of excess  $\text{Ca}^{++}$  and  $\text{Ba}^{++}$  showed much greater soly than was expected. The cause of this lack of agreement will be studied.  
W T H

Modified molybdenum method for determining phosphoric acid in mineral phosphates L V Vladimirov and L N Lobanov Zashchitaya Lab 4, 882–4 (1935) —A few minor modifications are made in the method of J O Handly  
Chas Blanc

Determination of traces of prussic acid in tissues G. Harker J Proc Roy N S Wales 68, 102–6 (1934) —During some work on cancers, it became necessary to det small quantities of HCN and the method of Chelle was tested (C A 13, 2194, 3107, 14, 507, 1274). Forty g of the shredded organ was mixed with 75 ml of water and 5 ml of  $\text{H}_2\text{PO}_4$ , heated and 50 ml of distillate collected. The distillate was placed in a flask, the HCN removed by a rapid stream of air, absorbed in 1 ml of 0.1 N KOH, after which the HCN was detected and detd either by the Prussian blue reaction or with the aid of ammoniacal AgI reagent. It was found that the use of  $\frac{1}{10}$  as much  $\text{H}_2\text{PO}_4$  and air free from  $\text{CO}_2$  increased the yield of HCN, but there was always some loss, probably from hydrolysis of HCN during the boiling, and it is best to assume that the yield is 55% of the truth. Expts were also tried in which the tissue was ground with sand and water, made slightly acid and air drawn through directly without distn. There was bad frothing which was overcome by adding a drop of capryl alc. With 0.008 to 0.096 mg of HCN, the yield improved steadily as the amt of HCN increased and varied from 49.4 to 84%. In this way the HCN is concd in 1 operation and the yields are better, except with less than 0.01 mg HCN.  
W T H

Electrocapillary method of qualitative analysis of cations of subgroup I, group IV. A F Orlenko J Gen. Chem. (U. S. S. R.) 5, 1091–2 (1935) —The electrocapillary method of passing a current at 80 v through a wet filter paper, at one end of which is placed a crystal of a reagent and at the other end a drop of the soln to be analyzed, was used for the detection of  $\text{Hg}^{++}$ ,  $\text{Cu}^{++}$ ,  $\text{Bi}^{+++}$  and  $\text{Cd}^{++}$ . The cations were introduced in the form of  $\text{HgCl}_2$ ,  $\text{CuSO}_4$ ,  $\text{Bi}(\text{NO}_3)_3$  and  $\text{Cd}(\text{NO}_3)_2$ , and as reagents  $\text{KI}$ ,  $\text{K}_2\text{Fe}(\text{CN})_6$ ,  $\text{C}_6\text{H}_5(\text{OH})_3$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$  and  $(\text{NH}_4)_2\text{S}$ , resp. were used. The relative mobilities of the cations were in the order  $\text{Cu}^{++} > \text{Bi}^{+++} > \text{Hg}^{++} > \text{Cd}$ , and of the anions in the order  $\text{I}^- > \text{Cr}_2\text{O}_7^{--} > \text{Fe}(\text{CN})_6^{--}$ . The ions move faster in an acid than in a neutral medium. It takes only 10–15 min to make a complete qual. analysis of the above cations by this method.  
S L Madorsky

Titrimetric determination of water and alcohols by their acid-catalyzed reactions with acetic anhydride in organic media. Gerrit Tonnesen and Margaret Philott J Am Chem. Soc. 57, 2136–9 (1935) —The method is based on the fact that the reactions of  $\text{Ac}_2\text{O}$  with  $\text{H}_2\text{O}$  and alcs in the absence of a catalyst are negligibly slow in org media, while in the presence of a strong acid, such as perchloric or 2,4-dinitrobenzenesulfonic acid ( $2-4 \times 10^{-3} M$  of the latter were used) the reactions are completed in less than 1 day. On titration with  $\text{NaOMe}$ ,  $\text{Ac}_2\text{O}$  consumes 1 equiv, while after hydrolysis it consumes 2 (cf. C A 27, 5355). After alcoholysis the titration value of  $\text{Ac}_2\text{O}$  is unchanged, but an unused excess can be detd by the increase in titration value after hydrolysis with excess  $\text{H}_2\text{O}$ , this permits sep. detn. of  $\text{H}_2\text{O}$  and alcs. ( $\text{MeOH}$  and  $\text{EtOH}$  were used). The method is applicable to nonacidic and nonbasic solvents that are inert against the reagents. Examples are given of detns in  $\text{CH}_3\text{CN}$ ,  $\text{Ft}_2\text{O}$ ,  $\text{C}_6\text{H}_6$  and  $\text{CHCl}_3$ . The error in concns. of 1% is less than  $\pm 1\%$ ,  
S L Madorsky

Determination of the reducing power of impure air. L. W. Winkler. Z. anal. Chem. 103, 183–6 (1935). —If the air of a badly ventilated room, such as that of a school or a theater, is chilled, say by passing some of it for some time through a 2-l. bottle placed in cracked ice, moisture will condense and the purity of the air can be estd by detg. how much alk.  $\text{KMnO}_4$  is required to oxidize the impurities. The no. of ml of  $N \text{ KMnO}_4$  required to oxidize 1 l. of condensed  $\text{H}_2\text{O}$  is defined as the reducing power of the air in degrees. To carry out the analysis, place 10 cc of alk.  $\text{KMnO}_4$  soln (0.1 N in  $\text{KMnO}_4$ , and 0.1 N in  $\text{NaOH}$ ) in each of two 50-ml Erlenmeyer flasks. To one flask add 1 cc of distd water and to the other 1 cc. of the condensed water. After 24 hrs at room temp make both solns acid by adding 1–2 ml of 10%  $\text{H}_2\text{SO}_4$ , add 0.05 g of KI and titrate the liberated  $\text{I}_2$  with 0.01 N  $\text{Na}_2\text{S}_2\text{O}_3$ . The latter soln will keep indefinitely if it contains 10 ml of purified isobutyl alc. per l. The reducing power of fresh air varied from 1.0° (after a spell of rainy weather) to 2.2° (cloudy day). The reducing power of the air of a movie theater was 18.2°.  
W T H.

Method of spectral analysis of complicated mixtures. B Ya Dan, I V Granovskii and E S Puzenkin. J. Gen. Chem. (U. S. S. R.) 5, 1093–7 (1935) —Standards for the quant spectroscopic analysis of the system Mn–Co and the effect of impurities, such as chlorides of Na, K, Ba, Al, Ni and Fe, singly or in pairs, on these standards, are indicated.  
S L Madorsky

The detection of strychnine in carcasses and corpses. Douw G Steyn Onderstepoort J Vet Sci 5, 130–74 (1935) —The factors affecting the taste, color and biol tests for strychnine are discussed. S was unable to detect a bitter taste in dilns of strychnine beyond 1 in 200,000 in distd. water when a standard quantity of 1 cc. of each diln. was placed on the tongue. A bitter taste could not be detected when only 1 drop of a 1 200,000 soln. was placed on the tongue. Wagner's reagent No 1 gave a distinct ppt. when 1 drop was added to 1 drop of a 1 20,000 soln of strychnine in distd water slightly acidified with  $\text{H}_2\text{SO}_4$ . Mayer's reagent was slightly less sensitive. Under the same conditions phosphotungstic, phosphomolybdic, picric and tannic acids did not give pos results with 1 4000 solns. of strychnine. For the biol test strychnine was best injected into white mice and frogs in the form of a physiol saline soln slightly acidified with  $\text{H}_2\text{SO}_4$ . As little as 0.008 mg. strychnine sulfate could be detected when it was injected intraperitoneally into white mice 3 weeks old weighing 10–12 g; white mice 14 days old weighing about 6 g. possessed the same degree of susceptibility per unit body wt. *Rana aqualems* possessed the same degree of susceptibility as white mice 3 weeks old but a 9-fold quantity of strychnine sulfate had no effect on *Xenopus laevis*. Mice reacted much more promptly than frogs to small amts. of strychnine. The symptoms of strychnine poisoning in white mice and *R. aqualems* are described. Factors responsible for the disappearance of strychnine from corpses and carcasses are discussed. Of 4 dogs killed with strychnine and exhumed 10 weeks later strychnine was detectable in 3 carcasses, while of 4 carcasses exhumed 18 weeks after death only 1 was pos. for strychnine. Strychnine was detectable in 4 of 8 carcasses of dogs exhumed 11 months after death. Methods of extg strychnine from carcasses and corpses and of purifying these exts are discussed. In fresh carcasses and corpses the most suitable organs for analysis for the presence of strychnine are liver, stomach, spleen, lungs, central nervous system and urine. In 2 of 3 dogs which had received strychnine as a tonic, strychnine was detectable in the liver and stomach (plus contents). A large no. of chem. substances which resemble strychnine chemically and biologically are discussed. A strychnine-like ptomaine was isolated from a decompd liver which was known not to contain any strychnine. This ptomaine had a bitter taste and gave a pos.  $\text{H}_2\text{SO}_4$ - $\text{K}_2\text{Cr}_2\text{O}_7$  test for strychnine. The results of taste, chem. and biol. tests with identified

and unidentified ptomaines are recorded. Twenty-seven references. K D Jacob

Microcrystallographic identification of blood spots in situ. Alejandro Raitzin. *Rev. asoc. med. argentina* 49, 1115-22 (1935).—By means of Leitz's ultrapak and the formation of cryst. hemin-HCl it is possible to detect blood spots in situ. E S G B

Microchemical analysis of organic compounds. M O Korshun. *Zarodskaja Lab* 4, 843 8 (1935).—A review with about 50 references. Chas Blanc

Determination of chromium in organic compounds. C Franklin Miller. *Chemist Analyst* 25, No. 1, 5-6 (1936).—Wet digestion of 10 g of sample with concd  $H_2SO_4$  and  $HNO_3$  is recommended. The clear, acid soln is poured into NaOH and the Cr oxidized to  $CrO_4^{--}$  with  $Na_2O_2$ . After the removal of excess peroxide, the Cr can be detd. iodometrically or, if only little is present, by the color resulting with diphenylpicarbazide. W T H

Use of Schiff's reagent in the determination of acetaldehyde in acetic acid. G V Zavarov. *Zarodskaja Lab* 4, 764-7 (1935).—Toprep Schiff's reagent, mix 150 cc of 0.1% fuchsian with 100 cc  $NaHSO_3$  soln (d<sub>15</sub> 1.308), 1000 cc  $H_2O$  and 15 cc  $H_2SO_4$ , and allow to stand for 24 hrs. Prep standard solns of 0.02% and 0.1% AcH in 50% alc. Prep AcOH free from AcH by refluxing 500 cc of glacial AcOH with 10 g of  $CrO_3$  for 1 hr and distg. the acid. Repeat the operation until the AcOH gives no color reaction with Schiff's reagent as described below. Place in a dry test tube 4 cc of 50% alc., 0.5 cc of AcOH to be tested and 1 cc of Schiff's reagent. Place in another tube 3.5 cc of 50% alc., 0.5 cc of one of the standard solns of AcH and 0.5 cc of the prepd AcOH. Shake well the 2 tubes and allow the contents to "age" for 30 min. Introduce into each tube 5 or 10 cc of  $H_2O$ , depending on whether 0.02% or 0.1% of the standard AcH soln was used, and compare in the Duboscq colorimeter. The method is accurate to 0.003% for 0.1% AcH in 95.5% AcOH. Chas Blanc

Alkalimetric determination of pentabromacetone in the analysis of citric acid. G A Korzhemovskii and Ya N. Rekeida. *Zarodskaja Lab* 4, 768-70 (1935).—In the detn of AcOH (I) in wines, milk, tobacco, etc., by the Kuntz method, based on Stahre reaction, by oxidation of I to  $CO(CH_3CO_2H)_2$  and conversion of the latter to pentabromacetone (II), a considerable saving of time is effected by titrating II with NaOH instead of drying it over  $P_2O_5$  in vacuo. Wash the ppt of II thoroughly (until wash waters are neutral to methyl orange), transfer it with the filter to a flask, dissolve in 20 cc alc., add 50 cc of 0.1 N NaOH (KOH), digest on a water bath at 85-90°

(not higher) for 30 min and titrate back with 0.1 N HCl in the presence of methyl red as indicator. In the sapon II undergoes a no. of conversions, from which was evolved an empirical equation for the calcn of II:  $a = 11.85b - 5$ , in which  $a$  is mg. II and  $b$  is cc 0.1 N NaOH. C B.

Naphthalene determination with picric acid. A. P. W Munch and R. Th. Heukens. *Chem. Weekblad* 32, 627-8 (1935); cf. C A 29, 6536<sup>3</sup>.—A reply to Pieters and Penners, cf. C A 29, 6023<sup>4</sup>. O W Wilcox

Estimation of biacetyl. Hans Schmalfuss and Hildegard Rethorn. *Z. Untersuch. Lebensmittel* 70, 233-40 (1935).—A method is described for the detn of biacetyl as Ni salt of dimethylglyoxime with an av. error of -0.2%. In this method, care is taken as to the degree and length of heating, degree of diln, content in bases and the volatility of the biacetyl. On the other hand, the content in hydroxylamine hydrochloride and NiCl<sub>2</sub> may be varied within wide limits. Sixty-one references. F L Dunlap

Microchemical determination of cholesterol, carbanide, glycerol, etc., by means of liquid crystals. Paul Gaubert. *Compt. rend.* 201, 1202-4 (1935).—A drop of the liquid for examn contg, e g, carbanide, glycerol or citric acid, with a crystal of cholesterol, is placed on a plate heated to the m. p. of cholesterol (148.5°). As the mixt. cools combinations of the carbanide, etc., with cholesterol form characteristic liquid crystals. Cf. C A 4, 138. C A Silberrad

Ultraviolet titration and its application in the analysis of volatile oils. Lawrence S. Malowan. *Rieschstoff-Ind* 10, 199-200 (1935).—M. utilizes the change in fluorescence properties of such compds. as quinine, esculin and umbelliferone and its substitution products when subjected to various  $pH$  values. H M. Burlage

Compn. and properties of K phosphotungstate [detection or detn of K] (Nikitina) 6. Color reactions of phenols with  $HNO_3$  (Malinovskii) 10.

Indicators. Raymond A A. Dry. Fr 787,604, Sept. 26, 1935. An indicator for colorimetric analysis is used in solid form, in the shape of a pencil which contains the reactive substance associated with a nonactive supporting material. Reactive substances used as indicators in this form include quinine, fluorescein, esculin,  $\beta$ -naphthol, umbelliferone,  $K_4Fe(CN)_6$  (to detect the presence of  $Cu^{++}$  and  $Fe^{+++}$ ),  $(AcO)_2Pb$  (to detect sulfides) and double iodides of Hg and Cu, or Hg and Ag (to detect variations of temp.).

## 8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND J. F. SCHLAIRER

The crystal form and molecular unit of nasygite. B. Gossner. *Zentr. Mineral. Geol.* 1935A, 321-7. —Nasygite is tetragonal or pseudotetragonal with  $a = 12.5$ ,  $c = 30.25$ . The results are somewhat uncertain, owing to the easy deformability of the crystals. While some analyses lead to the formula  $6Pb(S, Te)_2$ ,  $AcTe_2$ , no simple formula could be deduced for the Sb-bearing variety. Michael Fleischer

Significance of "filled" feldspar. H. P. Cornelius. *Schweiz. mineral. petrogr. Mitt.* 15, 4-30 (1935).—Plagioclase crystals "filled" with clinozoisite and (or) sericite or muscovite occur in certain granites, etc., of the Alps; occurrence of garnet, sillimanite or quartz as "filling" materials is doubtful. The plagioclase crystals, though retaining their form, are sometimes so much "filled" as to be indeterminate, in general the greater the proportion of anorthite the greater that of zoisite. The distribution of these "filled" plagioclases is stated and theories of their origin are discussed. C's view is that it is a deep-seated phenomenon, resulting from the action of  $K_2O$  and  $H_2O$  on plagioclase crystals under special conditions as to temp. and pressure during mountain-building processes, so that

optimal zones occur where these conditions were most favorable. C A Silberrad

Structure study of natural wollastonite. Max. Barnack. *Naturwissenschaften* 23, 770-1 (1935).—Wollastonite from Cziklova, Hungary, is monoclinic with  $a = 15.33$ ,  $b = 7.28$ ,  $c = 7.07$  Å,  $\beta = 95^\circ 24' 5''$ , 12 mols. of  $CaO$   $SiO_2$  per unit cell, space group  $C_{2h}^2$ . It is built up of  $(SiO_4)_4$  rings placed on top of each other in the  $b$ -direction and linked by the side O atoms. Triclinic wollastonite has thus undergone micro-twinning. B J C v d H

Chemical and x-ray investigation of amphiboles. II. Distribution of the analytical deficit. J. Jakob. *Schweiz. mineral. petrogr. Mitt.* 15, 146-52 (1935). Cf. *Ibid.* 11, 140 (1931).—Addnl. analyses of tremolite (3), actinolite (4), pargasite (3), common hornblende (1) and asbestiform hornblende (4) are given. All except the last show a deficit, up to a max. of 3.45%, hitherto attributed to mol O. This supposed excess O depends in no way on the presence of any particular cation, but only on the form. It is as a rule absent from asbestiform, i. e., fibrous ("one-dimensional") varieties, but never from compact ("three-dimensional") varieties, in which it is

suggested that the O is held by labile linkages stabilized through the more wide-reaching bonds of the "3-dimensional" state. C. A. Silberrad

Twins and trillings of rock-forming olivine. Conrad Burri *Schweiz. mineral. petrogr. Mitt.* 15, 160-7 (1935) —Replacement of Mg in forsterite by Fe to form fayalite alters the axial ratio very little, with a max. for 15 mols % of FeSiO<sub>4</sub>. The presence of Ca in the olivine mol. causes a much greater change, bringing the crystal nearer to hexagonal, with a corresponding greater tendency to form twins and trillings C. A. Silberrad

The influence of original composition upon the micas in the metamorphic rocks of the Hsianshuissu series, near Mt. Tahoshang, South Manchuria. Mititaka Sawatari. *Mem. Ryojun Coll. Eng.* 8, 67-84 (1935) (in English) —Muscovite samples taken from a 2-mica schist and from an argillaceous calc-schist showed  $\mu$  varying from 1.587 to 1.612 and 2V from 32° to 42°. Muscovite of high  $\mu$  and low V occurred in rock rich in Fe minerals, while the muscovite in Fe-poor rock had low  $\mu$  and high V values. Chem. compn. rather than grade of metamorphism was deduced as of most importance in influencing the optical properties of the muscovite, as well as of biotite.  $\gamma$  for the latter was 1.617-1.624 in calc-schist for which FeO/MgO was 1.04, and 1.642-1.647 in mica schist for which FeO/MgO was 1.63 P. S. Roller

The chemical nature of halloysite and the allophanoids Hugo L. Piotrowski. *Arch. mineral. soc. sci. Varsovie* 10, 1-89 (87-9 in French) (1934) —Halloysite (I) and allophanite (II) are found in aluminite mine north of the St. Cross mountains in Poland, forming veins in an argillaceous sandstone, named by P. halloysite-sandstone (III). Compn. of I in wt. percentage: SiO<sub>2</sub> 33.50-38.60, Al<sub>2</sub>O<sub>3</sub> 32.41-34.65, Fe<sub>2</sub>O<sub>3</sub> 0.06-0.60, FeO 0-0.02, MnO 0-traces, CaO 0-0.30, MgO 0-0.03, H<sub>2</sub>O 26.71-32.72, d. 2.039-2.108. II: SiO<sub>2</sub> 24.37-28.29, Al<sub>2</sub>O<sub>3</sub> 35.81, 37.16, Fe<sub>2</sub>O<sub>3</sub> traces-0.10, FeO 0, MnO 0-traces, CaO 0.64-0.72, MgO traces-0.02, H<sub>2</sub>O 24.80-33.57; d. 1.897-2.003. III: SiO<sub>2</sub> 71.01, CO<sub>2</sub> traces, TiO<sub>2</sub> 0.16, P<sub>2</sub>O<sub>5</sub> 0.22, Al<sub>2</sub>O<sub>3</sub> 10.25, Fe<sub>2</sub>O<sub>3</sub> 5.79, FeO 0.07, MnO 0.03, CaO 0.09, MgO 0.03, K<sub>2</sub>O 0.45, Na<sub>2</sub>O 0.07, H<sub>2</sub>O 12.12. The above compn. reveals undoubtedly that (I) is an aluminosilic acid, contg. in addn. to adsorbed water 2 mols. H<sub>2</sub>O combined chemically. From kaolin, I is distinguished only by its phys. state. The chem. nature of II is not evident J. Wiertelak

Polish bentonite from the vicinity of Krzemieniec, southeastern Poland. Zbigniew Sujkowski. *Arch. mineral. soc. sci. Varsovie* 10, 98-116 (115-16 in English) (1934) —The bentonite consists of a claylike yellow mineral, which swells readily in water. It has a waxy luster when dry and a soapy touch when wet. Its chief constituent (95%) is montmorillonite, a cryptocrystalline substance, having  $\mu$  1.520,  $d_{110}$  2.0745, birefringence 0.006-7, compn.: SiO<sub>2</sub> 48.85, Al<sub>2</sub>O<sub>3</sub> 19.04, Fe<sub>2</sub>O<sub>3</sub> 2.61, FeO 0.11, MnO 0.09, insol. matter 0.41%, corresponding to 96.07% of the first (3K<sub>2</sub>SO<sub>4</sub>·Na<sub>2</sub>SO<sub>4</sub>). The parameters, by x-ray powder method, are:  $a = 5.635$ ,  $b = 7.27$  Å. J. Wiertelak

Glaserite and syngenite of Stebnik, Poland. Antoni Lasziewicz. *Arch. mineral. soc. sci. Varsovie* 10, 117-21 (121 in French) (1934); cf. Kuznar, C. A. 28, 5010\* —Glaserite (aphtillalite) found in the salt mines of Stebnik in assoc. with syngenite and Glauber salt forms very pure prismatic crystals, as is evidenced by the compn. SO<sub>3</sub> 67.47, Cl 0.025, Ca 0.10, Mg traces, K 34.31, Na 7.36, H<sub>2</sub>O 0.09, insol. matter 0.41%, corresponding to 96.07% of the first (3K<sub>2</sub>SO<sub>4</sub>·Na<sub>2</sub>SO<sub>4</sub>). The parameters, by x-ray powder method, are:  $a = 5.635$ ,  $b = 7.27$  Å. J. Wiertelak

Contact metalliferous deposits E. Raguin. *Bull. soc. géol. France* [5], 4, 563-71 (1935) —From observations on mines in the southwestern U. S. it is concluded that there are the following varieties of contact deposits: contact stockworks, hypothermal and mesothermal con-

tact deposits, pyrometamorphic replacements, and diffuse impregnations of the eruptive rock itself. C. A. S.

Mineralized crust zones in Gold Coast Colony. T. Pickering. *Mining Mag.* 53, 329-36 (1935). —Geology and tectonics of areas contg. quartz reefs at Bogosu and in the vicinity are described. The horizon is a complex of lavas, dikes and sills, with occasional development of water-laid tuffs and phyllites, a younger system is characterized by arenaceous beds and conglomerates, and with subsidiary phyllites. Graphite is found to be associated with the ore horizon, and occurs as very fine films occupying slips and partings. For most quartz bodies in the area crushing and panning are of some value for rough guidance, but very little Au can be panned from the richest crush-zone material, at least so far as the oxidized material is concerned, the Au is so finely divided that in panning tests about 80% of the total is lost before a concentrate is obtained, and the remaining 20% is probably masked by Fe oxide films. In the crush zone in quartz reefs, quartz is developed strongly as stringers, veinlets, blebs and lenses, often of great size. Evidence is plentiful that graphite has acted as a precipitant for the Au. A. W. F.

Gold minerals of eastern Uregra (Kivu, Belgian Congo). René van Aubel. *Bull. soc. géol. France* [5], 4, 675-96 (1935), cf. C. A. 28, 5783\* —The Au occurs in veins in mainly biotite schists, assoc. with arsenopyrite and usually some pyrite and less often marcasite. The crystals of arsenopyrite are sometimes coated with a layer contg. scorodite and symplectite, with epidote and sericite. Black pleochroic (yellow to blue-green) tourmaline with some dravite is always present, and graphite frequently, especially at Mt. Kibikura. Accessory minerals are galena, sphalerite and chalcocopyrite. The veins also contain (besides quartz) biotite, muscovite, pyrrhotite, garnet, magnetite, and more rarely albite, apatite and tetradymite, and in fissures chlorite, sericite and kaolin. The surrounding schists are more or less metamorphosed, tourmaline having been formed at the expense of the biotite. The localization and origin of the deposits, which are essentially permarginate, are discussed. The Au is practically entirely recoverable by crushing and amalgamation. Traces of Cu occur in most veins, Bi sporadically, assoc. especially with tourmaline; Sn is very rare in the veins and apparently not as cassiterite, though alluvial cassiterite and tantalum-columbite occur under circumstances which seem to imply that their origin was the same as that of the auriferous group. Spectrographic examn. of native Au shows the presence of Ag and Fe, with a little Bi and Pb, less Ti, Zn, Sn and Cu and traces of B, Mo, Sb and As. C. A. Silberrad

Geological study at Cusi reveals new ore. Robert T. Donald. *Eng. Mining J.* 136, 614-17 (1935). —Cusi-huachic, located 75 miles southwest of Chihuahua City, Mexico, has been a producer of high-grade Ag ores for over 270 years. Pb, Cu, Ag and Zn have been produced. Investigation of wall rock alteration in old veins having barren outcrops gives an index for exploring deeper ore-bodies. W. H. Boynton

Platinum deposits of Kuznetzk-Altau. A. K. Kyuz. *Soviet. Zolotoprom.* 1935, No 5, 23-5 —A detailed map and geol. description of the Kuznetzk-Altau Au-Pt-bearing district. In some cases the Pt content is several % of that of the Au. S. L. Madorsky

The mineral resources of Uruguay. Juan P. Pitamiglio. *Rev. mensual asoc. rural Uruguay* 1935, No 1, 42-4 —Uruguay has deposits of Fe as oxide, sulfide and carbonate, Mn as the oxide, Pb as galena, Cu and auriferous quartz. Deposits and mining operations of the first 4 of these are discussed. Nonmetallic minerals occurring and mined are talc, kaolin, asbestos, emery and graphite. Sand and marble and other forms of CaCO<sub>3</sub> are quarried. Amethyst and agate occur. Other important materials are clays, sandstone, porphyry, granite, syenite, gneiss and slate. Colin W. Whittaker

Oblitic iron deposits in Franche-Comté. Ch. Fimaton. *Bull. soc. géol. France* [5], 4, 347-68 (1934). —Oolitic Fe ores have from time to time been worked in the Lias in Franche-Comté, but mainly at or near the outcrop.

Borings show that these deposits extend widely. They consist of one bed about 4 m thick, with usually a second and sometimes a third, each 2 m thick. Numerous analyses are given, the ore from the borings shows loss on heating 25-32,  $\text{Fe}_2\text{O}_3$  25-31,  $\text{CaO}$  21-30,  $\text{Al}_2\text{O}_3$  3-6,  $\text{SiO}_2$  5-14,  $\text{P}_2\text{O}_5$  0.68-1.02,  $\text{Mn}_2\text{O}_3$  0.15-0.28%. The ore as mined is a compact red rock with visible oolites and usually a calcareous cement. In one boring (Vy les-Filam) 22 m of bituminous marl was passed through at 300 m. When fresh this ignited easily in a flame, but yielded only 2-3% of oil on distn.

C. A. Silberrad  
Philippines a source of chrome ore Ralph Keeler. *Eng Mining J* 136, 612-13(1935)—The economic importance of deposits in Luzon is noted. The largest known body of Cr ore in the world is at Masinloc, Zambales.

W. H. Boynton  
Limestones of Canada, their occurrence and characteristics III Quebec M. F. Goudge. *Can Dept Mines, Mines Branch Rept No 755*, 274 pp (1935)

Alden H. Emery  
Deposits of potassium in the Ural-Emba district N. S. Kurnalov, N. I. Buyalov, D. I. Kuznetsov and I. L. Lepeshkov. *Kalsh (U S S R)* 1935, No 4, 11-26

A. Pestoff  
Potassium in the Iltetz region of Orenburg district A. A. Ivanov. *Kalsh (U S S R)* 1935, No 6, 4-7—K was found in a NaCl mine near Sol'-Iltetz, 72 km south of Orenburg on the Orenburg-Tashkent railroad A. P.

Petroleum geology of Gondwana rocks of southern Brazil Victor Oppenheim. *Bull Am Assoc Petroleum Geol* 19, 1725-1805(1935)—Analyses of bitumen, oil and gas are given, their genesis is discussed A. H. C.

Search for petroleum in Spanish Morocco. A. Marin, J. L. Pastora and J. de Laza. *Bull soc. géol. France* [5], 4, 649-73(1935)—An examn. of a tract of country between Larash and Kasr-el-Kabur, which seemed to possess a structure favorable for oil, has so far only shown a strong smell of petroleum in a native excavation for salt in Triassic marl at Dsar-Yadid C. A. Silberrad

Magmatic differentiation Paul Niggli. *Schweiz mineral petrog Mitt* 15, 153-9(1935)—Arguments based on the chem. compn. of many rocks (analyses given) and chem. reactions are adduced, disposing of 2 objections to N's differentiation theory C. A. Silberrad

Volcanic rocks of Nicaragua C. Burri and R. Sonder. *Schweiz mineral petrog Mitt* 14, 526-8(1935)—These rocks are first classified into Quaternary (I) and younger Tertiary (II). I are all basic types, olivine-basalt, gabbro, etc., II include liparite, quartz- and normal-dioritic andesites, and olivine basalt. All form a remarkably homogeneous province of purely Pacific character C. A. Silberrad

The magmatic rocks of the Bolivian Andes K. Smulikowski and Roman Kozłowski. *Arch. mineral. soc. Varsovie* 10, 122-242(1934) (in French)—An outline of the distribution and a micrographic study, with 19 plates. J. Wiertelak

Granite of Lys, Haute Garonne. D. Raguin. *Bull soc. géol. France* [5], 4, 421-30(1934)—The rock is a porphyroid biotite granite contg. large orthoclase crystals, and showing an apparent schistosity parallel with that of the schist inclusions. It contains dikes of tourmaline pegmatite and lamprophyre hornblende-andesite. In the granite-schist contact feldspathization has occurred, there is much chistolite slate, in places passing into leptynolite, and occasionally true gneiss contg. wavy orthoclase, myrmekite, biotite, muscovite and often andalusite, sillimanite and tourmaline C. A. S.

Authigenous tourmaline in sedimentary rocks L. Déverny. *Schweiz. mineral. petrog. Mitt.* 14, 528-9 (1935)—Several addnl. occurrences in the Alps of authigenous tourmaline in cellular dolomite and calcareous schist are described C. A. Silberrad

"Horizontal sandstone" of Mandingo plateau (French

West Sudan). glauconitic sandstone of Koulikoro, and surface coating of decomposed ferruginous rock. Paul Jodot. *Bull soc. géol. France* [5], 4, 369-74(1934).—The surface rock differs from ordinary latérite in contg. no  $\text{Al}_2\text{O}_3$ ; it consists of a certain amt. of quartz detritus from disintegration of the subjacent sandstone, recemented by hydrated  $\text{Fe}_2\text{O}_3$ , which is noncrystalline (stulponisiderite), and has been leached out of, and concd. from, the sandstone, in wet and dry seasons, resp. C. A. Silberrad

Flints, buhrstones and agates H. Douville. *Bull soc. géol. France* [5], 4, 537-44(1935); cf. C. A. 26, 4014— $\text{SiO}_2$  derived from sponges, radiolarians, etc., on their death, in the form of micelles, forms a pseudo soln. with the decomposing remains. In contact with other material, especially calcite, the  $\text{SiO}_2$  replaces it, as chalcedonite if the soln. is impure, as quartz if pure. Chalcedonite on deposition adsorbs the impurities. Hence an alternation of chalcedonite and quartz in layers of thickness varying according to the conditions from about 0.0012 mm upward, the extreme case being the geode, in which after the first deposition of chalcedonite all subsequently incoming soln. is freed from impurities. Occasionally the chalcedonite forms spherulites. The impurities consist of goethite or other hydrated  $\text{Fe}_2\text{O}_3$ , and a white material, probably kaolinite C. A. Silberrad

Some oolitic formations P. H. Derville. *Bull soc. géol. France* [5], 4, 771-5(1935)—Calcareous oolites occurring in the oolitic limestone of Carteret (Cotenin) are described, some are perfect, others apparently deformed before solidification C. A. Silberrad

Geology of glauconite E. Wayne Gallher. *Bull Am Assoc Petroleum Geol* 19, 1560-1601(1935).—In Monterey Bay, Calif., glauconite replaces biotite in sediments as they are followed off shore to deeper water.

In changing to glauconite, biotite loses some Al, K and Mg, it gains  $\text{H}_2\text{O}$  and most of the Fe is oxidized. Factors affecting these changes are (1) the alk. sea water and (2) conditions operating in the anaerobic black muds and oxidized layers overlying them. Slow sedimentation in parts of the bay provides a long diagenesis period for biotite alteration. The following regional occurrences of glauconite are explained by the fact that it forms from biotite: (1) Glauconite is rarely found in deep sea deposits, for most micaceous minerals come to rest around continental shores before reaching great depth. (2) It is found near land masses where exposed plutonic and metamorphic rocks contribute biotite to the adjacent ocean basin. (3) Glauconite compn., especially  $\text{K}_2\text{O}$  content, varies from place to place, depending on the  $\text{K}_2\text{O}$  in the biotite. (4) Glauconite is universally distributed, for it is formed from a prominent and widely distributed rock-forming mineral Alden H. Emery

Some metamorphic minerals in the Molasse A. v. Moos. *Schweiz mineral petrog Mitt* 14, 530-1(1935)—Occurrences in the Molasse of Switzerland of staurolite and kyanite (usually assoc.), andalusite, chloritoid and blue hornblende are described. Of the last-named all varieties including gastaldite, glaucophane and crossite occur. As inclusions one or more of zircon, rutile and epidote occur in the 2 first and the last C. A. S.

Rapid pyritization of organic remains near volcanic sulfur springs F. Bernauer. *Zentr. Mineral. Geol* 1935A, 343-4, cf. C. A. 29, 5780—Plant remains, floating in warm, sulfurous sea water off the island of Vulcano, were rapidly covered by a deposit of  $\text{FeS}_2$ , identified as marcasite by x-ray study. M. F.

Foundations of quantitative geochemistry. II Rare elements in meteorites V. M. Goldschmidt. *Fortschr. Mineral. Krist. Petrog* 19, 183-216(1935); cf. Noddack and Noddack, C. A. 24, 5546, 29, 707—A review. The av. contents of the silicate and metallic portions of meteorites are calcd. for a no. of the rare elements. Fifty-four references Michael Fleischer

## 9—METALLURGY AND METALLOGRAPHY

D J DFMOREST, OSCAR E HARDER AND RICHARD RIMBACH

Depression period well past for the rare metals and minerals Paul M Tyler *Mining Met* 17, 17 22 (1936).—Latest advances in the following are discussed: Sb, As, Be, Bi, Br, Cd, Ca, Cr, Co, Ga, Ge, In, Ti, Hg, Mo, K, Ra, rare earths, rhenium, Se, Na, Ta, Te, Th, Ti, W, V and Zr.

Metallurgical efficiency on the Sub Nigel G Carleton *S African Mining Eng J* 46, 197(1935).—The new reduction plant at the Sub Nigel Mine has reduced tailing values to 0.24 dwt per ton (formerly 0.37) for an extn of 98.5% (97.9% previously). Costs (including sorting and crushing) are 3s 3d per ton. About 55% of the ore value is extd on corduroy. Overflow from bowl classifiers is cyanide feed.

Principles of flotation IV. An experimental study of influence of sodium sulfide, alkalis and copper sulfate on effect of xanthates at mineral surfaces Ian W Wark and Alwyn B Cox *Am Inst Mining Met Engrs Tech Pub No 659, 20 pp* (1936), cf *C A* 29, 1049.—By measuring the influence of Na<sub>2</sub>S on the contact angle induced by xanthate between an air bubble and anglesite, cerussite, galena, sphalerite, pyrite, chalcocopyrite, bornite, covellite and chalcocite it is shown that: (1) Na<sub>2</sub>S is a depressant for sulfide minerals; (2) the concn of Na<sub>2</sub>S necessary to prevent contact is a function of the  $p_{H_2}$  value, (3) the hydrosulfide ion (HS<sup>-</sup>) is the effective depressant and there is, for each mineral, a critical HS-ion concn. just sufficient to prevent response to a given concn of K ethylxanthate, this crit value is not greatly influenced by the xanthate concn, (4) CuSO<sub>4</sub> influences the effect of Na<sub>2</sub>S on galena and chalcocopyrite, probably removing it from soln. as a ppt of CuS, (5) CuSO<sub>4</sub> must be stoichiometrically in excess of the sulfide if it is to activate sphalerite; (6) the combined influence of CuSO<sub>4</sub> and Na<sub>2</sub>S on pyrite is exceedingly complex; (7) anglesite responds to K ethylxanthate most readily between  $p_{H_2}$  0 and 11, and cerussite at  $p_{H_2} < 9$ , (8) Na<sub>2</sub>CO<sub>3</sub> increases the concn. of K ethylxanthate necessary to induce a response at surfaces of anglesite and cerussite; (9) controlled concns. of Na<sub>2</sub>S activate anglesite and cerussite so that they respond to lower concns. of xanthate, but excess of sulfide prevents a response; (10) cerussite responds more readily to xanthate after an initial treatment with Na<sub>2</sub>S, owing to the formation of a surface film of PbS, (11) the response of anglesite and cerussite to amylnxanthate is less influenced by Na<sub>2</sub>S than their response to ethylxanthate, (12) Na phosphate can be substituted for Na<sub>2</sub>S as an activator for anglesite and cerussite; (13) the test specimens of sulfide minerals consume very little sulfide or xanthate, but cerussite may consume appreciable amts.; under some conditions anglesite may consume a considerable fraction of the xanthate present; (14) sulfide is consumed in preference to xanthate by anglesite and cerussite; (15) because of the volatility of HS and the readiness with which Na<sub>2</sub>S is oxidized it would be difficult to control the sulfide concn. of flotation-plant circuits.

Alden H Emery  
The flotation of gold ore at the Bibiani mine W J. Cayer. *S. African Mining Eng. J* 46, 281-3(1935).—The old plant, using a battery of 50 stamps with subsequent amalgamation and cyanidation, gave sand tailings assaying 1.5 dwts. per ton and slimes contg. 4.2 dwts. due to Au in pyrite. A new plant consisting of a 5 × 10 ft. ball mill, simplex classifier, Dorr agitators and thickeners gave a 3.60 dwt. tailing from a 120 dwt ore. Later ball mill discharge was fed to corduroy tables, which recovered more than 1/2 the Au. The concentrate was roasted and charged back into the ball mill circuit. Extn was more than 81% at 40%-200 mesh. 1 in. grinding was uneconomical, even at 92% extn. A new flow sheet using flotation was adopted. Use of soda ash was not beneficial. CuSO<sub>4</sub> did not activate the pyrite. Cresylic acid gave no increase in extn. Starch to depress graphite decreased extn. Au recovery by flotation was 91% and that by cyanidation 97%.

Alden H. Emery

Flotation of Melent'evsk (gold) ores. S. I. Mitrofanov. *Soviet Zolotoprom* 1935, No 8, 43-5.—Details of grinding and of flotation by means of ethyl- and butyl-xanthates, together with pine oil. S. L. Madorsky  
Gold industry in U S S R during 1934. D. I. Shitenbok *Soviet Zolotoprom* 1935, No 1, 18-27.

S. L. Madorsky  
Method of estimating gold deposits G Levitov *Soviet Zolotoprom* 1935, No 6, 17-19.—A review.

S. L. Madorsky  
An investigation of gold-bearing ores at Trennill V G Agrenkov and K F Barusheva. *Soviet Zolotoprom* 1935, No 8, 35-43.—Reports of investigation of extn of Au by amalgamation and other methods from ore samples of the following sources: Laila, Alkabeke, Manka, Akdzhal, Riddersk, Kulundzhunsk and Maikin.

S. L. Madorsky  
Gold ores of Retiv and Aleksandrovsk veins I N Plaksin and S V Shibaev *Soviet Zolotoprom* 1935, No 6, 44-51.—Most of the Au in these veins is in the free state and only a small fraction in the form of sulfide. Free Au content in the ore from Retiv and Aleksandrovsk vein is 1.97 and 4.51 g per kg ore, resp. A mixt. of these ores was ground to a fine pulp and extd. by a cyanide method. Grinding the ore to 65 mesh gave an 85% Au extn by this method.

S. L. Madorsky  
Cyanidation (of gold ores) I V Paramonov. *Soviet Zolotoprom* 1935, No 7, 37-40.—A discussion. S. L. M.  
Occurrence of rare elements in gold slime. V. Zemel *Soviet Zolotoprom* 1935, No 7, 23-5.—Results of analysis of 42 samples of Au slime, collected from various Russian gold mines, are given. Among the elements found in the slimes were: Zr, W, V, Ti, Th and Mo.

S. L. M.  
Recovery of fine gold particles and treatment of slime. D. Kugushev *Soviet Zolotoprom* 1935, No 6, 23-4.—A discussion.

S. L. Madorsky  
Variety of improvements noted in concentration and milling. Charles E. Locke *Mining Met.* 17, 13-15 (1936).—The Hadel mill has been improved. Finer feed to ball mills is increasing. Advances and changes in design in various crushers, ball mills, screens, classifiers, thickeners, agitators, filters and flotation machines are described. For filtering small tonnages of concentrates in Au-flotation plants, small pan filters are used. A rubber-glass filter medium shows some advantages over canvas. Use of jugs of Lake Superior Fe ore is increasing. New flotation reagents are listed. Interesting developments in several plants are described briefly.

A. H. C.  
Present situation and the future of the aluminum industry in Japan Zentaro Nakanishi *Aluminium* 17, 610-14(1935).—The historical development of the Al industry since its inception is 1901 is described. Statistics on production and imports of raw materials and Al ingots and scrap are given, and the present situation and outlook are reviewed.

M. Hartenhem  
Progress in the field of beryllium extraction and the preparation of metallic beryllium, 1930-35. R. Strauss *Angew Chem* 48, 745-50(1935).—A summary covering the following subjects: decompn. of Be minerals with F compds., with alkalis, acids and Cl<sub>2</sub>, the sepn. of BeF<sub>2</sub> from the alkali fluoride in the soln. of the double fluoride, concn. of Be in the mineral, the prepn. of metallic Be and the prepn. of Be by means of vacuum distn. Thirty-nine references.

Karl Kammermeyer  
Recent trends in design and construction of copper concentrators in the Southwest. C. E. Rork. *Bur. Mines, Information Circ. No. 6860*, 16 pp (1935).  
A. H. E.  
Few changes in lead metallurgy reported. Carle R. Hayward. *Mining Met.* 17, 23(1936).—New developments in secondary Pb technology and modifications in the Betts electrolytic process are discussed briefly.

Alden H. Emery  
New methods for recovery of magnesium. Willy Machu *Metall u Erz* 32, 565-70(1935).—A discussion of



previously used methods for recovery of Mg and efforts to avoid formation of  $MgCl_2$  as an intermediate step. New methods for electrothermal reduction of  $MgO$  with  $C$  are described. H. Stoertz

Silver and silver pitchblende ore from Bear Exploration and Radium, Limited, Great Bear Lake, N. W. T. W. B. Tamm, et al. *Can. Dept. Mines, Mines Branch, Rept.*, No. 747, 30-43(1935).—About 90% of the Ag can be won by tabling and flotation or blanket concn. and flotation, but the tailings carry 100-200 oz. Ag per ton that can be recovered only by chem. treatment. Amalgamation recovered 91.5-93.7% of the Ag, flotation recovered only 27% of the Ag in the tailing and left a 100 oz. tailing. Cyanide extn. is high, but cyanide consumption is excessive. Tabling recovered 54.7% of the pitchblende in a concentrate contg. 39.09% pitchblende, but left a 3.06% tailing. Flotation showed little concn.

Origin and growth of graphite nuclei in solid and liquid iron solutions. H. A. Schwarz and Wolfram Ruff. *Am. Inst. Mining Met. Engrs. Tech. Pub. No. 655*, 17 pp. (1935).—In the white cast irons the no. of nuclei per unit vol. is not detd. by the melting and freezing conditions alone, but also by the temp. at which the nuclei are caused to grow. One sort of nodule that never grows to any considerable size is numerous, 10-20 times as frequent as the larger. These small nodules appear to increase in no. with time at graphitizing temp. The larger nodules do not alter greatly in no. with time but do change in size. At any given time, the size distribution of nodules is not very wide. The no. of larger nodules (and perhaps the no. of smaller ones) decreases as the graphitizing temp. decreases, and in a given Fe the max. size attained by nodules increases. The density of graphite packing in the nodules decreases as the graphitizing temp. increases; it is remarkably const. at a given temp. The no. of nodules is not solely a function of the graphitizing temp. Vestigial nuclei are of great importance in detg. the no. of nuclei that will grow into nodules. Graphitizing rate is enormously increased by previous quenching of white cast Fe from about 1000°, such treatment produces an enormous increase in no. of nodules. The no. of nuclei that will grow into nodules or mottles is a function of the materials of the charge, chem. compn., compn. of the melting furnace atm., melting and pouring temp., rate of cooling and temp. of germination of the graphite. A. H. E.

Separation of hematite by hysteretic repulsion. E. W. Schilling and Harwick Johnson. *Am. Inst. Mining Met. Engrs. Tech. Pub. No. 654*, 13 pp. (1935).—As the air gap between the plane on which the specular hematite was supported and the Fe core of the magnet was increased, the activity of the particles reached a max., then decreased. The speed of the moving particles increased as the frequency of excitation was increased to about 70 cycles per sec., then was const. As the relative amplitudes of vibration increased the wt. of hematite moved increased. Heat treatment of the hematite in a reducing atm. of  $H_2$  increased its activity. Alden H. Emery

Silvicultural damage from the arsenic acid in the white smelter smoke of the arsenic metallurgical plant F. Hilsch. *Tharandt forstl. Jb.* 85, 117-66(1934); *Rev. Applied Mycol.* 14, 725, *Biol. Abstr.* 9, 1001.—A summary of a century of research on As damage in German forests shows injury extends 2-3 km. from the As plant in the direction of the wind and is more severe on the outer fringes than inside the stands. Of hardwoods ash is most susceptible, then beech, birch, oak, and poplar. Of the conifers spruce is most susceptible, then fir, Scotch pine (*Pinus sylvestris*), white pine (*P. strobus*), Douglas fir (*Pseudotsuga taxifolia*), and larch. The toxic effect shows on browning or burning of the foliage. Oden E. Sheppard

The chlorine method for treating copper pyrites slag. D. M. Chuzhikov and Sh. Sh. Margolina. *J. Chem. Ind. (Moscow)* 12, 811-18(1935).—Slag contg. Cu and Zn is chlorinated for 2 hrs. at 100° with enough  $Cl_2$  to form  $CuCl_2$  and  $ZnCl_2$ . If the slag contains 10%  $H_2O$ , the heat of the reaction raises the temp. to 110-20° and no further heat need be applied. The presence of  $N_2$  and  $CO_2$  in the  $Cl_2$

has no effect on the reaction. The slag is then leached for 0.5 hr. with cold  $H_2O$ , and 93-5% of the Cu and 92-4% of the Zn are removed. The soln. is treated with  $CaCl_2$  to ppt. sulfates and so give purer products later. The Cu is pptd. by Fe, and the Zn by  $CaO$ . The slag can be used in the blast furnace. H. M. Leicester

Slag in the manufacture of ferromanganese. Maurice Dénbéré. *Chimie & Industrie* 34, 777-9(1935).—In the so-called "slag method" of producing Fe-Mn, the phys. and chem. study of the slags must be considered as being as important as that of the metal obtained. A characteristic example is given and discussed, showing how the study of the differences in color of a slag can furnish valuable indications on the mechanism of the working of the furnace. A. Papineau-Couture

Five years of progress in southern blast furnace practice. Francis H. Crockard. *Am. Inst. Mining Met. Engrs. Tech. Pub. No. 653*, 20 pp. (1935).—The major developments in southern blast-furnace practice during the past 5 yrs have been (1) reduction in ash in coke, (2) improved structure and porosity of coke, (3) sizing of coke and ores, (4) careful selection of ores from chem. standpoint, (5) wider stock lines, (6) improved offtakes and downcomers, (7) improved bosh design, (8) widespread use of McKee distributors, (9) introduction of machine-cast Fe and (10) improvement in phys. characteristics of Fe. Coke consumption has been reduced from about 3000 lb. per ton to about 2200 lb. and flue dust production from 250-300 lb. per ton of Fe to 50-100 lb. Chem. analysis of Fe has been improved. Furnaces are kept more uniform on the desired grades. Man-hrs. have been lowered through mechanization. A. H. L.

Dimensions and lines of modern blast furnaces. M. A. Pavlov. *Rev. mt.* 32, 451-65(1935).—A survey of the contemporary trend of design of blast furnaces in Europe and the United States. P. offers a design of the furnace which would give the best results on the basis of his wide experience. J. D. Gat

Nature, formation and removal of dust through the top of a blast furnace as a function of constitution of obolite ores. J. P. Arend, A. Jungblut and C. Aschman. *Rev. mt.* 32, 263-392(1935).—Minette ores were investigated. About one-third of dust is charged with the ore, the rest is formed in the furnace by attrition and shrinkage caused by dehydration. Dehydration is not a continuous function of the temp. One-fourth of the water is removed between 100° and 200°, one-half between 200° and 320° and the rest requires heating to 500°. Top-gas dust can be sep'd into three groups according to the magnetic properties, depending on the temp. levels to which the ore had time to settle in the furnace. Amount of dust removed is proportional to the kinetic energy of gases. J. D. Gat

The control of cupola operation. H. L. Campbell and John Grennan. *Iron Age* 136, No. 25, 18-23, 84-6 (1935).—Specific practical suggestions are given for improved cupola practice. F. G. Norris

Determination of the losses through burning in the cupola furnace by gas analysis. Try-Chalons. *Rev. fondre moderne* 29, 292-3(1935).—The method developed by McConachie (cf. *Foundry*, 63, 26(1935)) is explained and discussed. Although much more exact than methods which det. the losses in metal by weighing charge, product and slag, it is not considered accurate enough as it does not take account of the C of the combustion gases originating from the decompn. of the limestone (flux) and as the free O is not detd. exactly enough. However, the method is considered satisfactory for comparing the performances of furnaces. M. Hartenheum

Open-hearth roof temperatures. H. M. Schmitt. *Metals & Alloys* 6, 333-5(1935).—Four types of pyrometer installations are described, each having its own characteristics but all providing continuous records: (1) thermocouples imbedded under insulation, (2) thermocouples inserted in roof brick, (3) thermocouples mounted in refractory insert in water-cooled tuyère, (4) radiation pyrometer sighted into refractory tube mounted in the roof. Downt Schiaff

**Solution of the problem of the hot stoves.** Maurice Derclaye. *Rev. mét.* 32, 427-50 (1935).—Comprehensive mathematical analysis of the phenomena occurring in blast-furnace hot stoves. Double-zone stoves have a better temp. drop, which can be achieved in single-zone stoves by increasing the thickness of checker bricks. Parallel study of single- and double-zone stoves shows mathematically that the effects obtained are the same, but the single-zone type is less complicated. Heavier checkers in the upper zone can be dispensed with at the present because smaller bricks with the same mechanical and physical properties are already manifold. Efficiency of the stoves can be raised from the present 88.3 to 92% by preheating the air by passing it between a specially built outside and the usual shells of the stove. Frequency of reversals has a preponderant influence. Doubling this frequency will correspond, for example, to doubling the cond. of the bricks and their thickness. J. D. Gat.

**Changing to gas fuel improves Columbia tool steels.** Winfield Foster. *Ind. Gas* 14, No. 7, 9-10, 16 (1936).—The steel heating furnaces were altered for gas firing and equipped with low-pressure burners. Motor-operated proportioning valves are used to give the correct air and gas supply. Controls are located near the furnace, and permit automatic maintenance of desired temps. Decarburization of the surface of the steels has been reduced  $\frac{1}{2}$ , and scaling has been reduced  $\frac{1}{4}$ , so that the scale removed with the first hammer blows is only paper-thin. The thermal efficiency of the furnaces was increased approx. 50% by elimination of stack and infiltration loss, and greater temp. uniformity was secured. R. W. R.

**Applied spectroscopy in metal-working industries.** C. Heidhausen. *Mitt. Forschungsanstalten GHH-Konzern* 4, No. 3, 59-70 (1935).—The description of the spectrographic lab. of the MAN-Nuremberg serves to demonstrate how with simple and conveniently arranged app. spectroscopy can be used as an ordinary tool in exams and routine testing of materials, and also for detns in analyses. The principles of the measuring methods and the instruments are described. Six references.

M. Hartenheim

**The pressed-metal industry.** O. P. Hatton. *Iron Age* 136, No. 22, 20-1, 94-6; No. 21, 34-6, No. 26, 26-9 (1935).—Historical review. F. G. N.

**Treating metals and alloys.** Société d'exploitation des procédés Mahoux, Paris. *Oberflächen tech.* 12, 285-6 (1935).—The new process, Ger. 621,200 (C. A. 30, 10199), of treating, especially steels and Fe alloys, is based on the fact that materials upon heating are put in mol vibrations. It was found that the quality of the metallic structure is changed if in the metals or alloys under a heat-treatment, electromagnetic or mech vibrations of ultrasonic frequency are generated which practically do not contribute to the heating; this is of particular advantage for treatments of materials with low transformation temps, e. g., C steels at 500°, which must not be exceeded in the treatment. The frequency of the ultrasonic vibrations applied lies between 1 and 10 million per sec. (far beyond the audible range). In the presence of these oscillations cementing and nitriding can be done at lower temps. than usual, as C and N penetrate into the surface and harden it. M. Hartenheim

**Methods of testing embrittlement of metals.** P. B. Mikhailov-Mikheev. *Zavodskaya Lab.* 4, 940-50 (1935).—The results of tests of various steels are shown in graphs and are discussed. Chas. Blanc

**Apparatus for testing hardness of metals.** F. P. Marshanskii. *Zavodskaya Lab.* 4, 822 (1935).—Construction details with illustrations. Chas. Blanc

**Modern methods of testing fatigue in metals.** N. N. Afanas'ev. *Zavodskaya Lab.* 4, 951-8 (1935).—A discussion. Chas. Blanc

**High standards are maintained in heat-treating springs.** J. E. Coleman. *Industrial Gas* 14, No. 6, 7-8, 25 (1935).—A gas-heated continuous hardening furnace for springs is equipped with an alloy muffle and can also serve for drawing with a temp. range of 750-1750°F. Its rated capacity is 250 lb per hr. A quench chute is provided, sealed

from the outside air, the oil temp. being maintained automatically. The muffle atm. is obtained by burning 570 B. t. u. city gas with  $\frac{3}{4}$  cu. ft. of air per cu. ft. of gas, this amt. being insufficient for complete combustion, so that the resultant product is rich in CO and CO<sub>2</sub>. Automatic temp. control is used. After hardening, the work is washed and then tempered in a salt bath, after which it is washed again. R. W. Ryan

**Action of carbide etching agents.** R. Mitsche. *Arch. Eisenhüttenw.* 9, 311-12 (1935/6).—Expts with different alloy steels showed that to obtain a satisfactory carbide etching action the presence of an oxidizing agent besides hydroxyl ions is necessary. A soln. of KMnO<sub>4</sub> in KOH or NaOH is particularly useful. An explanation of this action is seen in that ferrous ions are going into the soln. from the baser ferrite under the action of the hydroxide, and the nascent H ions are discharged on the nobler cementite. The oxidizing agent oxidizes H to H<sub>2</sub>O and the Fe(OH)<sub>2</sub>, formed at first from the ferrous ions to Fe(OH)<sub>3</sub>, which ppts on the cementite and darkens it more or less according to the duration of the etching. Six references. M. Hartenheim

**Theory of the conversion of metallic mixed phases.** III. Phase diagram of partially oriented mixed phases. C. Borelius. *Ann. Physik* 24, 499-506 (1935) of C. A. 28, 50261.—The theory is extended and considered thermodynamically. Sample phase diagrams are shown. The properties of new alloys can be predicted. R. E. DeR.

**Method of polished etching for metallographic tests.** N. M. Zarubin and M. V. Sutin. *Zavodskaya Lab.* 4, 780-90 (1935).—It is considered that the method of chem. etching with subsequent burnishing of samples for metallographic examn. is preferable to any other procedure. Methods for prepn. of samples of metals unsuited for chem. etching because of their chem. or phys. nature are being investigated. Chas. Blanc

**Polishing in drums.** F. Schwarz. *Oberflächen tech.* 12, 243-6 (1935).—Polishing in drums is applied to glass articles, especially for Cu-, brass-, Ni- or Cd-plated ware, Cr-plated articles cannot be polished in this manner. Rusty or scaly articles are first scoured in a wooden, cylindrical drum with sand which has been wetted with water or dil. H<sub>2</sub>SO<sub>4</sub>. A polishing soln. for Al and Al alloys is made up of acid K fluoride 500 g., Na bisulfate 400 g., Neol 100 g. and water 100 l. For Fe and steel articles the soln. consists of soap flakes 4 kg., soda 2 kg., NaCN 0.5 kg. and water 100 l. For Cu and Cu alloys, soap powder 3 kg., potash 2 kg., NaCN 1 kg. and water 100 l., grain soap 4 kg., tartar 1 kg., Na bisulfate 1 kg. and water 100 l. Polishing with balls is used particularly for strongly profiled parts, the balls being of very hard, highly polished Cr steel of 0.5 to 10 mm. diam. The procedure and the design of the drums are described. Ten references. M. Hartenheim

**Hot-rolling of tubes of rectangular cross section.** G. A. Nedel'ko, G. P. Pishchikov and P. M. Shuvalov. *Dames* 1935, No. 7, 64-9. S. L. Madorsky

**Thin plates, their manufacture, surface treatment and working.** Koch. *Oberflächen tech.* 12, 291-2 (1935).—The mfg. processes of sheets of 0.5 mm. thickness and less for various industrial purposes and for fine mechanics, chem. treatment for surface quality, and requirements for deep-drawing are discussed. M. Hartenheim

**Effect of working conditions in the tinning process on the porosity of tinplate.** Fritz Peter and Geo. Le Cal. *Arch. Eisenhüttenw.* 9, 285-91 (1935).—The causes of porosity of tin coatings on steel sheets were investigated in the range from 30 to 110 g./sq. m. Porosity depends mainly on the purity of the surface of the sheet and on the thickness of the coating, then also on the pickling method and on the purity of the flux used. The H absorbed especially in pickling does not have as large an effect as is usually assumed. An abs. freedom from pores is at present not obtainable in com. grades of tin-plate in spite of recent improvements in the tinning process. Only by beginning at a thickness of Sn coat of 100 g./sq. m. can porous-free sheets be made under observations of all possible precautions. M. Hartenheim

Softening and recrystallization of pure aluminum H R Aluminum 17, 575-6(1935).—The expts of Calvet (C A 29, 68674) to det the effect of impurities on the velocity of softening (melting) are discussed. The velocity depends to a large degree on the purity of Al. The recrystn velocity is very high for the pure metal and increases rapidly with increasing temp. M H

Additions which reduce the attack of solutions on aluminum H Rohrig Aluminum 17, 559-62(1935).—The disintegration of Al by alkali solns is prevented not only by adds of Na silicate (as discovered in 1929) but also by other colloidal substances, as animal and vegetable glues. As an explanation of this protective action it is assumed that the colloids deposit on the metal surface because of their elec charge. Expts were made with nicotine and nicotine sulfate in HCl solns, dibenzyl sulfide in HCl soln, and several special solns of different origins with pure Al and Al alloys, the results are shown in tables and show remarkable reductions in the amt. of metal dissolved. A no. of industrial and domestic applications are described. M Hartenheum

The action of tap water on aluminum Tsunetaka Sasaki and Jitsusaburo Sameshima J Chem Soc Japan 56, 1353-8(1935).—Al dissolves noticeably on being boiled with tap water. This is attributed to the action of HCO<sub>3</sub> contained in the tap water. T K

Heat treatment of cast iron R G McIlwain Trans Am Foundrymen's Assoc 6, No 6, 27-40(1935).—Various desirable combinations of compn and heat treatment are enumerated with particular regard to specific practical applications. F G N

The warm strength of cast iron with particular consideration of stress-walled castings W H Uhitzsch and W Leineweber Arch Eisenhüttenw 9, 185-92(1935).—As discrepancies exist in the literature on the mech properties of cast iron, new technological, metallographic, phys and chem investigations were carried out which permit one to conclude that the strength up to 200° is affected, besides other causes, by the magnetic transformation of the cementite. Tensile tests with 13 different cast irons showed that Si, Ni, Cr and Mo have a smoothing effect on the fluctuations of the property curves, this seems to be due, for Ni and Si, to a reduced magnitude of cementite transformation, for Cr and Mo to a shifting of the transformation point towards lower temps. In general, it can be said that the behavior of castings under heat depends not only on the nature of the pig Fe used and the remelting process but also just as much on the molding and pouring processes and the interior stresses caused by the casting process. The investigation extended up to 750°, the tests results are illustrated by curves. Twenty-nine references. M Hartenheum

Study of natural and artificial aging of iron castings by the string method A I Ilnikov and V P Tarasova Zavodskaya Lah 4, 779-86(1935), cf. C A 29, 70044. —A discussion of practical methods of prepn of samples for acoustic measurements of the residual tensions by the method previously described. Chas Blanc

Structural changes in gray iron subsequent to annealing V S Prever Industrija mehanika 17, 978-82(1935).—An ordinary type of gray Fe was heated to different temps and for different lengths of time and the structural changes were observed in the microscope. No typical changes can, however, be stated, the complexity of the phenomena does not permit of a uniform explanation. Metallographic conditions will, in most cases, have to be examd micrographically. M Hartenheum

Factors affecting the structure and properties of gray cast iron A Di Giulio and A E White Trans Am Foundrymen's Assoc No 35-9, 35 pp (preprint)(1935).—The cast Fe used for this investigation was of the unalloyed automotive cylinder type contg total C 3.20, graphite C 2.76, Si 2.00, S 0.118, P 0.36 and Mn 0.48%. The beneficial effects of superheating begin at 1480° and reach a max at 1670°. The optimum range of pouring temp is 1470° to 1565°. Improved phys. properties and smaller and more nearly uniform graphite flakes are the result of pouring at temps lower than the

superheating temp. During the cooling before pouring the graphite flakes dissolve completely in the melt and are thus refined. Quenching molten cast Fe in H<sub>2</sub>O gives evidence that graphite will not entirely dissolve at the eutectic temp. If once dissolved, graphite cannot be pptd in the melt by cooling. Rules for improvement of the phys properties of unalloyed cast Fe are, the superheating temp should not exceed 1650°, too high superheat makes hard low-strength Fe; holding at a lower temp for a longer time will have almost as good an effect as superheat; pouring temp should be 1480°, charges with excessively coarse graphite flakes should be avoided, because these are more difficult to dissolve and may result in coarse flakes in the product. F G Norris

Impact resistance and other physical properties of alloy gray cast irons Garnet P. Phillips. Trans. Am Foundrymen's Assoc No 35-1, 18 pp. (preprint)(1935).—Samples from 38 cupola and 20 elec. furnace heats were tested to det transverse breaking load, deflection, and resilience, Brinell and Rockwell hardness, tensile strength, and repeated drop impact strength. Charpy and Izod tests were made on some of the compns. The transverse resilience bears a straight-line relation to the single-blow impact strength. The relation of resilience to repeated drop impact strength is not as definite. The relation between transverse strength and tensile strength is shown. The best impact resistance of the irons cast gray (not including austenitic Fe) was found in a 4 Ni-Mo iron of which the following compn is representative: total C 3.41, graphite C 2.69, combined C 0.72, Si 2.11, S 0.04, P 0.16, Mn 0.60, Ni 1.93, Cr 0.21 and Mo 0.90%. Austenitic Fe of low hardness has high impact resistance. The highest impact resistance is in samples cast white and annealed to give gray Fe. The compn. of the best sample is Si 1.33, total C 2.32, combined C 0.71, Mn 0.50, Cr 0.04, Mo 0.30 and Ni 0.19%. This sample had a resilience almost twice as great as the next best sample and 3 to 6 times as great as most of the samples. It did not break in any of the impact tests until after machining to less than standard section. F G Norris

The influence of phosphorus on the mechanical properties of gray cast iron at high temperatures Max Paschke and Friedrich Bischof Gieserei 22, 447-52(1935).—Eleven heats were melted in a 50-kg high frequency furnace and poured at 1290° into green sand molds to form bars 450 mm long and 30 mm diam. from which the test specimens were prep. The compn was approx 3.70% total C, 2.90% combined C, 1.90% Si, and 0.08% S with Mn varied in the range 0.50 to 1.50% and P in the range 0.14 to 1.22%. The tensile strength, impact strength, and Brinell hardness were detd at temps from 20° to 900°, 700°, and 600°, resp. Up to 500° there are max in the tensile strength curve at 0.33 and 0.88% P and a min at 0.58% P. The general trend is decreasing tensile strength with increasing P. At 700° the min is less pronounced and occurs at 0.64% P. At 900° increasing P has small effect on the tensile strength. For all compns the tensile strength and hardness are approx const. with temp. up to 500° above which they decrease markedly with increased temp. Up to 500° the hardness curve has min. at 0.58 and 0.69% P and max at 0.64 and 0.77% P. Increasing P from 0.88 to 1.22 has a small effect on hardness, which is in contrast to a marked decrease in tensile strength beyond 0.88% P. At 600° there is a min in the hardness curve at 0.69% P. The highest impact values are at 400° in specimens contg 0.69 and 0.77% P. There is no definite relation between impact strength and P content. Fourteen references. F G Norris

Wear resistance of white cast iron O W. Ellis, J. R. Gordon, and G S Farnham. Trans. Am Foundrymen's Assoc No 35-4, 19 pp (preprint)(1935).—A ball mill test using 1/4-in Si carbide grain for the grinding medium was used to study the effect of compn and structure on the wear resistance of cast-Fe rods and balls in ball mill service. Forty-seven alloys were prep. in 10 to 12-lb quantities by use of graphite crucibles lined with sillimanite or, in case of high-Mn melts, with mg-

nesia. Most of the alloys contained 2.5% total C, below 0.03 graphite C, 0.75% Si and 0.50% Mn. The higher the C, the lower the wear resistance of white cast Fe. Variation of Mn content up to 1% Mn does not affect wear resistance. Increase of Si from 0.75% to 1.50% has no effect in white cast Fe, but increases the wear resistance of mottled cast Fe. Variation in Cu above its soly limit (from 0.05% to 5.25% Cu was studied) has small effect on wear resistance. During the early stages of test the high-S alloys have high wear resistance. After the hard skin is worn away S is detrimental to wear resistance. Marks made by an elec. pencil stood out in relief at the end of the test, this indicated a local increase in wear resistance produced by partial decarburization and the formation of martensite. Differences in Vickers hardness do not indicate the relative wear resistance. F. G. N.

Cinematographic record of the  $\alpha \rightarrow \gamma$  iron transition, as seen by the electron microscope. W. G. Burgers and J. J. A. Ploos van Amstel. *Nature* 136, 721 (1935). — By use of an electron microscope with one magnetic lens, pictures were taken at 4 sec. intervals of the  $\gamma \rightarrow \alpha$  Fe transition, at 900°. G. M. P.

Experiments on the diffusion of carbon, silicon and manganese in solid and liquid iron. H. Paschke and A. Hauptmann. *Arch. Eisenhüttenw.* 9, 305-9 (1935/6). — The diffusion coeffs. for C, Si and Mn were detd. by the method of Graham (cf. C. 26, 5282) and by the method of infinitely long half-spaces (cf. *Sitz. Akad. Wiss. Wien* 79, 161 (1879)). The results agreed very well in the temp. range from 900° to 1250°. The diffusion of Mn in solid Fe is slower than that of C. In the liquid state of Fe, only very approx. figures could be obtained for Mn and Si. The coeffs. are given in curves in sq. cm./hr. as function of temp. Ten references. M. Hartenheimer.

Investigations into the phosphatic protection of iron obtained with the biphosphates of zinc, iron and manganese. O. Macchia and G. Boggio. *Industria meccanica* 17, 817-21, 906-9, 901-5 (1935). — A great no. of tests were made to find the principal factors which det. the protective action against atm. influences by phosphates with solns. of the biphosphates of Fe, Zn, Mn, Fe-Zn, Fe-Mn and Zn-Mn. The general conclusions of the results can be summarized as follows. Solns. of Zn biphosphate of about 3% offer a good protection but require too much time (85 min.). Solns. with 3.5% Mn biphosphate are good and more rapid. Fe biphosphate cannot be used, as it has no appreciable protective value. Fe-Zn biphosphate solns. of 3% concn. give a very high protective value. Zn-Mn biphosphate of 4% concn. and high in Mn biphosphate is satisfactory both from the point of view of protection and duration. Fe-Mn biphosphate gives the best results both from the viewpoint of behavior of the protective surface and from rapidity of treatment. The last one is recommended for all practical uses. Procedures are described, patents given and 41 references cited. M. Hartenheimer.

The reactions of iron with liquid zinc. W. Pungel, Erich Scheil and R. Stenkhoff. *Arch. Eisenhüttenw.* 9, 301-4 (1935). — The reactions taking place and the formations of alloys between Fe and Zn during the hot-galvanizing process were studied in the temp. range of 450°-500°. The attack of Fe by liquid Zn can occur in 2 different directions, according to whether the crystal FeZn<sub>13</sub> is or is not formed. If no FeZn<sub>13</sub> is formed, the attack is very strong and a loose crystal skeleton of FeZn<sub>13</sub> is formed so that Zn can combine continually by soln. The thereby originating layer of alloy grows vertically to the Fe surface and creates oddly shaped excrescences. The non-formation of FeZn<sub>13</sub> is, however, observed only up to a definite upper temp. The formation of this Fe-Zn alloy provides a tightly enveloping protective layer which reduces considerably the attack of the steel by the Zn bath. The relation between the chem. compn. of the steel or the Zn bath and the occurrence of FeZn<sub>13</sub> could not yet be explained sufficiently by the present observations. Five references. M. Hartenheimer.

The velocity of solution of iron in molten zinc. Herbert Grubisich. *Angew. Chem.* 48, 689-91 (1935). — The ac-

tion of pure Zn upon Armco iron was studied by measuring the amt. of Zn reacting with an Fe plate of known area. The temps. were kept const. within  $\pm 1.5^\circ$  and the exptl. results showed variations of 3-5%. The "velocity of soln." (1) increases only slowly (for a given test period) from 430° to 480°. The structure of the alloy layer shows a dense, compact cover on the iron basis, and exhibits distinct boundaries with the iron and the pure Zn layer. According to Daniels, the amt. of Fe dissolved is  $E = nt$ , where  $\log n = -(1256/T) + 2.705$ ,  $t$  is the time and  $T$  the temp. In this temp. range the growth of the thickness of the alloy layer behaves like that of dense metal oxide films. In the range of 480° to 520° I depends on the temp., Armco iron shows a definite max. at 495°. The alloy layer breaks up into a great no. of small crystals, which penetrate into the Zn layer, this eliminates the sharp boundary between the alloy and Zn layers. The migration of the crystals results in a decrease of the alloy layer thickness. The galvanization structure is very characteristic at the temp. of the max. The amt. of Fe dissolved in this range can be expressed by  $E = kt$ , the proportionality factor  $k$  depends on the temp., but could not be detd., as it varies greatly with the various kinds of iron examd. At 495° and 515° the linear increase of the dissolved Fe with  $t$ , up to the equal concn., could be observed. At temps. above 520° the structure of the alloy again becomes dense, this results in a decreased soly, and the thickness of the layer decreases greatly with increasing temps. The amt. of Fe dissolved is proportional to  $\sqrt{t}$ . Four other steels, with the amt. of C varying from 0.11 to 0.50%, and a Swedish iron (C = 0.03) were examd. likewise. A comparison of the soly. curves showed that the values almost coincide in the temp. ranges of 440-80° and 520-600°, while variations of 300% occurred at the curve max. at 600° (reaction time 1 hr.). No conclusions for the differences at the max. and the different behavior in the second temp. interval could be drawn from the chem. compn. A smooth "av. soly. curve" was established which can be expressed as  $E = 10^{-3} n + 0.0017 (t - 419) \times \sqrt{t} (f/1.7)$ , where  $T$  is the (const.) test temp., 419° the m. p. of Zn,  $t$  the extent of the test period and  $f$  the surface of the iron. The agreement of the calcd. and exptl. values is very satisfactory up to 600°, but above that temp. the calcd. values are considerably higher. Cold working of Swedish iron affected the soly, resulting in a broadening and simultaneous lowering of the max., and a shifting to a higher temp. as the amt. of cold work increased. Soly. curves and photomicrographs are presented. Twelve references. K. K.

Metallographic control of stamping sheet iron. A. M. Shirokov. *Zavodskaya Lab.* 4, 130-6 (1935). — Metallographic examn. satisfactorily shows the stamping properties of sheet iron as affected by the size and form of ferrite and cementite inclusions, contamination with slag and air bubbles, C contents, etc. Chas. Blanc.

Cause of longitudinal cracks on the surface of finished (steel) products. A. I. Nachal'nikov and Tz. N. Rafalovich. *Domes* 1935, No. 7, 17-26. — Cracks on the surface of steel products can be explained exclusively as a result of bursting of gas bubbles in the process of rolling in those cases where liquation or decarburized areas are found in the immediate neighborhood of these cracks. In the absence of such areas, the cracks can be explained either as due to bursting of gas bubbles, or to mech. strain, or to scratches resulting from rolling. S. I. M.

Cracks in round castings. A. Yakhkind and N. Gotgel'i. *Domes* 1935, No. 7, 26-32. — A study was made of the effect of the following factors on formation of longitudinal cracks in round steel castings: (1) diam. and thickness of casting, (2) C content, (3) rate of pouring the molten metal and (4) Si and S content. It is concluded that to avoid cracks, the rate and temp. of pouring should be low, particularly in castings with a diam. larger than 300 mm. The optimum rate of pouring is 1.3-1.5 ton/min. The no. of cracks increases considerably with increase of thickness. Low-C steels (up to

(0.25% C) have 3-4 times as many cracks as high-C steels (0.4-0.6%). S. L. Madorsky

**Cracks in forged pieces.** M. Ballay *Usine 44*, No. 46, 27-9 (1935).—The cause of fine fissures which sometimes are observed in large rolled or forged pieces and which are quite different from the dendritic and quenching cracks was investigated, but a satisfactory solution to the problem has not been found. The tendency to form these cracks (in French called "floccons") existed in increasing degree in C steels, Mn steels (1-1.5%), Mn-Si steels, steels with 1-2% Cr with or without Mo, Ni steel (1.5%), Cr-Ni steel and Cr-Ni-Mo steels in the order named. Steels made in the crucible or acid open hearth, and such made in high-frequency acid furnaces are less subject to these cracks than the basic open hearth or electric furnace steels. The tendency diminishes if temp. and velocity of pouring are reduced. Sufficiently slow cooling after forging is also important, about 1° per min. between 900° and 200° seems sufficient, which means about 10 hrs for this interval, and for very heavy pieces up to 80-100 hrs. The orientation of these cracks is perpendicular to the greatest deformation pressure and to the dendrites. Impurities do not seem to play any part. M. H.

**Determining purity of steel by deep-etching.** E. Houdremont and H. Schrader *Arch. Eisenhüttenw.* 9, 273-5 (1935).—In order to detect the impurities in steel, inclusions and segregations of P, by deep-etching, it has been found necessary to make this test only after heat-treatment, because in the untreated state the etching process may attack preferably the grain boundaries, especially in coarse grain. A correct etching shows in the longitudinal section mainly the P segregations, in the transverse section mainly the sulfide inclusions. The deep-etching test is, therefore, considered better suited to distinguish steels of different metallurgical production. The method is illustrated by numerous photographs of etchings demonstrating the different types of steel. Eight references. M. Hartenbaum

**Transformation of austenite and the theory of hardening of steel.** S. S. Shternberg *J. Tech. Phys. (U. S. S. R.)* 5, 467-82 (in Russian), *Tech. Phys. U. S. S. R.* 1, 395-603 (1935) (in English), cf. C. A. 29, 2422.—Expts with 0.7 and 1.3% C steel, high-speed steel, and Cr steel contg. 12% Cr and 2% Ni indicate that austenite decomposes in 3 ways. Between Ar' and 300° pearlite or troostite is formed, between 300° and Ar' cubic martensite is formed, and below Ar' tetragonal martensite. In the 3rd case, tension existing in the steel is alone sufficient to cause the transformation. Above Ar' the tensile stresses are insufficient and diffusion phenomena are required to initiate the decomposition. H. W. Rathmann

**The brittleness of steel at sub-zero temperatures and rapid-cooling brittleness of annealed steel.** Saburo Watanabe and Kiyochi Nagasawa *Tetsu-to-Hagane* 21, 890-7 (1935).—The brittleness of steels at sub-zero temp. was studied by means of Charpy's impact test. The impact values of the annealed C steels (0.04-0.38% C) drop between +25° and -20°, and those of quenched steel drop between +25° and -40°. From the result of the impact test at sub-zero temp. of several alloyed steels, it was found that steels contg. Ni show less brittleness than those contg. no Ni. The brittleness of steels at sub-zero temp. is not characteristic of a iron itself, but is due to the presence of carbides. By the impact test of C steels (0.10-0.26% C) which were annealed (920-870°), reheated (700-700° for 1 hr.) and then quenched in water, an extreme brittleness was found at room temp. This phenomenon was called "rapid cooling brittleness of annealed steel." This brittleness is attributed to the pptn. hardening of Fe<sub>3</sub>C at low temp. Tomoo Sato

**Effect of uranium on structure, hardenability and tempering permanence of unalloyed steels.** H. Benack and C. G. Holzschner *Arch. Eisenhüttenw.* 9, 193-200 (1935).—Alloying expts with ferrouanium proved that practical  $\gamma$  segregation free castings can be produced with a low-C, low percentage ferrouanium if it is added in small pieces to a well-densitized melt in the ladle. High-C ferrouanium with high U content is not

suitable because of its difficult soly. The exptl. castings could be forged satisfactorily up to 5% U. Two new constituents were observed in C steels with U addns which can be considered to be U carbides. Hardening and temper tests showed a certain soly. of the  $\gamma$ -solid soln. for U which, however, can be utilized in practice only to a limited degree, as melting phenomena occur at already fairly low temps. U improves uniform hardening over the whole vol. and reduces the sensitivity against overheating. U acts in the steel similarly to Cr as a special carbide-forming element, but improves the mech. properties of structural steels very little. Micrographs and curves illustrate the details of the expts. Eleven references.

M. Hartenbaum  
**The effect of special additions on the scaling of steel.** K. Kewt *Mitt. Kohle- und Eisenforschung G. m. b. H. 1.1*, No. 2, 9-23 (1935).—The scaling phenomenon of steels at temps. of 900°, 1000°, 1100° and 1200° was investigated with particular consideration of the part played by addns. The formation of scale by diffusion created sharply defined layers of scale which were analyzed individually. All alloying elements of steel, base as well as noble elements, accumulated at the border of the Fe scale. Al, Cr and Si which increase the resistance against scaling noticeably, and Ti, Be and V which increase it only slightly, form protective layers. The addn. of a nobler element, as Cu, Ni and Pt, did not give an improvement in resistance against scaling, Pt even lessened resistance. The investigation led to the conclusion that in order to obtain protection against scaling the new kind of crystal which is formed by the diffusion of the Fe atom, but not by that of the O atom, must possess a low diffusion velocity and be sol. in ferrous oxide, and besides must form a tight envelope around the Fe which also prevents the diffusion of Fe. The compn. of scale layers is shown in photographs, the tests and results are discussed. Twenty-six references.

M. Hartenbaum  
**Steels and alloys for permanent magnets.** André Michel *Usine 44*, No. 46, 27; *Rfr. met.* 32, 482-6 (1935).—A brief review of steels used at present for this purpose and their qualities and treatment. Fe-Co-Mo and Ni-Al steels are best suited according to the present state of knowledge.

M. Hartenbaum  
**Decarburization of some alloy steels by hydrogen.** W. Baukloh and H. Guthmann *Arch. Eisenhüttenw.* 9, 201-2 (1935/6); cf. C. A. 29, 2422.—Tests on decarburization in a H<sub>2</sub> atm. at 700-1000° showed an increase in the decarburization velocity for unalloyed steels above 700° with increasing C content, while below 700° it decreases with increasing C content. In Ni steels, the decarburization decreases with increasing Ni content, with a max. at about 850°. The effect of H on the favorable decarburizing action of Cr is very slight up to 0.9% Cr, but becomes fairly large for higher Cr contents. Mn steels show the least attack by H for about 0.5% Mn in the investigated temp. range. The decarburization velocity is reduced for Si, W, Mo, Sn and Cu, the latter is least attacked at about 0.65%. Increasing Si and W contents increases the resistance against H attack, while Mo shows an irregular behavior with a max. attack at 0.48% Mo. V alloy steels were decarburized to a greater degree than unalloyed steels; a steel with higher V content shows lesser decarburization. Five references. M. H.

**Effect of cold deformation and annealing on properties of chromium-molybdenum steel tubes.** Iz. N. Rafalovich. *Dones* 1935, No. 7, 32-56.—Samples of Cr-Mo steel tubes having an av. compn. of C 0.34, Mn 0.7, Cr 0.82, Mo 0.29, P 0.040 and S 0.045%, and crit. points Ac, 820° and Ac, 740°, were heated to various temps. and then slowly annealed in the furnace. The samples were then cold-rolled. Mech. properties and microstructure were investigated before and after cold-rolling. The cold-rolled samples were also studied in regard to recryst. by heat-treating them for varying periods at 600°, 650°, 700°, 750° and 800°. Change of mech. properties was found to depend only on the extent of deformation. Samples subjected to a preliminary heating to 750° suffered the least disturbance in structure during deformation.

tion. Numerous tables, graphs and photomicrographs are given. S. L. Madorsky

The carbon-oxygen equilibrium in molten steel. Sato Matoba. *Tetsu-to-Hagane* 21, 875-9 (1935).—Equil. of C and O in molten steel is expressed as follows, (2)  $\text{FeO}(s) + \text{CO} \rightleftharpoons \text{Fe} + \text{CO}_2$ , and (3)  $\text{C}(s) + \text{CO}_2 \rightleftharpoons 2\text{CO}$ , where  $\text{FeO}(s)$  and  $\text{C}(s)$  mean the FeO and C dissolved into the molten iron, resp. By melting of Fe in mixed gases of CO and  $\text{CO}_2$  at various rates, the above relations were investigated at 1570°, 1600° and 1620°. As long as the compn. of the gas mixt. remains unchanged, the soly. of C in the molten Fe in equil. with the gas mixt. decreases, while that of O increases as the temp. rises. The relations of temp. (T) and equil. const. (K) and changes of free energy ( $\Delta F^\circ$ ) were detd. as follows: For equil. (2),  $\log K_2 = 7.450/T - 4.60$ ,  $\Delta F^\circ = -34,000 + 21.31T$ , for (3),  $\log K_3 = -8.800/T + 7.40$ ,  $\Delta F^\circ = 10,242 - 33.84T$ . Tomoo Sato

Spectrographic examination of structure of alloys. Henri Triché. *Compt. rend.* 201, 1178-81 (1935).—The surface of the alloy (e.g., Al-Fe or Al-Cr) is etched by an oxidizing mixt. and a high frequency spark produced between its surface and a thin Au or Pt wire, the wire being moved about to prevent fusion. The sparks always start from certain fixed points on the surface of the alloy, these being crystals of definite compn. projecting above the general (oxidized and so nonconducting) surface. From the spectrum of the sparks the compn. of the crystals is deduced without their isolation. Thus with Al-Cr alloys contg. 2, 4, 4.2 or 17% Cr the same spectrum is obtained, this indicates the crystals contain approx. 18.2% Cr, in fair agreement with 21.4% required by  $\text{CrAl}_3$  (cf. Link and Freche, *C. A.* 28, 1970°). C. A. Silberrad

A new low-melting alloy. S. J. French. *Ind. Eng. Chem.* 27, 1464-6 (1935).—A quaternary alloy contg. 81.9% Lpowitz metal (50 Bi, 27 Pb, 13 Sn, 10 Cd) and 18.1% In proved to have a f. p. of 46.5°. Because of the high price of In, this low-melting alloy would cost, at present market prices, close to 5 dollars an oz. in small lots. The cost in large lots may be 1/2 of this figure. D. S.

Simple means of distinguishing different aluminum alloys. E. Zurbrugg. *Aluminum* 17, 531-3 (1937).—A simple method is described by which Mn, Mg and Ni can be detected even in very small amts. Mn is found by oxidation with  $\text{NH}_4$  persulfate in the presence of  $\text{AgNO}_3$ , Ni by dimethylglyoxime, and Mg by tetrahydroxyanthraquinone. The alloys of the type Al-Mg-Si, Al-Mg and Al-Mg-Mn which cannot be distinguished by these methods (and also not by those described by Zeilereder (*C. A.* 29, 1045°) and Boschard (*C. A.* 29, 2134°)) are distinguished from one another by hardening tests in conjunction with scratch or ball tests. Procedures are described. M. H.

The power consumption in forging and pressing aluminum alloys. A. v. Zeilereder and R. Irrmann. *Z. Metallkunde* 27, 145-8 (1937).—The general problem of working Al alloys is discussed, showing that the power requirements are much greater than for Cu alloys and comparable to those for Fe. Compression tests to show the effects of single components upon pure Al at temps. between 20° and 420° show that with adds. of up to 10% the power consumption increases more for Mg than Cu, while up to 2% Mg-Si has the least effect of any, com. alloys of the Al-Cu-Mg and Al-Mg-Si types both require more power than the above results would indicate, on account of their Mn content. Trip-hammer expts. on Fe at 900°, Avional at 420°, Fe at 1100°, Anticorodal at 180°, Fraca (57.7 Cu, 2.1 Pb, 40.2 Zn) at 700° and 99.5% Al at 500° showed the power requirements decreased in the order given. Die-forming of the same materials showed the importance of well-polished dies and forms, especially for the Al alloys. G. Derge

Testing of sea-water-resistant aluminum alloys in the north sea. H. Röhrig and W. Nicolini. *Aluminum* 17, 519-20 (1937).—Smooth, riveted and welded samples of pure (99.7%) Al, Albondur, Pantal, Hydronalium, and KS and PS sea-water Al alloys were exposed to the tides in the North Sea for 1 1/2 years. Surface quality, tensile

and shear strength and elongation were tested; the results are shown in tables and photographs. The BS and KS alloys and Hydronalium, both hard and soft, withstood perfectly the effects of the exposure for 1 1/2 years. The welded samples of pure Al (hard as rolled), Pantal and Hydronalium also did not change their properties. The riveted samples suffered very little; the shearing strength remained the same in all samples which were not corroded. Hard pure Al showed a loss of strength and elongation.

M. Hartenhein  
The effect of metals of the iron group on precipitation hardening of aluminum alloys of high purity. W. Koch and F. W. Nothling. *Aluminum* 17, 535-6 (1937).—The effect of the metals Fe, Cu, Ni and Mo on pptn. hardening was investigated on an Al-Cu alloy with 4% Cu by taking time-hardness curves at room temp., at 135° and at 200°. The results can be summarized briefly as follows: Test series with Fe. Quench hardness as well as pptn. hardness at all temps. decreases with increasing Fe content. This is ascribed to the fact that Cu is bound by the addn. of Fe and thus taken out of the solid soln. A ternary compd. is thereby formed,  $\text{Cu}_2\text{FeAl}_3$ . Test series with Co. Co is practically insol. in the Al-Cu alloy with 4% Cu in the solid state. It occurs as an intermetallic compd. of the probable compn.  $\text{Co}_2\text{Al}_3$ . At 525°, the quenching hardness shows an increase, partly because of the hardening actions of the pptns. and partly on account of an increase of the concn. of the solid soln. by removal of Al on account of the binary compd. Adds. of 0.1-0.2% Co impede the pptn. strongly at first; the increase of the pptn. hardness is about the same as that of the quench hardness. Test series with Ni. The view held by Bingham that Al-Cu-Ni alloys do not show pptn. hardening to any pronounced degree (*J. Inst. Metals*, 1926, 177) does not seem to be correct, as the tests with Ni: up to 2% show a slowly decreasing pptn. hardening with increasing Ni content, the decrease has a certain ratio to the quench hardness. The reason for the decrease is seen in the decrease of the amt. of Cu available for pptn. of the solid soln., and is due to formation of the ternary compd. Test series with Mo: The deciding influence in Al-Cu-Mo alloys is exerted by the originating Al-Mo compd.,  $\text{MoAl}_3$  or  $\text{Mo}_2\text{Al}_3$ . The increasing hardness after quenching from 300° is due to the hardening effect of the pptd. components. The results are represented in curves, all metals were added in amts. up to 2%. The curves which show maxima of pptn. hardness at different temps. are parallel among themselves, as also to the curve of quench hardness at 525°, except at very small amts. of addn. From this it is concluded that both quench hardness and aging and pptn. hardness are subjected in principle to the same effects, which view is also confirmed by examn. of the structures. For ternary alloys, it can be stated in general that the pptn. hardening is proportional to the Cu dissolved in the solid soln. According to whether Cu-free or Cu-contg. compds. occur, quench and pptn. hardness will be more or less affected. Sixteen references.

M. Hartenhein  
Transformations in copper-aluminum eutectoid alloys. I. Intermediate states in the hyperectoid alloys. G. Kurdymov and T. Steltelk. *Techn. Phys. U. S. S. R.* 2, 3-16 (in German). *J. Tech. Phys. (U. S. S. R.)* 5, 395-406 (1935) (in Russian).—Alloys contg. 10-15% Al were quenched from 600° to 930° and examd. by the Debye-Scherrer method. In alloys contg. less than 13% Al the  $\beta$ -phase changes to  $\beta'$ , in those contg. more than 13% Al, it changes to a  $\gamma'$ -phase. This  $\gamma'$ -phase decomposes in 2 distinct steps when heated. Decompn. starts at 300° and proceeds to a definite limit with the formation of  $\gamma$ . The remaining  $\gamma'$  decomposes only when heated to 425-50°, forming  $\alpha + \gamma$ . If the hyperectoid alloys are quenched in a salt bath above 300°, some  $\gamma$ -phase is pptd. from the  $\beta$ -phase. The remaining  $\beta$ -phase with reduced Al content is stable for a long time below 425°. If, however, the hyperectoid alloy is quickly cooled below 300°, the  $\gamma$ -phase is not pptd., and the  $\beta$ -phase will change to  $\gamma'$  at 170-200°. The transformation  $\beta \rightarrow \gamma'$  is irreversible and is comparable to

the austenite-martensite transformation in steels

H. W. Rathmann

The abnormal phenomena of cast copper-rich antimony-copper alloys during heating Yoshio Tanaka and Masakatsu Ito *J. Chem. Soc. Japan* 56, 1293-1300 (1935); cf. Taketani and Katori, *C. A.* 30, 654.—The abnormal thermal expansion of the alloy (Sb = 2-9%) was studied by means of differential expansion measurement with pure Cu as standard. With the decrease of Sb the abnormal thermal expansion near 490° and 490° decreases. The limit of the solid soly of Sb at 470° is estimated to be 9.5-9.75%.

T. Katsurai

The abnormal phenomena of cast copper rich magnesium-copper alloys during heating Euchi Koizumi and Takeluko Kawaguchi *J. Chem. Soc. Japan* 56, 1300-4 (1935), cf. preceding abstract.—The thermal change of the alloy (Mg = 0.2-10%) prepared by die casting has been studied. The alloy (Mg = 0.8%) shows noticeable expansion near 550°.

T. Katsurai

The influence of the third metals on the normalization of cast structures of bronze Saburo Katori and Yasushi Ogino *J. Chem. Soc. Japan* 56, 1305-13 (1935), cf. preceding abstract.—To the bronze (Sn = 10%), 0.5-5% Zn, Mn, Ag, Ni, Sb, Al, Si, Mg were added, resp., and with pure Cu as a standard the differential dilatation of the alloys has been studied. The presence of Zn seems to accelerate the normalization (homogenization) of the cast structure by annealing, while the others retard the normalization.

T. Katsurai

The formation of the graphite eutectic in the iron-carbon alloy Tomoo Sato and Seiji Nishigori *Tetsu-to-Hagane* 21, 887-93 (1935).—Three kinds of the pure white pig iron (3.54, 4.05 and 4.85% C) were melted in a high-vacuum furnace which was specially devised with W wire as heating element, and thermal analysis was carried out. The eutectic reaction in the pure Fe-C system takes place in 2 steps which differ by 7-10°. This phenomenon agrees well with Ruer and Goerens' result, and the change which takes place at the higher temp., in cooling, is due to the formation of the austenite-graphite eutectic, and that of the lower temp., to the austenite-Fe<sub>3</sub>C eutectic. In the specimens analyzed, 2 kinds of eutectics mentioned above were observed under the microscope. Therefore, the correctness of the double diagram of the Fe-C system was pointed out. By assuming that the degree of dissociation of Fe<sub>3</sub>C in the molten pig iron decreases as the amounts of some impurities (Cr, S, etc.) increases, the readiness of formation of graphite eutectic in the pig iron melted in high vacuum was explained. The mechanism of the formation of the mottled iron and reversed chill casting was explained by this assumption.

Tomoo Sato

Iron-carbon beryllium alloys Marcel Bailly *Compt. rend.* 201, 1124-6 (1935).—From 0 to 4% Be was added to cast irons containing 3-4.45% C, 0.25 Mn, 0.25 Si, 0.02 S and 0.05 P, they were then cast in bars 15 mm in diam. in stove sand. A series averaging 3.2% C and consisting (in absence of Be) of pearlite cementite showed on addition of increasing amounts (up to 3.85%) of Be first steps of graphite, then with more than about 1% Be, an increasing amount of a complex cementite containing Be, with more than about 2% Be this gradually disappeared until with 3.85% Be there was scarcely any, in place thereof was Be<sub>3</sub>C (perhaps containing a little Fe), which formed gray grains often with polygonal outlines attacked by boiling H<sub>2</sub>O, but preserved from oxidation when the alloy is decarburized at 950° by a film of oxide. There was little change, save a slight decrease with less than 1% Be, in hardness of the alloys as cast or after oil tempering at 950°, but with 1.43% Be a marked increase of about 80 and 50%, resp., followed by a decrease with more Be until with 3.85% the hardness scarcely exceeded that of the original cast Fe. Addition of Be caused little change in Ac.

C. A. Silberrad

The system iron-cobalt-cobalt silicide-iron silicide Rudolf Vogel and K. Rosenthal *Arch. Eisenhüttenw.* 9, 237-9 (1935).—The ternary system Fe-Co-Si was studied

thermally and microscopically in the range Fe-Co-CoSi-FeSi. The compounds FeSi and CoSi form an uninterrupted series of solid solutions. The existence of a ternary compound, FeCoSi, was established; 2 subregions are distinguished, Fe-Co-CoSi-Fe-CoSi-FeSi and FeSi-FeCoSi-CoSi-CoSi. The crystalline phenomena in the former partial range are detected essentially by 5 four-phase planes of which 2 are transition planes and 2 eutectic planes, while the fifth plane corresponds to a ternary eutectoid disintegration. In the second partial region, the pairs of the opposite border systems are very similar. Since the border systems FeSi-CoSi and CoSi-FeCoSi are uninterrupted series of solid solutions, the three-phase equilibria of the border system CoSi-CoSi go over into those of the system FeSi-FeCoSi. The diagram of state and reaction occurring in the various zones are discussed and illustrated by photographs of structures. M. Hartenhein.

The rolling and recrystallization structure of an iron-nickel alloy W. G. Burgers and J. L. Snoek. *Z. Metallkunde* 27, 153-60 (1935).—The (200) pole figure is determined for a 50% Fe-Ni sheet reduced from 0.35 mm to 0.1 mm by cold-rolling. This is found to be in agreement with the findings for other face-centered cubic metals and is best described as a scattering about all orientations for which a {135} plane is parallel to the plane of rolling, with a {325} direction in the direction of rolling. Recrystallization in H<sub>2</sub> at 1100° a preferred orientation is found with a {100} plane in the surface of the sheet and a {100} direction in the direction of rolling, and not more than 10° of scatter. The same textures were found for a 35% Ni alloy and were not altered by 2.5% Cu, but completely destroyed by small amounts of Co or Al. It is suggested that the differences in recrystallization texture found among face-centered cubic metals can be explained by differences in fragmentation processes which occur along slip planes during deformation. The anisotropy of magnetization was observed on the recrystallized foil and found to be in agreement with previous work, magnetic susceptibility least in the {100} direction.

G. Derge

The relation of the rolling and recrystallization textures of iron-nickel alloys to their magnetic properties Fr. Pawlek. *Z. Metallkunde* 27, 160-5 (1935).—Reversible Fe-Ni alloys were prepared by melting Swedish Fe with Mond Ni and by sintering carbonyl Fe and Ni, but no marked differences of behavior were found. All data are for a 40% Ni, 60% Fe specimen in the rolling direction. For 50, 80 and 98.5% reductions the normal combination of orientations was found with a {112} direction and a {110} plane in the direction and plane of rolling, resp., or a {111} direction and a {112} plane. With reductions of less than 90% the recrystallization structure was random, above this a texture appeared and became completely {100} in the rolling direction with {010} in the plane of rolling when annealing above 800°. Between this temp. and that of recrystallization, 500°, another undetected orientation was evident. Curves are shown for the magnetic and tensile properties after the above reductions and for annealing temps. between 20° and 1100°, all of which show sharp breaks corresponding to the recrystallization temps., with additional differences dependent upon whether or not the recrystallized material is randomly oriented. Annealing above 1100° produced exceptional grain growth and a distinctly new preferred orientation with a {100} direction in the rolling direction and a {120} plane in the surface. Subsequent cold rolling of recrystallized material with the cubic texture produced no change in this orientation with less than 80% reduction, though transition to the normal texture is complete with 90% reduction. Reannealing this originally cubic material produces no texture for reductions of less than 90%. Corresponding anomalies are found in the magnetic and tensile properties. Reduction of 50% produces the lowest residual magnetism, and special curves are shown for initial material of both the random and cubic types of orientation, the latter having a higher recrystallization temp. extending over a wider range, accompanied by anomalies in the recovery curves for the other properties.

G. Derge

Nickel alloys in the architectural field G. F. Geiger

and R. E. Case. *Ind. Eng. Chem.* 27, 1130-2(1935).—Color and compn. are discussed. P. S. R.

Investigation of special nickel silvers. D. G. Butomo *Metallogr.* 10, No. 4, 76-87(1935).—Alloys contg. Cu 81-83, Ni 14-5 and Al 1.5-3% were prep'd, heat-treated and exam'd for microstructure and mech. properties. When quenched from 800° to 900° these alloys form solid solns and are soft and ductile. After annealing the quenched specimens at 400° they become hard because of the pptn. of an Al comp'd. The annealed specimens had a tensile strength of 80 kg./sq. mm. and an elongation of 10%. H. W. Rathmann

Phase diagram and magnetic properties of platinum-iron alloys. I. Graf and A. Kussmann *Physik. Z.* 36, 544-51(1935).—The object of this work was (1) to det. the exact course of the magnetic properties, (2) to det. the concn. regions within which the intermediate phases exist and (3) to det. the relation between structure and magnetic properties. Sixteen alloys covering the entire concn. range were employed. For each sample the coercive force, the satn. value and the magnetic transformation temp. were det'd after annealing and after quenching from selected temps. up to 1100°. X-ray and micrographic methods were applied to structure and phase-existence detns. The temp. of the  $\alpha$ - $\gamma$  transformation of 1 e is gradually lowered by addn. of Pt up to 26 at. % where  $\gamma$ -1 e is stable at room temp. A very large hysteresis exists (0.600°) at this concn. Addn. of Pt up to 8.9 at. % (solid soln) increases the satn. values of  $\alpha$ -Fe by about 10%. The lattice parameter increases somewhat (from  $a_0 = 2.87$  to  $a_0 = 2.89$  Å). The alloy contg. 26 at. % Pt while in the  $\gamma$ -phase at room temp. can be transformed by cooling in liquid air. In the concn. field 35-65 at. % Pt the alloys quenched from temps. above 1200° exhibit micrographically a homogeneous  $\gamma$ -solid soln in which an oriented needle formation appears. X-ray structure diagrams indicate the presence of another lattice beside that of the  $\gamma$ -phase. This new phase is FePt and is body-centered cubic with  $a_0 = 2.88$  Å. The 2 sorts of atoms appear to be randomly distributed. At 50 at. % Pt FePt forms at temps. below 1200° and is ferromagnetic. The Curie point of FePt is about 450°. In the concn. field 30-70 at. % the alloys exhibit an extraordinarily wide hysteresis loop with coercive forces up to 1800 gauss at a remanence of 4000 C.G.S. and thus exhibit a higher magnetic energy content than any other known alloy. It is considered that this large amt. of energy (80,000 erg/cc) is intimately connected with the structural transformation. H. A. S.

Use of zinc alloys for water and gas pipes and distribution equipment in dwellings. R. Cazaud and H. Petot. *Génie. Civil* 105, 31(1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 67(June 29, 1935), of *C. A.* 29, 79231.—Upon exposure of Zn to air or water, the Zn becomes coated with a protective layer of carbonate. With Zn-Al alloys contg. 5-25% Al this film is very tenacious and forms rapidly. Such alloys consist of a mixt. of a solid and eutectic soln in different proportions and can be forged, cast, rolled, drawn and annealed at about 300°. The structure and mech. properties of the alloys can be improved by the addn. of traces of Mg, Sb and Ti. The alloys resist corrosion by waters contg. large doses of Cl and O<sub>2</sub>. They are very suitable for meters, as elec. reactions are reduced to a min. The e. m. f. developed between Ni and Zn is negligible. Pipes of 10-100 mm. interior diam. can be economically constructed from Zn-Al alloys. The amt. of Pb dissolved by water varies with the degree of aeration; some waters can carry 18-35 mg. of Pb per l. and Leipzig water up to 236 mg. Tests on guinea pigs show Al and Zn are much less toxic than Cu, and "total" alloys are 8 times less toxic than Cu. The Zn-Al alloys are very suitable for central heating systems and radiators as they conduct heat better than cast Fe and are unaffected by oil, hydrocarbons, alcs. and many chemicals. C. R. Fellers

Phase changes during aging of zinc-alloy die castings. II. Changes in the solid solution of aluminum in zinc and their relation to dimensional changes. M. L. Fuller and

R. L. Wilcox. *Am. Inst. Mining Met. Engrs., Inst. Metals Div., Tech. Pub. No. 657*, 13 pp (1935); cf. *C. A.* 28, 66824.—The dimensional changes that occur in Zn-alloy die castings (A. S. T. M. alloy No. 27) during aging and low-temp. annealings have been shown to be due to changes in the  $\alpha$ -phase. These changes are the segregation from, or the resoln. of  $\gamma$  in, the  $\alpha$ -phase. This relationship has been est'd semiquantitatively by a comparison of dimensional changes calcd. from x-ray data on the phase structure with those changes actually experienced. The evidence indicates strongly that the  $\beta$ -phase decompn. plays a minor part, if any, in the aging shrinkage of this alloy. Room-temp. aging of freshly cast material resulted in a segregation of  $\gamma$  from  $\alpha$  and an accompanying shrinkage. The annealing at 150° and 175° of castings that had been dimensionally stabilized at room temp. resulted in the resoln. of  $\gamma$  into  $\alpha$  and a corresponding expansion. Com. stabilizing annealing of 3 hrs. at 95° resulted in a more rapid depletion of the Al content of  $\alpha$  than takes place during room-temp. aging and a correspondingly more rapid shrinkage. Some further change in the  $\alpha$ -phase and further shrinkage took place during room-temp. aging following the annealing. Prolonged annealing at 95° (2 weeks) increased the extent of the aging effects during the subsequent room-temp. aging. C. L. Mantell

A study of lead bronzes with special consideration of manganese as an alloying element. J. Wecker and H. Nipper. *Z. Metallkunde* 27, 149-51(1935).—A survey of bearing bronzes shows that although Sn is required in high-Pb alloys, it is use objectionable because of the widening of the two-phase liquid region, the introduction of the  $\delta$  eutectoid, and the possible formation of stannic acid. A suitable substitute for Sn is sought in an alloy contg. 30% Pb. Ni reduced the Pb segregation but gave a soft alloy. Alkalies, alk. earths and P had no beneficial effects, while Co required prolonged high-temp. melting and gave poor phys. properties. Fifty-kg. melts contg. 63% Cu, 30% Pb, 2% Ni and 5% Mn showed no segregation under a variety of casting and cooling conditions, and no stannic acid was formed. The frictional and shrinkage properties for this Mn-Ni bronze were about the same as those for standard white bronzes, while the hardness, temp. coeff. of hardness and impact hardness were all much better than for standard Sn-bearing alloys. G. Dürge

Porous bronze. A. Chaplet. *Cuivre et laiton* 8, 502-3(1935).—Under certain conditions it may be desirable to have a porous metal as it has certain advantages, for instance, if the free surface of the metal is to be as large as possible so that a gas or a liquid can act upon it, or if lubricants have to be incorporated in the metal very intimately to reduce friction on the surface. Such metals are used for bearings, bushings, valve cocks, etc. They are made by alloying the metal with another one which crystallizes out in solidification and creates fine pores or interstices between the crystals, e. g., an alloy of 50% Pb and 50% Sb begins to solidify at 450° and to deposit Sb crystals. A com. porous bronze "olite" is made of a mixt. of 90% Cu powder and 10% Sn powder which is compressed in steel forms to the desired shape, it is then heated to a point intermediate between the m. ps. of the 2 metals when bronze is formed. The pores take up 40% oil in vol. so that automatic lubrication takes place. M. H.

The corrosion resistance of Hydronalium, especially to sea water. G. Siebel. *Aluminium* 17, 562-7(1935).—Because of their resistance to alkalies, the Mg-Al alloys possess an excellent corrosion resistance to sea water, which increases with increasing Mg content. The heterogeneous structure of the cast state of high-Mg-Al alloys is practically as resistant to sea water as the homogeneous structure. The wrought alloys of Mg-Al become very temp.-sensitive by cold-deformation, which results in a strong, intercryst. corrosion. A stable structure can, however, be obtained by heterogeneization below the demixing line. Addns. of Ca, Si, Zn, Ti and Cr strengthen the homogeneous state and render these alloys safer against corrosion. M. Hartenheim



The welding of chromium steels in chemical plant equipment J R Dawson *Can Chem. Met.* 19, No 12, 337-8 (1935) — The rods chosen in oxyacetylene welding should be as nearly as possible of the same compn. as the material being welded. A smaller flame should be used on stainless steels than on ordinary steels to avoid overheating of the weld. X-ray examn and the free-bend method of testing for ductility have been of great value in checking up on welded products. The selection of the correct steel and procedure for welding for each type of service are important. W. H. Boynton

Performance of refractories used in basic alloy steel production (Soler) 19 Influence of metals in bleaching (Pettinger) 25 Selection of lubricants for cold-rolling of strip steel (Moore) 22 Staybrite steel in the dyeing industry (Godber) 25 Philippines: a source of chrome ore (Keeler) 8 Production and use of high-grade Zn (Breyer) 13 Grinding (Uhlmann) 19 Fe-O diagram (Bogatzki) 2 Rotary-drum app for filtering and dewatering slimes such as those from ores (U S pat 2,024,358) 1. Sizing and segg fines from crude materials such as ores (U S pat 2,026,318) 1

Processing laterite ores Ernest W. Westcott (to Comstock & Westcott, Inc.) Brit 434,897, Sept. 11, 1935. See Fr. 775,161 (C. A. 29, 2139) and Can 353,342 (C. A. 29, 7928).

Roasted blende Bergwerksgesellschaft Georg von Giese's Erben. Ger. 611,684, Sept. 13, 1935 (Cl. 40a 2.20). The blende in a porous nonagglomerated form, easy to leach out, is produced by roasting a mixt. of 1 part of crude blende with 5-10 parts of completely roasted blende contg less than 5% of sulfidic S.

Roasting sulfide ores Edward P. Fleming (to American Smelting and Refining Co.) U. S. 2,026,621, Jan. 7, 1935. In roasting sulfide ores (such as those of Zn) in suspension, air and the ore are introduced tangentially into the upper portion of a preheated roasting chamber so as to establish a concurrent roasting zone, and a countercurrent roasting zone is established by introducing addnl. air into the lower portion of the chamber in countercurrent flow to the descending ore particles, resulting gases being withdrawn from a neutral zone at the interface of the concurrent and countercurrent treating zones. App. is described. U. S. 2,026,622 relates to similar app. and procedure in which the peripheral velocity of the introduced air and ore is progressively increased as the air-ore mixt. descends, so that solids are deposited close to the outer periphery of the chamber. Cf. C. A. 29, 3288.

Treating sulfide ores containing zinc, copper and iron Thomas A. Mitchell (to Hughes Mitchell Processes, Inc.) U. S. 2,025,068, Dec. 24. A sulfide ore such as one contg sulfides of Zn, Cu and Fe is mixed with a Pb compd. such as PbS or PbO proportioned for combining with the sulfate radical formed during a roasting operation, and the mixt. is roasted to form a product contg the oxide of the desired ore metal such as ZnO interspersed with Pb sulfate. Various details of treatment are described.

Fusing iron ores Karl Koller and Zsigmond v. Galocsy Fr. 787,484, Sept. 23, 1935. In fusing Fe ores or briquets contg ores and C in a blast furnace, a part of the C necessary for the operation and the O are introduced in the form of a mixt. of hot gases prepd in a combustion chamber connected with the furnace by complete combustion of a fuel with O or air enriched with O. A furnace is described.

Treating iron ores containing various companion metals Friedrich Johannsen (to Fried. Krupp Grusonwerk A.-G.). U. S. 2,026,683, Jan. 7. For reclaiming, from ferriferous raw materials such as ores, companion metals (such as Cr, Mn, Mo, etc.) reducible at higher temps than Fe, a mixt. of the material with a reducing agent such as comminuted waste coke is passed through a metallurgical furnace such as a rotary tubular furnace in which the charge is agitated and preheated up to temps at which Fe compounds present are converted into iron sponge (suitably to 600-900°), then further preheated (suitably to 900-1400°) to a

temp at which, while the companion metal compds. remain unreduced, the sponge iron is about to lump; a current of oxidizing gases is caused to react with the preheated charge so that a portion of the Fe of the sponge is burnt and addnl. heat is produced, sufficient for effecting the lumping of the iron sponge and for producing from the Fe oxides and the slag-forming constituents of the ore, which are fluxible by Fe oxide, slags contg. the companion metal compds. and Fe oxides sufficiently liquid as to sep from the lumping iron; the Fe oxides of the slags are reconverted into lumped iron, while the companion metal compds. remain unreduced, and the slags and lumped iron are jointly removed from the furnace and are subsequently sep'd from each other. App. is described.

Chloridizing zinc ores. Lafayette M. Hughes Brit 434,800, Sept. 6, 1935. In chloridizing a roasted sulfide ore contg ZnO, the major portion of the ZnO is first chloridized, e. g., at 80-110° in an atm. contg. Cl, HCl, etc., the ZnCl<sub>2</sub> being optionally removed, and the ore is then treated in the presence of Fe<sub>2</sub>O<sub>3</sub> with HCl gas in substantial absence of air and at a final temp. of below 90° to form cryst. FeCl<sub>3</sub> which is caused to react with the unchlorinated Zn to form FeCl<sub>2</sub> and more ZnCl<sub>2</sub>. To complete the formation of chlorides, air may be passed over the treated ore in counter-current at 250-350° to decompose the Fe chlorides and form Fe<sub>2</sub>O<sub>3</sub> and Cl, the waste gases from this stage being utilized in the 1st chloridizing step.

Recovering metals from oxides. Österreichisch-amerikanische Magnesit A.-G. (Konrad Erdmann, inventor) Austrian 143,621, Nov. 25, 1935 (Cl. 40a). A mixt. of a metal oxide and C is fed at regular intervals, in small quantities at a time, to a furnace heated to above the b. p. of the metal at the working pressure. The conditions are so selected that a rapid reaction occurs and accumulation of the charge in the furnace is avoided. Also, the furnace is preferably so constructed that by-products or impurities which remain solid under the working conditions are carried away by the gaseous and vaporized reaction products. The initial mixt. may be fed to the furnace in the form of small briquets. The process is particularly useful in the production of Zn, Cd and Mg. Plant including an elec. arc furnace is described.

Aluminum waste Metallurgische Erbslöh A.-G. Ger. 618,731, Sept. 14, 1935 (Cl. 40a 50.20). Al waste, especially foil, is pressed, or rolled to form a transportable mass.

Apparatus for making centrifugal castings such as those of different steels Hans Breitbart. U. S. 2,026,457, Dec. 31. Mech. and operative details.

Casting mold Heraeus-Vacuumschmelze A.-G. and Wilhelm Rohn Ger. 618,870, Sept. 17, 1935 (Cl. 31c 26.01). A mold for the casting is made by sintering powd. W or Mo or their carbides. An addn. of powd. low-melting metals such as Fe, Ni or Co may be given.

Aluminothermic process Wilhelm Sander (to Th. Goldschmidt A.-G.). U. S. 2,024,132, Dec. 10. Alloying constituents such as Mn, Ni, Cr, Ti, V or W are added to an aluminothermic mixt. in a crucible, in admixt. with inert, refractory, slag-forming substances such as Al<sub>2</sub>O<sub>3</sub> or slag, and the mixt. is applied as a packing layer about the tap hole of the crucible to prevent flow of the aluminothermic melt prior to completion of the aluminothermic reaction (the usual aluminothermic mixt. being added to the crucible above the packing layer). App. is described.

Metallurgical furnace Levi S. Longenecker. U. S. 2,024,649, Dec. 17. Various structural, mech. and operative details.

Open-hearth metallurgical furnace of the reversing type Wm. M. Henry (to American Steel and Wire Co. of N. J.). U. S. 2,025,165, Dec. 24. Structural features of a furnace with water-cooled fuel nozzles.

Furnace construction suitable for open-hearth furnaces Leon T. Petit. U. S. 2,024,595, Dec. 17. A construction is described of bricks so shaped and arranged as to provide internal undercut pockets which serve to hold in place an inner lining of material, such as chrome ore and MgO or the like, resistant to attack by basic substances when highly heated.

Rotating-drum furnace for metallurgical and chemical operations. Metallgesellschaft A.-G. (Kurt R. Göhre, inventors). Ger. 618,872, Sept. 17, 1935 (Cl. 40a 5 01).

Furnace for roasting ores, etc., comprising superimposed rotary and stationary hearths. Balz-Erzrohung G. m. b. H. Ger. 621,100, Nov. 2, 1935 (Cl. 40a 4 53). Means is described for cooling the upper hearths with air.

Rotating furnace for roasting ores. Fried Krupp Grusonwerk A.-G. (Walter Hartwig, inventor). Ger. 618,600, Sept. 11, 1935 (Cl. 40a 5 01).

Furnace for roasting sulfidic ores and foundry products. Soc. anon. la nouvelle Montagne Ger. 618,757, Sept. 16, 1935 (Cl. 40a 2 01).

Crucible furnace suitable for melting metals. Furman South, Jr., and Oscar E. Neuhausen (to Lava Crucible Co. of Pittsburgh). U. S. 2,024,707, Dec. 17. Various structural details.

Apparatus for fusing metals. Enrique G. Touceda (to Consolidated Car-Heating Co.) U. S. 2,024,615, Dec. 17. An app. is described in which a spectral image of an enclosed blank such as one in an electrically heated furnace is formed outside the enclosure to indicate the incidence of fusion.

Apparatus (with a vertical reaction chamber) for roasting fines such as those of pyritic ores. Wm. J. Burgoyne and Henry J. Cordy (to General Chemical Co.) U. S. 2,025,709, Dec. 31. The finely divided material is introduced into the upper end of the app. and suspended in an oxidizing gas. Various structural and operative details are described.

Furnace for heating metal strip before rolling. Demag A.-G. Ger. 621,199, Nov. 2, 1935 (Cl. 18c 6 60).

Oil-fired two-chamber furnace for annealing and hardening reamers and like tools. Carl Stiefel Ger. 621,040, Oct. 31, 1935 (Cl. 18c 5 10).

Annealing furnaces. Siemens-Schuckertwerke A.-G. (Johann Schnepf, Otto Günther, Rudolf Grundmann and Heinrich Wassner, inventors). Ger. 618,821, Sept. 16, 1935 (Cl. 40d 2 40). Rollers for conveying metal objects through annealing furnaces are made from steatite heated to 600-700°. The steatite may be mixed with kaolin or feldspar.

Conveyor shaft for high-temperature furnaces such as those used for heat-treating metals. Oliver K. Carpenter (to Duraloy Co.) U. S. 2,024,024, Dec. 10. Structural and mech. details.

Pyrometric device suitable for use with air mains of blast furnaces. Howard A. Byrns (to National Tube Co.) U. S. 2,025,015, Dec. 17. Structural details of a device with a thermocouple.

Carburizing box for use in heat-treating furnaces. Henry H. Harris U. S. 2,026,311, Dec. 31. End parts of a carburizing box are formed of castings of relatively high-Fe-content alloy and central portions of the box walls and bottom are formed of flexible sheet metal of relatively low-Fe-content alloy. Various structural details are described.

Slag deflector for furnaces. Fred J. Chatel. U. S. 2,026,270, Dec. 31. Structural details.

Blast-furnace slag. Veremigte Stahlwerke A.-G. (Josef Roll, inventor). Ger. 621,282, Nov. 4, 1935 (Cl. 80b 5 04). The mech. properties of blast-furnace slag are improved by introducing silica into the furnace at a point below the tuyères. This is preferably effected by blowing sand into the furnace.

Magnesium. Oesterreichisch-amerikanische Magnesit A.-G. Fr. 787,476, Sept. 23, 1935. Powd. Mg is wetted with hydrocarbon oils which are removed by heating in an inert or reducing atm. and metallic Mg is produced in a compact form by heating the mass to a temp. near the b. p. of Mg until the particles agglomerate, or by completely re-evap. and condensing.

Metallic magnesium. Fritz Hansgig (to American Magnesium Metals Corp.) U. S. 2,025,740, Dec. 31. Mg vapor such as that produced by the thermal reduction of MgO by C and H is condensed to obtain solid Mg in finely divided form and the latter is then heated with a nonoxidizing gas such as H to nearly the b. p. of metallic

Mg to effect coalescence of the Mg particles. Cf. C. A. 30, 715.

Apparatus for removing lead from zinc by distillation. The New Jersey Zinc Co. Ger. 618,820, Sept. 19, 1935 (Cl. 40a 34 80).

Zinc, tin and lead oxide "Sachtblehen" A.-G. für Bergbau und chemische Industrie (Adolf Krus and Waldemar Jensen, inventors). Ger. 618,685, Sept. 13, 1935 (Cl. 40a 41 01). Zn, Sn, PbO and Ag are obtained by heating slag contg. these and roasted pyrites in a converter with air-blast nozzles in its base. The slag is either molten or mixed with coke. Examples are given.

Apparatus for refining zinc by distillation. The New Jersey Zinc Co. Ger. 618,873, Sept. 17, 1935 (Cl. 40a 34 80).

Bearing materials. Leadzinc Co. Brit. 434,277, Aug. 26, 1935. In a composite bearing element having a facing member of a hardened Pb alloy contg. not less than 95% Pb bonded to a metallic body or backing member, the bonding medium or solder consists of a Pb alloy contg. 20-30% Sn. The Pb alloy of the facing member is hardened by the presence of Ca, Na or other alkali or alk. earth metals, "Safco" and "Iary" metals and "Bahnmetall" being especially suitable. The surface of the backing member is covered with a corrosive flux, e. g., an aq. soln. contg. 50-75% ZnCl<sub>2</sub>, and dipped in the molten solder which is maintained at 340-370°. The hardened Pb alloy is then cast against it at not less than 480°.

Producing pipe from skelp. Walter T. Mahla and James L. Mauthe (to National Tube Co.) U. S. 2,025,001, Dec. 17. In a process involving heating skelp in a furnace and then passing it through a welding bell, heated air and a mixt. of air and O are successively applied to the edges of the skelp after leaving the furnace and before passage through the welding bell, to avoid undue burning.

Pipes or tubes from skelp. Julius L. Sussman. U. S. 2,024,493, Dec. 17. App. and various operative details are described, involving the use of elec. current for heating and welding the skelp.

Pipes and tubes from pierced billets. Bryant Bannister. U. S. 2,025,140-7-8, Dec. 24. Various details of app. and operation.

Hard lining metal for lining steel tubes, etc. Frederick A. Kormann and Walter F. Hirsch (to Industrial Research Laboratories, Ltd.) U. S. 2,025,000, Dec. 24. Cast iron of a C content of 2-5% is melted in contact with borax and in the presence of Si and extraneous C and the material is maintained in molten condition for from about an hr. to several hrs. to cause absorption of B to the amount of 0.15-4.0% of the metal.

Metal surfaces. Bozel-Malétra (Société industrielle de produits chimiques) Fr. 780,225, Aug. 29, 1935. Metal surfaces are made resistant to wear and seizing by coating them with Co in any manner.

Apparatus for removing scale from hot wheels, etc., during rolling by scraping. Amos G. Cole U. S. 2,024,993, Dec. 17. Structural, mech. and operative details.

Pickling metal blanks. Walter Heimberger. Ger. 618,614, Sept. 12, 1935 (Cl. 48d 2 01); Fr. 786,191, Aug. 28, 1935. The pickling acid consists of a mineral acid of less than 5% concn. and contg. in soln. up to 5% of a salt with a different anion and whose cation is a less noble metal than that to be pickled. The bath is heated to above 50°. Thus Fe is pickled in a bath contg. HNO<sub>3</sub>, 1 and MgSO<sub>4</sub>, 2% or HCl or H<sub>2</sub>SO<sub>4</sub>, 1 and KNO<sub>3</sub> or NaNO<sub>3</sub>, 2%.

Treating metal sheets, etc. Bethlehem Steel Co. Brit. 434,113, Aug. 22, 1935. Tendency to flute or stretcher strain in annealed or normalized low-C steel sheets is lessened by subjecting them to mech. strain and preventing their reversion to their original state upon heating and subsequent cooling by subjecting them to mech. strain during the cooling while still above 700°F., preferably at about 750°F.

Nitriding metal articles. Lorenz Stammler (to Leeds & Northrup Co.) U. S. 2,025,134, Dec. 24.

for imparting high surface hardness with a smoothly continuous and substantially linear hardness-depth characteristic, the temp of the load in a nitriding atm is maintained substantially const at about 465-540° for a certain period and subsequently, during a substantial portion of the total nitriding period, is increased to about 600-650°. App is described for control of the process.

Rolling sheet metals Howard G. McIlvried (to American Sheet and Tin Plate Co.) U. S. 2,025,002, Dec. 17. In working a thin and relatively wide metal strip, it is moved through a single-stand cold rolling mill at a linear speed of at least 400 ft per min., the cross-sectional area of the strip is reduced at least 35% on its first pass, and is further reduced at least an additional 35% while confining the material to not more than three passes after the first pass. App is described.

Rolling rails, etc. Herman J. van Royen. Brit. 434,955, Sept. 12, 1935. Rails are made from steel with lamellar-pearlitic structure contg. C less than 0.9, Mn 0.6-0.8 and Mo up to 0.3%, the work cooling to about 1000° before the final rolling pass so that it leaves that pass at below 900° and retains said structure. The steel may contain Cr 0-0.5, W 0-0.7 and (or) V 0-0.2, in which case the Mn may be reduced to about 0.5%. Si 0.2-0.3 and P and S below 0.04% may also be present.

Preventing formation of blisters in heat treatment of aluminum and its alloys Philip T. Stroup (to Aluminum Co. of America). U. S. 2,024,751, Dec. 17. Blistering due to gas formation is prevented by treating the molten metal, prior to casting, with about 0.05-5.0% of Na fluoroborate.

Heat treatment of malleable iron castings Cyril S. Smith (to Battelle Memorial Institute). U. S. 2,024,014, Dec. 10. Malleable iron castings contg. C 0.5-5% are heated to about 700-850°, cooled at a rate greater than approx 25° per hr to about 400-600°, and, without further cooling, maintained at a temp within the latter range for a sufficient time (which may be about 4 hrs.) to produce a substantial increase in hardness. Cf. C. A. 29, 3641.

Heat treatment of chromium alloy steels Frederick M. Becket and Russell Franks (to Electro Metallurgical Co.) U. S. 2,024,561, Dec. 17. Ferritic steels contg. Cr about 4-30, C not over about 0.23% and Ti at least about 4 times the C content and not more than 4 times the C content plus about 1.5% are heated to about 700-1000°, held at such temp. for about an hr. and then cooled to room temp.

Treatment of metals or alloys, particularly steel, with heat and supersonic waves Soc. d'exploitation des procédés Mahoux. Ger. 621,200, Nov. 2, 1935 (Cl. 18c 8.50). See Fr. 695,045 (C. A. 25, 1792), 700,049 (C. A. 25, 3304), 38,999 (C. A. 26, 1565) and 39,915 (C. A. 27, 608).

Magnetic iron and steel Heihachi Kamura. U. S. 2,026,390, Dec. 31. Carbonless reduced iron is mixed with high-P reduced iron produced by H reduction from Fe ore high in P, and the mixt is melted with a small addn. of Si and Ti.

Cementing steel and iron Hugo Hanusch (to E. F. Houghton & Co.) U. S. 2,025,050, Dec. 24. A compn. molten at cementing temps. and contg. material capable of supplying C to the metal at such temps. is formed of an alkali metal cyanide such as NaCN about 8, NaCl about 62-65, KCl 18-19 and an activated C of low combustibility about 8%. An app is described.

Steel Earl C. Smith (to Republic Steel Corp.). U. S. 2,025,425, Dec. 24. After making Bessemer steel contg. about 0.05% Mn, about 1.25% of Mn is added to the steel while it is being poured into a ladle, and 4-7 lbs. of siderite per ton of steel is also added during the pouring, to produce a steel which can be readily machined.

Steel Mannesmannröhren-Werke (Wilhelm Bernatzky, inventor). Ger. 621,345, Nov. 6, 1935 (Cl. 18d 2.20). For making pressure weld containers, tubes, etc., required to withstand pressure, use is made of steel prep. by a fusion process and contg. C up to about 0.2 and W 0.1-0.7%.

Normalizing steel Wm. C. Chancellor (to National Tube Co.). U. S. 2,025,010, Dec. 17. The steel is heated to a temp. slightly above the crit. temp. range, cooled to a temp. below the crit. range and, during cooling through the crit. range and while above its transformation temp., is subjected to alternate tensile and compressive stresses effecting a slight reduction in area.

Dephosphorization and desulfurization of steel. Soc. d'électrochimie, d'électrometallurgie et des aciéries électriques d'Ugine. Fr. 790,380, Sept. 2, 1935. The steel is beaten up with a very oxidizing fluid slag contg. one or more free bases in large excess, the operation being carried out so as to obtain a reciprocal penetration and so that the slag retains its strongly basic character. The fluidity is obtained by adding fluxes such as fluorspar, SiO<sub>2</sub> or TiO<sub>2</sub>. Thus a flux may be used contg. lime 45, SiO<sub>2</sub> 20, Fe oxide 19, MnO<sub>2</sub> 6 and fluorspar 10%.

Working metals such as copper on steel Frank L. Antselt (to Copperweld Steel Co.) U. S. 2,026,605, Jan. 7. For making a coherent, integral mass of metal from a series of layers such as Cu-coated steel, portions of each layer are successively compacted to refine the grain structure, leaving intermediate portions in their original coarse grained state to anchor the layers together, and the layers are annealed so that the intrusive effect of the compacted portions extends to adjacent layers, wiping out boundaries and uniting the layers. App is described.

Annealing steel Vereinigte Stahlwerke A.-G. (Franz Eisenstecken, inventor). Ger. 621,346, Nov. 5, 1935 (Cl. 18c 8.80). In steel-annealing processes in which a nonoxidizing gas, e. g., coke-oven gas, is used to prevent oxidation, the gas is first passed at 250-400° over a catalyst contg. Cu and (or) Ni, and then dried. Traces of O in the gas are thus removed.

Stainless steel Fabien C. L. Rochat and Jules L. A. Rochat. Fr. 787,234, Sept. 19, 1935. The steel contains Fe 73.15-71.13, C 0.1-0.12, Cr 16-18, Ni 9, Co 0.5, Ag 0.25, Al 0.5 and Mg 0.5%.

Weld steel, I. G. Farbennad, A.-G. (Otto Heusler, inventor). Ger. 618,956, Sept. 19, 1935 (Cl. 18c 4). Small shot or puddled Fe is packed within the interstices filled with a mixt. of metal powder and a substance giving off an indifferent or reducing gas and pressed at welding temp. In an example, the filling mixt. may be powd. Fe obtained by decompn. of Fe oxide, chloride or carbonyl and NH<sub>4</sub>Cl or colophony.

High-chrome steel Lewis B. Lindemuth (to Moa Bay Co.) U. S. 2,026,183, Dec. 31. Chrome-iron is blown in a basic converter to a point where most of the Cr is accumulated in the slag as oxide, and a charge consisting of the converted slag and a sufficient quantity of the blown metal to produce the desired product is melted and refined under nonoxidizing conditions in a basic furnace in which reducing agents such as ferro-Si or Al are added to the slag to deoxidize the Cr in it.

Hard alloys. Anton Kratky. Austrian 133,627, Nov. 25, 1935 (Cl. 40b). In the manuf. of hard alloys by sintering hard carbides and (or) nitrides with 5-20% of an auxiliary metal of lower m. p., e. g., Fe, Co, Ni or Cr, use is made of a system where thin layers of the alloys are successively prep. and superimposed. A thin layer of the powd. alloy components is first spread on a support heated to a temp. somewhat below the m. p. of the auxiliary metal, e. g., 1000-1200°, and sufficient pressure is applied, e. g., by a roll or stamp, to form a coherent layer. Another thin layer of powd. alloy components is then applied, and the process is repeated until a mass of the required thickness is obtained. The products may be homogenized by heating them for some time. The process is effected *in vacuo* or in a reducing gas. The support may be of Fe, W or C, and may or may not, as desired, be removed from the finished products. App is described in which the alloy layers are built up on the inner surface of a rotating ring.

Hard alloys Alfred Kropf. Ger. 621,191, Nov. 2, 1935 (Cl. 40b 17). Addn. to 664,202 (C. A. 27, 915) and 611,934 (C. A. 29, 58037). Suitable materials are fused together to yield alloys of the compn. W 5-78,

Gold alloys W C Heraeus, G m b H. Ger 621,152, Nov 2, 1935 (Cl 40b 4). Hard alloys capable of being cast contain Au up to 20, Cd, Mo and (or) W up to 5, and Al, Mg, Sn, Zn, Cr and (or) Mn up to 10%, the remainder being Pd and Ag, with or without up to 3% of P or Si. The hardness of the alloys may be raised by chilling them from 900° or more and then reheating to 300-700°. Sp alloys are described.

Magnesium alloys Roy E Faine (to Magnesium Development Corp.) U S 2,026,549, Jan 7. Alloys which have good resistance to corrosion contain Mg together with Pb 0.5-22 and Cd 1-10%. U S 2,026,590 relates to alloys of Mg with Pb 0.5-22 and Al 1-15%. U S 2,026,591 relates to alloys of Mg with Pb 0.5-22, Al 1-15 and Sn 1-15%. U S 2,026,592 relates to alloys of Mg with Pb 0.5-22, Al 1-15, Mn 0.1-1 and Ca 0.1-2%. All these alloys are resistant to corrosion. Cf C A 29, 3640.

Nickel-chromium-calcium alloys David P. Moore U S 2,026,243, Dec 31. Alloys which may be used for lamp filaments or in making stainless steel contain Ni 33-95, Cr 2.5-33.3 and Ca 2.5-33.3%.

Coating metal sheets with tin Wm. E Taylor (to American Can Co.). U S 2,026,598, Jan 7. For removing impurities from oil used in a tin-coating app., the oil contg impurities is transferred to a cleaning device such as a filter, and the purified oil is heated and returned to the coating app. App is described.

Coating metal sheets by use of a molten tin bath Henry W Nieman (to Bethlehem Steel Co.) U S 2,025,768, Dec 31. In the use of a molten Sn bath, molten metal is continuously removed from the bath, cleaned and returned to the bath. App is described.

Testing and arranging magnetic materials such as steel blooms Wilbur H Armco (to Superheater Co.). U S 2,024,500, Dec 17. App and various operative details are described.

Welding electrodes The British Thomson-Houston Co Ltd Brit. 434,524, Sept 3, 1935. The electrodes are provided with a flux coating comprising a backing

member impregnated with a flux contg 10-60% of ilmenite (I). A suitable flux consists of I 21, feldspar 39, liquid Na silicate 31 and a deoxidizing material, e. g., Fe-Mn, 9%.

Coated welding rod Wilber B Miller (to Orwel Acetylene Co.) U S 2,024,991, Dec. 17. A core such as one of ferrous metal is provided with a coating contg slag-forming materials 65-98, a coal such as canal coal about 2-25% and a binder such as water glass.

Welding rod suitable for forming hard facings on metal articles Wm A Wissler and Wilber B Miller (to Haynes Stellite Co.). U S 2,024,992, Dec 17. A core of ferrous metal is provided with a coating contg flux material, metallic compds., carbonaceous material and metallic alloys formed of C 3-5, Mn 10-25, Si 0.1-3.0, Cr 30-45 with the balance principally Fe.

Rod suitable for welding with an oxyacetylene flame. John J Crowe (to Air Reduction Co.). U S 2,026,418, Dec. 31. An alloy is used contg C less than 0.3, Ni 1-4, V 0.05-0.5, Mn 0.4-1.2 and Si 0.1-0.45%.

Apparatus for electric welding of materials such as springs and electrical contacts Conrad L Pfeiffer (to Western Elec Co.) U S 2,024,597, Dec 17. Structural, mech and operative details.

Welding iron and steel alloys containing manganese. John H. Hall (to Taylor-Wharton Iron & Steel Co.) U S 2,026,407, Dec 31. Iron or steel alloys contg Mn 10-15% and C 1.0-1.4% are welded by use of a welding alloy of iron or steel contg Mn 10-15, Ni 1-5% and less C than the parent stock. U S 2,026,468 relates to a weld rod, for welding austenitic Mn steel articles, consisting of austenitic Mn steel having air toughening properties and contg Si about 0.5-2.5%.

Solder Carl Wunderle (15%) to Frank A Hunter). U S 2,025,323, Dec 24. Molten lead 12 oz in a crucible is treated with 0.25 gram of  $\text{NH}_4\text{Cl}$  and stirred to sep impurities such as compds of As, Fe and Cu which collect at the surface and are removed, Sn 3.13 oz. is added and Bi 0.87 oz is subsequently added.

## 10—ORGANIC CHEMISTRY

CHAS A ROUILLER AND CLARENCE J WEST

The action of thiocyanogen on unsaturated hydrocarbons A Krassulchik. *Ann combustibles liquides* 10, 923-87 (1935). —By double decompn. between  $\text{Pb}(\text{NO}_3)_2$  and  $\text{KSCN}$ , pure  $\text{Pb}(\text{SCN})_2$  is obtained which, after drying *in vacuo* over  $\text{H}_2\text{SO}_4$ , is suspended in  $\text{AcOH}$  made  $\text{H}_2\text{O}$  free by refluxing with  $\text{Ac}_2\text{O}$ . Upon addn of Br,  $(\text{SCN})_2$  (I) is formed, and its soln is filtered off. Solns 0.1 N or weaker are stable. Weighed amts. of unsatd hydrocarbons are dissolved in the  $\text{AcOH}$  soln of I, and after reaction, the excess I is titrated with  $\text{KI}$  and  $\text{Na}_2\text{S}_2\text{O}_3$ . With 300% excess I, the reaction is bimol and is practically complete in 10-40 hrs., with absorption from 60-95% of theoretical, depending on the hydrocarbon. Some hydrocarbons, like stilbene, do not react at all. The addn of I is not as regular as that of I, and is greatly influenced by traces of Cl or Br. The variation in rate of addn of I may be used to fractionate mixts of olefins. G. Calingaert.

Atomic equilibria in the molecules of hydrocarbons of the series  $\text{CH CCH}_2\text{R}$  E F Zeberg. *J. Gen. Chem. (U. S. S. R.)* 5, 1016-19 (1935), cf. *J. Russ Phys.-Chem. Soc.* 50, 161 (1918). —Various expts on the heating of  $\text{CH}_3\text{CICH}_2\text{CICH}_2\text{Ph}$ ,  $b_m$  136-8°,  $\alpha$  242-5°, and  $\text{CH}_3\text{Br-CHICH}_2\text{CH}_2\text{Ph}$ ,  $b_m$  137-8°, with alc. and solid KOH under pressure and in the water bath under atm and neg. pressures gave only methylphenylacetylene (I),  $b$  182-3°. The results indicate the instability of the intermediate phenylallene and the instantaneous isomerization of benzylacetylene to I.

Chas Blanc. Dimerization of 1,3 butadiene S Lebedev and S Sergienko. *Compt rend acad sci U. R. S. S. S.* 3, 79-82 (1935). —Fractionation of the dimer formed during the

thermal polymerization of 1,3-butadiene yields mainly 1-ethenyl-3-cyclohexene (I),  $b_m$  129.5-30.5°. A small amt of a monocyclic hydrocarbon isomeric with I is also obtained. Bromination of I yields 2 isomeric tetra-bromides, *cis* (II),  $m$  74-5°, and *trans* (III),  $m$  101.5-2.5°. The hydrocarbons generated from II and III proved identical with the original dimeric product and with I and its isomer show no tendency to further polymerization.

W J Peterson

The synthesis of the derivatives from 2,4-dichloro-2-butenes The use of by-products from the synthesis of chloroprene A L Klebanik and K K Cheyukhalova. *Sintet Kautchuk* 1935, No 6, 16-21. —(I) 2,4-Dichloro-2-butenes (I) was treated with a 15% excess of 24-50% alc. KOH, by stirring for 4 hrs on a water bath (60-70°). The product of reaction was sep'd from salts and fractionated *in vacuo* and was collected at 62-64° at 40 mm; it had the following consts.:  $d_{20}^{20}$  0.7923,  $n_D^{20}$  1.4382, mol refraction 36.03, Cl 26.07%, C 54.04%, H 8.01%, O 11.78,  $\text{LiO}$  99.70% of theoretical value, formula  $\text{C}_4\text{H}_5\text{OCl}$  (Et ether of 2-chloro-2-buten-4-ol); it did not polymerize. (2) I was treated with 15% excess of 15-20%  $\text{Na}_2\text{CO}_3$ , by stirring for 2 hrs on a boiling water bath. The product of reaction is a chloro alc. (chlorobutenol) (II), it was dist'd at 90-110° (yield 70-80%), and  $b_m$  92°,  $d_{20}^{20}$  1.106,  $n_D^{20}$  1.4652, mol refraction 26.64, Cl 32.8%, C 45.33%, H 6.47%, O 15.40%, formula  $\text{C}_4\text{H}_5\text{OCl}$ , it did not polymerize. (3) To establish the alc. group in II a xanthate was obtained by adding the theoretical amt of KOH to the II with stirring for 30 mins and heating on a water bath. KOH was in a suspension; with the addn. of a 20% excess of  $\text{CS}_2$ , a cryst

ppt. of xanthate was formed, the latter was dried in air or at const. temp. of 30–40°. The reaction proceeded as follows:  $\text{CH}_3\text{CCl}(\text{CH}_2\text{CH}_2\text{OH}) + \text{KOH} + \text{CS}_2 \rightarrow \text{KSCCOCH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{H}_2\text{O}$ . The resulting xanthate was sol in water and alc.; it contained Cl 17.6%, S 31.20%, at 142° it changed from yellow to a darker color, it can be used for flotation (equals the K Et xanthate) (4). The water soln. of KOH (15% in excess) was treated with II, heated on a water bath, and I was gradually added with stirring. The reaction was completed in 4 hrs. The product of reaction was washed with water, dried over  $\text{CaCl}_2$  and distd. *in vacuo* (yield 80–90%). The consts. of the resulting dichlorodibutyl ether were  $b_p$  142°, Cl 36.9%, d<sub>4</sub> 1.171,  $n_D^{20}$  1.4560, mol. refraction 50.11, formula  $\text{C}_8\text{H}_{16}\text{OCl}_2$ . The product did not polymerize (5). I was passed through a china pipe over silica gel at 245–75°, over Glukhovskaya clay at 240–60° and 250–300° and over molten KOH at 285–450°. In all cases chloroprene was obtained (yield 17–24%), with the following consts.  $b_p$  26°, d<sub>4</sub> 0.9375,  $n_D^{20}$  1.4570.

A Pestoff

Synthesis of  $\alpha,\beta$ -dichlorobutadiene and its polymerization. A. I. Klebanik, A. S. Volkenshtein and A. P. Orlova. *J. Gen. Chem. (U. S. S. R.)* 5, 1255–67 (1935). Attempts to obtain  $\text{CH}_2=\text{CHCl}(\text{CHCl})_2$  (I) from  $\text{CH}_2=\text{CHCl}$  (II) directly with  $\text{SbCl}_5$  in the presence of  $\text{SbCl}_3$  (cf. *Chem. Ind.* 25, No. 1, 22 (1931)) and by treating II with a soln. of  $\text{CuCl}_2$ ,  $\text{CuCl}$  and  $\text{NH}_4\text{Cl}$  or  $\text{FeCl}_3$ ,  $\text{CuCl}$  and  $\text{NH}_4\text{Cl}$  resulted in little I and considerable tetramer and tetrachloride. The best results were obtained by converting II with  $\text{NaClO}$  to  $\text{CH}_2=\text{CHCl}(\text{CHCl})_2$  (III) and this with HCl to I under the following optimum conditions: III, b 55–7°, d<sub>4</sub> 1.021, d<sub>20</sub> 1.0034,  $n_D^{20}$  1.4669, M. R. 23.61, exaltation 0.64, was obtained in 57% yield by mechanically shaking for 16 hrs. II with 5%  $\text{NaClO}$  contg. 15%  $\text{NaOH}$ . Several months after this work was completed Jacobson and Carothers (*C. A.* 28, 95) reported that by a similar process they obtained III in 10% yield, possessing extremely explosive properties. This may be explained by contamination of their product with Cl derivs. of  $\text{C}_4\text{H}_6$ , I,  $b_p$  60–65°, d<sub>4</sub> 1.207,  $n_D^{20}$  1.5078, M. R. 30.47 (obs.), 29.472 (calc.), exaltation 0.99, was obtained in 56% yield when 179 g. HCl (d 1.19) said with HCl to 50–3% concn. was treated with 80 g. III, 23.2 g.  $\text{CuCl}$  and 4 g.  $\text{NH}_4\text{Cl}$  and then allowed to stand for 16 hrs. I is spontaneously polymerized to a rubber-like polymer which on vulcanization gives an ebonite-like substance. Twenty literature references. C. B.

Organic vanadium compounds. I. A. V. Kirsanov and T. V. Sazonova. *J. Gen. Chem. (U. S. S. R.)* 5, 956–62 (1935). (iso-AmO) $\cdot$ VO reacted with  $\text{PhMgBr}$ , giving Ph $\cdot$  and some org. V compounds which could not be isolated (cf. Vernon, *C. A.* 25, 5897). The interaction of  $\text{MeMgI}$  and (iso-AmO) $\cdot$ VO produced  $\text{C}_4\text{H}_9$  and no org. V compounds ( $\text{BrO}$ ),  $\text{VOH}$ , ( $\alpha\text{-HOC}(\text{H}_2\text{CO}_2)\text{VOH}$ , ( $\alpha\text{-H}_2\text{N}(\text{H}_2\text{CO}_2)\text{VOH}$  and ( $\text{CH}_3\text{CO}_2$ ) $\cdot$ VOH were prepd. by the interaction of a  $\text{H}_2\text{SO}_4$  soln. of V+++ with the Na salts of the acids. Chas. Blanc.

The reversible splitting of organomercuric cyanides with hydrogen chloride. Edward Carr. *Iowa State Coll. J. Sci.* 10, 61–3 (1935).—The electron-sharing ability of org. radicals (cf. *C. A.* 21, 2637) may serve as a basis for systematizing org. chemistry. This ability can be measured by ionizations or equilibria. Kharasch defines electronegativity on the basis of irreversible splitting of organo-Hg compds. (cf. *C. A.* 26, 1589). Both  $\text{R}_2\text{HgCN}$  and  $\text{RHgR}$  were split with HCl. The former, a reversible reaction, measures a different property of the RHg bond than does the latter, an irreversible reaction. Decomposition temps were found by heating to darkening. Comparison of results shows that thermal decompn. and irreversible decompn. with HCl measure the stability of the C-Hg bond. F. E. Brown.

Production of decylaldehyde. O. Osipova. *Masloboino Zhivoe Delo* 11, 378–9 (1935).—Distn. of fusel oils gives about 3% of a residue contg. 13% fatty acids with about 60% capric acid. Passing a vapor mixt. of capric and

formic acids over  $\text{MnO}$  at 330–75° resulted in 70–8% decylaldehyde. Chas. Blanc.

Production of nonylaldehyde and nonyl alcohol. R. Shagalova. *Masloboino Zhivoe Delo* 11, 452–3 (1935).—The Hesse method of production of nonylaldehyde (I) and nonyl alc. (II) by fusing undecylenic acid with KOH to give pelargonic acid (III), reducing the Et pelargonate with Na in abs. alc. to II and oxidizing the latter to I, was modified by reducing III in the presence of a  $\text{MnO}$  catalyst to I of high purity with 60–5% yield. The method of reducing I to II is being investigated. C. B.

Production of hydrocitronellal. O. Khol'mer. *Masloboino Zhivoe Delo* 11, 379–80 (1935).—The bisulfite compd. of citronellal was hydrated and then decompd. with NaOH, giving 50% of pure hydrocitronellal. Chas. Blanc.

Addition of hydrogen bromide to triple and double bonds. Undecyanoic, undecenoic and 10,11-epoxyundecenoic acids. P. L. Harris and J. C. Smith. *J. Chem. Soc.* 1935, 1572–6, cf. *C. A.* 29, 7278<sup>1</sup>.—Passing HBr-air through  $\text{CH}_3\text{C}(\text{CH}_3)_2\text{CO}_2\text{H}$  (I) in  $\text{C}_6\text{H}_6$  contg.  $\text{Br}_2/\text{H}$  for 3 hrs. at 0° gives as the main product 11-bromo-undecenoic acid (II), in the presence of  $\text{PhNH}_2$ , 33, 27 and 18% of I remained after 3, 5.5 and 8 hrs., resp., the main fraction by, 151–7° and m. 12.1°. Identification of the products of the reactions depends on the constitution of the Br acid, m. 41.5°, assumed by Kraft and Seldis (*Ber.* 29, 2232 (1896)) to be *trans*-II. The following considerations give a clear indication of the constitution. The product of incomplete catalytic reduction does not contain 10-bromoundecenoic acid (III) and although the 11-isomer was not isolated, its presence was indicated by the cryoscopic behavior of the mixt., complete reduction gives undecenoic acid, the Br atom in III is very difficult to reduce. The formation of solid solns. with the 11-Br acid and of a eutectic system with III indicates great similarity of structure with II. Addn. of HBr to undecenoic acid (IV) in  $\text{CS}_2$ ,  $\text{CHCl}_3$  and  $\text{CCl}_4$  is sensitive to "oxidants", unless traces of  $\text{EtOH}$  are removed from  $\text{CHCl}_3$ , the solvent acts as an antioxidant and gives mainly III. In the absence of  $\text{EtOH}$ ,  $\text{Br}_2/\text{H}$  has an immediate catalytic effect on addn. to IV. 10,11-Epoxyundecenoic acid (V), m. 45.5°, results in 25–50% of  $\text{Br}_2/\text{H}$  from IV and  $\text{Br}_2/\text{H}$  in ligroin at 30°; HBr gives a mixt. of bromohydrins. From a mixt. of IV and V in ligroin, HBr at 0° gives the 11-Br deriv. of IV. In these reactions none of the catalysts is effective in the absence of mol.  $\text{O}_2$ . V is not an intermediate in the formation of the 11-Br deriv. of IV. C. J. West.

Preparation in a high state of purity of long-chain compounds, in view of the determination of their energy contents. I. Oleic acid. L. Keffler and J. H. McLean. *J. Soc. Chem. Ind.* 54, 362–77 (1935); cf. *C. A.* 29, 6209<sup>1</sup>.—The thorough purification of oleic acid is described. The Pb salt method fails to remove the final 3–4% of satd. impurities. The use of  $\text{Pb}(\text{OAc})_2$  in large excess of the quantity required for the pptn. of the satd. acids does not render the sepn. more effective. The recrystn. of the Li salts from 5 vols. of 80% alc. can remove completely the higher unsatd. impurities, but 10–12 recrystns. are needed to secure that result. The low-boiling as well as the high-boiling satd. esters which contaminate the esters of oleic acid can be removed to within 0.2–0.3% by repeated fractional distn. under very much reduced pressure. The higher unsatd. esters cannot be removed thus. In the case of esters of oleic acid which have "aged" by keeping for long periods, complete (although possibly very wasteful) recovery may be effected by fractional distn., especially when combined with a preheating treatment at a temp. just below the b. p. of the ester. Oleic acid free from linoleic acid, and the esters prepd. from it, are much less susceptible to rancidity changes than samples which contain impurities of a highly satd. nature. Improvements in the app. required for the most effective fractional distn. of fatty esters are described. W. J. Peterson.

Oleic acid. T. P. Hilditch. *Chemistry & Industry* 1935, 1009.—Keffler and McLean (preceding abstr.) are

criticized for their failure to acknowledge the work of others in the prepn of pure oleic acid. Brocklesby and Denstedt (C. A. 25, 5582) have described and illustrated a form of Bahl receiver which is the same in principle as that presented by K. and M. W. J. Peterson.

**Relation between acidity and tautomerism IV.** The role of the cyano group. F. Arndt, H. Scholz and Frobel. *Ann.* 521, 95 (1935), cf. C. A. 29, 1816<sup>1</sup>—NCCCH<sub>2</sub>CO<sub>2</sub>Me (I) contains 1–32% of enol (Br titration), depending upon the temp (–12° to 20°), with CH<sub>3</sub>N<sub>3</sub> it yields 2 Me derivs, m 76° (II) and 97–8° (III), the latter being NCC(CMeOMe)CO<sub>2</sub>Me (Schmitt, *Bull. soc. chim.* 31, 341 (1904)), both give NCC(CMeOMe)CO<sub>2</sub>Me with 2 N NH<sub>4</sub>OH, heating II at 130–5° for 3.5 hrs gives III, indicating cis-trans isomerism. p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>CH<sub>2</sub>CN (IV) gives no color with FeCl<sub>3</sub>, does not add Br and is sol in 2 N Na<sub>2</sub>CO<sub>3</sub> only on heating, it does not react with CH<sub>3</sub>N<sub>3</sub> on heating for 14 days, but in MeOH it reacts slowly (12 days), giving C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>CH<sub>2</sub>CN, b<sub>p</sub> 200–5°, m 60°, and C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>CH<sub>2</sub>CN CH N NMe,

m 162°. The Na deriv of IV with ClCO<sub>2</sub>Me gives *Me tosylcyanoacetate* (V), m 70–80°, which is strongly acidic, gives a pos FeCl<sub>3</sub> reaction and adds Br, the enol content varies from 48 to 91%, with CH<sub>3</sub>N<sub>3</sub> there results from 3 g. V at –50° 0.1 g. C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>C(CO<sub>2</sub>Me)C NMe, m 125–7°, and 0.9 g. C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>CMe(CN)CO<sub>2</sub>Me, m 101°; the 1st but not the 2nd decolorizes Br-MeOH soln. The Na salt from 19.5 g IV and 7.9 g AcCl in Et<sub>2</sub>O give 7.9 g of 1-tosyl-1-cyanoacetone (VI), m 122–3°, with 30–65% enol form, with CH<sub>3</sub>N<sub>3</sub> at –10° there result C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>C(CN)CMeOMe, m 124°, and C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>CMe(CN)Ac, m 150–1°; both are insol in cold alkali; the former is sol. in hot 2 N KOH, giving VI. H<sub>2</sub>C(CN)<sub>2</sub> does not react with FeCl<sub>3</sub>, Br or CH<sub>3</sub>N<sub>3</sub> in MeOH. The Na deriv. from 19.5 g IV and 10.6 g BrCN give 8.1 g *tosylal nonstole*, m 101–2°, with 45–88% enol form, at –50° CH<sub>3</sub>N<sub>3</sub> yields a small quantity of C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>CMe(CN)<sub>2</sub>, m 103°, at –10° C<sub>7</sub>H<sub>5</sub>SO<sub>2</sub>C(CN)C NMe is probably formed but was not isolated. The Na compd of CH<sub>2</sub>(CN)<sub>2</sub> and ClCO<sub>2</sub>Me give *Me dicyanoacetate*, m 65°, with 26–60% enol form, CH<sub>2</sub>N<sub>3</sub> at –50° does not give a cryst. compd; the liquid is a mixt of O- and N-Me derivs. 1,1-Dicyanoacetone, m 141°, has 1.4 to 3.3% enol form, p-nitrophenylhydrazine, reddish brown, m 144° (decompn), CH<sub>2</sub>N<sub>3</sub> gives a brown-yellow oil, a mixt of O- and N-Me derivs. The acidifying effect of the CN group is the strongest of all the C-contg simple substituents and follows the SO<sub>2</sub>R group. It also shows electromeric action, as well as the form of the "enotrophic effect" in a "conjugation partner." In both respects it is less effective than the CO group but more so than the CO<sub>2</sub>R group. The group CHCN can be transformed into the enamide group (C C NH) but a conjugation-partner is necessary, this may be a 2nd CN group, an ester CO group or a NO<sub>2</sub> group. The C C NH group behaves toward FeCl<sub>3</sub>, Br and CH<sub>3</sub>N<sub>3</sub> like the enol group. C. J. West.

**The catalytic dehydrogenation and dehydrogenation of alcohols.** N. N. Motovilova. *J. Chem. Ind. (Moscow)* 12, 1184-8 (1935).—A review. H. M. Leicester.

**The synthesis of esters by the dehydrogenation of alcohols at ordinary pressure.** B. N. Dolgov, M. M. Koton and S. L. Lei chuk. *J. Chem. Ind. (Moscow)* 12, 1060–73 (1935).—When EtOH is passed at 275° and a rate of 15 cc. per hr over a Cu catalyst prepd from Cu(OAc)<sub>2</sub> and contg 0.2% Ce, it gives 33% of EtOAc. If the gas mixt. is passed 4 times over the catalyst at 250°, the products are 49% EtOAc, 20% AcOH and 15.5% AcH. Long columns of catalyst give better yields of ester. The catalyst is very stable and can be regenerated easily by passing air and then H<sub>2</sub> over it. Poor results are obtained if the catalyst is powd. or is deposited on asbestos, but if it is deposited on granular C, it works well. The addn of H<sub>2</sub>O to the EtOH used lowers the yield of ester and raises that of the aldehyde. AcOH and AcH deactivate the catalyst, while EtOH, EtOAc and MeCH(OEt), and BuOAc (by-products of the reaction) have no effect on it. It is therefore best to keep a slight excess of EtOH

always present in the reaction chamber. BuOH and iso-BuOH undergo an analogous reaction to yield about 19% of the corresponding esters. Mixts of alcs do not give good yields of esters. The mechanism and possible uses of these reactions are discussed. I. M. Leicester.

**The synthesis of ethylene glycol.** N. Wada and M. Sato. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 497–500 (1935), cf. C. A. 29, 7913<sup>1</sup>—The prepn of glycol by the chlorohydrin process gives better results than by the chloride process, the former giving a yield of 71% and the latter 43%, based upon the CaH<sub>2</sub> 1 xptl results are presented. Karl Kammermeyer.

**Trichloro hydroxy aliphatic amines.** F. D. Chattaway and P. Witherington. *J. Chem. Soc.* 1935, 1623–4—Reduction of Cl<sub>3</sub>CCH(OH)CH<sub>2</sub>NO<sub>2</sub> (C. A. 29, 7277<sup>1</sup>) with Sn and HCl gives γ,γ,γ-trichloro-α-amino-β hydroxy propane, m 123°; HCl salt, m 235° (decompn), oxalate, m 220° (decompn); sulfate, m 255° (decompn); N-Ac deriv, m 154°, O,N-di-Ac deriv, m 99°. KCN gives trichlorohydroxypropylurea, m 175°, γ,γ,γ-Trichloro-α-amino-β hydroxypentane, m 141°, HCl salt, m 231° (decompn); oxalate, m 232° (decompn); sulfate, m 235–10° (decompn), N-Ac deriv, m 109°; O,N-di-Ac deriv, m 136°; the urea m. 205° (di-Ac deriv, m. 175°). C. J. West.

**Derivatives of carbamylcholine chloride.** Alberto Ercole. *Ann. chim. applicata* 25, 263–73 (1935)—Carbamylcholine chloride is a somewhat more powerful blood-pressure-reducing drug than acetylcholine, but is more toxic. Therefore, several derivs have been prepd in an attempt to retain the advantages of the former, while reducing its toxicity. The following representative derivs were prepd: MeCOHNCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>Cl (I) is prepd by adding CH<sub>3</sub>(OH)CH<sub>2</sub>Cl to MeCOCN with cooling. It crystallizes as white needles m 73–4°. By heating I for 30 hrs at 100° with 18% Me<sub>2</sub>N in C<sub>2</sub>H<sub>5</sub>MeCOHNCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>NMe<sub>2</sub>Cl (II) is formed as fine hygroscopic needles. When H<sub>2</sub>NCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>Cl (III) is suspended in water, acidified slightly, and then CH<sub>3</sub>O added, the compd. CH<sub>3</sub>H<sub>2</sub>NCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>Cl<sub>2</sub> (IV) is formed as white plates, m 148°. CH<sub>3</sub>H<sub>2</sub>NCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>NMe<sub>2</sub>Cl<sub>2</sub> (V) is prepd from IV by treating with Me<sub>2</sub>N just as II, above, is prepd from I. The chloroplatinate of V forms yellow-orange crystals with 2 H<sub>2</sub>O, m 230°. When III is treated directly with CH<sub>3</sub>O and concd HCl, an oil [CH<sub>3</sub>NCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>Cl<sub>2</sub>] (VI) forms, which cannot be crystd. Treated with Me<sub>2</sub>N VI also forms a choline deriv [CH<sub>3</sub>NCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>NMe<sub>2</sub>Cl<sub>2</sub>] (VII) which is amorphous. Its chloraurate and chloroplatinate are also amorphous. When III is added to MeNHPh a colorless oil, MePhNCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>Cl (VIII) is formed, b<sub>p</sub> 165°, 290–300° (decompn), d<sub>4</sub> 1.196. With Me<sub>2</sub>N VIII gives a choline MePhNCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>NMe<sub>2</sub>Cl (IX). It is an oil whose chloroplatinate m 222°, and chloraurate m 190°, both are readily crystd. When III is heated with KCN, and then CH<sub>3</sub>(OH)CH<sub>2</sub>Cl added, and the heating continued 10 hrs longer on an oil bath at 130°, white crystals of H<sub>2</sub>NCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>Cl<sub>2</sub> (X) are formed, m 202°. With Me<sub>2</sub>N, X gives the choline deriv H<sub>2</sub>NCO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>NMe<sub>2</sub>Cl<sub>2</sub> (XI), as white, very hygroscopic crystals. It is very stable even in boiling water. The chloraurate m 240° (decompn), the chloroplatinate decomposes at 248°. V is about 0.5 as effective as acetylcholine as a blood pressure-reducing agent, but is only 1/3 as toxic and is the most promising of these derivs. The cholines show decreasing activity in comparison with carbamylcholine, thus, XI is only 1/250th as active. A. W. Conter.

**Phenomena of migration in the course of the hydrolysis of the 3 mixed diesters of orthophosphoric acid.** Passage of α- to β glycerophosphates and vice versa. O. Bailey and Jacques Gaumé. *J. pharm. chim.* 22, 32–32 (1935); cf. C. A. 28, 5498<sup>1</sup>, 6105<sup>1</sup>, 29, 1388<sup>1</sup>, 3991<sup>1</sup>, Levene and Raymond, C. A. 29, 1067<sup>1</sup>.—A summary and discussion. S. Waldbott.

**Esterification of hydroxy acids and polyalcohols.** R. Ciocca and A. Sempron. *Ann. chim. applicata* 25, 319–23 (1935)—The principle of Thielpappe (cf. C. A. 28, 108<sup>1</sup>) has been extended to the esterification of HIO acids

and polyols. Instead of  $\text{H}_2\text{SO}_4$ , a sulfonic acid such as  $\text{PhSO}_3\text{H}$ ,  $\text{MeC}_6\text{H}_4\text{SO}_3\text{H}$ ,  $\text{C}_6\text{H}_5\text{SO}_3\text{H}$  and camphorsulfonic acid, was used. The Et esters of lactic, tartaric, citric and nicotinic acid, and glycol and glycerol stearates were prepd. with yields up to 95%. The formates cannot be prepd. by this method as  $\text{HCO}_2\text{H}$  is decomposed by  $\text{CaC}_2$ .

A. W. Conner

**Action of normal solutions of sulfuric acid and sodium hydroxide on glycoyl I** S. Yauchnikov, *J. Gen. Chem. (U. S. S. R.)* 5, 967-9 (1933), cf. *J. Russ. Phys.-Chem. Soc.* 52, 147 (1920).—Further tests disclosed that the action of  $\text{N H}_2\text{SO}_4$  and  $\text{N NaOH}$  decreases the formol-titrated  $\text{N}$  not only in a dipeptide (glycylglycine) but also in its component amino acid (glycoyl). This may be explained by the formation of an anhydride or a polymer, and in case of an amino acid by the formation of a dipeptide.

Chas. Blanc

**Interaction of glycoyl and glycylglycine with nicotine** I. S. Yauchnikov, *J. Gen. Chem. (U. S. S. R.)* 5, 970-1 (1933), cf. *C. A.* 28, 790<sup>1</sup>.—The results of Sørensen titration showed that glycoyl and glycylglycine in the presence of  $\text{CH}_2\text{O}$  are capable of combining up to 70% of nicotine.

Chas. Blanc

**Optical rotatory dispersion in the carbohydrate group VI** The amide rotation rule I. L. Harris, I. L. Hirst and C. L. Wood, *J. Chem. Soc.* 1935, 1658-62, cf. *C. A.* 29, 3933<sup>1</sup>.—This study was made to det. whether the amide rotation rule depends upon control of the sign of the induced activity by the configuration of the 2nd C atom. Data are given for Me *d*-dimethoxysuccinate (I), *d*-dimethoxysuccinamide (II) and its Me ester (III), 1,2,3,5-trimethylarabonamide (IV) and *d*-1,6-dimethylmannonamide (V). In II, which follows the amide rule, the rotation is definitely controlled by the induced dissymmetry and can be represented by a 1-term equation of the Drude-Natanon type. The absorption band giving rise to the rotation is situated at  $\lambda$  2300. Exactly similar considerations apply to III and IV. V does not obey the amide rule in  $\text{H}_2\text{O}$  but follows it in EtOH, in  $\text{H}_2\text{O}$ , the observed rotation is governed by the induced dissymmetry, the sign of the induced term is pos. and the rotation therefore fails to obey the amide rule at any wave length. In this amide the normal action of the  $\alpha$ -HO group has been altered by the influence of the neighboring groups to such a degree that the total effect produced by coupling with the CO group gives a pos. partial rotation in place of the expected neg. term. The study of I shows that between  $\lambda$  6709-2613 the rotation of the ester in  $\text{H}_2\text{O}$  is given by the equation  $\alpha = 4.5283/(\lambda^2 - 0.07) - 2.0129/(\lambda^2 - 0.05)$ , in which the 2 frequency const. are identical with those used by Bruhat for tartaric acid. In I the sign of the induced term is neg. and is opposite to that of the corresponding term in II, hence the rotation of the amide has the same sign as that of the ester in the visual region. The paper contains a discussion of the interpretation of dispersion equations. Within the limits of exp't error, the equation for sucrose,  $\alpha = 7.4645/(\lambda^2 - 0.0213)$ , can be replaced by  $\alpha = 7.4645/[\lambda^2 - 0.0212 + 0.0001 \lambda^2/(\lambda^2 - 0.0212)]$ , the half width of the band ( $\lambda_1$ ) having the reasonable value of 100 Å.

C. J. West

**Structure of hexamethylenetetramine** Mieczyslaw Dominikiewicz, *Arch. Chem. Farm.* 2, 78-129 (1935) (in German) (1935).—Of all proposed formulas for hexamethylenetetramine (I) only those of Duden and Scharf (*Ann.* 288, 247 (1895)) and Delépine (*C. A.* 6, 622) are discussible, although none explains satisfactorily the reactions and paramount properties of I. The results of Duden and Scharf do not justify by any means the assumption of a trimethylenetetramine ring as a fundamental structural unit in I. D's results disprove also the existence of such derivs.: Addn of I (14 g.) to  $\text{Ac}_2\text{O}$  (10.5 g.) in EtO (150 cc.) gives in the cold pentamethylenediacetyl-

1 *di*-Bz deriv. could not be obtained, since it is benzoylated immediately farther to trimethylenetetrabenzoyltramine (II) of the formula  $\text{Bz}_2\text{NCH}_2\text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{NBz}_2$ , m. 218-19°,

colorless, insol. in  $\text{H}_2\text{O}$ , increasingly sol. in  $\text{C}_6\text{H}_6$ , EtO, EtOH, CHCl<sub>3</sub>,  $\text{CHBr}_3$ , AcOH. Another product of benzoylation is dimethylene-N,N'-bis(methylenedibenzoyl)-

2 *aminediamine*,  $\text{Bz}_2\text{NCH}_2\text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{NCH}_2\text{NBz}_2$ , colorless, m. 267.8°, insol. in  $\text{H}_2\text{O}$ , soly. increasing in  $\text{C}_6\text{H}_6$ , EtO, cold AcOH, CHCl<sub>3</sub>,  $\text{CHBr}_3$  and hot AcOH. A 3rd addn product of the benzoylation is the hexamethylenetetraminebenzoyldibenzoyl, of the formula  $\text{Bz}_2\text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{NBz}_2$ , m. 218-19°, easily sol in

3 EtOH, CHCl<sub>3</sub> and  $\text{CHBr}_3$ , less sol. in EtO and AcOEt. On boiling with an acid it degrades to HCHO, BzOH and an NH<sub>4</sub> salt. Benzoylation of I in CHCl<sub>3</sub> in the presence of pyridine gives chlorobenzoylhexamethylenetetramine,  $\text{C}_6\text{H}_5\text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{NBz}$ , m. 183-9°, and an addn product, dichloropyridinedibenzoylhexamethylenetetramine (III),

4  $\text{Bz}_2\text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N}(\text{NCICl}_2\text{H}_5) \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{NBz} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N}(\text{NCICl}_2\text{H}_5)$ . Thus it is evident that also dichlorobenzoylhexamethylenetetramine,  $(\text{CH}_2)_6\text{N}_4\text{C}_2\text{H}_4$ , is formed during the reaction. III is decomposed by water to HCl salt of I, m. 185°, in addn during this decomn. another product of benzoylation is formed, viz. dimethylene-N-methylenedibenzoylamine - N' - methylenebenzoylaminediamine,  $\text{Bz}_2\text{NCH}_2\text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{NCH}_2\text{NBz}$ , m. 235-6°. Benzoylation of I in

5 presence of  $\text{Na}_2\text{CO}_3$  gives II, in a pure state, m. 218-19° (Duden and Scharf's compd. is impure, m. 220-1°). On the basis of the above expts. the formula:

6  $\text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N} \begin{array}{c} \text{CH}_2 \\ \text{CH}_2 \end{array} \text{N}$  is attributed to I. J. Wiertelak

**Trimethylenetriallyltramine and its mercury derivatives** M. Dominikiewicz, *Arch. Chem. Farm.* 2, 160-4 (1935) (German summary).—Allyltramine in EtO treated with an  $\text{CH}_2\text{O}$  condenses to trimethylenetriallyltramine (I),  $(\text{CH}_2)_3\text{N}(\text{CH}_2\text{CH}_2\text{NCH}_2)_3$ , b. 138-41°, colorless liquid, with a spermiac odor, sol. in all proportions in  $\text{H}_2\text{O}$  and most org. solvents. Picrate, yellow, m. 139°, sol. in hot  $\text{H}_2\text{O}$ , does not decompose on alkalization. Mercuration of I with  $\text{Hg}(\text{OAc})_2$  in MeOH gives the *Hg* deriv.,  $(\text{CH}_2)_3\text{NCH}_2\text{CH}(\text{OMe})_2\text{CH}_2\text{Hg}(\text{OAc})_2$ . This compd. is sol. in NaOH, and on satg. the soln with  $\text{CO}_2$  trimethylene-glycidylmercury-β-methoxy-α-propyltramine,  $\text{C}_6\text{H}_5\text{O}_2\text{N}(\text{CH}_2)_3\text{Hg}$ , is pptd. as a yellow powder. Its alk. solns. are quite stable. J. Wiertelak

**Condensation of α-formyl 1,2-dicarboxylic esters with esters of α-halo-substituted acids** M. N. Shchukina and N. A. Preobrazhenskii, *Ber.* 68B, 1991-8 (1935).—As already stated (*C. A.* 28, 4039<sup>1</sup>), this condensation was studied for the purpose of prepg. homoparaconic acids.  $\text{RO}_2\text{CCH}_2\text{CH}(\text{CO}_2\text{R})\text{CHO} + \text{R}'\text{CH}(\text{CO}_2\text{R}) + \text{NaOEt} \rightarrow \text{RO}_2\text{CCH}_2\text{CH}(\text{CH}(\text{CO}_2\text{R})\text{CHO}) + \text{H}_2 \rightarrow \text{O CO CHR}'\text{CH}(\text{CH}_2\text{CO}_2\text{R})\text{CH}_2$ . As a matter of

fact, EtO<sub>2</sub>CCH<sub>2</sub>CH(CO<sub>2</sub>Et)CHO (I) with  $\text{CH}_2\text{ClCO}_2\text{Et}$  gives NaCl and the ester EtO<sub>2</sub>CCH<sub>2</sub>CH(CO<sub>2</sub>Et)CHOC<sub>2</sub>H<sub>5</sub>CO<sub>2</sub>Et (II), and EtO<sub>2</sub>CCH<sub>2</sub>CH(CO<sub>2</sub>Et)CHO (III) with EtCHBrCO<sub>2</sub>Et yields an isomeric ester EtO<sub>2</sub>CCH<sub>2</sub>CH(CO<sub>2</sub>Et)CHOC<sub>2</sub>H<sub>5</sub>CO<sub>2</sub>Et (IV). III with  $\text{ClCH}_2\text{CO}_2\text{Et}$  similarly gives the ester EtO<sub>2</sub>CCH<sub>2</sub>CH(CO<sub>2</sub>Et)CHOC<sub>2</sub>H<sub>5</sub>.

186-7° (yield 30%), colorless prisms, sol. in  $\text{H}_2\text{O}$ , EtOH and CHCl<sub>3</sub>, less sol. in EtO, AcOEt and  $\text{C}_6\text{H}_6$ . An analogous

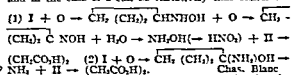
CO-Et (V). II and IV are entirely distinct from each other, hence the condensation does not follow the course of the Frankland and Duppa synthesis, which would have given the same ester,  $\text{ROCCHEIC}(\text{CO}_2\text{R})(\text{CH}_2\text{CO}_2\text{R})\text{CHO}$ , in both cases; the acids (VI and VII) obtained by hydrolysis and decarboxylation of II and IV are likewise different. II, IV and V do not show free aldehyde properties, they do not color fuchsin- $\text{SO}_2$  and form no semicarbazone even on long standing. On boiling with  $\text{NH}_3\text{-AgNO}_3$ , however, they form an intense Ag mirror in 5-10 min. and they immediately decolorize Br water. The free acid of II shows the same qual reactions. It is therefore assumed that O-alkylation of the hydroxymethylene form of the formylsuccinic ester occurs in the condensation. As there was a possibility of an Erlenmeyer condensation of the aldehyde group with the halo-substituted ester to form a glycidic acid ester, 2 esters of the latter type were selected for comparison. *di-trans*-Ethylene oxide-dicarboxylic ester forms no Ag mirror with  $\text{NH}_3\text{-AgNO}_3$ , does not decolorize Br water, and does not color fuchsin- $\text{SO}_2$ , and pilopylethylene oxide-dicarboxylic ester likewise does not color fuchsin- $\text{SO}_2$  or decolorize Br water, although it does form a Ag mirror when boiled with  $\text{NH}_3\text{-AgNO}_3$ . In the sapon of glycidic acid esters,  $\text{CO}_2$  is split off and the oxide group rearranges to carbonyl, where VI and VII contain no C O groups and are unsat. Knunyantz and Gershuk (C. A. 29, 6882) assign the oxide ester structure to IV and the 4-ketohexane-1,2-dicarboxylic acid structure to VII because VII gives succinic acid on oxidation. But succinic acid is an unavoidable oxidation product of O-alkylated formylsuccinic acids and S. and P. have obtained it in the oxidation of VII with all.  $\text{Ag}_2\text{O}$ , the ease with which the oxidation occurs is in itself evidence against the keto structure for VII. In boiling acid aq. soln. the ether union in these unsat. esters is quite stable, many hrs. boiling with 10% (CO-H), gives chiefly VI and VII. Boiling 10% HCl, however, ruptures the ether union, giving  $\text{HOCH}_2\text{CO}_2\text{H}$  (or  $\text{EtCH}(\text{OH})\text{CO}_2\text{H}$ ) and  $\text{HOCH}_2\text{CH}_2\text{CHO}$  (or the Et homologue), isolated as the semicarbazone. A characteristic property of II, IV and V is their intense light blue fluorescence. II (yield, 54%),  $b_p$  270°,  $n_D$  1.495°. VI,  $b_p$  202-4°,  $n_D$  1.02-3°. Et succinic acid hemiacetaldehyde semicarbazone, m. 161-2° (decomp.). IV,  $b_p$  187°,  $n_D$  211.5°,  $d_4^{20}$  1.0900,  $n_D^{20}$  1.4600. VII,  $b_p$  182-4°,  $n_D$  211-12°,  $d_4^{20}$  1.1386,  $n_D^{20}$  1.4646. C. A. R.

Tautomerism of the products of condensation of aromatic aldehydes with barbituric acid. Mięczyński Krakowski Arch. Chem. Farm. 2, 164-70 (1935) (German summary).—It is attempted to ascertain whether condensation products of methyl- and ethyl-vanillin are suitable for the distinction of the 2 kinds of vanillin. To this purpose vanillin (7.6 g. in 30 cc. EtOH) is refluxed for 1 hr. with barbituric acid (16.4 g. in 60 cc. EtOH), giving a theoretical yield of *C-methylbarbituric acid*,  $\text{C}_8\text{H}_{10}\text{O}_4\text{N}_2$  (I), orange, difficultly sol. in H<sub>2</sub>O with yellow color, m. 263°. Bourbonal (ethylvanillin) gives under identical conditions *C-bourbonalbarbituric acid* (II),  $\text{C}_{10}\text{H}_{12}\text{O}_4\text{N}_2$ , similar properties. Piperonal gives *C-piperonalbarbituric acid*,  $\text{C}_{11}\text{H}_{12}\text{O}_4\text{N}_2$ , yellow, insol. in H<sub>2</sub>O and Et-O, decompd. at 200°. Veratraldehyde, treated similarly with barbituric acid, gives *C-veratralbarbituric acid*,  $\text{C}_{12}\text{H}_{14}\text{O}_4\text{N}_2$ , orange-yellow, insol. in H<sub>2</sub>O and Et-O, difficultly sol. in EtOH, decompd. at 270°. I and II in alkalis turn orange because of formation of a quinoid grouping in the ring of the aldehyde residue. In the 2 other compds. such grouping is not possible; hence their solns. do not change color. J. Wiertelak

Hydrolysis of cellulose and its oligosaccharides. Karl Freudenberg and Gunnar Blomqvist. Ber. 68B, 2070-82 (1935).—The structures and configurations of the oligosaccharides of the cellulose series (cellobiose, -triose, -tetrose) have now been fully cleared up; they all belong to the  $\beta$ -series. If cellulose, as is now generally assumed, is the end product of the same series, its properties should agree with those called by extrapolation from the properties of the oligosaccharides. Expt. has shown that the yield of biose from cellulose is in harmony with the as-

sumption that all the glucose units in cellulose are combined in the same way as in cellobiose, and that the kinetics of the degradation of cellulose can be explained on the same assumption. There is a simple relationship between the optical rotations of cellulose and its oligosaccharides, the mol. rotation of cellobiose differing from those of the triose and tetrose by units corresponding to the mol. rotation of 1 and 2 glucose units, resp., of the polysaccharide. In addition to the calcd. and found values for triose and tetrose derivs. in 50%  $\text{H}_2\text{SO}_4$ , water and  $\text{CHCl}_3$  already published (F. Tamm, Cellulose, Lignin, Berlin, 1933), the following new values are given (calcd. values in parentheses): cellobiose in 51%  $\text{H}_2\text{SO}_4$ , for  $m$  578,  $64^\circ$  (50°);  $\alpha$ -acetylcellobiose,  $\text{CHCl}_3$ ,  $m$  589,  $71^\circ$  (73°);  $\beta$ -acetylcellobiose,  $\text{CHCl}_3$ ,  $m$  589,  $-57.5^\circ$  (-56°); cellobiotetrose, 51%  $\text{H}_2\text{SO}_4$ ,  $m$  578,  $42^\circ$  (38.5°). The close agreement between the calcd. and observed values is perhaps the best proof thus far obtained that the assumption of  $\beta$ -unions exclusively in cellulose is correct. The study of the kinetics of the degradation of cellulose made when only cellobiose was known has now been amplified by a study of the triose and tetrose. As expected, the velocity of the cleavage increases with time; the initial velocities are intermediate between those for cellobiose and cellulose, the values for  $k \times 10^4$  in 51%  $\text{H}_2\text{SO}_4$  being as follows at 18° and 30°, resp.: Cellulose 0.505, 2.34; tetrose 0.506-0.520, 3.65-3.80; triose 0.630-0.640, 4.50-4.60, biose 1.07, 6.94. The values here given for cellulose and the biose were detd. with greater accuracy than those previously reported. The activation heats ( $U$ ) and steric factors ( $A \times 10^{-10}$ ) calcd. for the 4 ones from the above values are: biose 27,300, 3.4; triose 28,600, 18; tetrose 28,900, 24; cellulose 29,800, 67. Full details of the measurements are given. The velocity of cleavage is affected greatly by small changes in the acid concn.,  $k \times 10^4$  in 65%  $\text{H}_2\text{SO}_4$  at 18° being 2.5 for cellulose and 5.50 for the biose. C. A. R.

Oxidation of cyclobutylamine. N. Ya. Dem'yanov and Z. I. Shuškina. J. Gen. Chem. (U. S. S. R.) 5, 1213-25 (1935).—Study of the behavior of simple aliphatic amines to various oxidizing agents in connection with the tendency of the simplest cycles to polymerization was begun with the oxidation of cyclobutylamine (I) to cyclobutanone (II). For the prepn. of I, b. 80-4°,  $\text{Br}(\text{CH}_2)_4\text{Cl}$  was condensed with Na methylmalonate giving 40% di-*Me* tetramethylene-1,1-dicarboxylate, b. 220-6° (Kuzhner, J. Russ. Phys.-Chem. Soc. 37, 507). The free acid on distn. gave 71% tetramethylenedicarboxylic acid, b. 180-91°. This with  $\text{PCl}_5$  gave 84.4% of the acid chloride, b. 137-9°, which in abs. Et-O with dry  $\text{NH}_3$  gave 82.9% of the amide. This heated for 5 min. with 10% excess of NaClO, obtained by the method of Graebe (Ber. 35, 43, 2753 (1902)), gave 78.2% I. The oxidations of I with  $\text{CrO}_3$ ,  $\text{CrO}_4$  +  $\text{H}_2\text{SO}_4$ ,  $\text{KMnO}_4$  and ozonized air produced poor yields of II (semicarbazone, m. 200-1°). Catalytic oxidation of I by the method of Taube and Schönwald (Ber. 39, 178 (1906)) gave with Cu 18.6% II and with Os-O<sub>4</sub> about 25% II. As by-products were formed cyclobutyl ketoxime, m. 54-6°, and succinic acid. The results confirm the complex mechanism of the oxidation of amines observed by other investigators, and in the case of I can be tentatively thus conceived



(2)  $\text{I} + \text{O} \rightarrow \text{CH}_2(\text{CH}_2)_2\text{C}(\text{NH}_2)\text{OH} \rightarrow \text{NH}_2 + \text{II} \rightarrow (\text{CH}_2)_4\text{CNOH}$ . Chas. Blane. Action of trimethylene bromide on acetmedicarbonylic ester. A new and more convenient method of synthesis of ethylcyclohexanone-2,6-dicarboxylate. P. C. Guha and N. K. Seshadriengar. Current Sci. 4, 155 (1935); cf. C. A. 28, 7252.  $\text{CH}_3(\text{CH}_2)_3\text{Br}$  and the Na deriv. of  $\text{CO}(\text{CH}_2\text{CO}_2\text{Et})_2$  (II), suspended in dry  $\text{CH}_2\text{Cl}_2$ , were heated for nearly 100 hrs. at 140-50°. The reaction mixt. was sepd. into gasoline-sol. (II) and gasoline-insol. (III)



aminocrotonate (I), this also results by adding the amine, heated to about 200°, to 4 mols. boiling  $\text{AcCH}_3\text{CO}_2\text{Et}$  (reactions A and B), to obtain a lepidine deriv., it is necessary to use a sealed tube.  $\text{m H}_2\text{NC}_6\text{H}_4\text{NHAc}$  gives Et  $\beta$ -3-acetamidophenylaminocrotonate (II), m 92°,  $\text{p-C}_6\text{H}_4(\text{NH}_2)$ , by reaction A gives the  $\beta$ -isomer of I, m 135°, in a quant. yield,  $\text{EtOH-FeCl}_3$  gives a dark red color, Knorr's compd., m 176°, could not be obtained (Ber 17, 545 (1884), 19, 3303 (1886)).  $\text{EtOH-HCl}$  gives  $\beta$ -C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)HCl, the same compd. resulted in reaction B.  $\beta$ -H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHAc gives the 4-isomer of II, m 185°, reaction B gives the same compd. but heating in a sealed tube at 140° for 5 hrs gives  $\text{p-C}_6\text{H}_4(\text{NHAc})$ ; Diamidine gives Et 3,3'-dimethoxybiphenylene-4,4'-bis- $\beta$ -aminocrotonate, cream, m 132-140°, toluene gives the 3,3'-di-Me analog, m 129-30°. Benzidine gives Et biphenylene-4,4'-bis  $\beta$ -aminocrotonate, m 99-6-100°, but the main product was the compd.  $\text{C}_6\text{H}_5\text{O}_2\text{N}_2$ , m 134°, which appears to be a double compd. of 2 mols of 4-H<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>-NH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et with 1 mol of ( $\text{p-C}_6\text{H}_4$ -NHCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et), with  $\text{Ac}_2\text{O}$  it yields Et 4-acetamidobiphenyl-4'- $\beta$ -aminocrotonate, m 201°. All the esters are readily hydrolyzed to their parent bases by cold dil. mineral acids but none of them could be converted into a quinoline deriv.

C. J. West

Asymmetric platinum atom VII. A new kind of optically active compound Hans Reihlen, Gerda Seipel and Erwin Weinbrenner. Ann. 520, 256-59 (1935); C. A. 29, 6163. —(—)-H<sub>2</sub>NC(CH<sub>2</sub>)<sub>2</sub>NH<sub>2</sub>(phen) (1.32 g) in 20 cc. Et<sub>2</sub>O, added to 4.5 g. K<sub>2</sub>PtCl<sub>6</sub> in 30 cc. H<sub>2</sub>O at 40° with stirring, gives 98% of dichloro-(—)-phen-platinum-2 (I), yellow, difficultly sol. in boiling H<sub>2</sub>O. Refluxing 4 g. I and 1.56 g.  $\alpha,\alpha$ -dipyridyl (dipy) in 61 H<sub>2</sub>O for 4 hrs gives 62% of (—)-phen-dipy-platinum-2 chloride (II),  $\text{C}_{10}\text{H}_{14}\text{N}_4\text{Cl}_2\text{Pt}$ , pale yellow, with 2 or 3 mols. H<sub>2</sub>O, [M]<sub>D</sub> 89°, chloroplatinate, yellow. II (0.45 g) and 0.31 g. Na bromocamphorsulfonate (III) (mol ratio 1:1) in H<sub>2</sub>O, give 85% of the chloride (+)-bromocamphorsulfonate (IV), with 3 mols. H<sub>2</sub>O, [M]<sub>D</sub> 363°, this is termed the racemic form. With a mol ratio of 1:2, there results 85% of an active isomer (V), with 3 mols. H<sub>2</sub>O, [M]<sub>D</sub> 392°, [ $\alpha$ ]<sub>D</sub> 25-9°, the intermediate glass in the prepn. of V is nearly pure racemic di-(+)-bromosulfonate (VI), with [M]<sub>D</sub> 622°, addn. of III to the mother liquor of V gives the active isomer (VII) with 2 mols. H<sub>2</sub>O, [M]<sub>D</sub> 604°. The nitrate (VIII) from IV or VI (91% yield), [M]<sub>D</sub> 88.5°, that (IX) from V or VII has [M]<sub>D</sub> 119°. Shaking 0.6 g. VIII with H<sub>2</sub>O at 10-15° for 15 hrs gives 0.3 g. IX, the filtrate contains VIII, the sol part from IX also has the higher rotation.  $\alpha$ -Phenanthroline and K<sub>2</sub>PtCl<sub>6</sub> give 92% of dichloro- $\alpha$ -phenanthroline platinum 2, yellow, (—)-phen- $\alpha$ -phenanthroline-2 chloride, with 2 mols. H<sub>2</sub>O, pale yellow, [M]<sub>D</sub> 119°, chloroplatinate, red, dibromocamphorsulfonate, with 2 mols. H<sub>2</sub>O, [M]<sub>D</sub> 670°. Phenyl-ethylendiamine ethyldiamine platinum-2 chloride was sepd into 2 fractions, [M]<sub>D</sub> 132° (difficultly sol.) and 120° (easily sol.). the iodide has [M]<sub>D</sub> 139° and 110.5°; the perchlorate, [M]<sub>D</sub> 109° and 142° (easily sol. fraction); the stodeo perchlorate has [M]<sub>D</sub> 142°.

C. J. West

Stereochemistry of azobenzene. IV. Eugen Muller and Rolf Illgen. Ann. 521, 72-80 (1935), cf. C. A. 27, 2141. —Nitration of hydrattic acid gives 30% of the  $\text{p-NO}_2$  deriv. (I), m 87°, reduction of the Na salt with Zn in NH<sub>4</sub>Cl gives the  $\text{p-hydroxylamino}$  deriv. (II), m 155-8°;  $\text{FeCl}_3$  oxidizes this in EtOH at 0° to the  $\text{p-NO}_2$  deriv. (III), pale yellow, m 93° (30% yield), a mixt. of II and III gives the azoxy deriv. (IV), m 198° (83%). I may be resolved by quinine, (+)-I, m 88.5°, [ $\alpha$ ]<sub>D</sub> 10.9° (0.092 g in 25 cc. MeOH); (—)-I, m 86.5°, [ $\alpha$ ]<sub>D</sub> -11.2° (0.039 g in 25 cc. MeOH); (+)-II, m 155-8°, [ $\alpha$ ]<sub>D</sub> 9.6° (0.573 g in 25 cc. MeOH); (—)-II, [ $\alpha$ ]<sub>D</sub> -10.6° (0.0052 g in 5 cc. MeOH); (+)-III, m 91°, [ $\alpha$ ]<sub>D</sub> 14.1° (0.1083 g in 25 cc. MeOH); (—)-III, m 92°, [ $\alpha$ ]<sub>D</sub> -14° (0.0848 g in 25 cc. MeOH). (—)-II and (+)-III give IV, optically inactive; this also results from (+)-II and (—)-III. (+) II and (+)-III give a (+) (+)-IV, [ $\alpha$ ]<sub>D</sub> 14.5° (0.2516 g in 25 cc. MeOH).

C. J. W.

Azo group as a chelating group. I. Metallic derivatives of  $\alpha$ -hydroxy azo compounds. Marjorie Eilkins and Louis Hunter. J. Chem. Soc. 1935, 1594-1610. —Metallic salts of the  $\alpha$ -hydroxy azo compds. have been prepd. with a view

of detecting coordination of the type  $\text{C}_6\text{H}_5\text{O}-\text{N}=\text{N}-\text{Ar}$

The formation of coordinated salts appears to be common to all such compds. and it is believed that their stability is due to their chelated structure, since salts of the  $\text{p-isomers}$  are extremely unstable. Benzeneazo- $\text{p-cresol}$  and excess  $\text{Cu}(\text{OAc})_2$  in 50% aq. EtOH give cupric  $\beta$ -benzeneazo- $\text{p-tolyl}$  oxide, brown, m 230°,  $\text{Ni}$  salt, black with green reflex, m 216°,  $\text{Co}$  salt (formed in presence of  $\text{H}_2\text{O}_2$ ), dull black, m 202°, 3- $\text{p-toluene}$  analogs:  $\text{Cu}$ , red-brown, m 242°,  $\text{Ni}$ , dark olive-green, m 242-3°,  $\text{Co}$ , jet-black, m 201-1°, 3- $\text{m-toluene}$  analogs:  $\text{Cu}$ , dark red-brown, m 221°,  $\text{Ni}$ , dark green, m 214°,  $\text{Co}$ , black, m 187°, 2,4-Bisbenzeneazophenoxides:  $\text{Cu}$ , chocolate-brown, m 243°,  $\text{Ni}$ , black, decompn. 295-310°,  $\text{Co}$ , black, m 180-5°, 1-Benzeneazo- $\beta$ -naphthorides:  $\text{Cu}$ , red-brown, m 295°,  $\text{Ni}$ , dark green, m 290°,  $\text{Co}$ , brownish black, m 221°, 2-isomers:  $\text{Cu}$ , dark brown, m 237°,  $\text{Ni}$ , light olive-green, m 213°,  $\text{Co}$ , dark purplish brown, m 199°. Decompn. of the metallic complexes by cold acid to the original azo compds. supports the view that all stable azo compds. are *trans*.

C. J. West

Oxidation of hydrohalic salts of phenylhydrazine in the presence of copper salts. A. B. Bruler and L. Z. Soborovskii. J. Gen. Chem. (U. S. S. R.) 5, 1024-8 (1935). Gatterman and Heile (Ber. 25, 1074 (1921)) obtained good yields of PhCl, PhBr and PhI by the oxidation of the resp. hydrohalic salts of PhNHNH<sub>2</sub> (I) with 4 mols. of  $\text{CuSO}_4$ . Treating  $\text{CuCl}_2$  in 12% HCl in the cold, with stirring, with an equimol. amt. of I resulted in a complete decolorization of the soln. and pptn. of white, cryst. I. HCl  $\text{CuCl}_2$  (II). The reaction is conceived as a partial oxidation of I with the formation of  $\text{CuCl}$  which reacts with I, giving II. This theory is supported by a nearly 100% yield of II obtained by the interaction of  $\text{CuCl}$  and I. The slight variations in the compn. of II obtained are caused by its instability to atm.  $\text{O}_2$ . Thus, II in HCl on exposure to air or addn. of  $\text{O}_2$  is rapidly decompd. with the formation of PhCl, but can be preserved for months by keeping it in hermetically sealed containers. The probable mechanism of the oxidation of I in HCl to PhCl with an excess of  $\text{Cu}^{++}$  salts consists of preliminary formation of II which on further oxidation forms PhN<sub>2</sub>Cl and this with  $\text{CuCl}$  gives PhCl according to the Sandmeyer reaction.

Chas. Blanc

$\alpha$ - and  $\text{p-Tolylstannonic}$  acids. K. A. Kocheskhov and M. M. Nad. J. Gen. Chem. (U. S. S. R.) 5, 1158-67 (1935); cf. C. A. 23, 5172; 23, 22934. —The following new stannanes and stannonic acids and their derivs. are described.  $\text{p-(MeC}_6\text{H}_4)_2\text{SnCl}_2$  (I), m 49-50°, and  $\alpha\text{-(MeC}_6\text{H}_4)_2\text{SnCl}_2$  (II), m 49-50°, were obtained by heating  $\text{SnCl}_4$  with  $\text{p-}$  and  $\alpha\text{-(MeC}_6\text{H}_4)_2\text{Sn}$ , resp., on a water bath and then at 200-5° for 2 hrs. I gave with 4 mols.  $\text{HgCl}_2$  in alc.  $\text{p-MeC}_6\text{H}_4\text{HgCl}_2$  m 233°, and with 1 mol.  $\text{HgCl}_2$  and  $\text{KOH}$   $\text{p-(MeC}_6\text{H}_4)_2\text{Hg}$ , m 243.5°.  $\text{p-MeC}_6\text{H}_4\text{SnCl}_2$  (III), b<sub>p</sub> 163-3°, d<sub>4</sub> 1.7512, resulted in 40% yield from 3 mols. of freshly prepd.  $\text{SnCl}_4$  and 1 mol. of  $\text{p-(MeC}_6\text{H}_4)_2\text{Sn}$  on heating the mixt. in a sealed tube at 210-15° for 2 hrs. III was also obtained in 45% yield by heating an equimol. mixt. of I and  $\text{SnCl}_4$  for 2 hrs., first at 100° and then at 210-15°.  $\alpha\text{-MeC}_6\text{H}_4\text{SnCl}_2$  (IV), b<sub>p</sub> 157-5°, d<sub>4</sub> 1.7719, in  $\text{H}_2\text{O}$  with  $\text{H}_2\text{S}$  gave 100%  $\alpha\text{-(MeC}_6\text{H}_4)_2\text{SnS}_2$ , yellow, amorphous and infusible powder. IV gave  $\alpha\text{-MeC}_6\text{H}_4\text{HgCl}_2$  m 146°. III mixed with pyridine in Et<sub>2</sub>O gave III.2Py.  $\alpha\text{-MeC}_6\text{H}_4\text{SnO}_2\text{H}$  (V) and the  $\text{p-isomer}$ , infusible at 295°, were prepd. by the interaction of III and IV, resp., with 20%  $\text{KOH}$ , neutralizing the soln. with  $\text{AcOH}$  and then with  $\text{CO}_2$  to a neutral reaction, dissolving the ppt. in alc. and evapn. the filtrate at 40-55° *in vacuo*.  $\alpha\text{-(MeC}_6\text{H}_4)_2\text{Hg}$ , m 107°, was prepd. from IV in 5 N KOH with an equimol.

amt. of  $\text{HgCl}_2$ . V oxidized with  $\text{K}_2\text{Fe}(\text{CN})_6$  is completely decolorized, with sepn. of nearly 100%  $\text{K}_2\text{S}_2\text{O}_8$ . C. B.

**Derivatives of allyl ethers of phenols mercaptated in the nucleus.** A. N. Nesmeyanov and R. Kh. Shatzkaya. *J. Gen. Chem.* (U. S. S. R.) 5, 1248-52 (1935).— $\text{C}_6\text{H}_5\text{OC}_3\text{H}_5\text{I}_2\text{HgCl}$  (I) was prepared in 3.5 g yield (40%) by refluxing a mixt of 8 g  $\alpha$ - $\text{NaOC}_3\text{H}_5\text{I}_2\text{HgCl}$  (II) in 25 cc  $\text{Me}_2\text{CO}$  with 6 g allyl bromide (III) for 7 hrs. A prolonged heating gave  $\alpha$ - $(\text{C}_6\text{H}_5\text{OC}_3\text{H}_5)_2\text{Hg}$  (IV), m. 50.5–60.5°. IV was also obtained by substituting allyl chloride for III in the above rxn., and by treating 1 g I with 1 g  $\text{SnCl}_4$  in 20%  $\text{NaOH}$  ( $\text{C}_6\text{H}_5\text{NC}_3\text{H}_5\text{I}_2\text{HgBr}$ , m. 120–15°, was obtained (I) by treating 30 g II in 40 g  $\text{C}_6\text{H}_5\text{N}$  at  $-10^\circ$  with 11.2 g III, (2) by mixing in the cold 3 g  $\text{HgBr}_2$  in  $\text{C}_6\text{H}_5\text{N}$  (satd.) with an equal vol. of III, and (3) from 1,2- $\text{HOC}_3\text{H}_5\text{I}_2\text{HgCl}$  in  $\text{C}_6\text{H}_5\text{N}$  with III.  $\beta$ - $(\text{C}_6\text{H}_5\text{OC}_3\text{H}_5)_2\text{HgCl}$  (V), m. 101–15°, was obtained in 2.5 g yield (57%) by treating an emulsion of 15 g  $\text{PhOC}_3\text{H}_5$ , 9 cc. of 80%  $\text{AcOH}$  and 37 cc.  $\text{H}_2\text{O}$  with 6.5 g  $\text{HgO}$  in form of a paste, pouring the hot mixt into a boiling soln. of 50 g  $\text{NaCl}$ , boiling for 2–3 min. and allowing the mixt to cool. V, treated with Na stannite as above, gave  $\beta$ - $(\text{C}_6\text{H}_5\text{OC}_3\text{H}_5)_2\text{Hg}$ , m. 108.5–10°.  $\text{C}_6\text{H}_5\text{OC}_3\text{H}_5\text{I}_2\text{HgCl}$ , m. 161.2°, was prepd from 5 g  $\text{C}_6\text{H}_5\text{OC}_3\text{H}_5$  and  $\text{HgO}$  by the method described for the prepn. of V. The structures of some of these compds. are being investigated. Chas. Blanc.

**Decolorization of indigotin by alkyl and aryl sulfonic acids.** Max Bazken and Fritz Scholtz. *Ber.* 68B, 2045 (1935).—Hitherto it had not been possible to decolorize indigotin in water with alkyl and aryl sulfonic acids as with aldehydo sulfonic acids (C. A. 21, 3170). B and S have now established the conditions under which the reduction can be effected with these acids also. The free sulfonic acids themselves cannot be used, as they decompose above 100° before they exert a reducing action. Salts must be used, at 180° and above, best in glycerol. Under these conditions they behave like, e. g., rongite. Aq. neutral indigotin (1.7 g indigo per l.), boiled with a few particles rongite, is decolorized after a time, and on cooling and shaking with air the blue color is immediately restored. The neutral Na and Zn salts of  $\text{RSO}_3\text{H}$  (R = Me, 1,1', $\beta$ , $\alpha$ - and  $\beta$ - $\text{C}_6\text{H}_4$ ) behave in exactly the same way in glycerol at 180°. and as do also  $\beta$ - $\text{MeC}_6\text{H}_4\text{SO}_3\text{H}$ ,  $\alpha$ - $\text{H}_2\text{NC}_6\text{H}_4\text{SO}_3\text{H}$ , benzanthrone- $\beta$ -sulfonic acid and  $\beta$ - $\text{C}_6\text{H}_4(\text{CO})_2\text{C}_6\text{H}_4\text{SO}_3\text{H}$ . C. A. R.

**Color reactions of phenols with nitrous acid.** M. S. Mahmood. *J. Gen. Chem.* (U. S. S. R.) 5, 1247–8 (1935).—A discovery that phenols on the addn. of highly dil.  $\text{HNO}_2$  give nearly identical color reactions from brown to yellow without the formation of a blue tint, observed by Castiglioni (C. A. 27, 2078) and other investigators, led to a study of the influence of the following factors on the coloring of phenols by  $\text{HNO}_2$ : the degree of  $\text{HNO}_2$  diln., the position of the OH groups in phenols, and the presence of other than OH groups in phenol. The procedure consisted of adding 2–3 drops of strong  $\text{H}_2\text{SO}_4$  and 2–3 drops of 1% phenol in  $\text{H}_2\text{O}$  to 2 cc. of a nitrite soln. Trihydric phenols gave coloring of the highest intensity.  $\text{PhOH}$ , cresol, carvacrol,  $\alpha$ - and  $\beta$ - $\text{HOC}_6\text{H}_4\text{NO}_2$ , phenetole and anisole gave no color reaction even with 10%  $\text{HNO}_2$ . A closer position of the OH groups to each other in a mol. increases the color sensitivity of phenols: pyrocatechol is colored by 0.004%  $\text{HNO}_2$ , while hydroquinone begins to give color with 0.02%  $\text{HNO}_2$ . Pyrogallol (1%) gives color reaction with 0.001%  $\text{HNO}_2$ , while phloroglucinol and hydroxyhydroquinone give no color at this concn. of  $\text{HNO}_2$ . Di- and tri-hydric phenols with the 1 OH acetylated give no color reactions. Free  $\text{HNO}_2$ , nitrates,  $\text{HClO}_3$ ,  $\text{HClO}_2$ , and other oxidants give no similar reactions with phenols. Chas. Blanc.

**Iodination and nitration of diphenyl ether-aldehyde.** K. H. Slotta and K. H. Soremla. *Ber.* 68B, 2059–66 (1935).—In the Gattermann method of prepn.  $\beta$ - $\text{PhO-C}_6\text{H}_4\text{CHO}$  (I) (C. A. 1, 820), when applied to large quantities of materials, recombination reduces the yields to 20%. This has been found to be due to the use of the large excess (5 mols.) of  $\text{HCN}$ , by using 1.3–1.8 mols

1  $\text{HCN}$  per mol.  $\text{Ph}_2\text{O}$ , 70–80% I can be obtained and purification through the bisulfite compd. is not necessary. Introduction of I into I must be effected with reagents which do not alter the CHO group. Of the various methods tried, only the use of  $\text{ICl}$  and  $\text{HIO}_3$  was successful. There was thus obtained in good yield  $p$ -(4'-iodophenyl)-benzaldehyde (II), the structure of which was established by oxidation to the acid. The addn. of  $\text{HIO}_3$  seems to be specific, since without it no or only very little iodination occurs. The most successful of the various methods tried for prepn.  $\beta$ -(2'- $\text{O-NC}_6\text{H}_4\text{O}$ ) $\text{C}_6\text{H}_4\text{CHO}$  (III) by nitration of I consisted in dropping I at 15–20° into concd.  $\text{HNO}_3$  (d. 1.140), which yielded about 50%  $\beta$ -(4'- $\text{O-NC}_6\text{H}_4\text{O}$ ) $\text{C}_6\text{H}_4\text{CHO}$  (IV) along with III. Nitrating acid ( $\text{HNO}_3 \cdot \text{H}_2\text{SO}_4 = 1:2$ ) gave 70–80% 2',4'-dinitro-4-iodobenzaldehyde diphenyl ether (V).  $\beta$ -(2',4'- $\text{O}_2\text{N}$ ) $\text{C}_6\text{H}_4\text{O}$  $\text{C}_6\text{H}_4\text{CHO}$  (VI), from 2,4- $\text{O}_2\text{N}$  $\text{C}_6\text{H}_4\text{Cl}$  and  $\beta$ - $\text{HOC}_6\text{H}_4\text{CHO}$ , when nitrated like I above, gave V, that the new  $\text{NO}_2$  group is in the 2-position was established by the fact that V is different from the 2',4',6'-trinitro compd. (VII) prepd from puryl chloride and  $\beta$ - $\text{HOC}_6\text{H}_4\text{CHO}$ . This was confirmed by cleavage of the corresponding acid prepd. by oxidation of V with  $\text{CrO}_3/\text{AcOH}$ . In the product, 3-nitro-4-(2',4'- $\text{O}_2\text{N}$ )benzoic acid (VIII), the O-bridge, evidently weakened by the 2- $\text{O-NO}_2$  groups, is ruptured by boiling dil. alkalis with formation of 2,4- $\text{O}_2\text{N}$  $\text{C}_6\text{H}_4\text{O}$  and 1,3- $\text{HIO}_2(\text{O}_2\text{N})\text{C}_6\text{H}_4\text{CO}_2\text{H}$ . I, b. 174.6°, 170–82°, 1.33–5, 101.3° under 12, 14, 18 and 22 mm. n.p. II (yld. 81% from I in  $\text{AcOH}$  at 50° slowly treated with slightly more than 1 mol.  $\text{ICl}$ , then heated 20 hrs. at 110–20° with hourly addns. of finely powd.  $\text{HIO}_3$  (about 5 g in all for 50 g I) and purified through the bisulfite compd.), light yellow, m. 65–7°, phenylhydrazone, yellowish white, m. 150°. acid,  $\text{IClC}_6\text{H}_4\text{CO}_2\text{H}$ , m. 215°. I was unchanged by 1- $\text{HIO}_3$  in boiling 1- $\text{HIO}_3/\text{H}_2\text{O}$ , and  $\beta$ - $\text{IClC}_6\text{H}_4\text{O}$  in  $\text{C}_6\text{H}_5$  with  $\text{AlCl}_3$  and  $\text{HCN}$  gave only  $\text{PhO}$  and I, with no II. IV, m. 107–8°, phenylhydrazone, light yellow, m. 160°. III, m. 84–6°, acid, m. 143°. V, m. 100°. phenylhydrazone, red, m. 190–2°. VI (90% yld.), whitish yellow, m. 128° becomes discolored in the light; phenylhydrazone, yellow leaflets with metallic luster, m. 225°. VIII, greenish white, m. 205–7°. C. A. R.

6 The salts of 1,2,4-phenoldisulfonic acid. J. Šuráček. *Chem. Listy* 29, 213–5 (1935).—S. prepd. and analyzed the crystals of Li, Na, K, Be, Mg, Zn, Cd, Ca, Sr, Ba, Cu, Mn, Ni, Co and Al 1,2,4-phenoldisulfonates. In comparing these salts with those prepd. with  $\text{PhSO}_3\text{H}$ ,  $\beta$ - $\text{MeC}_6\text{H}_4\text{SO}_3\text{H}$ ,  $\beta$ - $\text{HOC}_6\text{H}_4\text{SO}_3\text{H}$ , 1,2,4- $\text{Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{SO}_3\text{H}$ , and 1,2,4- $\text{HOC}_6\text{H}_3(\text{SO}_3\text{H})_2$ , S. finds that the no. of mols. of water of crystn. becomes larger and more variable as the structure of the anion becomes more complicated and complex. Placing Be and Mg into the subgroup containing Zn and Cd of the periodic system is erroneous, for the no. of mols. of the water of crystn. attached to the Be, Mg and Zn, Cd salt becomes more variable and divergent as the anion becomes more complicated and as the sulfonic acid becomes more distinctive in its chem. behavior. Be 1,2,4-phenoldisulfonate just as Be  $\alpha$ -naphthalenesulfonate forms tetrahydrates. T. M.

7 Metallic derivatives of 2-nitroso-5-methoxy- and of 3-chloro-2-nitroso-5-methoxyphenol. Herbert H. Hodgson and Walter E. Batty. *J. Chem. Soc.* 1935, 1617–19.—In the following X =  $\text{CH}_3\text{O}$  and Y =  $\text{Cl}$ ,  $\text{O}_2\text{N}$ . The following are probably coordination compds.  $\text{HgV}_2$ , deep red microcrystals, m. 201°;  $\text{HgI}_2$ , bright red microcrystals,  $\text{AlVCl}_3$ , light yellow, darkens in air;  $\text{AlVCl}_3$ , very light yellow prisms  $\text{SnVCl}_3$ , red-orange microplates;  $\text{SnVCl}_3$ , brown-red plates,  $\text{Fe}(\text{OEt})_3$ , dark green microneedles;  $\text{FeV}_2$ , brown;  $\text{Fe}(\text{OEt})_3$ , dark green needles;  $\text{FeV}_2$ , brown. CoV, red microcrystals; CoV, bright red micrograins.  $\text{NiV}_2$ , microcrystals;  $\text{NiV}_2$ , bright red. The  $\text{NiV}$  compd. with  $\alpha$ - $\text{HOC}_6\text{H}_4\text{CHO}$ , light yellow; the  $\text{KX}$  compd. with  $\alpha$ - $\text{HOC}_6\text{H}_4\text{CHO}$ , light yellow. Non-coordinated compds.:  $\text{NaV}$ , bright green;  $\text{NaV}$ , bright green;  $\text{KX}$ , bright green;  $\text{KX}$ , bright green;  $\text{AgV}$ , red-brown microcrystals;  $\text{AgV}$ , brown microcrystals;  $\text{ZnV}_2$ , light yellow-brown, flat prisms;  $\text{ZnV}_2$ ,

light yellow prisms,  $CdX_2$ , maroon macroprisms;  $CdY_2$ , maroon plates  $PbX_2$ , dark yellow-brown,  $PbY_2$ , dark brown  $MnX_2$ , brown prisms,  $MnY_2$ , dark brown prisms  $UO_2X_2$ , yellow-brown,  $UO_2Y_2$ , yellow-brown microplates. The structure of these compounds is discussed.

C. J. West

Mechanism of the oxidation of *o*-toluenesulfonamide to saccharin O Yu Magidson and I G Zil'berg *J Gen. Chem. (U S S R)* 5, 920-3 (1935) —In the preliminary study of the mechanism of oxidation of *o*- $MeC_6H_4SO_2NH_2$  (I) to saccharin, I was oxidized with  $CrO_3$  in  $Ac_2O$  at from  $-5^\circ$  to  $30^\circ$  with and without the addition of a little  $H_2SO_4$ . By the method of fractional neutralization and  $Et_2O$  extrn of the mother liquor, the following 4 compounds were isolated *o*- $MeC_6H_4SO_2NHAc$ , m 132-4°; (with

$H_2SO_4$ ) acetosaccharin  $OC_6H_4SO_2NAC$ , m 195-7° (cf *Ber* 29, 1050), a compd, m 174-6°, easily oxidized with  $KMnO_4$  to saccharin and which is, probably, either

$AcOCH_2C_6H_4SO_2NHAc$  or  $AcOCH_2C_6H_4SO_2NAC$ , and a compd, m 103-5°, which was not identified. C. B.

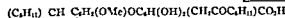
Walden inversion reactions of the *p*-toluenesulfonic and the *p*-toluenesulfonic esters of ethyl *d*- $\beta$ -hydroxy- $\beta$ -phenylpropionate Joseph Kenyon, Henry Phillips and Gerald R. Shutt *J Chem Soc* 1935, 1063-8, cf. *C. A.* 29, 7272° — $PhCH(OH)CH_2CO_2H$  (I) may be resolved by brucine, *d*-I, m 115°,  $[a]_{D}^{25}$  18.9°,  $[a]_{D}^{25}$  23.4°,  $[a]_{D}^{25}$  33.4° (EtOH, c 5.00); brucine salt, m 145°,  $[a]_{D}^{25}$  37.85°,  $[a]_{D}^{25}$  45.8° (EtOH, c 5.00); *l*-I, m 115-16°,  $[a]_{D}^{25}$  -19° (EtOH, c 5.130); *Et* ester (II) of *d*-I, *b*<sub>1</sub>, 90-1°, *d*<sub>1</sub><sup>1</sup>, 1.058, *n*<sub>D</sub><sup>20</sup> 1.5125,  $\alpha_{D}^{25}$  19-17°,  $\alpha_{D}^{25}$  23.39°,  $\alpha_{D}^{25}$  31.52°, with  $EtI$  and  $Ag_2O$  II yields *Et* *d*-(+)- $\beta$ -ethoxy- $\beta$ -phenylpropionate (III), *b*<sub>1</sub>, 85-6°, *n*<sub>D</sub><sup>20</sup> 1.4983, *d*<sub>1</sub><sup>1</sup>, 1.067,  $\alpha_{D}^{25}$  7.59°. The *Ac* deriv of II *b*<sub>1</sub>, 102-3°, *d*<sub>1</sub><sup>1</sup>, 1.088, *n*<sub>D</sub><sup>20</sup> 1.5168,  $\alpha_{D}^{25}$  1.32°,  $\alpha_{D}^{25}$  1.66°,  $\alpha_{D}^{25}$  2.79°, the formoxy deriv *b*<sub>1</sub>, 95-7°, *d*<sub>1</sub><sup>1</sup>, 1.123,  $\alpha_{D}^{25}$  1.5333,  $\alpha_{D}^{25}$  2.47°,  $\alpha_{D}^{25}$  2.98°,  $\alpha_{D}^{25}$  4.39°. II and *p*- $MeC_6H_4SOCl$  give the *p*-toluenesulfonate deriv (IV), yellow, could not be distd,  $\alpha_{D}^{25}$  3.77°, *n*<sub>D</sub><sup>20</sup> 1.5286, *d*<sub>1</sub><sup>1</sup>, 1.178°, heating with  $Ac_2O$  and  $AcOH$  gives the *Ac* deriv, while  $HCO_2H$  gives the formoxy deriv IV is converted into *l*-(+)- $PhCH(Cl)CH_2CO_2Et$  (V) by  $Cl$  in  $CHCl_3$  ( $\alpha_{D}^{25}$  -1.57°),  $Cl$  in  $H_2O$  ( $\alpha_{D}^{25}$  -1.30°),  $HOCl$  ( $\alpha_{D}^{25}$  -0.72°) and  $ICl$  ( $\alpha_{D}^{25}$  -2.27°).  $Br$  in  $CHCl_3$  or  $H_2O$  gives *Et* *l*-(-)- $\beta$ -bromo- $\beta$ -phenylpropionate, *b*<sub>1</sub>, 82-4°, *n*<sub>D</sub><sup>20</sup> 1.5124,  $\alpha_{D}^{25}$  -1.43°.  $CNCl$  yields the *p*- $CN$  deriv, *b*<sub>1</sub>, 78-9°, *n*<sub>D</sub><sup>20</sup> 1.5243, *d*<sub>1</sub><sup>1</sup>, 1.811,  $\alpha_{D}^{25}$  -0.38°,  $\alpha_{D}^{25}$  -0.53°,  $\alpha_{D}^{25}$  -0.92°. IV and  $HNO_3$  gives *l*-(-)- $PhCH(OH)CH_2CO_2Et$ ,  $\alpha_{D}^{25}$  -2.57°. Refluxing III in  $EtOH$  ( $N$  atm.) for 48 hrs gives *l*-III with  $\alpha_{D}^{25}$  -3.73°, while  $EtOH$  contg.  $LiCl$  and  $K_2CO_3$  gives *d*-III with  $\alpha_{D}^{25}$  4.91°. Oxidation of IV with  $H_2O_2$  gives the  $\beta$ -*p*-toluenesulfonyloxy deriv (VI), which could not be distd,  $\alpha_{D}^{25}$   $EtOH$ - $K_2CO_3$  gives *l*-III with  $\alpha_{D}^{25}$  -5.12° and  $EtOH$ - $LiCl$ - $K_2CO_3$  gives *d*-III with  $\alpha_{D}^{25}$  4.36°. V with  $EtOH$ - $K_2CO_3$  gives *d*-III with  $\alpha_{D}^{25}$  0.23°. IV and  $HI_2O$  in  $AcOH$  give the *l*-*Ac* deriv,  $\alpha_{D}^{25}$  -0.85°; addn of  $LiCl$  to this reaction gives a mixt of the *Cl* deriv with  $\alpha_{D}^{25}$  -1.3° and the *Ac* deriv with  $\alpha_{D}^{25}$  -1.23°. *d*-(+)- $PhCH(OH)CH_2CO_2Et$  yields (-)- $PhCH(Cl)CH_2CO_2Et$  with  $PCl_5$  ( $\alpha_{D}^{25}$  -1.59°),  $PCl_5$  +  $C_2H_5N$  ( $\alpha_{D}^{25}$  -2.40°),  $PCl_5$  ( $\alpha_{D}^{25}$  -1.04°),  $PCl_5$  +  $C_2H_5N$  ( $\alpha_{D}^{25}$  -2.69°),  $SOCl_2$  +  $C_2H_5N$  ( $\alpha_{D}^{25}$  -2.21°), while  $SOCl_2$  alone gives the *d*-*Cl* deriv with  $\alpha_{D}^{25}$  2.87°.  $PBr_3$  gives the *l*-*Br* deriv with  $\alpha_{D}^{25}$  -5.08°, while with  $C_2H_5N$   $\alpha_{D}^{25}$  is -7.25°. The configurational relations of these compounds are discussed and illustrated with charts. C. J. West

Addition of halogens to unsaturated acids and esters. V. The bromination of *m* methoxycinnamic acid and its ethyl ester J. Idres Jones and T. Campbell James *J. Chem. Soc.* 1935, 1600-4 —*m*- $MeOC_6H_4CH=CHCO_2H$  (I) and *Br* in  $AcOH$  in the dark give a quant. yield of the 6-*Br* deriv. (II).  $Br$  in  $CCl_4$  or  $CHCl_3$  at  $0^\circ$  in the dark gives the same deriv. In boiling  $CCl_4$  in bright sunlight there results  $\alpha$ , $\beta$ -*dibromo*- $\beta$ -3-methoxyphenylpropionic acid (III), m 167°, the rate of addn in ice-cold  $CCl_4$  in incident sunlight is very slow. addn of *Br* was also observed

in boiling  $CS_2$ , in boiling  $AcOH$  in sunlight the product was almost pure II. In boiling  $CHCl_3$  the product was a mixt of II and III, with II predominating. I and *Br* in boiling  $MeOH$  gave 6,3 *Br*( $MeO$ ) $C_6H_3CH=CHCO_2Me$ . The *Et* ester of I and *Br* in cold  $AcOH$  or  $CHCl_3$  in the dark give the 6-*Br* deriv., while in  $CCl_4$  in strong sunlight there results the *Et* ester of III, m 58°. II and *Br* in  $CCl_4$  in sunlight gave the  $\beta$ -6-*Br* deriv of III, cream, m 163°. III (20 g) with 2 mols  $EtOH$ - $KOH$  for 2 days at 25° gives 2.4 g of the  $\alpha$ -*Br* deriv. (IV) of I, m 122°, and 9.8 g of  $\alpha$ -*bromo*-*m* methoxyphenylpropionic acid (V), pale yellow, m 91°. V is transformed into IV by sunlight or by heating the solid at 150° for several hrs. IV is considered to have the *trans*- and V the *cis*-configuration. The ester of III gives a larger proportion of IV. These acids may be sepd as the *Ba* salts, that of IV being less sol. in  $H_2O$ . IV and 2 mols  $EtOH$ - $KOH$  boiled 4 hrs, give 96% of *m*-methoxyphenylpropionic acid (VI), m 109°. VI and I in  $K_2CO_3$  give  $\alpha$ , $\beta$ -*disudo*-*m*-methoxycinnamic acid, yellow, m 142°. Catalytic reduction of VI yields *m*-methoxyphenylpropionic acid, m 109-10°, the 6-*Br* deriv, m 133°, forms by the action of *Br* in cold  $CHCl_3$  in the dark. C. J. West

Spectroscopic investigations of amino acids and amino acid derivatives. I. Ultraviolet absorption spectra of *l*-tyrosine, *d*l phenylalanine and *l* tryptophan Katherine Feraud, Max S. Dunn and Joseph Kaplan *J Biol Chem* 112, 323-8 (1935) —The ultraviolet absorption spectra of *l*-tyrosine, *d*l-phenylalanine and *l* tryptophan resemble those of their corresponding aromatic nuclei, hydroxyphenyl, phenyl and indole. The aliphatic side chains serve merely to produce alterations in the positions of the absorption bands. The accurate detn of either *l*-tyrosine or *d*l-phenylalanine in a protein or a mixt of amino acids is feasible only in the absence of the other form because of the overlapping spectra of these 2 amino acids. A. P. Lohrhop

Lichen substances. LIX. The nonexistence of  $\gamma$ -collatolic acid Yasuhiko Asahina and Tokujiro Fujikawa *Ber.* 68B, 2020-1 (1935); cf *C. A.* 29, 7963° —It had been reported that the *Me* ester of  $\alpha$ -collatolic acid (I) is isomerized on sapon. with alkali to a  $\beta$ -acid (II), m 162°, whereas on standing 2 days in pyridine it changes into a 2nd isomeric  $\gamma$ -acid (III), m 140-1° (*C. A.* 29, 3066°). It has since been found that I changes completely into II on long standing in  $NaHCO_3$ . II is best characterized through its *Me* ester, m 75°. Permethylations of I and II with  $CH_3N$  gives the same *d*-*Me* ether *Me* ester (IV), m 114°. The supposed III proved to be a nonhomogeneous substance, consisting of I admixed with more or less I. On repeated crystn from benzene it yields pure II and  $CH_3N$  converts it almost quantitatively into IV. III should therefore be stricken from the literature, and there remains for II only the structure  $CO$  O C-



LX. Microphyllinic acid and its cleavage products *Ibid* 2022-6 —Having secured a larger amt of the lichen, A and F have been able to ext. enough microphyllinic acid (I) (*C. A.* 29, 2524°) to make some of its derivs. Treated in  $AcOH$  suspension with a drop of concd  $H_2SO_4$ , I gives anhydroacetilmicrophyllinic acid, m 98°, insol in  $NaHCO_3$ ,  $Na_2CO_3$  and cold  $KOH$ , gives no color with alc.  $FeCl_3$ . Sapon of I with cold  $l$   $KOH$  yields olivetonide *p* *Me* ether (II), m 57°, and olivetonic acid, m 150-60°, sol without color in  $NaHCO_3$ ,  $Na_2CO_3$  and  $KOH$  and giving a violet color with alc.  $FeCl_3$  and a red color with bleaching powder (*Me* ester, m 85-6°). *Me* ester *d*-*Me* ether of I, from I with  $Ag_2O$  and *MeI* in ether (excess of  $CH_3N$  decomposes I), m 89-90°, insol. in cold  $KOH$ , gives no color with alc.  $FeCl_3$ ; with boiling 95%  $HCO_2H$  it gives olivetonide *d*-*Me* ether, m 94°, and olivetonide  $\alpha$ -*Me* ether (III), m 146-7°, which gives no color with alc.  $FeCl_3$  or bleaching powder and dissolves without color in  $KOH$ . *p*-Acetylolivetonic acid, from olivetonide and  $Ac_2O$ , m 53°, insol. in  $Na_2CO_3$ , difficultly sol. in alkalis, gives a violet color with  $FeCl_3$ .

and no color with bleaching powder, yields with MeI and Ag<sub>2</sub>O in ether the *o*-Me ether, m. 84°, also obtained by acetylation of III. II, best prep'd. from olivetonide and ClH<sub>3</sub>N<sub>3</sub>, m. 57°, gives a violet color with alc. FeCl<sub>3</sub>. *o*-Ac deriv., m. 60–1°, gives no color with alc. FeCl<sub>3</sub>. Olivetonic acid *p*-Me ether, from II and aq. KOH, m. 94–5°, gives a violet color with alc. FeCl<sub>3</sub>, is readily converted back into II by boiling HCO<sub>2</sub>H. Me ester, m. 80°. Olivetonic acid *o*-Me ether, from III and aq. KOH, m. 119–20°, gives no color in alc. with FeCl<sub>3</sub> or bleaching powder. LXI. Olivetonic acid 3. Ibid 2026–8—Olivetonic acid (I), identical in all respects with that obtained from *Alectoria salazana* Gyalnik (C A 26, 3244, the lichen is incorrectly named in this paper, see J Jap Bot. 10, 18 (1934)), has been isolated from *Parmelia olivetorum* Nyl. It m. 150–1°, gives in alc. with FeCl<sub>3</sub> a violet, with bleaching powder a red color, and is slowly colored lemon-yellow and then green when moistened with Ba(OH)<sub>2</sub>. Me ester, from the acid tritied in ether with CH<sub>3</sub>N<sub>3</sub>, yellow color with subsequent rapid decolorization, m. 134°, gives in alc. with FeCl<sub>3</sub> a violet, with bleaching powder a blood-red color. Me ester *tri*-Me ether, from the acid with Ag<sub>2</sub>O and MeI in ether, in 74.5%, is hydrolyzed by 5% alc. KOH to olivetonic acid *di*-Me ether, m. 93° (giving no color with alc. FeCl<sub>3</sub> or bleaching powder), and olivetolcarboxylic acid *o*-Me ether, m. 105°.

C. A. R.  
Synthesis of coumarins from phenols and  $\beta$ -ketonic esters. III. Use of various condensing agents. Duhkharan Chakravarti J Indian Chem Soc 12, 530–9 (1935); cf. C A 26, 434—Various condensing agents have been investigated as substitutes for P<sub>2</sub>O<sub>5</sub> for the synthesis of chromones but all attempts failed since coumarins were invariably formed. In Pechmann's reaction H<sub>2</sub>SO<sub>4</sub> may be replaced by P<sub>2</sub>O<sub>5</sub> with advantage, especially in the case of polyhydroxy phenols, when better yields and purer products are obtained. In Bulow's reaction the condensation of phenols with  $\beta$ -diketones may be effected with advantage by using H<sub>2</sub>SO<sub>4</sub> or better, P<sub>2</sub>O<sub>5</sub>. The addn. of 30 cc. of H<sub>2</sub>PO<sub>4</sub> to a mixt. of 10 cc. of resorcinol (I) and 8 cc. of AcCH<sub>2</sub>CO<sub>2</sub>Et (II) produced, on standing, 50% of  $\beta$ -methylumbelliferone (III), m. 185–6°, Ac deriv., m. 150°. I and II condensed in the presence of AcONa, B<sub>2</sub>O<sub>3</sub>, EtONa on heating to give 72, 50 and 64% yields of III. The condensation of I with EtO<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>AcCO<sub>2</sub>Et (IV) in the presence of H<sub>2</sub>PO<sub>4</sub> or P<sub>2</sub>O<sub>5</sub> gave 7,8-dihydroxy-4-methylcoumarin-3-acetate, m. 163°, identical with a specimen prep'd. in the presence of H<sub>2</sub>SO<sub>4</sub>. With orcinol and IV, Et 5-hydroxy-4,7-dimethylcoumarin-3-acetate, m. 194–200°, was similarly prep'd. Pyrogallol and IV in the presence of H<sub>2</sub>SO<sub>4</sub> yielded Et 7,8-dihydroxy-4-methylcoumarin-3-acetate, m. 180°. With H<sub>2</sub>PO<sub>4</sub>, AcONa or EtONa as condensing agents, mixts. of phenols and  $\beta$ -ketonic esters gave the following coumarins: 5-hydroxy-4,7-dimethyl, m. 250°, 7,8-dihydroxy-4-methyl, m. 235°, 5,7-dihydroxy-4-methyl, m. 255°, 7,8-dihydroxy-3-phenyl-4-methyl, m. 268°, 7-hydroxy-4-phenyl, m. 210°, 7,8-dihydroxy-4-phenyl, m. 188°, 7-hydroxy-3-benzyl-4-methyl, m. 221°, 7-hydroxy-3,4-dimethyl, m. 250°, and 4,6-dimethyl, m. 150°. The condensation of  $\alpha$ - and  $\beta$ -C<sub>10</sub>H<sub>7</sub>OH with II in the presence of H<sub>2</sub>PO<sub>4</sub> or NaOAc gave 4-methyl-1,2- $\alpha$ -naphthopyrone, m. 168°, and the corresponding  $\beta$ -naphthopyrone, forming the styrene deriv., m. 193°, with BzH<sub>2</sub>. IV Coumarins from 4-chloro- and 2-nitroresorcinols. Duhkharan Chakravarti and Baidyanath Ghosh Ibid 622–6.—The generalization made by Clayton (C A 2, 1966) as to the hindering and total inhibition of Pechmann's reaction by the introduction into the phenolic nucleus of halogen atoms and neg. groups, resp., has been tested by an investigation of the condensation of chloro- (I) and nitroresorcinol (II) with alkylacetoacetic esters in the presence of H<sub>2</sub>SO<sub>4</sub> and of P<sub>2</sub>O<sub>5</sub>. I, m. 88.5–9°, b. 250°, condensed in the presence of H<sub>2</sub>SO<sub>4</sub> with AcCH<sub>2</sub>CO<sub>2</sub>Et to form 6-chloro-7-hydroxy-4-methylcoumarin, m. 280°, Ac deriv., m. 168°. On changing the condensing agent to P<sub>2</sub>O<sub>5</sub>, the identical coumarin and no chromone was formed (C A 9, 2762). I condensed with the Me, Et, Pr, iso-Bu, PhCH<sub>2</sub>,  $\alpha$ -Cl derivs. of acetoacetic ester and also with (HO<sub>2</sub>CCH<sub>2</sub>)<sub>2</sub>-

CO, EtO<sub>2</sub>CCH<sub>2</sub>CHAcCO<sub>2</sub>Et, and BzCH<sub>2</sub>CO<sub>2</sub>Et in the presence of H<sub>2</sub>SO<sub>4</sub> or even of P<sub>2</sub>O<sub>5</sub> to give good yields of the following 6-chloro-7-hydroxy-4-methylcoumarins: 3-Me, m. 248° (Ac deriv., m. 170–1°); 3-Et, m. 257–8° (Ac deriv., m. 145°); 3-Pr, m. 230° (Ac deriv., m. 137°); 3-iso-Bu, m. 199°; 3-benzyl, m. 249°; 3-Cl, m. 154° (Ac deriv., m. 192°), and the 6-chloro-7-hydroxycoumarin-4-acetic acid, m. 210°; Et 6-chloro-7-hydroxy-4-methylcoumarin-3-acetate, m. 174°, and 6-chloro-7-hydroxy-4-phenylcoumarin, m. 258–60°. I condensed with HO<sub>2</sub>CCH<sub>2</sub>CH(OH)CO<sub>2</sub>H in the presence of H<sub>2</sub>SO<sub>4</sub> to form 6-chloroumbelliferone, m. 271° (Ac deriv., m. 160°), as readily as resorcinol under the same conditions. II condensed with AcCH<sub>2</sub>CO<sub>2</sub>Et and MeCH<sub>2</sub>COCH<sub>2</sub>CO<sub>2</sub>Et in the presence of H<sub>2</sub>SO<sub>4</sub> to give 8-nitro-7-hydroxy-4-methylcoumarin, m. 256° (Ac deriv., m. 193°), and 8-nitro-7-hydroxy-3,4-dimethylcoumarin, m. 260° (decompn.) (Ac deriv., m. 246°), in 60 and 15% yields, resp. No condensation was achieved with the ethyl-, propyl- and butyl-acetoacetic esters and it was not possible to isolate any product with P<sub>2</sub>O<sub>5</sub>, due to the formation of resins. Thus a substituent in the acetoacetic ester mol., however heavy or complex, does not in any way favor the formation of a chromone even in the presence of P<sub>2</sub>O<sub>5</sub>. The violet fluorescence of the umbelliferones in alk. soln. is appreciably increased by the introduction of Cl into the pyrone ring but is destroyed completely by the presence of the NO<sub>2</sub> group. C. R. Addnall.

Bitter principles of calomel roots. III. K. Feist, E. Kuntz and R. Brachvogel. Ann 521, 184–8 (1935); cf. C A 29, 7339.—This is largely a discussion of the differences in the results of previous work by F. and those of Wessely (C A 29, 6242); these are probably due to the fact that W used an impure product. In an expt. on the action of P-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>Me upon calumbin in 5% EtOH-KOH, there resulted a comp'd termed carbonylo-V-calumbin, C<sub>21</sub>H<sub>12</sub>O<sub>6</sub>, m. 191–5° (decompn.); loss of CO<sub>2</sub> gives iso-V-calumbin. C. J. West.

Sulfur compounds of terpenes. VI. Action of gold chloride on C<sub>10</sub>H<sub>16</sub>S (=C-S-C≡ type), Akita Nakatsuchi. J. Soc. Chem. Ind., Japan 38, Suppl. binding 511–12 (1935); cf. C A 27, 3927.—Upon mixing C<sub>10</sub>H<sub>16</sub>S (I) and AuCl<sub>3</sub> in a mol. ratio of 4:1, at 30°, C<sub>10</sub>H<sub>16</sub>S.AuCl<sub>3</sub> (II) was obtained as yellowish crystals, and with liberation of HCl the crystals changed to a colorless liquid. In 45 days, 60% Cl was liberated and C<sub>10</sub>H<sub>16</sub>S.AuCl<sub>3</sub> (III) was formed. The reaction velocity between I and II was measured, and it was found that the reaction may be considered pseudo monomol., if a large excess of I is present. AmOH (IV), PhNO<sub>2</sub> (V), limonene (VI) and I, resp., were added to II dissolved in CHCl<sub>3</sub>, and upon standing at 50° for 44.75 hrs. the following amts. of metallic Au were obtained: IV 2.60%, V 1.80%, VI 18.42% and I 0%. VII. Action of gold chloride on C<sub>10</sub>H<sub>16</sub>S (=C-S-C≡ type) at higher temperatures. Ibid 512–13.—Data are reported on the stability of II and III in presence of I at 100–20°. A large excess of I inhibited the reduction to metallic Au. III was stable at 100° with excess of I, but decomposed rapidly at 120°. III in I was heated at 109–111° for 6 hrs. in presence of colophonium, ester gum and ethylcellulose, resp., and an increase in reduced Au was noted. Ten mols. of I and 1 mol. of AuCl<sub>3</sub> at 110° gave a grayish, oily liquid, which yielded an olive greenish ppt. after the volatile liquid was dist'd off and the residue had been treated with alc. This comp'd contained 71.35% Au and 8.11% S, was sparingly sol. in Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, AmOH, but sol. in CHCl<sub>3</sub> and CS<sub>2</sub>, and decomp'd at 166°. K. K.

Synthesis of *dl*-piperitone (*dl*- $\Delta^1$ -*p*-menthen-3-one). James Walker. J. Chem. Soc. 1935, 1585–6—C<sub>10</sub>H<sub>16</sub>CH<sub>2</sub>Ac (5 g.) and 8.1 g. of AcCH(isoPr)CO<sub>2</sub>Et with EtONa in EtOH, allowed to stand 20 hrs. at room temp. and then refluxed 7 hrs., give 3.6 g. of *dl*- $\Delta^1$ -*p*-menthen-3-one-4-carboxylate, b<sub>p</sub> 155–60°, n<sub>D</sub><sup>20</sup> 1.425; this is unchanged after refluxing with 20% HCl for 20 hrs., but with 3 mols. 20% MeOH-KOH in 12 hrs. 74% (26% overall) of *dl*-piperitone results. This is the 1st complete synthesis of this comp'd. C. J. West.



on contact with Na a considerable e m f, though with the compds. possessing no mobile H this e m f. is very small. In the presence of NaI the e m f. of the compds. incapable of changing to the enol (or analogous) form either increases many times or remains nearly unchanged. To the first group belong ketones ( $\text{Ph}_2\text{CO}$ ), nitriles ( $\text{PhCN}$ ) and heterocyclic derivs of the pyridine group. To the 2nd group belong some compd. esters (e g, nitrates). The intermediate position is occupied by  $\text{PhNO}_2$  and iso- $\text{AmNO}_2$ . Of interest is the small increase of the e m f. shown by  $\text{BzOEt}$ , this indicates a poorer reactivity of the ester C O than the ketone C O (cf. Trosov, *et al.*, *C A* 22, 2535). The e m f. of compds with a OH group on the addn. of NaI is either not affected (acids and some alcs, such as allyl alc and cyclohexanol) or is changed considerably, but less than, e g., with the compds. incapable of enolization of ketones. This may be explained by the decreased tendency to complex formation with NaI in the absence of a double or triple bond. The double bond of acids is, evidently, little active. The effect of the C double bond in a mol of alc is little pronounced. Pyrrrole gave no increase of activity. Compds with C O, C N and  $\text{NO}_2$  groups and H atoms in the vicinity of the unsatd group showed either a considerably increased e m f. (cyclohexanone, methyl oxide) or very little or none at all ( $\text{MeCN}$ ,  $\text{MeNO}_2$ ,  $\text{AcH}$ ). This may be conditioned by a different degree of enolization and by the different reactivities of the possible tautomeric forms. The difference in the effect of NaI on  $\text{AcH}$  and  $\text{BzH}$  is very great, because  $\text{AcH}$  can exist in 2 hydric forms  $\text{CH}_2$ ,  $\text{CHOH}$  and  $\text{MeCOH}$  (bivalent C), while  $\text{BzH}$  cannot change to the 1st form and does not evidently exist in the 2nd form, which follows from the absence of paraaldehyde polymerization.  $\text{MeNO}_2$  behaved in the same not as a nitro compd. but as an acid, viz.,  $\text{CH}_2$  -  $\text{N}(\text{O})\text{H}$ . The results clearly show that the reactivity of some unsatd groups, particularly C O and C N, to Na increases greatly on the addn. of NaI. In this the compds. contg. these groups differ sharply from the compds. with an active H. It is also shown that the activity of C O group depends greatly on its position in the mol. It is especially active in ketones and aldehydes but not in compd. esters and acids. The  $\text{NO}_2$  group in nitro compds. behaves differently from that in nitric esters. It suggests itself that this method can find application for the identification of various unsatd groups in org. compds. and even for the detn. of their positions in the mol.

Chas. Blanc

Hydrogen reactivity in the complex compounds of alcohols with unsaturated organic compounds. B. V. Trosov and L. P. Kulev *J. Gen. Chem. (U. S. S. R.)* 5, 1233-9 (1935); cf. *C A* 29, 2054<sup>1</sup> and preceding abstr.—The influence of mol. structure, such as the chain length, chain branching, double bond and  $\text{C}_6\text{H}_5$  ring, on the H reactivity in alcs. detd. by the e m f. of the reaction with Na, was studied by the complex formation  $\text{PhOH}$ , iso- $\text{PhOH}$ ,  $\text{BuOH}$ ,  $\text{MeCOH}$ , cyclohexanol,  $\text{MeC}(\text{OH})\text{Et}$  and  $\text{PhCH}_2\text{CH}_2\text{OH}$  were mixed in various proportions with  $\text{Et}_3\text{N}$ ,  $\text{C}_6\text{H}_5\text{N}$  and  $\text{PhNO}_2$  and made up to 6 cc. with  $\text{C}_6\text{H}_6$ . Conclusions.—The primary satd alcs. either give no complex compds., or the latter have but little effect on the e m f. of the reaction of these alcs. with Na. The secondary satd. alcs. show no max. above the e m f. observed for a given alc. Tertiary alcs. showed insignificant e m f. in the reaction with Na with and without the addn. of  $\text{Et}_3\text{N}$ . With  $\text{C}_6\text{H}_5\text{N}$  and  $\text{PhNO}_2$  they formed complexes highly active to Na, increasing the e m f. more than 10 times ( $\text{MeCOH}$ ). Of the secondary alcs. cyclohexanol and its homologs are exceptions, acting as tertiary alcs. by increasing considerably the e m f. on the addn. of the unsatd. compds. (*C A* 25, 894).  $\text{PhCH}_2\text{CH}_2\text{OH}$  behaves somewhat similarly to phenols, increasing slightly the e m f. on the addn. of  $\text{C}_6\text{H}_5\text{N}$  and  $\text{PhNO}_2$ . The satd. alcs. with  $\text{PhNH}_2$  and  $\text{C}_6\text{H}_5(\text{NH}_2)$  showed no increase of the e m f., while aromatic alcs. gave a small max.

Chas. Blanc

Oxidation of diphenylpyruvic acid. Jules Jarrousse (*compt. rend.* 201, 676-7 (1935))— $\text{PhCH}(\text{C}(\text{OH})(\text{CO}_2\text{H}))$ -

$\text{CHPhCOCO}_2\text{H}$  (I), treated in the cold with alk.  $\text{KMnO}_4$ , gives an unstable acid (II), extd. with  $\text{Et}_2\text{O}$  from the acidified reaction mixt. and  $\text{PhCH}(\text{COCHPhCHO})$  (III), m. 114° (semicarbazone, m. about 200° (decompn.)). The formula  $\text{PhCH}(\text{CHPhCOCO}_2\text{H})$  has been assigned to II as a result of the oxidation of the tert. alc. group of I. II easily loses  $\text{CO}_2$  in alk. soln. to give III. III gives reactions characteristic of aldehydes. On oxidation with  $\text{I}_2$  a small amt. of an unstable acid with higher mol. wt. than III is obtained.

Rachel Brown

Derivatives of benzoylbenzoic acids. I. 3-Methyl-2-(2'-hydroxybenzoyl)benzoic acid, 3-methyl-2-(4'-hydroxybenzoyl)benzoic acid and 3-methyl-2-(4'-chlorobenzoyl)benzoic acid. Mosuke Hayashi and Shinzo Tsuruoka *J. Chem. Soc. Japan* 56, 1031-4 (1935).—Condensation of 3-methylphthalic anhydride (I) and  $\text{PhOH}$  gave 2-hydroxybenzoylbenzoic acids, m. 220-1° (II) and m. 197-8° (III). On treating them with concd.  $\text{H}_2\text{SO}_4$  and letting stand II gives an isomer (IV), m. 141-2°, and III gives an isomer (V), m. 183-4°. Judging from the fact that treating 3,2-MeBzC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H with concd.  $\text{H}_2\text{SO}_4$  changes it to its 6-Me isomer, II is 3-methyl-2-(2'-hydroxybenzoyl)benzoic acid, IV is the 6-Me isomer of II, III is 3-methyl-2-(4'-hydroxybenzoyl)benzoic acid and V is the 6-Me isomer of III. The product from the condensation of I and  $\text{PhCl}$  is probably 3-methyl-2-(4'-chlorobenzoyl)benzoic acid, m. 173-4°. II 3-Methyl-2-benzoylbenzoic acid and 6-methyl-2-benzoylbenzoic acid *Ibid.* 1084-92.—That 6- (I) and 3-methyl-2-benzoylbenzoic acids (II) are isomers due to the change of positions of the CO-H and Bz groups and not stereoisomers is proved by the following method. The condensation product from 3-methylphthalic acid and  $\text{C}_6\text{H}_5$  is treated with aq.  $\text{Na}_2\text{CO}_3$ , the insol. portion gives 1-methyl-2,3-dibenzoylbenzene, m. 116-17° (and not diphenylmethylphthalide), and the sol. portion gives benzoyltoluic acids,  $\text{C}_6\text{H}_5\text{CO}_2\text{H}$ , from which I, m. 126.5-7.5°, and II, m. 171-2°, are sep'd. Heating of II in concd.  $\text{H}_2\text{SO}_4$  at room temp. gives no change but when heated at 100-5° it gives an isomer I. Oxidation of I and II with  $\text{KMnO}_4$  gives diphenyl ketone-2,6-dicarboxylic acid, m. 225-6°, and the 2,3-isomer, m. 121-5°, resp. III 3(6p)-Nitro-2-benzoylbenzoic acid, 3(6p)-nitro-2-(2'(4'p)-hydroxybenzoyl)benzoic acid, 3(6p)-nitro-2-(2'(5'p)-dimethylbenzoyl)benzoic acid and 5(4'p)-nitro-2-(2'(5'p)-dimethylbenzoyl)benzoic acid. Mosuke Hayashi, Shinzo Tsuruoka and Akio Nakayama *Ibid.* 1093-1101.—Condensation of 3-O-N $\text{C}_6\text{H}_4(\text{CO})_2\text{O}$  (I) and  $\text{C}_6\text{H}_5$  by the Friedel-Crafts method gave 2,3(6'p)-Bz(O $\text{N}$ ) $\text{C}_6\text{H}_4\text{CO}_2\text{H}$  (II), m. 236-7°; the m. p. of II was not changed by heating in 10 vols. concd.  $\text{H}_2\text{SO}_4$  at room temp. for 17 hrs. or at 100-5° for 45 min. 2,3-AcO(NO $\text{C}_6\text{H}_4$ ) $\text{C}_6\text{H}_5\text{CO}_2\text{H}$  in a similar manner gave 2,6(3'p)-Bz(O $\text{N}$ ) $\text{C}_6\text{H}_4\text{CO}_2\text{H}$ , m. 160-1°. 4-NO $\text{C}_6\text{H}_4(\text{CO})_2\text{O}$  and *p*-xylene gave 4(5'p)-nitro-2-(2'(5'p)-dimethylbenzoyl)benzoic acid, m. 191.5-2.5°. The other compds. in the title are not obtained in pure state. K. Kutsuta

Substitution in polycyclic systems. I. Nitration of fluorene and 9-bromofluorene. S. V. Anantakrishnan and E. D. Hughes. *J. Chem. Soc.* 1935, 1607-9.—Dinitration of fluorene confirms the results of Courtot (*C A* 25, 508). Mild nitration of 9-bromofluorene gives the 2-NO $\text{C}_6\text{H}_4$  deriv. (I), m. 145°; oxidation gives 2-nitrofluorenone. More vigorous action of  $\text{HNO}_3$  in  $\text{Ac}_2\text{O}$  gives a mixt. of 60% of the 2,7-di-NO $\text{C}_6\text{H}_4$  deriv. (II), m. 255-60° (decompn.), and 20% of the 2,5-di-NO $\text{C}_6\text{H}_4$  deriv. (III), m. 199°, sep'd. by  $\text{Me}_2\text{CO}$ . Nitration in  $\text{AcOH}$  is complicated by oxidation and nuclear bromination and gives II and a ketone,  $\text{C}_{14}\text{H}_9\text{O}_2\text{NBr}$ , m. 230°, which may be 7-bromo-2-nitrofluorenone (Schmidt and Bauer, *Ber.* 38, 3755 (1905)). Nitration of I gives a mixt. of II and III.

C. J. West

Influence of poles and polar linkings on the course pursued by elimination reactions. XXIII. Stable derivatives of the trivalent-carbon compound of Ingold and Jessop. E. D. Hughes and K. I. Kurryan. *J. Chem. Soc.* 1935, 1609-11; cf. *C A* 29, 5073<sup>1</sup>—The compd. ( $\text{C}_6\text{H}_5$ ) $\text{CSMe}_2$ , isolated by Ingold and Jessop (*C A* 24, 588), proved to be unstable; the NO $\text{C}_6\text{H}_4$  derivs

have now been studied. 9-Bromo-2-nitrofluorene (I) and  $\text{Me}_2\text{S}$  in  $\text{PhNO}_2$  give 2-nitrofluorenyl-9-dimethylsulfonium bromide (II), m 135° with decompn to I and  $\text{Me}_2\text{S}$ , which also results on crystn from dil  $\text{EtOH}$ . The picrate in 185° (decompn). I with alkali in  $\text{H}_2\text{O}$ ,  $\text{EtOH}$  or  $\text{Me}_2\text{SO}$  gives dimethylsulfonium 9-(2-nitrofluorenyl)ide (III), dark purple needles, slowly decompn on standing and more rapidly on heating, it does not m 300°, on heating in  $\text{MeNO}_2$ , 2,2'-dimitrobis(phenylethylene) results. Fluorenyl-9-dimethylsulfonium picrate and  $\text{HNO}_3$  at 0° give the 2,7-di- $\text{NO}_2$  deriv, yellow, m 180° (decompn).  $\text{HBr}$  in  $\text{AcOH}$  gives 9-bromo-2,7-dinitrofluorene (IV), bromide, from V and  $\text{HBr}$ , m 230° (decompn.) to IV and  $\text{Me}_2\text{S}$ , it also decomposes on crystn from  $\text{EtOH}$ . Dimethylsulfonium 9-(2,7-dinitrofluorenyl)ide (V),  $\text{KMnO}_4$  like crystals, changing to brick-red on heating and finally decompn with a flash of light, at 150° it evolves  $\text{Me}_2\text{S}$ , boiling  $\text{MeNO}_2$  gives 2,2',7,7'-tetranitrobis(phenylethylene), m above 300°. 9-Bromo-fluorene and  $\text{Me}_2\text{S}$  give fluorenyl-9-dimethylsulfonium bromide, m 134-5° (quant yield), picrate, m 143°, heating with  $\text{H}_2\text{O}$  for 30 min gives  $\text{Me}_2\text{S}$  and 9-fluorenyl alc. Alkali gives a very unstable black ppt, which yields  $\text{Me}_2\text{S}$  at room temp.

**C J West**  
Action of bromine on 2- and 4-nitro-1-naphthylamines. Raphael Conden and Joseph Kenyon. *J. Chem. Soc.* 1935, 1596-7. —4,1- $\text{C}_{10}\text{H}_7(\text{NO}_2)\text{NH}_2$  (I) and  $\text{Br}$  in  $\text{CHCl}_3$  gives 2,4-dibromo-1-naphthalenediazoperbromide (II), which decomposes at 128-36° to give 1,2,4- $\text{C}_{10}\text{H}_4\text{Br}_3$  (III), heating with  $\text{C}_6\text{H}_5\text{N}$  also gives III. In  $\text{AcOH}$  I and  $\text{Br}$  give principally 2-bromo-1-nitronaphthylamine (IV) and some II, IV reacts with  $\text{Br}$  in  $\text{CHCl}_3$  or  $\text{AcOH}$  to give II.  $\text{Ac}$  deriv of IV, yellow, m 235-6°. 2,1- $\text{C}_{10}\text{H}_7(\text{NO}_2)\text{NH}_2$  and  $\text{Br}$  give the 4  $\text{Br}$  deriv., reacting with excess  $\text{Br}$  to give II, decompd by boiling  $\text{AcOH}$  to III.

**C J West**  
The preparation of Neville-Winter acid from  $\alpha$ -chloronaphthalene. N N Voroshilov, Jr, and P V Karish. *Russ. gen. mat. color* 39, 373-8(1935). —See C A 29, 2530.

**Halsey E Sillman**  
Biaryls and their derivatives. IV. Oxidation of 2-naphthol-6-sulfonic acid and 2,6-dihydroxynaphthalene. I S Ioffe and S G Kuznetsov. *J. Gen. Chem.* (U S S R) 5, 877-85(1935), cf C A 28, 2709. —The oxidation of 2,6- $\text{HO}_2\text{C}_6\text{H}_3\text{SO}_3\text{H}$  (I) by  $\text{FeCl}_3$  in  $\text{H}_2\text{O}$  at elevated temps gave 2,6- $\text{HO}_2\text{C}_6\text{H}_3(\text{SO}_3\text{H})\text{Cl}$  and no 2,2'-dihydroxy-1,1'-binaphthyl-6,6'-disulfonic acid (II) as was expected (cf C A 28, 1691). At 20-5° the reaction proceeded normally, giving 90% II after 20 days of interaction. II, heated with 25%  $\text{H}_2\text{SO}_4$  at 170-80° for 12 hrs, gave 2,2'-dihydroxy-1,1'-binaphthyl, m 216°. II, fused with  $\text{KOH}$  at 330° for 15 min, produced 47.7% of 2,6,2',6'-tetrahydroxy-1,1'-binaphthyl (III), m 318-20° (uncor.) (decompn). 2,6- $\text{C}_{10}\text{H}_6(\text{OH})_2$  (IV) gave with 1 mol of  $\text{FeCl}_3$  III and with 1.5 mols of  $\text{FeCl}_3$  the tetramer consisting of 4 IV groups with mol wt 650 (cf *Sals*, *Ber* 39, 300b(1906), Willstätter and Parnas, *Ber* 40, 1406 (1907)). II is highly sol and cannot be sepd with  $\text{NaCl}$ . The sepn was effected by making the reaction mixt. slightly alk. with  $\text{Ba}(\text{OH})_2$ , neutralizing the filtrate with  $\text{AcOH}$ , evapn the soln to a small vol and filtering from  $\text{BaCl}_2$ . The filtrate was treated with  $\text{Ba}(\text{OAc})_2$  and the I in the soln was removed by coupling it with the theoretical amt of  $\beta$ - $\text{O}_2\text{NC}_6\text{H}_4\text{N}_2\text{Cl}$ . The filtrate was treated with the exact amt of  $\text{H}_2\text{SO}_4$  and the filtrate from  $\text{BaSO}_4$  was repeatedly evapd to dryness, giving II 73% (dil alc.). V. Optical activity of 2,2'-dihydroxy-1,1'-binaphthylsulfonic acids. I S Ioffe and I V Grachev. *Ibid* 560-5. —The dibrucine salts of the 6,6'- and 7,7'-disulfonic acids of 2,2'-dihydroxy-1,1'-binaphthyl were sepd by fractional crystn. into the diastereomeric dibrucine salts of 1- and 4-acids. The latter by the action of alkali sepd the optical antipodes, 1- and 4-acid. These show optical rotation only in the form of the Na salts in alk. soln, and none in a free state or in acid soln. The antipodes of the 7,7'-disulfonic acid are more stable than the 6,6'-acid. The racemization of the 2 acids proceeds more rapidly in an alk. soln than in an acid one.

**VI. Oxidation of 2-naphthol-3-carboxylic acid** I. S Ioffe and I Z Smolnitskaya. *Ibid* 1215-9. —The oxidation of 2,3- $\text{HO}_2\text{C}_6\text{H}_3\text{CO}_2\text{H}$  (I) with 3 mols of  $\text{FeCl}_3$  proceeded with the formation of 2,2'-dihydroxy-1,1'-binaphthyl-3,3'-dicarboxylic acid (II), m 331°, and 1,2,3- $\text{Cl}(\text{HO})_2\text{C}_6\text{H}_3\text{CO}_2\text{H}$  (III), m 231°, at a ratio depending on the conditions of the reaction. In aq medium the Na salt of I gave 90% II and 6% III, while free I under these conditions produced 60% II and 25% III. Similar results were obtained by working in dil alc. and  $\text{AcOH}$ . With a large excess of  $\text{HCl}$  no II and 60% III were formed. I (5 g), heated with 2.5 g of dry  $\text{CuO}$  in 10 cc.  $\text{PhNO}_2$  at 210-20° for 6 hrs, gave 2.5 g II. II is not affected by boiling with  $\text{FeCl}_3$  or by  $\text{CuO}$  in  $\text{PhNO}_2$  at 200-40°. At 300° the latter procedure results in the cleavage of  $\text{CO}_2$  and formation of dihydroxybinaphthyl, m 229°. Cyclization of II with the formation of a perylene ring was effected by heating a mixt. of 10 g of Pb salt of II with 40 g of anhyd  $\text{AlCl}_3$  at 150° for 1 hr. The product, m above 330°, is a dye for animal fibers, giving in acid bath an orange dyestuff changing to a beautiful brown-chocolate after mordanting with  $\text{K}_2\text{Cr}_2\text{O}_7$ . The presence of a perylene ring was demonstrated by the formation of perylene on distn with  $\text{Zn}$  dust. The product is, probably, 1,12-dihydroxyperylene-2-carboxylic acid or 1,12-perylenequinone-2-carboxylic acid. VII. Oxidation of 2-hydroxyanthracene. I S Ioffe. *Ibid* 1210-12. —The oxidation of 2-hydroxyanthracene with  $\text{FeCl}_3$  in alc. or  $\text{AcOH}$  resulted in the formation of 2-hydroxy-1,1',9,9'-bianthrylene oxide (I) and a brown compd of unknown structure. I is unstable, changing at 200° to the brown compd, m above 300°. I gives with  $\text{HCl}$  the oxonium salt and with  $\text{AcO}$  in dry pyridine the  $\text{Ac}$  deriv, m 247-50° (uncor.).

**Chas Blanc**  
Halochromism of 5-benzoyl-1,4-naphthohydroquinone. Roland Schell, Joachim Donat, Sigrud Hass and Alfred Keller. *Ber.* 68B, 2034-9(1935). —1-Benzoylanthraquinone is converted by  $\text{Al}$  powder in concd  $\text{H}_2\text{SO}_4$ , the anthraquinone by concd  $\text{H}_2\text{SO}_4$  (or other strong acids) alone, through the oxanthrone into the emerald-green sulfate of the violet 2-phenyl-6,7-benzoylene- $\beta$ - $\beta'$ -benzofuran (C A 27, 3210). It was of interest to determine whether the  $\text{C}_{10}\text{H}_6$  analogs, 5-benzoyl-1,4-naphthohydroquinone (I) and the hydroquinone (II), would likewise give the halochromic sulfate of a deeply colored 2-phenyl-6,7-acrylene- $\beta$ - $\beta'$ -benzofuran. The brownish yellow soln of I in concd.  $\text{H}_2\text{SO}_4$  on heating with  $\text{Al}$  powder rapidly becomes a vivid ruby-red (violet in thin layers), with bright blue fluorescence. A similar soln results immediately when II is introduced into concd.  $\text{H}_2\text{SO}_4$  and the same results are obtained with hot 70%  $\text{HClO}_4$  instead of  $\text{H}_2\text{SO}_4$ . If in acetone is treated with 70%  $\text{H}_2\text{SO}_4$  or  $\text{HClO}_4$  or with  $\text{HCl}$  gas there soon sep. dark violet (red by transmitted light) crystals with strong golden luster which form in concd.  $\text{H}_2\text{SO}_4$  and hot  $\text{HClO}_4$  solns of the same ruby-red color and blue fluorescence as those obtained directly from I and II. The solid salts dissolve in org. solvents more or less readily with blue color, but the solns, especially in alc., soon become discolored, the solns in acetone, alc. and concd.  $\text{H}_2\text{SO}_4$  are immediately decolorized by water with formation of a yellow ppt. From this behavior it must be assumed that the solid salts are not halochromic salts of an acrylenebenzofuran but of II. The sulfate and chloride are not suitable for setting this point by detg. the amt of II set free by water, for they contain more  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$  than corresponds to a mol ratio of 1 and less than 1.2. A perchlorate of the compn  $\text{C}_{19}\text{H}_{10}\text{O}_4\text{ClO}_4$  can readily be obtained, however, and when it is heated in acetone contg.  $\text{NaOAc}$  and treated with hot water it yields 80% of the calcd amt. of pure II, thus proving that it is a halochromic salt of II. In addn to the open structure,  $(\text{HO})_2\text{C}_{10}\text{H}_6(\text{OPh})_2$ , II can have the lactol structure, and certain phenomena in the anthracene series speak in favor of this structure for II itself and for its halochromic salts. 6-Methyl-5-benzoyl-1,4-naphthohydroquinone, m 148°, from 1,2- $\text{C}_{10}\text{H}_6\text{Br}_2\text{Me}$  and  $\text{CrO}_3$  in  $\text{AcOH}$  (18% yield), 2,6-Di- $\beta$ -homolog, m 109°, obtained in 32% yield from 2,6-di-

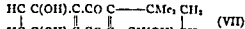
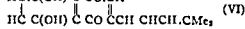
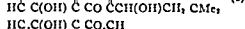
methyl-1-benzoylnaphthalene, b.p. 218–60°, m. 83–4° (prepd. from  $\text{C}_{10}\text{H}_7\text{Me}$ ,  $\text{BzCl}$  and  $\text{AlCl}_3$ ). Bi-2-(1,4-dihydroxynaphthyl), from the biquinonyl in  $\text{AcOH}$  at 35° with 7n dust, colorless needles, begins to decompose 100°, blackens 250°, sensitive to the air and soon assuming a bluish tinge even when well dried; solns. in org. solvents become violet when heated in the air and on cooling deposit black-violet needles of the quinoxaline, which begins to decompose about 200° C. A. R.

Constituents of natural phenolic resins III Synthesis of dehydro-“sulfite liquor lactone” dimethyl ether and some observations on the structure of podophyllotoxin Robert D. Haworth, Thomas Richardson and Geo. Sheldrick *J. Chem. Soc.* 1935, 1576–81, cf. C. A. 29, 5100°. The proposed structure for dehydro-“sulfite liquor lactone” (I) has been confirmed by its synthesis. Li  $\beta$ -(3,4-dimethoxybenzoyl)propionate m. 57–8°,  $\text{HCO}_2\text{Et}$  with Na in  $\text{C}_6\text{H}_6$  give the  $\beta$ -hydroxymethylene deriv. m. 114–16° (red color with  $\text{FeCl}_3$ ) and  $(\text{CO}_2\text{Et})_2$  gives Li  $\beta$ -(3,4-dimethoxybenzoyl)  $\Delta^2$ -crotonolactone- $\gamma$ -carboxylate, yellow, m. 151–4°, recognizable reduction products of these esters could not be prepd. 3,1-(MeO) $_2$ - $\text{C}_6\text{H}_3\text{CH}_2\text{C}(\text{CO}_2\text{H})\text{CH}_2\text{COC}_6\text{H}_3(\text{OMe})_2$ -3,4(3,4)-formalin and 10% NaOH give 2,7 of  $\beta$ -(3,4-dimethoxybenzoyl)- $\alpha$ -(3',4'-dimethoxybenzylidene)- $\beta$ -methylpropionic acid, m. 157–8°, MeOH acid with  $\text{HCl}$  gives a mixt. of Me 6,7-dimethoxy-1-(3',4'-dimethoxybenzylidene)-2-chloromethylnaphthalene-3-carboxylic acid, m. 171–7° (free acid (II), m. 241–5° and then 291–5°), and the lactone of  $\beta$ -(3,4-dimethoxybenzoyl)- $\alpha$ -(3',4'-dimethoxybenzylidene)- $\beta$ -chloromethylpropionic acid (III), yellow, m. 183–4°; boiling MeOH gives the  $\beta$ -methoxymethyl analog, yellow, m. 145°; II, heated in camphor at 200–10° for 10 min, yields 4',5',6,7-tetramethoxybenzo-3,4-fluorene-1-carboxylic acid, cream, m. 303–5° (Me ester, m. 202–4°). Heating II with 10% NaOH for 1 hr. at 100°, hydrolysis of the Me ester of II with 10% MeOH-KOH, or boiling III with MeOH-HCl in  $\text{CHCl}_3$  for 6 hrs., followed by hydrolysis with 10% MeOH-KOH, acidification of each of the reaction products and lactonization by heating at 100° for 0.5 hr., gives I (lactone of 6,7-dimethoxy-1-(3',4'-dimethoxybenzylidene)-2-hydroxymethylnaphthalene-3-carboxylic acid), m. 215–16°; the overall yield is practically quant. The application of the above synthesis in the prepn. of substances related to podophyllotoxin has also been investigated. 3,4,5-(MeO) $_3$ - $\text{C}_6\text{H}_2\text{COC}_6\text{H}_3(\text{CO}_2\text{Et})_2$  (83 g.),  $\text{BrCH}_2\text{CO}_2\text{Et}$  and  $\text{Fe}(\text{OAc})_3$ , refluxed in EtOH for 16 hrs., give, after hydrolysis, 4,2 g. of  $\beta$ -(3,4,5-trimethoxybenzoyl)propionic acid, m. 121–2°; the Na salt with piperonal and  $\text{Ac}_2\text{O}$  gives the  $\gamma$ -lactone, yellow, m. 101–2°, of  $\beta$ -(3,4,5-trimethoxybenzoyl)- $\alpha$ -(3',4',5'-trimethoxybenzylidene)propionic acid, m. 183–4°;  $\text{HClHO}$  gives the  $\beta$ -methylene deriv. m. 169–70°. 6,7-Methylenedioxy-1-(3',4',5'-trimethoxybenzylidene)-2-chloromethylnaphthalene-3-carboxylic acid, does not m. 300°; the lactone of the 2-hydroxymethyl analog, m. 258–6°, on oxidation yields 6,7-methylenedioxy-1-(3',4',5'-trimethoxybenzylidene)naphthalene-2,3-dicarboxylic acid, characterized as the anhydride, pale yellow, m. 293–300°, and the di-Me ester, m. 206–7°, dehydroanhydripropodipodophyllin gives the same acid. C. J. West.

By-products formed in the Friedel-Crafts synthesis of ketones from acid chlorides and phenolic esters Synthesis of ketones of naphthalene series. P. I. Popov *J. Gen. Chem.* (U. S. S. R.) 5, 996–92 (1935).—In the synthesis of ketones from phenolic esters and acid chlorides with  $\text{AlCl}_3$  in  $\text{CS}_2$ , the ether ext. of ketones, obtained by the decolor. of the reaction product with  $\text{H}_2\text{O}_2$ , was washed with dil. NaOH and the alk. ext. acidified, producing always a crystallizable oil. The amt. of this by-product is greater the longer the reaction. It was postulated that these alkali-sol. products are  $\beta$ -HO ketones formed by sapon of the alkyl ketones by the excess of  $\text{AlCl}_3$ .  $\text{AcC}_6\text{H}_4\text{OMe} + \text{AcC}_6\text{H}_4\text{OH}$ . In the synthesis of 1,4-MeOC $_6\text{H}_3$ Ac there was isolated 1,2,4-HOC $_6\text{H}_3$ Ac, m. 179–40° and no 1,4-HOC $_6\text{H}_3$ Ac. In a similar expt. with the production of 1,4-LiOC $_6\text{H}_3$ Bz from  $\text{BzCl}$  and  $\alpha$ -C $_6\text{H}_4\text{OH}$  with  $\text{AlCl}_3$ , the alk. extra. produced 1,4-

HOC $_6\text{H}_3$ Bz, m. 164–5°, and 1,2,4-HOC $_6\text{H}_3$ Bz (I), m. 138–9°. The formation of I may be best explained by the partial sapon of the ether to  $\alpha$ -C $_6\text{H}_4\text{OH}$  by  $\text{AlCl}_3$ , which with  $\text{BzCl}$  forms  $\text{C}_6\text{H}_4\text{OBz}$ . This under the action of  $\text{AlCl}_3$  is rearranged partly to 1,4-HOC $_6\text{H}_3$ Bz (Rosenmund and Schurr, C. A. 22, 1570) and 1,2-HOC $_6\text{H}_3$ Bz. The latter in the presence of  $\text{BzCl}$  and  $\text{AlCl}_3$  is converted to I. The ability of  $\alpha$ -naphthol 2 ketones to give naphthol 2,4-diketones was demonstrated by the condensation of 1,2-HOC $_6\text{H}_3$ Ac with  $\text{BzCl}$  and  $\text{AlCl}_3$  in  $\text{CS}_2$  to 1,2,4-HOC $_6\text{H}_3$ AcBz, m. 131–2°. Chas. Blanc.

Constitution of alkannin, shikonin and alkannan. Hans Brockmann. *Ann.* 521, 1–47 (1935).—Alkannin (3-(2-methyl-5-hydroxypenten-5-yl)-5,8-dihydroxy-1,4-naphthoquinone),  $\text{C}_{18}\text{H}_{16}\text{O}_4$  (I), from *Alkanna tinctoria*, brown-red with Cu ester, m. 118° (all m. ps. cor.),  $[\alpha]_D^{25} -157^\circ$  ( $\text{C}_6\text{H}_6$ ),  $-227^\circ$  ( $\text{CHCl}_3$ ), contains 3 active H atoms, I sublimes unchanged at 140–50° at 0.001 mm Hg, the rotatory power is independent of the concn of the soln. “Alkannin” Merck (20 g.) gives 2.5 g. pur. I. I contains about 0.1% of alkannan (3,4-hexylnaphthazarin) (II), m. 97–8°, optically inactive, sep'd by adsorption on kieselguhr. Shikonin (III) (Majumdar and Kuroda, *Acta physiochim.* 1, 43 (1922)) is the optical antipode of I and has  $[\alpha]_D^{25} 135^\circ$  ( $\text{C}_6\text{H}_6$ ); the absorption spectra of I and II are identical, O $_2$  gives 1 mol.  $\text{Me}_2\text{CO}$ . Dry distn. of I gives 1-methylquinizarin, which was synthesized by passing air through an alk. soln. of the condensation product of diacetylnaphthazarin and piperonylene (heated 1 hr. at 100°). Zn distn. of I gives  $\text{C}_6\text{H}_4$  and  $\alpha$ - and  $\beta$ -methylanthracene. I yields a tri-Ac deriv. (IV), yellow, m. 132°,  $[\alpha]_D^{25} -110^\circ$  ( $\text{C}_6\text{H}_6$ ); di-Bz deriv., yellow, m. 174–5°. IV with O $_2$  gives 1,4-dihydroxyphthalic acid, I gives  $\text{Me}_2\text{CO}$ ,  $\text{KMnO}_4$  gives only AcOH and malic acid, I and MeOH-HCl at 20° for 15–18 hrs. give the Me ether, brown-red, m. 195°, contains 2 active H atoms and is optically inactive. The Me ether (V) of III also m. 105° and does not depress the m. p. of the deriv. of I. The Li ether of I, brown-red, m. 83°, the Pr ether, m. 57–8°. A mixt. of 50 mg. of III and 40.5 mg. of I in  $\text{C}_6\text{H}_6$  + benzene give a racemic I, m. 148°. Catalytic reduction of I with 1 mol.  $\text{H}_2$  gives only I; with 3 mols.  $\text{H}_2$  there results II, whose di-Ac deriv., yellow, m. 99–101°; reductive acetylation of II yields a tetra-Ac deriv.,  $\text{C}_{18}\text{H}_{16}\text{O}_4$ , m. 170°. Oxidative degradation of II gives is- $\alpha$ - $\text{AmC}_6\text{H}_4\text{CO}_2\text{H}$ , whose  $\beta$ -bromophenacyl ester, m. 75°; the corresponding ester of isocaproic acid, m. 75°. The dihydro deriv. of V, red, m. 50–1°; oxidation with  $\text{KMnO}_4$  in Me $_2\text{CO}$  gives  $\alpha$ -methoxyisocaproic acid, b.p. 123–4°, prepd. from the  $\alpha$ -Br deriv. (b.p. 84–90°) and MeONa; oxidation gives isocaproaldehyde, whose 2,4-dinitrophenylhydrazone, yellow, m. 95°. I and 2 N NaOH, heated 2 hrs. at 100° in a N atm, gives anhydroalkannin,  $\text{C}_{18}\text{H}_{14}\text{O}_4$  (VI), dark red, m. 155°.  $\text{PrOH-HCl}$  gives 15% yield; catalytic reduction gives II. I and  $\text{ZnCl}_2$  in  $\text{C}_6\text{H}_6$  give cycloalkannin (VII),  $\text{C}_{18}\text{H}_{18}\text{O}_4$ , red, m. 79–80°,  $[\alpha]_D^{25} -69.2^\circ$  ( $\text{C}_6\text{H}_6$ ); the cyclo deriv. of III, m. 70–80°,  $[\alpha]_D^{25} 51.2^\circ$  ( $\text{C}_6\text{H}_6$ ). The racemate m. 86°.



C. J. West  
Substitution in arylsulfon-1- and -2-naphthalides. Raphael Conden and Joseph Kenyon. *J. Chem. Soc.* 1935, 1091–6.  $\alpha$ -C $_6\text{H}_4\text{NH}_2$  and  $\text{m-O}_2\text{NC}_6\text{H}_4\text{SO}_2\text{Cl}$  in  $\text{C}_6\text{H}_5\text{Mg}$  give  $\text{m-nitrobenzenesulfon-1-naphthalide}$  (I), m. 162–4°. I (30 g.) with concd.  $\text{HNO}_3$  in AcOH gives 24 g. of the 2,4-di- $\text{NO}_2$  deriv., yellow, m. 185–8°; cold



fuming  $\text{HNO}_3$  with or without  $\text{AcOH}$  gives the 2,4,5-tri- $\text{NO}_2$  deriv. (II), m. 215° (decompn). 5-O-N- $\alpha$ - $\text{C}_6\text{H}_4\text{NH}_2$  and m-O- $\text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{Cl}$  give 5-nitro-1-m-nitrobenzenesulfonaphthalide, m. 208-10°, nitration gives I and Br in  $\text{CHCl}_3$ , refluxed 1 hr, give the 4-Br deriv., m. 174-6°, hydrolysis gives 4-Br- $\alpha$ - $\text{C}_6\text{H}_4\text{NH}_2$ , further action of Br in  $\text{C}_6\text{H}_5\text{NH}_2$  gives the 2,4-di-Br deriv., m. 232-3°, also prep. from m-O- $\text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{Cl}$  and 2,4-Br- $\alpha$ - $\text{C}_6\text{H}_4\text{NH}_2$ ; 4 Nitro-1-p-toluenesulfonaphthalide and Br in  $\text{C}_6\text{H}_5\text{NH}_2$  give the 2 Br deriv., yellow, m. 193-5°; no reaction occurs in boiling  $\text{CHCl}_3$ . p-Toluenesulfonaphthalide (III) (20 g.) and concd  $\text{HNO}_3$  give only 0.5 g. of the 2- $\text{NO}_2$  deriv., deep yellow, m. 154°. Bromination of  $\alpha$ - $\text{C}_6\text{H}_4\text{NH}_2$  gives the theoretical yield of the 2,4-di-Br deriv. 2,4-( $\text{O}_2\text{N}$ ) $_2$ - $\alpha$ - $\text{C}_6\text{H}_4\text{OH}$  and fuming  $\text{HNO}_3$  give a mixt. of 2,4,5- and 2,4,7-tri- $\text{NO}_2$  derivs III and Br in  $\text{CHCl}_3$  give the 1-Br deriv., m. 100°. Reduction of 1,3,2-Br $_3\text{C}_6\text{H}_3\text{NH}_2$  with Sn and concd  $\text{HCl}$ -EtOH gives 3-bromo-2-naphthylamine, m. 173°. Ac deriv., m. 177°. p-MeC $_6\text{H}_4\text{SO}_2\text{Cl}$  gives 3-bromo-2-p-toluenesulfonaphthalide, pale yellow, m. 127-9°;  $\text{HNO}_3$  in AcOH gives the 1- $\text{NO}_2$  deriv. (IV), pale yellow, m. 237-9° (decompn), this also results on bromination of the 1- $\text{NO}_2$  deriv. of III, reduction with Sn and  $\text{HCl}$  or Zn and AcOH gives 3-bromo-2-p-toluenesulfonaphthalenediamine, m. 185°. Hydrolysis of IV gives 3-bromo-1-nitro-2-naphthylamine (V), orange, m. 105°. Ac deriv., yellow, m. 136°, 3-bromo-1-nitro-2-naphthol, yellow, m. 131° (decompn). Reduction of V gives 3-bromo-1,2-naphthylenediamine, m. 85° (decompn) (quinazoline deriv., yellow, m. 195-9°). 1,6-Dinitro-2-p-toluenesulfonaphthalide gives the 3-Br deriv., pale yellow, m. 228-31° (decompn), hydrolysis yields 3-bromo-1,6-dinitro-2-naphthylamine, golden, m. 238-41°. Ac deriv., m. 273-7° (decompn). III in  $\text{C}_6\text{H}_5\text{NH}_2$  with I,  $\text{ICl}$  or  $\text{ICl}_3$  gives the 1-I deriv., m. 126-7°. 3-Iodo-2-p-toluenesulfonaphthalenyl, m. 114-15°, the 4-p-toluene isomer, m. 109-15°, 4-iodo-2-p-toluenesulfonaphthalene, m. 127-32°. C. J. W.

Constitution and reactivity XIV. Reaction kinetics of the sulfonation by oleum and the properties of different concentrations of fuming sulfuric acid. Karl Lauer and Ryohei Oda. *J. prakt. Chem.* 144, 32-40 (1935), cf. C. A. 29, 6216f.—Sulfonation of anthraquinone (I) with  $\text{H}_2\text{SO}_4$  at various temps and concns gave the following results at 130°, 140° and 150° for  $k \times 10^4$ , calcd. for a pseudomonomol reaction:  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{O}$ , 479, 1210, 2510, 1,289%  $\text{SO}_3$ , 854, 1696, 3340, 3.2%  $\text{SO}_3$ , (1380), (2890), 5913, 5%  $\text{SO}_3$ , 40.1, 91.5, 213, 8.5%  $\text{SO}_3$ , 804, 1583, 3851, 19.9%  $\text{SO}_3$ , 2542, 5062, 9350. Since equimol. quantities of  $\text{SO}_3$  were used, the mol ratios of  $\text{H}_2\text{SO}_4$  to I to  $\text{SO}_3$  varied from 3.2:1 to 39:1. However, this appeared to have no effect on the value of  $k$ . Sulfonation of I with  $\text{H}_2\text{SO}_4$  has an activation heat of about 40,000 cal. and an action const. of  $300-1000 \times 10^{10}$ . With  $\text{SO}_3$  the values are 22,000 cal. and  $0.1 \times 10^{10}$ . Oleum with 3-10%  $\text{SO}_3$  contains  $\text{SO}_3$  in a specially inactive form. This accounts for the observation that the reaction of sulfonation of I with 2-3% oleum at 140° soon comes to a standstill. C. J. West.

Benzanthrone derivatives III. The autocyclizable dihydrobenzanthrone E. Clar. *Ber.* 68B, 2069-70 (1935), cf. C. A. 26, 594f.—It had been found that benzanthrone (I) in alk. or mineral acid soln is reduced to 1,10 trimethylene-9-hydroxyphenanthrene (II). No dihydrobenzanthrone could be isolated as intermediate product in these reductions, but this gap has now been filled. With Zn dust and AcOH I gives in excellent yield a dihydro compd (III), whose interesting properties explain the earlier failures to isolate it. The colorless, well-cryst. III is very sensitive to air, so that all operations must be carried out in a  $\text{CO}_2$  atm. It also shows the properties of a biradical, it takes up exactly 1 mol. O in xylene, instantly decolorizes 2 atoms Br and is disproportionated by strong acids or weak alkalis, or by heating to 150°, into I and II. The oxidation is markedly accelerated by light and proceeds with greatest velocity (in about 5 min.) in AcOH, but in this solvent only about 1.6 atoms O is taken up. The xylene and AcOH mother

liquors nevertheless show for days the properties of peroxides (liberation of I from KI, decolorization of  $\text{KMnO}_4$ ). In xylene the O is apparently given up to the solvent and does not act further on unchanged III, while in AcOH  $\text{H}_2\text{O}_2$  is split off and forms I with II still present.  $\beta$ -5-1-bromobenzanthrone also yields a dihydro deriv. (IV) with similar properties. The detn. of the structure of III is rendered very difficult by the sensitivity of III to acids and heat, so that it can undergo almost no reaction without alteration of the skeleton of double bonds. Short action of  $\text{Ac}_2\text{O}$  gives an acetate not sensitive to air. Of the possible formulas for III,



seems the most probable. The course of the reduction of I is further confirmation of the conception of a reactive diyl state for I, which would explain its behavior as an unsat. ketone. Especially worthy of note is the appearance of an orange-red intermediate stage in the reduction of I, as well as in the oxidation of III in solid state or in soln (not in xylene, however). This intermediate stage is also observed when I is reduced to III with Zn dust, pyridine and AcOH according to Kuhn and Winterstein (C. A. 27, 723). Since no pinacol of I can be obtained by any reduction method, it may be that the orange-red intermediate stage represents a disocd. pinacol, a monohydro compd or quinhydrone. Attempts to prep such a compd from equal mols. of I and III gave an orange-red product which, although homogenous in appearance, melted considerably lower (120-5°) than its components, on oxidation it consumed exactly half as much O as III, but not enough of the oxidation product (also formed in small amt. in the oxidation of III in xylene) was obtained for study. A radical monohydric compd. can therefore be present only in very small concn. in equi. with I and III. III, tables, quickly becoming orange-red in the air and brown-yellow in the light, sol. in concd.  $\text{H}_2\text{SO}_4$  with orange-red color and fluorescence, m. 150-2° on rapid heating under  $\text{CO}_2$  in a sealed capillary (the m. p. falls to 132° when the detns are repeated). Acetate of III, m. 159-61° (decompn.) under  $\text{CO}_2$  (sealed tube), 154-5° in air. IV, m. 167° (evolution of  $\text{HBr}$ ). C. A. R.

Condensation of phthalic anhydride with  $\alpha$ -octahydrophenanthrene E. de Barry Barnett, N. F. Goodway and C. A. Lawrence. *J. Chem. Soc.* 1935, 1684.— $\alpha$ -Octahydrophenanthrene and  $\text{C}_6\text{H}_4(\text{CO})_2\text{O}$  with  $\text{AlCl}_3$  in  $\text{C}_6\text{H}_2\text{Cl}_4$  give 90% of  $\alpha$ -1,2,3,4,5,6,7,8-octahydrophenanthrylbenczoate (I), m. 200°, 4,5-C $_6\text{H}_4(\text{CO})_2\text{O}$  yields the 4',5'-di-Cl deriv., m. 276° (decompn.). Reduction of I with Zn, NaOH and  $\text{NH}_4\text{OH}$  by heating 15 hrs at 100° gives  $\omega$ -1,2,3,4,5,6,7,8-octahydrophenanthryl- $\alpha$ -toluene, m. 182°, cyclized by concd  $\text{H}_2\text{SO}_4$  to octahydro-1,2,3,4-dibenzanthrone, pale yellow, m. 200°, AcO and  $\text{C}_6\text{H}_5\text{NH}_2$  give octahydro-1,2,3,4-dibenzanthranol acetate, yellow, m. 214°,  $\text{CrO}_3$  in AcOH gives octahydro-1,2,3,4-dibenzanthraquinone, yellow, m. 234°; reduction gives octahydro-1,2,3,4-dibenzanthracene, pale yellow, m. 129°.

C. J. West. Constitution of vitamin D $_2$ . A Windaus and W. Thiele. *Ann.* 521, 160-75 (1935).—The acetate from 15 g. vitamin D $_2$  (I) and 6 g. maleic anhydride, refluxed 4 hrs in benzene, give an amorphous product which, neutralized with 10% KOH in MeOH, acidified with AcOH, esterified with  $\text{CH}_3\text{N}_3$  and crysd. from MeOH, yields 6-7 g. of di-3- $\alpha$ -vitamin D $_2$  acetate maleate (II),  $\text{C}_{28}\text{H}_{44}\text{O}_4$ , m. 141°,  $[\alpha]_D^{20}$  170.8°, adds 2 atoms O on titration with  $\text{MeOH-KOH}$ . The mother liquor, hydrolyzed with  $\text{MeOH-KOH}$ , acetylated and esterified, yields the  $\beta$ -isomer (III), m. 94-5°,  $[\alpha]_D^{20}$  -158.3°, adds 3 atoms O. These compds distil at 0.01 mm. without decompn. The dihydro deriv. of II in 112°,  $[\alpha]_D^{20}$  167.6°, and adds 1.16-1.33



differ markedly from *O*-methyl- and *O*-ethylcarbostyryl and VI. This shows that it is possible to distinguish between hydroxyquinoline and quinolone derivs by use of absorption spectra. C. J. West

Action of sodium methylate on ethyl isatin-*N*-acetate. N. I. Putokhin. *J. Gen. Chem.* (U. S. S. R.) 5, 1176-84 (1935).—Gabriel and Colman (*Ber.* 33, 980 (1900)) showed that the interaction of MeONa with phthalylglycine ester results in isomerization and cyclic increment with the formation of Me 4-hydroxyisocarbostyryl-3-carboxylate. Radelescu (*C. A.* 21, 1647) and Gheorghiu (*C. A.* 28, 2709) obtained dihydroxy-naphthalene derivs by a similar isomerization of indandione derivs with EtONa. Study of this type of reaction was begun with the easily isomerized isatin and its homologs by the action of MeONa on

$\text{C}_6\text{H}_4\text{CO CO NCH}_2\text{CO}_2\text{Et}$  (I). As the following scheme shows, the reaction proceeds in 2 directions: one leading to XI, and the other to a quinoline ring. Usually the 2 reactions take place simultaneously with either of the 2 reactions predominating, depending on the conditions of the process used. Of the 3 possible structures of the isomerized compd., convertible to VIII, IV and V are more acceptable as relatively more stable, while VI is likely to be a labile form isomerizing to XI. This may in part explain the presence of XI in the reaction mixts. X was also isolated. (I)  $\text{I} + \text{MeONa} \rightarrow \text{EtO}_2\text{CCH}_2\text{NHCH}_2\text{COC}$

$(\text{OMe})_2\text{ONa}$  (II)  $-2\text{MeOH} \rightarrow \text{C}_6\text{H}_4\text{CO CO}(\text{ONa})\text{C}$

$(\text{CO}_2\text{Et})\text{NH} + \text{NaOH} \rightarrow \text{C}_6\text{H}_4\text{CO CO}(\text{ONa})\text{C}(\text{CO}_2\text{Na})\text{NH}$  (III)  $+ \text{HCl} \rightarrow \text{C}_6\text{H}_4\text{C}(\text{OH})\text{C}(\text{OH})\text{C}$

$(\text{CO}_2\text{H})\text{N}$  (IV),  $\text{C}_6\text{H}_4\text{CO CO}(\text{OH})\text{C}(\text{CO}_2\text{H})\text{NH}$  (V) or

$\text{C}_6\text{H}_4\text{CO CO CH}(\text{CO}_2\text{H})\text{NH}$  (VI)  $+ \text{H}_2 \rightarrow \text{C}_6\text{H}_4\text{CH}$

$(\text{OH})\text{CH}(\text{OH})\text{CH}(\text{CO}_2\text{H})\text{NH} + \text{H}_2 \rightarrow \text{C}_6\text{H}_4(\text{CH}_2)_2$

$\text{CH}(\text{CO}_2\text{H})\text{NH}$  (VII)  $+ \text{CaO} \rightarrow \text{C}_6\text{H}_4(\text{CH}_2)_2\text{NH}$  (VIII).

(2)  $\text{I} + \text{NaOH} \rightarrow \text{NaO}_2\text{CCH}_2\text{NHCH}_2\text{COC}(\text{OH})\text{ONa} \rightarrow \text{H}_2\text{O} \rightarrow \text{NaO}_2\text{CCH}_2\text{NHC}_6\text{H}_4\text{COC}(\text{OH})\text{Na}$  (IX)  $+ \text{HCl} \rightarrow$

$\text{HO}_2\text{CCH}_2\text{NHC}_6\text{H}_4\text{COC}(\text{OH})\text{H}$  (X)  $\rightarrow \text{H}_2\text{O} \rightarrow \text{C}_6\text{H}_4\text{CO CO N}$

$\text{CH}_2\text{CO}_2\text{H}$  (XI). A mixt of 4 g I, m 114°, (cf P, C 23, 2970), in 33 cc of dry MeOH with 0.8 g MeONa in 32 cc MeOH in a rubber stoppered flask was digested at 75-80° for 3-4 hrs until the formation of a ppt. had begun. After cooling the mixt was treated again with 0.6 g MeONa in 10-12 cc MeOH and digested as above for 4 hrs, giving 2 g III. It was identified as the mono-

and di-Ag salt, and as  $\text{C}_6\text{H}_4\text{CO CO}(\text{OEt})\text{C}(\text{CO}_2\text{Et})\text{NH}$ , m 119-20°.

I treated as above but with double the amt. of MeONa at 80-100° formed IX. IX in H<sub>2</sub>O treated with HCl gave XI, m 206° (cf Langenbeck, C 22, 2749).

II, carbonylating at 230°, was obtained as III on heating the mixt for 3 hrs to an incipient ppt. (without the 2nd addn of MeONa). Two g IV (V or VI) with 40 cc HI (d 1.55) and 1 g of red P, refluxed for 8-10 hrs (or preferably at 150° in a sealed tube), gave 0.7-0.8 g VII, which on distn with CaO formed VIII, b 247-8°.

Chas. Blanc

Arsenic derivatives of carbazole. S. M. Sherin and A. Ya. Berlin. *J. Gen. Chem.* (U. S. S. R.) 5, 938-42

(1935).—Carbazole-3-arsenic acid,  $\text{HN C}_6\text{H}_4\text{C}_6\text{H}_4\text{AsO}_2(\text{OH})_2$  (I), m 346-7°, was obtained by the following reaction:  $(\text{C}_6\text{H}_5)_2\text{NH}$  (II)  $+ \text{HNO}_3 \rightarrow (\text{C}_6\text{H}_5)_2\text{NNO} +$

$\text{HNO}_3 \rightarrow \text{O}_2\text{NC}_6\text{H}_5\text{C}_6\text{H}_5\text{NNO} + \text{H}_2 \rightarrow \text{H}_2\text{NC}_6\text{H}_5\text{C}_6\text{H}_5\text{N}$

$\text{H}(\text{III}) + \text{H}_2 \rightarrow \text{CIN}_2\text{C}_6\text{H}_5\text{C}_6\text{H}_5\text{NH} + \text{Na}_2\text{AsO}_2 \rightarrow \text{I}$ . III was obtained in 80% yield by the method of Lindemann

(C 18, 2705). A mixt. of 35 g III, 48 cc HCl (d 1.175) and 400 of H<sub>2</sub>O was treated with 13.3 g NaNO, in H<sub>2</sub>O and directly neutralized with a cold NaOH soln.

The soln was slowly poured at room temp., with stirring, into the soln. of 29.2 g As<sub>2</sub>O<sub>3</sub>, 46.6 g Na<sub>2</sub>CO<sub>3</sub>, 100 cc H<sub>2</sub>O and 30 cc 10% NH<sub>4</sub>CO<sub>3</sub>. After a continued stirring for 2 hrs and standing overnight, the mixt. was boiled with animal C and the filtrate acidified, giving 27% of crude I. This was purified by converting it with boiling

Na<sub>2</sub>CO<sub>3</sub> into  $\text{HN C}_6\text{H}_4\text{C}_6\text{H}_4\text{AsO}(\text{OH})\text{ONa} \cdot 5\text{H}_2\text{O}$  and

decompd with dil HCl.  $\text{HN C}_6\text{H}_4\text{C}_6\text{H}_4\text{AsCl}_2$  (IV), m 150°, was prepd when 4 g I was dissolved in a mixt. of 20 cc of concd. HCl, 20 cc alc. and a few drops of 10% I soln and the mixt treated at room temp. with a SO<sub>2</sub> current for 30 min. The ppt. was washed with 20% HCl and dried *in vacuo*. IV in alc. treated with an equal vol of hot concd HCl and the crystals extd with Li<sub>2</sub>O gave

68% II.  $\text{HN C}_6\text{H}_4\text{C}_6\text{H}_4\text{As}(\text{OH})_2$ , m. 207-9°, resulted when 0.7 g IV in Me<sub>2</sub>CO was treated with concd. NH<sub>4</sub>OH and the mixt. dild with H<sub>2</sub>O. Chas. Blanc

Constitution of the dihydroxy derivative of diphenylene oxide obtained from resorcinol. Kazuo Hata, Kiyoharu Tateatsu and Benno Kubota. *Bull. Chem. Soc. Japan* 10, 425-32 (1935).—Acylation of *p*-anisidine with Ac<sub>2</sub>O gives *p*-acetylansidine (I), m. 127-7.5°. I, heated to boiling with 11% HNO<sub>3</sub>, yields *o*-nitro-*p*-acetylansidine (II), m. 117°. Sapon of II with alc. KOH gives *o*-nitro-*p*-anisidine (III), m. 122-3-3.0°. Diazotization of III and treatment with KI soln gives *m*-nitro-*p*-iodoanisole (IV), m. 62°. IV, heated with Cu-bronze at 130-170° for 1 hr., gives 2,3'-dimethoxy-4,4'-dimethoxybiphenyl (V), m. 136-7°. Reduction of V with SnCl<sub>4</sub> in glacial AcOH said with HCl gives 2,2'-diamino-4,4'-dimethoxybiphenyl (VI), m. 110-5-11°. Addn. of 50% CuSO<sub>4</sub> soln. to a soln of diazotized VI during steam distn gives 2,7-dimethoxydiphenylene oxide (VII), m. 150°, in the distillate. The residue remaining in the distn. flask is 2,7-dimethoxyphenazone, m. 197°. VII, refluxed 15 hrs with HI, gives 2,7-dihydroxydiphenylene oxide (VIII), m. 241-1.5°. The properties of VIII derived from *p*-anisidine are perfectly coincident with those of VIII obtained from resorcinol. W. J. Peterson

Reply to a remark of E. Späth and F. Kuffner. I. <sup>1</sup> (hydroxyphenyl)ethylamines and their transformations

III. Synthesis of benzylisoquinolines under physiological conditions. Georg Hahn and Otto Schöck. *Ber.* 68B, 2019 (1935); cf S and K, C. A. 29, 7960.

C. A. R

Photoreaction in tropical sunlight. Braj Kishore Malaviya and Sukbhabshun Dutt. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 4, 319-20 (1935).

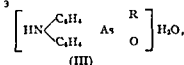
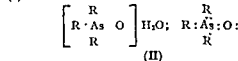
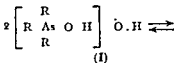
The action of strong sunlight on a large no. of org. compds was studied by the following general method. Solns, usually 2%, in H<sub>2</sub>O, dil HCl, dil NaOH or EtOH, made from carefully purified compds, were placed in 1-4 conical flasks of clear Jena glass, which were filled to the neck and loosely covered with glazed paper. These were then exposed to full sunlight and were not disturbed, except to replace evapd solvent, until no further action took place or it was seen that no action was going to occur.

The products were then isolated and identified if possible. A soln of PhNH<sub>2</sub> in 0.2 N HCl first reddened, then after 2 days a ppt started to form. The action was complete in 135 days. This ppt. was identified as phenazine, m 170°, by its properties and a mixed m. *p*-MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave 1,5-dimethylphenazine, m. 150°, complete in 120 days. *m*-MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave 2,6-dimethylphenazine, m. 156°, in 71 days. By analogy *p*-MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave 3,7-dimethylphenazine (7) in 123 days. This product did not melt but gave characteristic phenazine tests and the correct percentage of N. PhNMe<sub>2</sub> showed no change after 141 days. 1,3,4-Xylydine gave only a trace of ppt. in 121 days. *o*-C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave *o*-dimethylphenazine (7), no m. p., in 107 days. *p*-C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave *p*-dimethylphenazine (7), m. 240°, in 110 days. *o*-N<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> was unaffected in 125 days. *p*-ONC<sub>6</sub>H<sub>4</sub>NMe<sub>2</sub> in aq soln was oxidized to *p*-nitrodiphenylamine, m. 220°, in 42 days; in dil HCl it gave an unidentified ppt., which kept forming

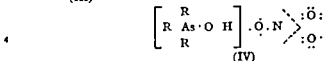
for over 111 days. Benzidine gave an unidentified phenazine deriv. in 84 days. Phenol, quinol, and  $\alpha$ - and  $\beta$ -naphthol in aq. or dil. NaOH solns. gave similar dark unidentified ppt. in 64 to 121 days. Resorcinol, pyrocatechol and pyrogallol were unchanged after 156 days. A 1% soln. of  $\alpha$ -C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> in dil. HCl gave 2,3-diaminophenazine-HCl in 125 days. Aq.  $m$ -C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> gave 2,6-diaminophenazine, m. 128°, in 28 days.  $p$ -C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> in dil. HCl gave 3,6-diaminophenazine (?), m. 130°, in 106 days.  $p$ -H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NMe<sub>2</sub> in dil. HCl in 120 days gave an intensely violet soln. which was shown to contain an unidentified azine dye. Aq.  $p$ -H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHAc gave diacetyl- $p$ , $p'$ -diaminodiphenylamine, m. 219°, in 81 days. 1,8-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> in dil. HCl in 87 days gave a brown ppt. which was probably *peri*-dinaphthalene azoxide, since it gave azine reactions.  $o$ -HOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> in aq. soln. in 94 days gave a red compd., C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub>, identical with one described by G. Fischer (*J. prakt. Chem.* [2], 19, 318 (1879)).  $m$ -HOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave an unidentified brown ppt. in 62 days.  $p$ -HOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> in 70 days gave a violet ppt. whose properties indicated that it was  $p$ , $p'$ -dihydroxydianilinoindophenol.  $m$ - and  $p$ -HOC<sub>6</sub>H<sub>4</sub>NMe<sub>2</sub> were unchanged in 125 days.  $o$ -MeOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> in dil. HCl gave 1,5-dimethoxyphenazine, no m. p., in 78 days.  $p$ -MeOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave 3,7-dimethoxyphenazine,  $o$ -LOc<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave 1,5-dithoxyphenazine,  $p$ -LOc<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> gave 3,7-dithoxyphenazine, and 2,4-HOC<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> gave an unidentified black ppt. which had the properties of a phenazine deriv. H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H in dil. HCl gave phenazine-1,5-dicarboxylic acid, m. higher than 300°, in 120 days.  $m$ -H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H in dil. NaOH was unchanged, but  $p$ -H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H gave the Na salt of  $p$ , $p'$ -azobenzene-dicarboxylic acid, di-Et ester, m. 111°. Vanillin, 2,4-(OH)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO, and  $p$ -Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO were unchanged. Aq.  $p$ -OHC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> in dil. HCl gave  $p$ , $p'$ -dialdehydazobenzene, m. 239°, in 123 days. Aq. 1% eosin was decolorized in 75 days and gave HBr and a ppt. of 2,4-dibromobenzoic acid, m. 218°. Aq. 1% erythrosin was decolorized in 120 days to give III and by analogy, 2,4-diodobenzoic acid (?), no m. p.  $p$ -H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHAc in dil. HCl in 75 days gave an unidentified ppt., m. 180°, which reduced NH<sub>4</sub>AgNO<sub>3</sub>. Gallacetophenone in dil. NaOH was unaffected. Ph<sub>3</sub>HNH<sub>2</sub> evolved N<sub>2</sub> and in 56 days gave an unidentified, apparently heterocyclic N<sub>2</sub> compd., m. 128°. 2-Aminothiazole in dil. HCl gave a trace of ppt. which apparently was a phenazine deriv. Aq. thiocarbamide gave S and carbamide in 46 days.  $\alpha$ -Benzil dioxime in dil. NaOH gave 3,4-diphenylfuran, m. 94°, in 35 days. Sterile aq. 1% maleic acid gave pyruvic acid in 123 days. Sterile citric acid was unaffected. Tartaric acid, while mainly unchanged, gave a trace of a solid with strong aldehydic or ketonic properties. Erucic acid in dil. LiOH gave dihydroxyerucic acid, m. 118°, in 38 days. Aq. 1% maleic acid was completely converted into fumaric acid in 50 days. Na cinnamate was 17.2% converted into allocinnamic acid in 50 days. Aq. itaconic, citraconic, and tiglic acids and Na oleate and brassydate were not inverted after 75 days.

John E. Milbery  
Dihydroxy derivatives of ternary arsesines, arsonium bases and their salts. G. A. Razuvaev, V. S. Malinovskii and D. A. Godina. *J. Gen. Chem.* (U. S. S. R.) 5, 721-7 (1935); cf. Razuvaev and Malinovskii, *C. A.* 29, 6803f.—The di-HO derivs. of ternary arsesines were obtained by the following reactions:  $R_3AsX_2 + 2RMgX \rightarrow R_3As + 2MgX_2$ ;  $R_3AsX + RMgX \rightarrow R_3As + MgX_2$ ;  $R_3As + I_2 + 2H_2O \rightarrow R_3As(OH)_2 + 2HI$  ( $R = Me$  or  $Ph$ ;  $X = Cl$  or  $I$ ). The reaction with the dihydrophenarsine series proceeds analogously. All di-HO derivs. on drying over P<sub>2</sub>O<sub>5</sub> in *vacuo* give oxides. Trimethyl- and dimethylphenyl-arsines give the corresponding oxides. These compds. form hygroscopic crystals, sol. in H<sub>2</sub>O and alc. They give with HNO<sub>3</sub> monobasic salts, R<sub>3</sub>As(OH)ONO<sub>2</sub>, and with H<sub>2</sub>SO<sub>4</sub> (Ph<sub>3</sub>AsOH)<sub>2</sub>SO<sub>4</sub>. In distinction from the described derivs. of the fatty-aromatic series, the oxides and dihydroxides of 10-R-9,10-dihydrophenarsine are little or not at all hygroscopic and are considerably more difficultly sol. in H<sub>2</sub>O and alc. (cf. R. and M., *C. A.* 25, 1831). They give with HNO<sub>3</sub> no

salts but are very easily nitrated (cf. R. and M., *C. A.* 28, 4059f). This indicates a greater mobility of H in the C<sub>6</sub>H<sub>5</sub> nuclei of these derivs. of dihydrophenarsine than in that of methylphenylarsine. The structure of the dihydroxides, oxides and their salts may be thus formulated.

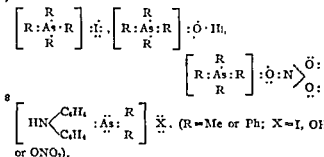


(III)



(IV)

Formula I agrees with the ability of the dihydroxides to form monosubstituted salts, the difficulty in forming neutral salts and the evident inequality of the 2 OH groups or the 2 halide atoms in the corresponding dihalide derivs. of ternary arsesines (cf. Michaelis, *Ann.* 321, 162 (1902); Steinkopf and Schwen, *C. A.* 16, 1407 (1921)). Formula II is substantiated by the easy conversion of the dihydroxides into oxides and the even dehydration indicates the presence of the H<sub>2</sub>O of hydration and not of constitution. It is possible that the compds. I and II exist in a state of mutual equil. Formula III for the HO derivs. of dihydrophenarsazines is based on their easy dehydration, inability to form salts and the relatively poor soly. in H<sub>2</sub>O. The dihydroxides of dihydrophenarsazine and their derivs. are more toxic than the corresponding derivs. of fatty-aromatic arsesines. The arsonium bases and their salts were obtained by the following reactions:  $R_3As + MeI \rightarrow R_3MeAsI$ ;  $2R_3MeAsI + Ag_2O \rightarrow 2R_3MeAsOH + 2AgI$ ;  $R_3MeAsI + AgNO_3 \rightarrow R_3MeAsONO_2 + AgI$ . The reaction with the heterocyclic arsesines proceeds analogously. The following structure of these arsonium derivs. is based on their high degree of dissociation and salt formation:



For the production of arsine derivs. R<sub>3</sub>As was oxidized with I in alc.-H<sub>2</sub>O to R<sub>3</sub>As(OH)<sub>2</sub>; this on heating with HNO<sub>3</sub> was converted into R<sub>3</sub>As(OH)ONO<sub>2</sub>. The arsonium compds. were prepd. by treating R<sub>3</sub>As in the cold with MeI. The R<sub>3</sub>MeAsI in H<sub>2</sub>O, refluxed with Ag<sub>2</sub>O, gave RMeAsOH and with aq. AgNO<sub>3</sub> RMeAsONO<sub>2</sub>. The filtrates from Ag were evapd. to 2-3 cc. and dried in a vacuum desiccator over P<sub>2</sub>O<sub>5</sub> to a const. wt. The dihydroxides and their nitrate salts were similarly isolated, the solns. were freed from HI with Ag<sub>2</sub>O or AgNO<sub>3</sub>. All these reactions, except the formation of ternary arsesines,

gave theoretical yields with 70-80% of pure products. MeAs was prep'd in a H atm. The EtO soln. was distd. in a CO<sub>2</sub> atm into a receiver contg a reagent to produce the desired arsine deriv. The EtO soln of MeAs treated with Br soln and reworked gave MeAsO and not MeAs(OH); MeAs(OH)ONa, m 127° MeAsI, m 328°. MeAsONa, m 268-70° MeAsOH could not be isolated. PhMeAs was obtained in 60% yield. PhMeAs(OH), m 157-61°, gave PhMeAsO. PhMeAs(OH)ONa, m 149-50° PhMeAsOH, m 106-16°, is highly hygroscopic. PhMeAsONa, m 195-6°. PhMeAs is an oil. PhMeAs(OH), is highly hygroscopic. PhMeAs(OH)ONa, m 106-7° PhMeAsI, m 211-13°. PhMeAsOH, m 129-35°, is highly hygroscopic. PhMeAsONa, m 149-51°. PhAs(OH), m 115-16°. PhAs(OH)ONa, m 165°. PhMeAsI, m 176°. PhMeAsOH, m 124-6°. PhMeAsONa, m about 100°. (PhAsOH)SO<sub>3</sub>, m 195°. Derivs of methylcyclohexylarsine (C<sub>6</sub>H<sub>11</sub>)<sub>3</sub>MeAs, b<sub>m</sub> 170° (C<sub>6</sub>H<sub>11</sub>)<sub>3</sub>MeAsI, m 186-7° (C<sub>6</sub>H<sub>11</sub>)<sub>3</sub>MeAsOH could not be isolated (C<sub>6</sub>H<sub>11</sub>)<sub>3</sub>MeAsONa, m 147° Chas Blanc

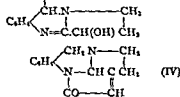
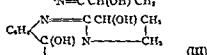
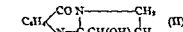
Ergometrine Harold W Dudley *Proc. Roy Soc (London)* B118, 478-84 (1935), cf C A. 29, 7988<sup>2</sup>. Ergometrine is a white alkaloid, freely sol in the simple alcs, AcOEt, MeCO and MeCOEt, sparingly sol in dichloroethylene and C<sub>6</sub>H<sub>6</sub>, and very sparingly sol in CHCl<sub>3</sub>. It crystallizes from 400 parts C<sub>6</sub>H<sub>6</sub> as slender white needles, and from MeCOEt as stout prisms, m 162-3° (decompn). A soln. in 40 parts AcOEt at -4° deposits thin, white, lustrous plates, m 160-1°, free from solvent of crystn, on concn *in vacuo*, the mother liquor deposits needles which dissolve on warming, on cooling to room temp diamond-shaped plates deposit contg. 0.5 mol AcOEt of crystn and m 130-2° with effervescence but no darkening. The alkaloid tends to darken in the air, contains an indole nucleus (pos. reactions with glyoxylic acid, p-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, and FeCl<sub>3</sub>), has the formula C<sub>17</sub>H<sub>23</sub>N<sub>3</sub>O<sub>2</sub>, is moderately sol in H<sub>2</sub>O to a d-rotatory, alk (to litmus), blue fluorescent soln. which oxidizes and becomes brown on exposure to the air. In alc the alkaloid has [α]<sub>D</sub><sup>25</sup> 40.25°, [α]<sub>D</sub><sup>18</sup> 59.2°, its soln in CHCl<sub>3</sub> is slightly l-rotatory. Calcd from values for its salts in aq soln, the ergometrine anion has [α]<sub>D</sub><sup>20</sup> 70-1°. Ergometrine forms stable salts with HCl, HBr, oxalic and picric acids. Ergometrine-HCl, needles, m 245-6° (decompn). Ergometrine-HBr, needles, m 236-7° (decompn). Ergometrine oxalate, needles, m 193° (decompn). Ergometrine picrate exists as yellow hydrated needles, m 148° (decompn), and red anhyd columns, m 188-9° (sudden decompn), which are interconvertible. Ergometrine, ergotocine and ergobasine apparently are identical.

Joseph S Hepburn

An alkaloid from *Trichodesma incanum* D C I G Men'shikov and V Rubinshtein *Ber* 68B, 2039-44 (1935).—Ext'n of the plant with 95% alc, contg 1% NH<sub>3</sub>, gave about 0.1% crude bases from which was isolated an alkaloid *trichodesmine*, C<sub>17</sub>H<sub>23</sub>O<sub>2</sub>N (I) (14 g from 20 kg dried plants), which contains 2 HO groups, forms a cryst methiodide and does not react with HNO<sub>3</sub> in the cold. Hot 10% NaOH hydrolyzes it to an unsatd base C<sub>15</sub>H<sub>19</sub>O<sub>2</sub>N (II), dl-lactic acid (isolated as the quinine salt), and iso-BuCOMe. The base II, *trichodesmine*, has the same m p as the heliotridane (III) obtained by sapon of heliotrine (C A 26, 4818), but the rotation and the much lower mixed m p show that II and III are not identical. II is readily hydrogenated by the Adams method to a satd HO-contg base, *hydroxytrichodesmine*, C<sub>17</sub>H<sub>25</sub>O<sub>2</sub>N (IV). In this respect and in the m p and rotation, II is very similar to the retronecine (V) formed by sapon of retrorsine (Barger, *et al*, C A. 29, 1829<sup>2</sup>), but no V was available for direct comparison. From the character of IV, M and R are inclined to believe that II and V are also not identical. B describes the retronecinal obtained by hydrogenation of V as an oil, while IV, which has the same compn, is a solid m. 92-4°. IV is also different from the isomeric hydroxyheliotridane (C A. 29, 5451<sup>2</sup>) obtained by hydrogenation of heliotrine. IV with hot concd H<sub>2</sub>SO<sub>4</sub> loses 1 mol. H<sub>2</sub>O to form an unsatd.

base, b. 164-7°, obtained in too small amt for precise characterization. To compare the O free skeleton of II with that of III, VI was hydrogenated by Adams' method, yielding a small amt. of a said. base, b. 165-9°, whose picrate, m. 236° (decompn), and methiodide, carbonizing above 300°, proved to be identical with the corresponding derivs. of heliotridane. It is therefore quite probable that II and III differ in the position of the HO groups and perhaps of the double bond, but have the same C skeleton. iso-BuCOMe can be formed in the hydrolysis of II only by ketone cleavage of the corresponding β-ketonic acid, this may be either iso-PrCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H or iso-BuCOCH<sub>2</sub>CO<sub>2</sub>H. I is therefore a complex ester of II (contg. 2 HO groups) and 2 acids, 1 of which is dl-lactic acid and the other is 1 of the 2 ketonic acids above. Since I gives no color with FeCl<sub>3</sub>, it is probable that the lactic acid esterifies the enolic HO of the tautomeric form of the ketonic acid. I m 160-1° (decompn), reacts strongly alk., to litmus in water, instantly decolorizes KMnO<sub>4</sub> in dil H<sub>2</sub>SO<sub>4</sub>, [α]<sub>D</sub><sup>25</sup> 38° (10% alc. soln); methiodide, m. 202° (decompn). II m. 117-18°, instantly decolorizes KMnO<sub>4</sub> in 10% H<sub>2</sub>SO<sub>4</sub>, [α]<sub>D</sub><sup>25</sup> 50° (10% MeOH soln); picrate, yellow, m 142 2 5°. Picrate of IV, yellow, m. 211 5 15° (decompn). C. A. R.

The oxidation products of vasicine with hydrogen peroxide T. P. Ghose, S Krishna, K. S. Narang and J. N. Ray. *Current Sci* 4, 158-9 (1935).—Vasicine (I) reacts with 30% H<sub>2</sub>O<sub>2</sub> (contg 3% H<sub>2</sub>O) as stated in C. A. 27, 510) to give II, m. 214°, and III, m. 168°.



Although Morris, Hanford and Adams (C A. 29, 4365<sup>2</sup>) were unable to obtain III, G, K, N and R. obtained it repeatedly. III was oxidized almost quantitatively to II. The structure of the acyl deriv. of I, m. 164°, is most probably IV.

Rachel Brown

Occurrence of epiquinine and epiquinidine in cinchona bark. Chemistry of quinuclidine Wilhelm Dirscherl and Heinrich Thron *Ann* 521, 48-71 (1935).—Quinuclidine (I), a non-cryst brown smp obtained from the ext'n. of cinchona bark after the known cryst. alkaloids have been removed, analyzes approx. for C<sub>17</sub>H<sub>23</sub>O<sub>2</sub>N<sub>2</sub>, contains 10.06% MeO and has a mol. wt. in C<sub>6</sub>H<sub>6</sub> of 311-35, by titration 363.6. Oxidation with hot KMnO<sub>4</sub> in H<sub>2</sub>SO<sub>4</sub> gives 44% of quinic acid; at 5° there results 57% of HCO<sub>2</sub>H. Catalytic reduction yields a *dihydro deriv*, a brown resin, analyzing for C<sub>17</sub>H<sub>25</sub>O<sub>2</sub>N<sub>2</sub>, which does not yield a compd with Hg(OAc)<sub>2</sub>. Purification of I by means of Hg(OAc)<sub>2</sub> gives 65% of the vinyl bases, with [α]<sub>D</sub><sup>25</sup> 50° (N HCl) or 59° (EtOH), oxidation of this product yields 68% HCO<sub>2</sub>H. Heating 10 g of purified I with 6.05 g 80% H<sub>2</sub>SO<sub>4</sub> 2 hrs at 140° gives 8.8 g quinuclidotouine (II), yellow resin, [α]<sub>D</sub><sup>25</sup> 16.6° (N HCl); with (CO<sub>2</sub>H)<sub>2</sub> there results from 40 g II 15.2 g of quinuclidine oxalate, m. 156°, soly. in H<sub>2</sub>O 1:20, [α]<sub>D</sub><sup>25</sup> 21.6° (N HCl), [α]<sub>D</sub><sup>25</sup> 25° (EtOH-CHCl<sub>3</sub>, 1:2); that from quinine m. 166-7°, [α]<sub>D</sub><sup>25</sup> 24° (EtOH-CHCl<sub>3</sub>). II and BzCl in C<sub>6</sub>H<sub>6</sub> give a *Bz deriv*, yellow resin, sol. in 20 parts Et<sub>2</sub>O, its *isomeric deriv* (45 7 g) with 5% NaOH at 45° gives 16.3 g. of *benzoylmerquinoidinecarbinole* (III), light yellow resin, and 16.5 g of quinic acid III (1 g) and aq Ba(OH)<sub>2</sub>, refluxed 20 hrs, give 0.83 g of merquinoidine sulfate.

acetylmerocinnone, m. 110–11°. The  $\beta$  ester of merocinnone, b. 119–21°,  $[\alpha]_D^{25}$  43.33° (HCl soln, m. 108°,  $[\alpha]_D^{25}$  32.5°). III with HCl and  $HgCl_2$ , heated 6 hrs. at 200°, yields  $\beta$ -collidine. Purified I with EtOH-HCl yields about 50% of a mixt. of about equal parts of epiquinone and epiquimidine; this is the 1st time these epiquinone compounds have been found in nature. C. J. West

**Rearrangement of hydrocinnoline by esterification in two stages** Rufin Ludwiczakówna and Jerzy Szuzylo *Arch. Chem. Farm.* 2, 190–202 (1935) (German summary)—A rearrangement of hydrocinnoline (I) by esterification in 2 stages leads in the 1st step to  $\beta$ -toluene-sulfonylhydrocinnoline,  $C_6H_5SO_2NHC_6H_4CH_2CH_2N$  (II), obtained by interaction during several days at room temp. of I and  $\beta$ -MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>Cl in pyridine, prisms, m. 160–7° (decompn.), slightly sol. in H<sub>2</sub>O, easily sol. in org. solvents,  $[\alpha]_D^{25}$  52.5° (in 96% EtOH). The ester is saponified in the usual way with 4% NaOH, whereby no hetero hydrocinnoline (III) is obtained. The 2nd step is the formation of benzoylhydrocinnoline,  $C_6H_5NHC_6H_4CH_2CH_2N$  (C(OBz)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>N, from II and BrOK by boiling during 95 hrs. in abs. EtOH, short prisms, m. 141–2°, easily sol. in common org. solvents, insol. in H<sub>2</sub>O,  $[\alpha]_D^{25}$  –21.8° (m. 96% EtOH). This ester is saponified by prolonged heating in alc. NaOH, concd. HCl or preferably in dil. HCl, whereby BrOH and III are obtained in theoretical yield, colorless prisms, m. 201–2°, difficultly sol. in Et<sub>2</sub>O and AcOEt,  $[\alpha]_D^{25}$  154.5° (in 96% EtOH). III is analogous as regards sign and value of rotation to the epiquinone obtained previously (C. A. 27, 2958). J. Wiertelak

**Synthesis of hydrastinine chloride on a technical scale** 1. Gryszkiewicz-Trochimowski *Arch. Chem. Farm.* 2, 148–60 (1935) (German summary)—The prepn of hydrastinine chloride (I) on a tech. scale proceeds as follows: heptopropin is condensed with malonic acid in pyridine and piperidine at 40–50° to form methylenedioxybenzoic acid (yield 90%). The latter is reduced with Na-Hg (or less economically with H in presence of Pd or Pt) in alk. medium to methylenedioxyhydrocinnamic acid (II) (yield 90%). The chloride is obtained by treating II, m. 125–6°, may be easily recrystd. from water. The amide of II, treated in the cold with hypochlorite and heated subsequently with 40% NaOH, gives homopiperonylamine, b. 140–1°, (yield 80%). The resulting amine, condensed with BzH, is methylated with Me<sub>2</sub>SO, and the product hydrolyzed immediately to *N*-methylhomopiperonylamine (III) and BzH, these 3 reactions being performed in one step. III is a colorless liquid, b. 141–2°, yield 80%. Boiling III with HCO<sub>2</sub>H and a trace of pyridine during 5 hrs. gives *N*-formylmethylhomopiperonylamine, and the latter boiled with SOCl<sub>2</sub> gives I, m. 210°, in 75% yield. J. Wiertelak

**Sophoricoside, a new heteroside from the fruits of Sophora japonica** L. C. Charaux and J. Rabaté *J. pharm. chim.* 21, 546–54 (1935)—From 1 kg. of the green pods of *S. japonica*, 15 g. sophoricoside (I) was obtained in small white prisms, m. 297–8°,  $[\alpha]_D^{25}$  –32.2° in pyridine soln. contg. 10% H<sub>2</sub>O, as it is very feebly sol. in H<sub>2</sub>O, alc. and AcOH. By dil. acids or emulsin, I is hydrolyzed to *D*-glucose and sophorol (II) identical with genistein (from *Genista tinctoria*; Perkin and Newbury, 1899). Upon fusion with alkali, I forms phloroglucinol and  $\beta$ -HOC<sub>6</sub>H<sub>3</sub>(CH<sub>3</sub>)<sub>2</sub>CO<sub>2</sub>H. Constitution of genistein. *Ibid.* 22, 32–3 (1935)—Baker and Robinson (C. A. 20, 190, 21, 246), restudying genistein and prunellol (Immemore, C. A. 5, 567), found them to be identical, an isoflavone, C<sub>15</sub>H<sub>10</sub>O<sub>6</sub>. This (revised) formula for II leads to C<sub>15</sub>H<sub>10</sub>O<sub>6</sub> for I, it agrees better than the old formula (C<sub>15</sub>H<sub>10</sub>O<sub>6</sub>) with the data obtained for it. The genisteoside obtained by Waltz from *Soja hispida* (C. A. 25, 5675) has the same compn., but its m. p. 254–6° is much lower. S. W.

**Chemical examination of Glycosmis pentaphylla and the constitution and synthesis of its active principle** Sukhshun Dutt. *Proc. Acad. Sci. United Provinces Agra Oudh, India* 5, 55–60 (1935).—A compd. glycosmin, C<sub>15</sub>H<sub>10</sub>O<sub>6</sub>, m. 169°, was isolated from a benzene ext. of the plant leaves. The rotation of a 5% soln. in EtOH

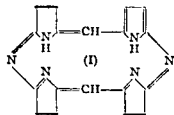
shows  $[\alpha]_D^{25}$  –30°. It yields salsylaldehyde and veratric acid on oxidation with acid KMnO<sub>4</sub>. It was proved to be identical with veratroylsalicyl prep. synthetically.

C. E. P. Jeffreys

**Toad poisons. I. Selenium dehydrogenation of cinobufagin** R. Teschesche and Hans-A. Ofse. *Ber.* 68B, 1908–2000 (1935)—Although the work of Wieland and his co-workers (C. A. 29, 4371), Jensen and Chen (C. A. 24, 4851) and Kondo and Ikawa (I, C. A. 29, 7341) has made it very probable that the toad poisons are derivs. of hydrogenated cyclopentanophenanthrene, it had hitherto not been possible to obtain conclusive evidence of this. Wieland and Hesse by dehydrogenation of bufotalin with Se obtained, instead of methylcyclopentanophenanthrene (I), some other hydrocarbon, probably chrysene. T and O have now, however, obtained a little I (5 mg.) by dehydrogenation of cinobufagin (5 g.) at 270–335°. The I so obtained m. 125–6°, does not depress the m. p. of the Diels I, and agrees with the latter in  $n_D^{20}$  (1.5538  $\pm$  6 and 1.6753  $\pm$  6 for  $\alpha$  and  $\beta$ ) and in its absorption. Wieland's theory of the success was probably due to the use of too high a temp. in the dehydrogenation. C. A. R.

**Porphyrias. XXXV. Synthesis of porphyrin** Hans Fischer and Wilhelm Gleim *Ann.* 521, 157–60 (1935); cf. C. A. 29, 5451°— $\alpha$ -Pyrrolealdehyde (20 g.) in 40 cc. EtOH, added dropwise during 2 hrs. to 200 g. 100% boiling HCO<sub>2</sub>H and refluxed 36 hrs., gives 17 mg. porphyrin, dark red leaflets, gradually darkens about 400°, with acid no 1.7 and contg. 2 active H atoms. Spectra are given for solns. in C<sub>6</sub>H<sub>6</sub>N-Et<sub>2</sub>O, HCl and dioxane and for the Cu salt in C<sub>6</sub>H<sub>6</sub>N-Et<sub>2</sub>O. The principal reaction product is a resinous black product. C. J. West

**Synthesis of diminoporphyrins** I Hans Fischer, Hans Haberland and Adolf Müller *Ann.* 521, 122–8 (1935).—5,5'-Dibromo-4,4'-dimethyl-3,3'-dipropionic acid-pyromethene-HBr (5 g.) and 150 cc. 25% NH<sub>4</sub>OH, after 4 weeks, give 150 mg.  $\beta,\beta$ -diminoporphyrin (I),



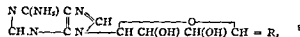
violet needles, m. 308–400° (Pregl block). The yield is smaller with dil. NH<sub>4</sub>OH. It is easily sol. in alkali with a blue color (no fluorescence). The HCO<sub>2</sub>H soln. is blue-green, that in HCl is green. The Cu salt m. over 300° and is sol. in C<sub>6</sub>H<sub>6</sub>N with a blue color. The tetra-*Me* ester, prepd with CH<sub>3</sub>N, forms violet needles, m. 250° (Cu salt, violet, m. 312°). C. J. West

**Syntheses of benzoylporphyrins** Hans Fischer and Kurt Hansen. *Ann.* 521, 128–36 (1935)—2,3-Dimethylpyrrole (5 g.) and 5 g. N<sub>2</sub>CHCO<sub>2</sub>H with a little Cu powder give 3.5 g. of *Et*, 2,4-dimethylpyrrole-5-acetate (I), yellow, b. 142°, the 2-methyl-3-ethyl analog (II), pale yellow, b. 143°. 2-Methyl-3-ethylpyrrole (3 g.), HCN and HCl give 2.6 g. 2-methyl-3-ethylpyrrole-5-aldehyde (III), m. 109° (alazine, yellow, m. 162°). 2,5-Dimethyl-3-carbethoxypyrrole, BzCl and AlCl<sub>3</sub> in CS<sub>2</sub> give the 4-*Bz* deriv. (IV), m. 135°. Refluxing the ester with 10% NaOH yields 2,4-dimethyl-3-benzoylpyrrole-5-carboxylic acid, m. 194° (decompn.); at 200° this gives, nearly quant. 2,4-dimethyl-3-benzoylpyrrole (V), m. 130°, which gives a strong red color with 1 hrlich reagent. 2,3-Dimethylpyrrole-5-aldehyde (VI) (0.5 g.), 0.8 g. V, 3 cc. abs. EtOH and 0.5 cc. 48% HBr, heated 5 min., yield 55% of 3',4,5,5'-tetramethyl-4'-benzoylpyromethene-HBr (VII), m. 268° (decompn.); Br in AcOH gives the 3-*Bz* deriv., red, decompn. 193°. VI and 2,4-dimethyl-3-acetylpyrrole give the 4'-*Ac* analog of VII, decompn. 212°; Br splits off a portion of the Ac group. V and III give 3',5,5'-trimethyl-4-ethyl-4'-benzoylpyromethene-HBr, red.

dish yellow, m. 224° (decompn), 3-Br deriv, brownish red, decomp 202°. The 4'-Ac analog, prisms with a blue luster, m. 214°, nearly quant yield, again Br gives an unsatisfactory product. 2,3,4-Trimethylpyrrole-5-aldehyde and V give 91% of 3,3',4,5,5'-pentamethyl-4'-benzoylpyrromethene-HBr, yellow, m. 224° (decompn), 2,4-Dimethyl-3-ethylpyrrole-5-aldehyde and V give 89% of 3,3',5,5'-tetramethyl-4-ethyl-4'-benzoylpyrromethene-HBr (VIII), m. 211° (decompn) V and 3-methylpyrrolepropionic acid-2-aldehyde give 92% of 3,3',5'-trimethyl-4'-benzoylpyrromethene-4-propionic acid HBr, yellow, m. 204° (decompn), 5-Br deriv (IX), no definite m. p. Heating V with 90% HCO<sub>2</sub>H and adding 48% HBr give 75% of 3,3',5,5'-tetramethyl-4,4'-dibenzoylpyrromethene HBr (X), decomp 225°, it also results in poor yield with AcOH-Br, also from 2,4-dimethyl-5-carbethoxypyrrole-3-aldehyde and V with HBr in EtOH, the free methine, golden yellow, m. 209°. VIII (2 g.) and 3 g. 5,5'-dibromo-4,4'-dimethylpyrromethene-3,3'-di-propionic acid HBr (XI) with 10 g. succinic acid (XII), heated 2 min in lots of 1.5 g. with 2 g. XII, give, after esterification, 40 mg (1%) of di-Me 2,3,5,5'-tetramethyl-1-ethyl-4-benzoylpyrrolin-6,7-dipropionate, m. 263° (cor.), the hemin, C<sub>44</sub>H<sub>50</sub>N<sub>4</sub>FeCl, forms dark needles, there is also formed about 2% of mesoporphyrin 13. X and XI with XII give a very poor yield of di-Me 2,3,5,5'-tetramethyl-1,4'-dibenzoylpyrrolin-6,7-dipropionate (di-Me ester of dibenzoyldeuteriopyrrolin 13) (XIII), reddish brown, m. 252° (cor.), the hemin (XIV), bluish black, m. 290°. 7% HCl exts a very small quantity of a porphyrin, m. 183.5° (cor.) and 1% HCl exts a porphyrin, m. 257° (cor.), XIII also results in a still smaller yield from 5-bromo-3,3',5'-trimethyl-4'-benzoylpyrromethene-4-propionic acid-HBr and XII XIV (10.5 g.), BzO and SnBr<sub>4</sub> at 70° give 100 mg benzoylpyrrophenin Me ester 15 (XV), m. 313° (cor.), acid no 7, 10% HCl gives benzoylpyrrophenin 15 (XVI), does not m. 320°, stable to boiling HCl, even after addn of HBr, and to melting with m-C<sub>6</sub>H<sub>4</sub>(OH), XV yields a hemin (XVII), black prisms, and a Cu salt, rhombic prisms XVI yields an oxime, bluish red, m. 244°. Reduction of XVI with KOH in abs EtOH by heating 4 hrs gives a hydroxybenzylpyrrophenin, dark red, does not m. 300°. Me ester, bluish red, m. 206° (cor.), the behavior with Ac<sub>2</sub>O, (EtCO)<sub>2</sub>CO and concd H<sub>2</sub>SO<sub>4</sub> is described. Reduction of XVII with Na and iso-AmOH yields a chlorin.

C J West

The constitution of adenylypyrophosphoric and of adenosinediphosphoric acid K Lohmann *Biochem Z* 282, 120-3(1935)—The facts that the rate of hydrolysis of 2 mols H<sub>2</sub>PO<sub>4</sub> by boiling dil acids corresponds with that of inorg pyrophosphate, that inorg pyrophosphate is formed by Ba sapon, that hypoxanthine derivs are formed by HNO<sub>3</sub>, and that complexes with Cu salts are obtained, as well as the elec titration curves before and after hydrolysis, all lead to the conclusion that adenosine triphosphate (I) and adenosine diphosphate (II) have the following constitutions



then I is RCH<sub>2</sub>OPO(OH)OPO(OH)OPO(OH)<sub>2</sub> and II is RCH<sub>2</sub>OPO(OH)OPO(OH)<sub>2</sub>.

S Morgulis

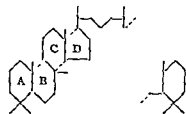
The phytosterolin in wheat germ H A. Ichiba *Sci. Papers Inst Phys Chem Research (Tokyo)* 28, 124-7(1935), cf C A 25, 2434—The phytosterolin was a mixt. of the glucosides of β- and γ-sitosterol.

J J. Willaman

Phytosterol of wheat-germ oil A. Ichiba *Sci. Papers Inst Phys Chem Research (Tokyo)* 28, 112-23 (1935)—Dihydrostosterol and 4 isomeric stosterols were obtained by fractionating the Ac deriv. with MeOH-Et<sub>2</sub>O mixts α-Sitosterol and its Ac deriv. in 134-5°, 116-7°, [α]<sub>D</sub> -22.7°, -23.6°. β-Sitosterol, Ac and Bz derivs, m. 136-7°, 122-3°, 140-5°, [α]<sub>D</sub> -31.5°, -36.7°, -12.3°. γ-Sitosterol, Ac and Bz deriv. m. 147°, 143°, 152°, [α]<sub>D</sub> -42.8°, -47.7°, -19.4°. δ-Sitosterol, Ac

and Bz deriv, m. 140-7°, 113.5-14.5°, 157-8°, [α]<sub>D</sub> -23.9°, -24.4°, -16.0°. The mother liquors from these contained an unidentified hydrocarbon J J W.

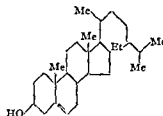
Relation between the cholesterol and carotene structures W. M. D. Bryant *Chemistry & Industry* 1935, 907.—The C skeletons of α- or β-carotene can be more conveniently arranged to yield the cholesterol structure than can squalene. The following scheme of cyclization is suggested.



Ring closure is thought of as beginning at the right of ring A, already present in carotene, rings B, C and D forming successively in a manner characteristic of the higher terpenes. The formation of a 5- instead of a 6-membered ring is attributed to the presence of the 4 unbranched C chains formed by the sym. union of 2 partially cyclized phytyl chains in carotene. It seems possible that this departure from the more common consecutive form of isoprene catenation is responsible for the preferential formation of a 5-membered ring. The aliphatic side chain of cholesterol is formed by cutting off the last 2 isoprene units of the sym. carotene structure.

W. J. Peterson

Structure of sitosterol Mihail Vangelovici and Barbu N Angelescu *Bul soc chim Romania* 17, 177-90 (1935)—Cholesterol, heated gently for 2 hrs with an excess of citraconic anhydride, gives cholesterol citraconate (I), m. 195°. Sitosterol, similarly treated, gives sitosterol citraconate (II), m. 169°. The following derivs of chloro-β-cholestanone (III) are prepd.: oxime, m. 178°, semicarbazone, m. 155°, nitrophenylhydrazone, m. 207°. Nitration of sitosterol chloride with concd HNO<sub>3</sub> and NaNO<sub>2</sub> at room temp gives nitrodehydrocholesterol chloride (IV), m. 118°. Reduction of IV with Zn and AcOH gives chloro-6-sitostanone (V), m. 112°, oxime, m. 180°, semicarbazone, m. 207°, nitrophenylhydrazone, m. 188°. Reduction of III with Na + Hg in alc gives iso-6-cholestanone (VI), m. 97°, oxime, 157°. V reduced in the same manner gives 6-sitostanone, m. 77°, oxime, m. 173°. Oxidation of V with HNO<sub>3</sub> in AcOH gives a chloro dibasic acid (VII), C<sub>27</sub>H<sub>44</sub>ClO<sub>4</sub>, m. 277°. VII, heated with Ac<sub>2</sub>O, gives the anhydride. Sitosterol acetate oxidized with CrO<sub>3</sub> in AcOH gives Me<sub>2</sub>CO, indicating that the lateral chain in sitosterol is possibly MeCHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>. Reduction of III and V by Clemmensen's method yields isocholesteryl chloride, m. 110°, and sitosterol chloride, m. 108°, resp. In view of existing facts the following is suggested as the probable formula of sitosterol



W J Peterson

Synthesis of compounds related to the sterols, hile acids and estrus-producing hormones VII. A. Cohen, J. W. Cook and C. L. Hewett *J Chem Soc* 1935, 1633-7; cf C A 29, 4372—1-Keto-2-methyl-1,2,3,4-tetrahydrophenanthrene (method of prepn given), CH<sub>3</sub>CHCH<sub>2</sub>MgBr, PhOMe and Et<sub>2</sub>O, boiled 3 hrs, and the residual oil

boiled 0.5 hr. with 98%  $\text{HCO}_2\text{H}$ , give 2-methyl-1-allyl-3,4-dihydrophenanthrene,  $b_p$  177-0° (picrate, crimson, m 75-8°); with  $\text{AcOH-HCl}$  1 hr. at 100° there results 2-methyl-1-propylphenanthrene, m 65° (picrate, yellow, m 121.5-2°); sym-trinitrobenzene compd., pale yellow, m 131°; Se at 320° for 15 hrs. gives the same compd 2-Methyl-1- $\Delta^1$ -butenyl-3,4-dihydrophenanthrene (I),  $b_p$  155-60° (picrate, dark red, m 77-8°), in large scale expts there results some 2-methyl-3,4-dihydrophenanthrene (sym trinitrobenzene compd., yellow, m 155-7°).  $\text{H}_2\text{SO}_4$  in  $\text{AcOH}$  appears to convert I into an isomer (?), which gives chrysene with Se. The Grignard reagent from  $\beta$ -5-tetralylethyl chloride and cyclohexanone give 1-( $\beta$ -5'-tetralylethyl)cyclohexanol,  $b_p$  175-80° (3,5-dinitrobenzoate, yellow, m 124-5°).  $\text{KHSO}_4$  gives 1-( $\beta$ -5'-tetralylethyl)- $\Delta^1$ -cyclohexene,  $b_p$  140-50°. Cyclization gives a mixt of sat'd hydrocarbons, which, on complete dehydrogenation with Se or Pt black, yields chrysene and 4,5-benzohydroindene 1 spirocyclohexane,  $b_p$  140°, m 56-7° (picrate, orange, m 125-6°). The quantity of chrysene obtained shows that not more than 10% of the original mixt consists of dodecahydrochrysene and although octahydrochrysene was isolated after partial dehydrogenation, the small yield precluded the use of the method for preparative purposes. C J West

Tea leaves III Chem constitution of tannin in tea leaves (Oshima) 12 Homogenization with amines of some amine-water systems (Mertztin, Ust-Kachintzev) 2 Catalytic action of Japanese acid clay 1 Condensation of benzyl chloride and benzene (Kuwata) 2 Raman effect and org chemistry (Bourguet, Piaux) 3 Physicochem. studies of organometallic and furan compds (Cathin) 2 Catalysis with org fibers (Bredig, et al) 2 Reaction mechanism study—action of fused Na amide on mono-, di- and trimethylamines (Fry, Culp) 2 Quantum mechanics and kinetics of org chem reactions (Hindewood) 2 Dissoc const's and rotations of some  $\alpha$ -substituted ethylamines (Burch) 2

Oxygenated organic compounds from hydrocarbons Orlando R. Sweeney, Frank C. Vilbrandt, Henry H. Beeson and Howard A. Montgomery (to Hanlon-Buchanan, Inc.) U. S. 2,024,954, Dec. 17 For sepg constituents of a freshly prep'd. vapor-phase mixt of oxygenated org compds. contg aldehydes such as may be formed by the oxidation of a petroleum fraction, the vapors are brought into contact with a phenolic material such as  $\text{PhOH}$  with which at least one of the constituents, such as an aldehyde reacts to form a condensation product adapted for use as a plastic, and the remaining constituents are sepd. from the condensation product

Hydrating olefins Standard Oil Development Co. Fr. 793,041, Aug. 26, 1935. An olefin is caused to pass in a continuous manner into intimate contact with a catalyst in dil. aq. soln., in a series of steps, at a temp. below 350° and increased pressure. A partial pressure of water is maintained, by adding water, so as to prevent any appreciable concn. of the catalytic soln., the olefin being thus hydrated to form an alc., and the catalytic soln. is withdrawn continuously and the alc. recovered by distn. at a pressure below that at which the reaction is carried out. App. is described.

Hydrogenation of tertiary alkyl-phenols to form hydroaromatic alcohols Herman A. Brunson and Lloyd W. Covert (to Rohm & Haas Co.). U. S. 2,020,668, Jan. 7. Tertiary alkylated phenols the tertiary alkyl group of which contains at least 8 C atoms such as  $\alpha,\alpha,\gamma,\gamma$ -tetramethylbutylphenol, *tert* isododecylphenol, *tert*-isohexadecylphenol and the corresponding cresols, xylenols, resorcinols, guaiacols, and naphthols, obtainable, resp., from the tertiary olefins such as diisobutylene, triisobutylene, tetraisobutylene,  $\beta$ -dibutylene, and diisopentylene, by condensation with phenols in the presence of acidic catalysts, preferably  $\text{H}_2\text{SO}_4$ , upon treatment with H under high pressure and at elevated temps. in the presence of active, finely divided hydrogenation catalysts such as finely divided Ni, give the corresponding nuclear

tertiary alkyl hydroaromatic secondary alcs. They are colorless, practically odorless liquids or low-melting waxes which are insol. in water but readily sol. in petroleum oils, fatty glycerides and in most org. solvents, and are miscible and compatible with rubber, paraffin, natural and synthetic resins, cellulose esters and cellulose ethers. Because of their high b. ps, low vapor pressure, pale color, light-fastness, freedom from rancidity, they are useful as plasticizers for coating and molding compns., and as lubricants for textile fibers or for fine mechanisms such as watches. They are useful as intermediates for prep. long-chain *tert*-alkyl adipic acids by oxidation with  $\text{HNO}_3$ , for example, also in preparing esters, or as dye intermediates, as softening agents in rubber compns., and as ingredients for cosmetics, soaps, and insecticides.  $\alpha,\alpha,\gamma,\gamma$ -Tetramethylbutylcyclohexanol m 56° and  $b_p$  140-2°. *tert* isododecylcyclohexanol  $b_p$  180-200°. Mixed *tert*-isohexadecylcyclohexanols solidify from a viscous oily liquid to a waxy mass.  $\alpha,\alpha,\gamma,\gamma$ -Tetramethylbutylmethylcyclohexanol is a colorless viscous oil. Various examples with details are given.

Aldehydes I G Farbenind A.-G. (Ulrich Hoffmann, inventor) Ger. 618,972, Sept. 19, 1935 (Cl. 12a, 17 03). Addn to 528,822 1,2-Oxides are isomerized with more than two C atoms in the mol in the gaseous phase with the aid of a surface catalyst of silicate or  $\text{SiO}_2$  gel. The oxides may be used alone or mixed with chlorohydrins or glycols. Thus, propylene oxide vapor mixed with steam is heated over a  $\text{SiO}_2$  gel contact catalyst to 300° to give a 70% yield of propionaldehyde.

Ketones Rheumische Kampfer-Fabrik G. m. b. H. (Siegfried Skrap, inventor) Ger. 621,454, Nov. 7, 1935 (Cl. 12a 20 01). Ketones are produced when aromatic or heterocyclic compds., excepting compounds of the pyridine series, are strongly heated with org. acids or their anhydrides in the presence of a catalyst of large surface area. Thus,  $\text{C}_6\text{H}_5\text{I}$  and  $\text{BzOH}$ , boiled for 4 hrs. in the presence of an Al hydrosulfate, yield  $\beta$ - $\text{C}_6\text{H}_5\text{I}$ ,  $\text{COPH}$ , m 82°. Hydroxy ketones may be obtained either as described above or by heating the corresponding phenol esters under similar conditions. Thus, 1-hydroxy-2-benzoylnaphthalene, m 65°, is obtainable by heating either  $\alpha$ -naphthol and  $\text{BzOH}$  or  $\alpha$ -naphthyl benzoate to 230-250° in the presence of an Al hydrosulfate. Examples are given also of the prepn. of phenolphthalein from phthalic anhydride and  $\text{PhOH}$ , a phenylidihydrocoumarin from cinnamic acid and  $\text{PhOH}$ , a 1-aminonaphthyl phenyl ketone, m 80°, from  $\alpha$ -naphthylamine and  $\text{BzOH}$ , a benzoylindole, m 114°,  $b_p$  100-210°, from indole and  $\text{BzOH}$ , a benzoylthionaphthene, m. about 235°, from thionaphthene and  $\text{BzOH}$ , and a mixt. of butyrylthiophenes,  $b_p$  180°, from thiophene and  $\text{PrCOOH}$ .

Cyclic ketones Schering-Kahlbaum A.-G. Brit. 475,330, Sept. 19, 1935 Addn to 415,259 (C. A. 29, 813). This corresponds to Fr. 44,912 (C. A. 29, 6246).

Esters of hydroxy acids. Carbide & Carbon Chemicals Corp. Fr. 787,812, Sept. 30, 1935. The H of each carboxylic group of esters of hydroxy carboxylic acids is replaced by an alkoxy alc. radical, e. g., the radical of a monoalkyl ether of an alkylene glycol and more particularly of ethylene glycol or diethylene glycol and in each hydroxy group is substituted a radical of an aliphatic monocarboxylic acid. Examples are given of the prepn. of bis(butoxyethyl) tartrate diacetate,  $b_p$  236°, tris(methoxyethyl) citrate acetate, decomposes on heating and  $\beta$ -ethoxyethoxyethyl lactate butyrate.

Fatty acid esters of symmetrical dialkyl ethylene glycols Virgil L. Hansley (to E. I. du Pont de Nemours & Co.) U. S. 2,025,684, Dec. 21. Fatty acid esters, of a sym. dialkyl ethylene glycol having more than 12 C atoms per mol, such as the diacetate of sym. diundecylethylene glycol (m. about 34°), etc., are produced by reducing a fatty acid acylon (as by use of H under pressure in the presence of a Ni catalyst) to produce a glycol and then esterifying the glycol with an aliphatic acid. The acyloins of caprylic, lauric, myristic or stearic acid or the like may be used. Numerous examples with details of procedures are given.



**Monalkyl ethers of aromatic polyhydroxy compounds.** Karl Marx, Hans Wesche, Karl Bittner and Hans Baenger to General Aniline Works). U S 2,024,534, Dec. 17. 1 or the production of a monalkyl ether such as guaiacol, reaction is effected between an alkyl halide such as chloromethane and an anhyd acid alk earth metal salt of an aromatic polyhydroxy compd such as the acid Na salt of 1,2-dihydroxybenzene suspended in an inert org liquid such as xylene (suitably by heating to about 240°).

**Halogenated ethers.** Norman D Scott (to E. I. du Pont de Nemours & Co.) U S 2,024,749, Dec. 17. See Brit 423,520 (C A 29, 43747)

**Halogen compounds.** N V de Bataafsche Petroleum Maatschappij. Fr 787,520, Sept. 23, 1935. Products which may be used for purifying mineral oils and gases and for making resins are prepd by treating monohalides of the vinyl type with an aq soln of halogen, a hypo-halogenated acid, hydrogen halide or org hypohalites. Examples are given of the prepn of chlorobutane ( $\text{CH}_3\text{COCHClCH}_3$ , from 2-chloro-2-butene), chloroacetaldehyde ( $\text{CH}_2\text{ClCHO}$ , from vinyl chloride), *tert*-dichlorobutyl alc (from isocetyl chloride), 2,3,3-trichloro 2-methyl-1-phenylpropane, 3-chloro-2-(chloromethyl)-1-phenyl-3-propene and 1,2-dibromo-3-propanol.

**Polyhalogenated hydroxy compounds and hydrocarbons.** Herbert P A Groll and Geo Hearne (to N. V. de Bataafsche Petroleum Maatschappij). Brit 435,096, Sept. 13, 1935. Unsaturated monohalides contg at least 4 C atoms and the halogen linked to a satd C atom are caused to react with hypohalous acids, aq halogen solns, solns of the said acids and H halides, or alkyl or aralkyl hypohalites in the presence of  $\text{H}_2\text{O}$ , to give polyhalogenated compds. The unsatd halides and reaction product should not be permitted to come into contact with free or undissolved halogen, the soln of halogen in  $\text{H}_2\text{O}$  is therefore preferably effected in a sepg stage. Among 7 examples, (1) a cyclic system comprising reaction chamber, a separator stage for breaking up emulsions in the reaction products and an absorption column for satg circulating  $\text{H}_2\text{O}$  with Cl is employed, the system is charged with  $\text{H}_2\text{O}$ ,  $\text{CCl}_4$  and *n*-butenyl chloride (I). These are agitated in the reaction chamber and the  $\text{H}_2\text{O}$  circulated through the system, Cl being introduced into the column continuously and I into the reaction chamber.  $\text{H}_2\text{O}$  is introduced to maintain the HCl concn at 0.7 N. A portion of the circulating liquid is intermittently withdrawn to maintain the vol const. The product is sepd into 2 layers and the non-aq phase, together with an  $\text{Et}_2\text{O}$  ext of the other, distd to remove  $\text{Et}_2\text{O}$  and  $\text{CCl}_4$ . The main product is a dichloro-*tert* butyl alc. and (2) isopentenyl chloride is treated with 0.5 N  $\text{HClO}_4$  to give a dichloro-*tert*-amyl alc.

**Halogenated indanthrenes.** James Ogilvie (to National Aniline & Chemical Co.) U S 2,026,647, Jan. 7. In the halogenation of an indanthrene, as in the production of a mono- or poly-halogenated deriv, Cl or Br is caused to react with a suspension of the starting material, such as indanthrene, in a chlorobenzene such as  $\text{C}_6\text{H}_5\text{Cl}$ .

**Purifying chlorinated hydrocarbons.** Edgar C Britton, Gerald H Coleman, John W Zimba and Ldwrd C Zuckerman (to Dow Chemical Co.) U S 2,025,924, Dec. 24. A chlorinated aliphatic hydrocarbon material such as  $\text{C}_2\text{Cl}_4$  is freed from impurities such as unstable toxic compds which cannot be removed by washing with an aq alk soln by treating the material with  $\text{NH}_3$  or a primary amine such as triethylamine, a secondary amine contg not more than one aryl substituent linked with the basic N atom, a tertiary alkyl amine, a satd ring N base, or a pyridine base, and then fractionally distg.

**Methyl chloride.** Hans Klein and Conrad Pfandlfer (to I. G. Farbenindustrie A.-G.) U S 2,026,131, Dec. 31. See Fr 788,481 (C A 29, 4809)

**Halogenated acyclic hydrocarbon derivatives containing fluorine.** Herbert W Daudt, Mortimer A Youker and Harry H Reynolds (to Kinetic Chemicals, Inc.) U S 2,024,095, Dec. 10. In fluorinating a halogenated acyclic hydrocarbon deriv. contg a halogen other than F, with substantially anhyd  $\text{HF}$ , the reaction is effected in the presence of an Sb halide such as a fluorochloride

1 contg a controlled and substantially const F content (as by use of an Sb fluorochloride contg F 4-13% in producing  $\text{CF}_3\text{Cl}$  from  $\text{HF}$  and  $\text{CCl}_4$  at a temp. of about 45-95°)

**Organic fluorine compounds.** I. G. Farbenindustrie A.-G. Fr 786,112, Aug. 27, 1935.  $\text{HF}$  is caused to act, if necessary in the presence of an inert diluent, on compds contg at least 3 C atoms and at least 1 triple bond. Thus, 9,10-difluorostearic acid, m 70-4°, is prepd from stearic acid in  $\text{CH}_2\text{Cl}_2$ .

**Chlorination derivatives of naphthalene.** Ernest R Hanson and Sanford Brown (to Haloway Corp.). U S 2,025,742, Dec. 31. A liquid neutral Cl substitution product of naphthalene which exhibits no cloudiness and is transparent at -5° and contains up to 30% of Cl is produced by distg a liquid impure chlorinated naphthalene having a sp. gr. of 1.25 or less at 150° under a pressure of 35 mm Hg or less and collecting a fraction having a color lighter than 2.

**Dehalogenating organic halides.** Herrick R Arnold and Wilbur A. Laner (to E. I. du Pont de Nemours & Co.). U S 2,025,032, Dec. 24. Reactions such as the production of  $\text{C}_2\text{H}_4$  from  $\text{CH}_2\text{Cl}_2$  are effected by the action of an excess of H in the presence of a catalyst essentially comprising a sulfide of a metal from group 6, sub-group A of the periodic system such as Mo and Cr sulfides at a temp. of 200-600° (suitably about 400° under 200 atm pressure). Several examples are given.

**Trialkyl phosphates.** Consortium fur elektrochemische industrie G m b H. Fr 787,438, Sept. 23, 1935. See Brit. 433,927 (C. A. 30, 7329)

**Aliphatic sulfur compounds.** Firm. Alexander & Posnansky. Fr 787,810, Sept. 30, 1935. S is combined with aliphatic compds. contg unsatd bonds by submitting them to the simultaneous action of  $\text{H}_2\text{S}$  and S. A basic substance such as  $\text{NH}_3$ , hydrazine, amines, piperidine, substituted guanidines and thioam sulfides may be used as a carrier for the S. The process may be used for vulcanizing rubber and for making a clear solid or liquid facts. If polysulfides of H are used, e. g., in making facts, a min amt. of a stabilizing substance such as a chlorinated aliphatic hydrocarbon is added.

**Organic disulfides.** I. G. Farbenindustrie A.-G. Brit 435,113, Sept. 16, 1935. See Fr. 772,002 (C. A. 29, 14369).

**Organic disulfides from mercaptobenzothiazoles.** Silesta, Verein Chemischer Fabriken. Fr 786,126, Aug. 27, 1935. See Ger 613,068 (C. A. 29, 54609)

**Anthraquinone compounds and dyes.** Soc pour l'ind. chim. & Bâle. Fr 786,105, Aug. 27, 1935. Heterocyclic compds of the bianthraquinonyl and dianthrene series are prepd by treating bianthraquinonyl compds, which contain in the 2 position at least 1  $\text{NH}_2$  group or group capable of being transformed to  $\text{NH}_2$  by a sapon agent, if necessary after treatment with reducing agent and if necessary after subsequent oxidation, with condensing agents so that both 2 positions are joined by a bridge contg N. The products may be halogenated, and if compds acylated in the 2 position are used the acyl group may be sapon before or during the condensation. Examples are given of the prepn of 2-phthalimido-1,1'-bianthraquinonyl (I), 2-phthalimido-(II) (by treating I with Cu in  $\text{H}_2\text{SO}_4$  at a low temp.), 2-phthalimido-2'-methyl-, 2,2'-diphthalimido-, 2-amino-, 2,2'-diamino- and 2,2'-amino-meso-benzodanthrone, 2-phthalimido- (by oxidizing II with  $\text{MnO}_2$ ), 2-phthalimido-2'-methyl- and 2,2'-amino-meso-naphthodanthrone and an *allo*-pyridino-meso-benzodanthrone.

**Diazo compounds.** I. G. Farbenindustrie A.-G. Brit 434,725, Sept. 9, 1935. See Ger 611,299 (C. A. 29, 40247).

**Water-soluble diazomino compounds.** Miles A. Dahlen (to E. I. du Pont de Nemours & Co.) U S 2,025,095, Dec. 24. Compds capable of stabilizing diazotized primary arylamines against reaction with typical coupling components in a neutral or alk. medium are formed by coupling a primary arylamine which may contain in the aryl nucleus one or several N-oxochrome substituents.

but which does not contain a S-auxochrome, through the diazo group to the N of an isonitrobenzene-mono- or -di-sulfonic acid in a neutral or alk. medium, as by coupling diazotized 4-chloro-2-aminoanisole through the azo group to N of isonitrobenzenesulfonic acid. Numerous examples and details of procedure are given.

**Hydrogenating phenolic compounds** Yorkshire Tar Distillers Ltd and Edward B Maxted. Brit 435,102, Sept. 13, 1935. In the conversion of cresols and other phenolic compds into hydrocarbons by treatment with H<sub>2</sub>, a catalyst consisting of, or contg., a metallic thiomolybdate, e. g., of Co, Cr, Sn or Bi, is used. The use of thiomolybdates contg. a volatile cation, and of other thiomolybdates that are converted into sulfides at the reaction temp., is disclaimed.

**Quaternary ammonium compounds** I G Farbenindustrie A.-G. Brit 434,602, Sept. 5, 1935. See Fr 769,444 (C A 29, 30389).

**Benzanthrone derivatives** E I du Pont de Nemours & Co. Brit 435,321, Sept. 19, 1935. See U S 1,965,855 (C A 28, 54714).

**Dibenzanthrone derivatives** I G Farbenindustrie A.-G. (Karl Köberle, Hugo Wolff and Emil Krauch, inventors). Ger 621,475, Nov. 7, 1935 (Cl 225 3 00). Dibenzanthrone or a substitution product is treated at 20–90° with aq. HNO<sub>3</sub> of 10–75% concn, preferably 35–60%. Nitrodibenzanthrones free from OH compds are obtained. Sp. processes are described.

**Bs-2-Azabenzanthrone derivatives** I G Farbenindustrie A.-G. (Friedrich Ebel and Otto Bayer, inventors). Ger 621,455, Nov. 7, 1935 (Cl 12p 5). Compds of the formula ACONHC(H<sub>2</sub>)COOR, where A is 1-anthraquinonyl and R is H or an alkyl or aryl group, are heated alone or in the presence of a condensing agent, e. g., Ac<sub>2</sub>O, NaOH, AlCl<sub>3</sub> or silica gel. The product is Bs-1-hydroxy-Bs-2-azabenzanthrone, m. 334°, or a deriv thereof in which the COOR group from the starting material is substituted in the Bs-3-position. Mixts of these products may also be obtained. Sp. processes are described. The starting materials are obtainable by condensing anthraquinone-1-carboxylic halides with glycine or its esters, and details are given of the prepn of 1-anthraquinonylglycine Et ester, m. 187–9°, and the free acid, m. 257°.

**Phthaloylfluorene derivatives** I G Farbenindustrie A.-G. (Paul Nawatsky and Rudolf Robl, inventors). Ger 621,474, Nov. 7, 1935 (Cl 225 2 07). *In*-Phthaloylfluorene (I) (C A 28, 10377), or a substitution product unaltered at the CH<sub>2</sub> group, is treated with an aldehyde or a ketone or a reagent which reacts like an aldehyde or a ketone under the reaction conditions. The reaction may be effected by boiling the reagents together, if desired in a solvent, e. g., pyridine. Products of unspecified constitution, useful as vat dyes or intermediates, are obtained. Examples are given of the manuf. of products from I and BzH<sub>2</sub> (or PhCHCl<sub>2</sub>), PhCOPh, anthraquinone-2-aldehyde and its 1-chloro-, 1-bromo- and 1-amino derivs., and glyoxal sulfate.

**Azo-, amino- and acylamino-N-nitroamines** Société pour l'ind. chim. à Bâle. Brit 434,917, Sept. 11, 1935. N-Nitroamines of primary aromatic amines of the C<sub>6</sub>H<sub>5</sub> series, nitrated in the nucleus, are treated with reducing agent in neutral or alk. medium so that reduction of the nuclear NO<sub>2</sub> occurs in stages to the azo or amino compd, the latter of which may be further acylated. Among examples, the Na salts of the nitroamine of 1-amino-4-nitro-2,5-dimethylbenzene, obtainable by treating this compd. with NaNO<sub>2</sub> and oxidation with hypochlorite, and the nitroamine of 1-amino-2,5-dichloro-4-nitrobenzene are reduced in alk. soln with Na<sub>2</sub>S to give the corresponding amino-mono-N-nitroamino compds., the N-nitroamine of 1-amino-2-methyl-1-nitrobenzene is reduced by Zn dust in NaOH to give the dinitroamine of 4,4'-diamino-3,3'-dimethylazobenzene. The last compd. may be printed with 2,3-hydroxyphenothic anilide and nitrite and developed with acid to give a brownish violet dye; the 1,4-amino-N-nitroamines may also be mono- and tetra-azotized, the 1-N-nitroamino-2-methyl-4-amino-2,5-dimethylbenzene (II) giving, on diazotization and printing as

above, a red-brown tint on cotton and, with the use of double quantity of nitrite and development, a blue print. 1-Nitro-5-methylbenzotriazole is obtained from I (prepd. as above) on treatment with NaNO<sub>2</sub> and dil. acid. The benzoylation of 1-N-nitroamino-4-amino-2,5-diethoxybenzene is also described.

**Bromomethylbenzanthrones** Soc. pour l'ind. chim. à Bâle. Fr 787,175, Sept. 18, 1935. Br is caused to react in the presence of H<sub>2</sub>SO<sub>4</sub> and water (the water being at least 5 and not more than 20%) on 2-, 5-, or 6-methylbenzanthrone or the mixt of methylbenzanthrones obtained after eliminating the 2-methylbenzanthrone from the mixt of methylbenzanthrones prepd. from 2-methylanthraquinone.

**Terpenes** Soc. alsacienne de produits chimiques. Fr 780,437, Sept. 3, 1935. Mixts of bornyl and fenchyl ethers contg. terpenes and resulting from the etherification of pinene by AcOH are submitted to direct sapon, the AcONa formed is sepd. by decanting and boric acid is added to form bornyl and fenchyl borates, the terpenes remaining unchanged. The terpenes are removed by distn and the borates are sapond with water.

**Xanthates** Wilhelm Hirschkind (to Great Western Electro-Chemical Co.) U S 2,024,023, Dec. 17, 1935. In the production of xanthates such as Na ethyl xanthate, K ethyl xanthate, K butyl xanthate or Na amyl xanthate, if the alkali metal alcoholate used as initial material for reaction with CS<sub>2</sub> is free from water the reaction proceeds approx. quantitatively without formation of any considerable quantity of by-products even without cooling but permitting the reaction temp. to rise even to about 80°. Several examples are given. U S 2,021,925 relates to similar production of xanthates from anhyd. alkali metal alcoholates such as those of tertiary, secondary or dihydric alcs., fatty alcs., terpenes, etc., by reaction with CS<sub>2</sub> without need of cooling. U S 2,024,024 (Wilhelm Hirschkind, Wm D Ramage and Harry Bender (to Great Western Electro-Chemical Co.)) relates to xanthate production by reaction of an anhyd. alcoholate with CS<sub>2</sub> in the presence of an unhydrated desiccant such as anhyd. Na<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>CO<sub>3</sub>, CaO, CaSO<sub>4</sub>, or Na<sub>3</sub>PO<sub>4</sub>, from which the xanthate formed can be subsequently leached with a solvent such as C<sub>6</sub>H<sub>6</sub> or toluene. Various examples are given.

**Organic acids from alcohols and carbon monoxide**. John C. Woodhouse (to E. I. du Pont de Nemours & Co.) U S 2,025,676, Dec. 24, 1935. Production of an acid such as HOAc from an alc. such as MeOH is effected in the presence of a catalyst comprising a halogenated aliphatic monocarboxylic acid such as monochloroacetic acid or an aliphatic acyl halide such as acetyl chloride by heating under pressure and in contact with activated charcoal. U S 2,025,677 relates to the generally similar production of acids such as propionic, butyric or valeric acid from olefinic hydrocarbons such as C<sub>3</sub>H<sub>6</sub>, C<sub>4</sub>H<sub>6</sub> or C<sub>5</sub>H<sub>6</sub> by reaction with steam and CO in the presence of similar catalysts.

**Polyhydroxy carboxylic acids** I Hoffmann-La Roche & Co. A.-G. Ger. 618,907, Sept. 19, 1935 (Cl. 12a. 11). Uronic acids, their esters, uronides, or salts are treated with H under pressure at temps. not exceeding 140°, in the presence of usual hydrogenation catalysts. Thus, an aq. soln. of d-galacturonic acid is given an addn. of an alc. suspension of Ni-kieselguhr gel catalyst and treated with H in an autoclave to give l-galactonic acid. Similarly d-glucuronic acid gives l-gluconic acid. Other examples and structural formulas are given.

**Lactic acid** Geza Braun (to Standard Brands Inc.) U S 2,024,565, Dec. 17, 1935. A suitable carbohydrate material such as sucrose is heated with a strong alkali such as Ca(OH)<sub>2</sub> at a temp. of 200–250° and under a pressure of at least 20 atm. Ca lactate formed may be treated with ZnSO<sub>4</sub> to form Zn lactate from which free lactic acid may be recovered. Various details of procedure are described.

**p-Diketocamphanecarboxylic acid and hydroxyketocamphanecarboxylic acid** Kenzo Tamura, Gyoikuji Kihara, Yasuhiko Asahina and Morizo Ishidate. U S 2,026,280, Dec. 31, 1935. A compn. contg. these compds. is

produced by introducing  $\text{CO}_2$  into  $\beta$ -diketocamphane in the presence of an alkali metal such as Na

1,3 Dibromo-2-aminoanthraquinone-6- or -7-sulfonic acid Georg Kranzlein, Martin Corell and Wilhelm Schösch (to General Aniline Works) U S 2,025,169, Dec 24 2-Aminoanthraquinone is disulfonated by heating to about  $150^\circ$  with  $\text{H}_2\text{SO}_4$  contg  $\text{SO}_3$ , and a dibromo deriv. is produced by treating the product, in aq soln., with Br at about  $40-50^\circ$  U S 2,025,170 relates to the production of 2-amino-3-bromoanthraquinone-6- or -7-sulfonic acid by heating 2-amino-3-bromoanthraquinone with  $\text{H}_2\text{SO}_4$  contg  $\text{SO}_3$  at a temp. of  $125-30^\circ$ . These products may be used as dye intermediates

Acetylene from methane and gases containing methane. Martin Banck (to Ruhrchemie A.-G.) U S reissue 19,794, Dec 24 A reissue of original pat. No. 1,773,611 (C A 24, 4994)

Acetaldehyde from acetylene I G Farbenind. A.-G. (Walter Rosinsky and Paul Baumann, inventors) Ger 621,202, Nov 4, 1935 (Cl 12a 7 02). Addn to 620,402 (C A 30, 737) See Brit 393,690 (C A 27, 5751-2).

Dimethylamine from monomethylamine Commercial Solvents Corp Ger 621,214, Nov 1, 1935 (Cl 12q 1 01). See L S 1,926,691 (C A 27, 5756)

Trimethylamine I G Farbenindustrie A.-G. Brit 435,200, Sept 17, 1935 See Fr 752,212 (C A 29, 6907) and Ger 615,527 (C A 29, 8007)

Trimethylamine, trimethylamine I G Farbenindustrie

1 A.-G. Brit. 435,404, Sept. 19, 1935  $\text{NMMe}_2$  and  $\text{NEt}_3$  are obtained by passing vaporized  $\text{NHMe}_2$  or  $\text{NH}_3$  over a dehydrating catalyst at raised temp. and preferably at raised pressure. Among examples,  $\text{NHMe}_2$  is passed at  $380^\circ$  and 200 atm. over alumina gel and the products are condensed under 10 atm. pressure, a mixt. of  $\text{NH}_3$  and methylamines contg 61% of  $\text{NMMe}_2$  is obtained and is sep'd by fractional distn. under pressure

Vinyl alcohol Chemische Forschungsgesellschaft m. b. H. Brit. 434,580, Sept. 4, 1935 This corresponds to 1r 781,503 (C A 29, 6907)

Ketene Consortium für elektrochemische Industrie G. m. b. H. Brit. 435,210, Sept. 17, 1935 See Fr. 747,483 (C A 29, 4329)

$\alpha$ -Naphthol Wm J. Cotton (to National Aniline & Chemical Co.) U S 2,025,197, Dec. 24 Naphthalene is sulfonated under such conditions that there is produced a reaction mass contg cryd. 1-naphthalenesulfonic acid and  $\text{H}_2\text{SO}_4$ , but contg less than 2% of 2-naphthalenesulfonic acid, the cryd. 1-naphthalenesulfonic acid is sep'd and fused with caustic alkali

2-Amino-1,1'-bianthraquinonyl Soc. pour l'ind. chim. à Biele. Fr 786,363, Sept. 2, 1935 An almost equimol. mixt. of 1-halo-2-acylaminoanthraquinone and 1-haloanthraquinone is submitted to a treatment with dehalogenating agents and the products thus obtained are treated with saponig. agents.

## 11—BIOLOGICAL CHEMISTRY

PAUL E. ROWE

### A—GENERAL

ARTHUR W. DOX

The protein of Bence-Jones L. Meyler *Nederl. Ind. d. Geneeskunde*, 79, 5712-15 (1935) —Bence-Jones protein, found in the urine in myeloma, ppt's at  $60^\circ$  and redissolves at  $100^\circ$  M preps a protein with similar properties from bone marrow The fluid pressed from the bone marrow of calves is mixed with 2 vols  $\text{EtOH}$ , the ppt is ext'd with water and filtered To the clear filtrate, which contains 0.3 to 0.4% protein, some  $\text{NaCl}$  and  $\text{AcOH}$  are added This soln., when heated to  $60^\circ$ , turns turbid, when heated to  $100^\circ$  it becomes clear It is ppt'd by  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{AcOH}$ , sulfosalicylic acid and picric acid, two parts of sat'd  $(\text{NH}_4)_2\text{SO}_4$  will ppt it, also an excess of sat'd  $\text{NaCl}$  in acid soln. It gives pos. biuret-, Millon- and xanthoproteic reactions Moreover, the protein from bone marrow, like Bence-Jones protein, is more readily dissolved at  $100^\circ$ , if, in an acid soln., a salt is added,  $\text{CaCl}_2$  is more effective than  $\text{NaCl}$ , also  $\text{Na}_2\text{SO}_4$ ,  $\text{Na}$  citrate and urea have this action After injection of the bone-marrow filtrate into rabbits, Bence-Jones protein appears in the urine, although it is sometimes difficult to detect Since Bence-Jones protein has been found in the urine in leucemia and emphysema, it also ext'd pos. obtained from emphysema, and lymphocytes from leucemic patients, using the same technic as for bone marrow In this case also, a protein with properties like those of Bence-Jones was found Protein from other sources, or various other organs, when ext'd., fails to show this phenomenon R. Beutner

Action of amylases on starch H. C. Gore *Ind. Eng. Chem.* 28, 86-8 (1936) —A discussion of the present status of the problem of the action of amylases on starch A brief description is given of the 3 types of diastatic activity of plant amylases These 3 types are discussed under the general headings of "saccharogenesis" or attack on raw starch, "liquefying power" and "saccharification" or Lintner activity Three quant. methods for measuring diastatic activity are contrasted Seventeen references F. L. Dunlap

Advances in the colloidal chemistry of protoplasm during the last ten years. I. W. W. Lepeschkin *Protoplasma* 26, 470-94 (1935) —Eighty six references F. L. D.

Constitution of cytochrome C Jean Roche and Marie T. Bénévise *Bull. soc. chim. biol.* 17, 1473-93 (1935) —Hemin from hog blood was repeatedly reduced by  $\text{Na}_2\text{SO}_3$  and oxidized by  $\text{K}_3\text{Fe}(\text{CN})_6$ , forming "hematin C" Cytochrome C was prep'd from beef yeast. The absorption spectra of the pyridine hemochromogens derived from cytochrome C and hematin C are almost identical Absorption spectra of several related compds. were also det'd Hematin C, unlike other hematin, does not combine with globins to form methemoglobins It does unite with globins to form parahematin, which yield hemochromogens when reduced None of the hemochromogens of hematin C, protein or nonprotein, exhibits the non-autoxidizability characteristic of cytochrome C. Fourteen references L. E. Gilson

The initial phosphorylations of glycogen J. K. Parasz and T. Baranowski *Compt. rend. soc. biol.* 129, 397-10 (1935), cf. C. A. 29, 8012 —Expts with muscle ext. showed that the free  $\text{H}_2\text{PO}_4$  groups liberated during the fission of adenosinetriphosphoric acid (I) attach themselves by preference to the glycogen but some are hydrated to form free phosphate Fructosediphosphate ester is formed in the reaction between glycogen and I. and this reaction proceeds independently of the simultaneous esterification of the glycogen by the free phosphate The last-named reaction produces only monophosphate ester. L. E. Gilson

Spectrophotographic determination of phenol in different media G. Barac *Compt. rend. soc. biol.* 129, 520-2 (1935), cf. C. A. 29, 1117 —A very dil. soln. of  $\text{PhOH}$  in  $\text{Et}_2\text{O}$  shows 3 narrow absorption bands at 2670, 2740 and 2807 Å The mol. extinction coeff. in  $\text{Et}_2\text{O}$  is 2400 for 2807 Å The Beer-Lambert law holds good to within 1% for such solns  $\text{PhOH}$  was added to blood and plasma, the mixts were defecated with  $\text{H}_2\text{WO}_4$  and an almost quant. recovery of the  $\text{PhOH}$  was made by extg. the filtrates with  $\text{Et}_2\text{O}$ . State of phenol added to blood *Ibid.* 522-3 — $\text{PhOH}$  added to whole blood divides itself equally between plasma and corpuscles When added to the sep'd corpuscles 80% can be recovered after defecation with  $\text{H}_2\text{WO}_4$  and 100% after defecation with  $\text{CCl}_4\text{-CO}_2\text{H}$ , showing that it is not destroyed by the corpuscles  $\text{PhOH}$  is not fixed by the lipides of the plasma and can be sep'd from plasma by ultrafiltration L. E. Gilson

Inactivation of adrenaline by methylglyoxal, glyceraldehyde and acetaldehyde. J. Toscano Rico and A. Malafaya Baptista. *Compt. rend. soc. biol.* 120, 545-6 (1935); cf. *C. A.* 29, 4385<sup>1</sup>, 30, 516<sup>1</sup>.—At  $pH$  6.6 or above a 1/50,000 soln. of adrenaline was inactivated by methylglyoxal in concns. greater than 0.04 M, by glyceraldehyde at 0.04 M but not at 0.01 M, and by AcCl at 0.0025 M concn. The Na salts of phosphoglyceric and pyruvic acids had no inactivating effect. The inactivation of adrenaline by acetaldehyde verified by experiments with various organs containing smooth muscle. A. Malafaya Baptista. *Ibid.* 547-50. L. F. G.

Action of magnesium on the aspartase system. Kurt P. Jacobson and Fernando Belo Pereira. *Compt. rend. soc. biol.* 120, 551-4 (1935).—The enzymic formation of aspartic acid from fumaric acid and  $NH_4$  salts is discussed. When a nonproliferating culture of *Escherichia* was used as a source of aspartase the reaction fumaric acid  $\rightleftharpoons$  aspartic acid reached a definite equil. The addn. of a Mg salt to 0.1 M concn. caused a considerable shift of the equil. point toward the fumaric acid side. L. F. G.

Absence of any correlation between the lactoficification reaction and protein unbalance of [human] blood serum. G. Lefrou and L. Aulfrét. *Compt. rend. soc. biol.* 120, 514-16 (1935). L. E. Gilson

Swelling of structured proteins. The influence of the reticular tissue on the swelling of collagen in water and hydrochloric acid. Dorothy Jordan-Lloyd and R. H. Marriott. *Proc. Roy. Soc. (London)*, B118, 439-45 (1935), cf. *C. A.* 29, 949<sup>1</sup>.—The reticular tissue, which invests the collagen fibers in the tendons in the tail of the rat, undergoes mech. rupture and rolls back to form reticular garters or rings when the collagen fibers swell and contract in length. Young tendons usually swell less than adult tendons. In HCl solns. with  $pH$  less than 2, young and adult tendons swell similarly, and to a greater extent than anticipated from their behavior in more dil. solns. Joseph S. Heplurn

Bound water and phase equilibria in protein systems. egg albumin and muscle. T. Moran. *Proc. Roy. Soc. (London)* B118, 548-59 (1935).—The chemically bound  $H_2O$  is approx. the same (0.26 g. per g. of protein) in native egg albumin and in egg albumin denatured by heat and by urea; the  $H_2O$  held more loosely at high activities is less in the denatured protein. The eutectic point of muscle is approx.  $-37.5^\circ$ ; its bound  $H_2O$  is small, and is 0.40 g. per g. of dry solids at intermediate and high activities of  $H_2O$ . Joseph S. Heplurn

Oxidation of hemoglobin to methemoglobin by oxygen. II. The relation between the rate of oxidation and the partial pressure of oxygen. J. Brooks. *Proc. Roy. Soc. (London)* B118, 660-77 (1935), cf. *C. A.* 25, 5903<sup>1</sup>.—The oxidation of hemoglobin to methemoglobin at  $pH$  6.69 and a temp. of  $30^\circ$  was unimol. with respect to substrate concn. The only detectable reaction was formation of methemoglobin. The rate of oxidation was proportional to the concn. of reduced hemoglobin and to a function of the O pressure. Apparently O reacted at the same rate with all unoxxygenated ferro-radicals in the system. Joseph S. Heplurn

The lipochrome of higher animals and of man. L. Zechmeister and P. Tuzson. *Naturwissenschaften* 23, 840-5 (1935).—A review of the chem. and phys. properties of vegetable lipochromes found in animal tissue, methods for their isolation, course of decomposition or deposition in the animal metabolism, etc. B. J. C. van der Hoeven

The active group of heparin. Sune Bergström. *Naturwissenschaften* 23, 706 (1935).—The heparin substance active in inhibiting blood clotting is a chondroitinpolysulfuric acid. Sulfuric acid esters of several polysaccharides were tested for heparin action. cellulose, chitin, starch, glycogen, gum arabic, yeast, nucleic acid and chondroitin sulfuric acid were heated with chlorosulfonic acid in pyridine and treated by the method of Gebauer, et al. (*C. A.* 24, 1501<sup>1</sup>). All preps. showed a heparin effect; the one from cellulose (3 sulfate groups) had an activity equal to that of com. heparin preps. Evidently natural heparin is active through its sulfate group, like-

wise its metachromatic violet color with toluidine blue depends on it (Lison, *C. A.* 29, 4033<sup>1</sup>). Thymonucleic acid has no heparin effect; the polysaccharide sulfuric acid from *Chondrus crispus* is active. The cellulose deriv. is toxic, 200 mg. causes pleural hemorrhage in rabbits.

B. J. C. van der Hoeven  
Heavy hydrogen in biology. G. v. Hevesy. *Naturwissenschaften* 23, 775-80 (1935).—A review of expts. on the behavior of  $D_2O$  in living organisms: algae growth, frog life, development of frog eggs,  $D_2O$  as indicator for  $H_2O$  metabolism, diffusion through tissue, etc.

B. J. C. van der Hoeven  
Transformation of rutin into brown pigment by tobacco enzymes. C. Neuberg and M. Kolbe. *Naturwissenschaften* 23, 800-1 (1935).—The slightly sol. rutin (quercetin-rhamnogluconide) is transformed by tobacco enzyme into a sol. brown pigment, one of these products, a sol. glucoside, hydrolyzed by HCl, gives a slightly sol. aglucon. The expts. were made with a 0.5-1.0% suspension of tobacco leaves or a 1:10 H<sub>2</sub>O dry prepn. of fresh leaves with 0.1-0.2 g. rutin per l. The brown coloration is intensified by using 0.003%  $H_2O_2$  instead of  $H_2O$ . Under those conditions the insol. rutin disappears in a few min. Boiled enzyme is inactive.

B. J. C. van der Hoeven  
The enzymic activity of egg white—its bearing on the problem of watery whites. I. van Manen and Claude Rimmington. *Onderstepoort J. Vet. Sci.* 5, 329-41 (1935).—The enzymic activity of solns. of thick and thin albumin from normal and watery egg whites was studied, by use of a microtitration method to det. peptide cleavage in terms of the increase in amino N. Thick or thin albumin alone at  $pH$  values ranging from 5.5 to 8.5 undergoes no autolysis at  $37^\circ$  and protein-splitting enzymes cannot be detected at any  $pH$  value by the addn. of gelatin as a substrate. The presence in egg white of at least 2 crepsin-like enzymes having different  $pI$  optima (approx. 5.5 and 7.0-8.0) was demonstrated. It seems unlikely that these enzymes play any part in the development of watery whites. The work of Balls and Swenson (cf. *C. A.* 28, 3493<sup>1</sup>) is criticized on account of the insufficient sensitivity of the method employed and the fact that it is such as would fail to distinguish between an increase in acid groups due to lypolytic and due to proteolytic activity. Moreover, the use of casein as a substrate is incapable of differentiating protease from creptic activity. K. D. Jacob

Invertase. II. Purification of invertase by adsorption methods. Noboru Taketomi. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 579 (1935); cf. *C. A.* 28, 6738<sup>1</sup>.—The expts. were made with an aq. ext. of takadiastase and 3 other enzyme solns. purified by different methods. As adsorbents the following were used: kaolin (I), kiesguhr (II), silica gel (III), Japanese acid clay (IV), activated clay (V), cholesterol (VI),  $Al(OH)_3$  (VII) and active carbons (VIII). Invertase was scarcely adsorbed by I, II and III, and only slightly more by IV. V adsorbed it well and can be used for the purification. When a suitable amt. of V was mixed at  $30^\circ$  with the enzyme soln. contg. about 25 vol. % EtOH, invertase was adsorbed and most of the amylase present in the liquor. The 2 enzymes can be sep'd in this way. VI adsorbed invertase, amylase and proteins quite well, but not selectively. The adsorbed enzymes were readily sep'd. from the adsorbent by dissolving VI in  $H_2O$ . VII behaved like VI in regard to adsorption. VIII showed varying adsorptive powers. Coloring matter could be removed from the enzyme soln. by a pretreatment with a small amt. of C, while invertase and amylase were scarcely adsorbed. The latter could then both be completely adsorbed by adding a suitable amt. of active C, but a sep'n. of invertase and amylase by this active C adsorption method was difficult. K. K.

A potentiometric study of hepatoflavins. F. J. Stare. *J. Biol. Chem.* 112, 223-9 (1935).—The oxidation-reduction potentials of hepatoflavins have been measured at  $pH$  values from 0.32 to 13.38 by titration with reducing agents. Definite branching of the titration curves in the more acid regions into 2 1-valent curves is indicated. Graphic analysis indicates ionization consts. at  $pH$  values

about 6.8 in the reductant and about 9.6 in the oxidant. An  $E_0$  value of 0.188 v. was obtained which is rather close to the value reported for lactoflavin. A. P. Lothrop

A crystalline by-product obtained in the large scale extraction of theelin and theolol. A. W. Dox, W. G. Bywater and F. H. Tendick. *J. Biol. Chem.* 112, 425-30 (1935).—A hitherto unknown constituent of pregnancy urine has been sepd. from the crude ext. in the concn. extn. of theelin and theolol. The cryst. substance,  $C_{21}H_{29}O_5N_3$ , is alkali-sol., resistant to hydrolysis and gives no reaction for proteins, amino acids or purines. Three active H atoms are present, 1 of which is easily ethylated to give a monoethyl, alkali-insol. deriv. An acid,  $C_{21}H_{27}O_5N_3$ , results from oxidation with chromic acid. The substance shows no hormone activity and is toxic to mice in doses of 0.0055 g. per g. of body wt. A. P. Lothrop

Photochemical processes in biology. A critical review. I. The principal photochemical reactions and the mechanism of their production. Giovanni De Toni. *Biochim. terap. sper.* 22, 547-55 (1935). A. E. M.

Enzymic synthesis of phosphocreatine by de-esterification of phosphopyruvic acid. H. Lehmann. *Biochem. Z.* 281, 271-91 (1935).—The synthesis of phosphocreatine from phosphoglyceric acid, discovered by Parnas in muscle poisoned with  $CH_3CO_2H$ , has been studied on dialyzed muscle exts. This occurs especially well in old, long-dialyzed exts., in which there is no more spontaneous dephosphorylation, but only in the presence of adenylic acid or of adenosinetriphosphoric acid (ATP). The re-esterification takes place in 2 stages: (a) Phosphopyruvic + adenylic acid  $\rightarrow$  ATP + pyruvic acid, (b) ATP + creatine  $\rightarrow$  phosphocreatine + adenylic acid. The adenylic acid thus acts as a catalyst and promotes the esterification of 100 times its quantity of creatine. The Mg ion is necessary for both stages. Considerable amts. of phosphocreatine can thus be prepd. in the pure state. Phosphopyruvic acid is more effective for this reaction than phosphoglyceric acid. The same can be accomplished in synthesis of arginine phosphate in crab muscles. The synthesis of phosphocreatine with ATP and with adenylic acid forms a reversible system, the equil. depending upon the H ion concn., excess of creatine and the removal of adenylic acid. In an alk. medium phosphocreatine accumulates and ATP disappears, but in a neutral reaction the process is reversed, and this can be changed again by making the reaction alk. S. Morgulis

Energy relations in enzymic lactic acid formation and in the synthesis of phosphagen. O. Meyerhof and W. Schulz. *Biochem. Z.* 281, 292-305 (1935).—The anaerobic heat restitution in active muscle consists of the difference between the exothermic lactic acid formation and the endothermic phosphocreatine synthesis. If 2 mols. phosphocreatine is synthesized for 1 mol. lactic acid, this would be represented by -22,000 g.-cal. and +16,000 g.-cal., besides the heat of neutralization, 6000-8000 g.-cal. per mol. lactic acid. The anaerobic restitution of heat is, therefore, a pos. value of about 10% the initial heat production. The direction of the reaction, however, depends not on the heat but on the change in free energy, and this cannot be measured owing to the lack of information with regard to active concns. in the living muscle. In expts. with the reaction system: phosphopyruvic acid + creatine  $\rightarrow$  creatinephosphoric acid + pyruvic acid - (about) 3000 g.-cal. are involved, but the similar reaction: phosphopyruvic acid + arginine  $\rightarrow$  argininephosphoric acid + pyruvic acid is thermally practically neutral. S. Morgulis

Ultrafiltration through Cellophane under high pressure as a means of purifying enzymes and of determining their particle size. G. Gorbach and K. Nitsche. *Biochem. Z.* 281, 309-91 (1935).—Ultrafiltration under 100 atm. through Cellophane yields highly purified enzyme preps. The Cellophane is cut in round pieces, left in distd.  $H_2O$  for 24 hrs. and tested with Congo red soln. in the pressure-filtration app. The rate of filtration is about 10-15 cc. per hr. The detn. of particle size is discussed. S. M.

Optical activity of horse globin hemochromogen and of hemoglobin dissolved in 0.25 N sodium hydroxide, with special reference to its sulfur content. Istvan Simonovits

and George Balassa. *Biochem. Z.* 281, 333-8 (1935); cf. C. A. 30, 741<sup>1</sup>.—Horse globin hemochromogen in a concn. of 0.16% in 0.25 N NaOH has  $[\alpha]_D^{25} = -48.2^\circ$  and  $[\alpha]_D^{25} = -45.2^\circ$ , while globin + hematin (hemoglobin) under similar conditions has  $[\alpha]_D^{25} = -45.7^\circ$ . Neither concn. nor recrystn. affects the sp. rotation, which seems to depend upon the S content of the preps. S. Morgulis

Theory of the mitogenetic radiation. V. Yeast as detector of mitogenetic rays. M. Mosseva. *Biochem. Z.* 281, 349-57 (1935).—Discussion of methodological points. S. Morgulis

Protease action on protein of ungerminated cereal grains and its effect on the amylolytic power. Tadeusz Chrzastewski and Josef Jankowski. *Biochem. Z.* 281, 408-19 (1935), cf. C. A. 29, 1110<sup>1</sup>.—The effectiveness of the amylase contained in various cereal grains can be increased by proteases (trypsin, pepsin, papain, rennin) to different degrees. The increase in the amylase activity depends partly upon the nature of the protein and the extent of its hydrolysis. Trypsin produces the strongest effect. However, the increase in amylase effectiveness is greater in seeds of high amylase content (wheat, rye, barley) than of a low content so that the protease action is due primarily to formation or liberation of amylase mobilizing factors, the eluto-substances, kinases, etc., rather than the destruction of the proteins. The effect is manifested principally in the saccharifying ability of the amylase. S. Morgulis

The chemical groups of proteins which show affinity for polysaccharides. VI. The role of lysine, leucine, phenylalanine, cystine, tryptophan, asparagine, aspartic and glutamic acids. St. J. v. Przytycki, H. Rafalowska and J. Cichocka. *Biochem. Z.* 281, 420-2 (1935); cf. C. A. 29, 8026<sup>2</sup>.—Expts. with various amino acids seem to indicate that in the formation of the polysaccharide-protein symplexes the following groups do not play a part:  $COOH$ ,  $COO^-$ ,  $CONH_2$ ,  $CH_3CH_2NH_2$ ,  $CSSC$ ,  $iso-Bu$ ,  $Ph$  or indolyl. VII. The nature of the polysaccharide-guanidine combination. W. Giedroyc, J. Cichocka and L. Myskowski. *Ibid.* 422-30 (1935).—Arginine and tyrosine manifest strong affinity toward, and form compds. with, polysaccharides. The combination is thus ascribed to the  $RC_6H_4OH$  or  $CNHC(=NH)NH_2$  groupings, which are widely distributed in the various proteins. S. M.

Hydrolysis of lactose by enzymes of liver and kidney. Carl Neuberg and Eduard Hofmann. *Biochem. Z.* 281, 431-7 (1935).—Expts. demonstrate the presence of strong lactase activity of preps. from liver and kidney. Especially strong activity has been found in the autolyzates of horse kidney. S. Morgulis

Hydrolysis of  $\beta$  glucosides and of conjugated glucuronates by enzymes of liver and kidney. Eduard Hofmann. *Biochem. Z.* 281, 438-43 (1935).—Autolyzates, dry alc. ether exts. and dry acetone exts. of kidney and liver were used. The enzymic hydrolysis of salicin,  $\beta$ -naphthylglucuronic acid,  $\beta$ -phenoxylglycoside, galactoside and glucoside was studied. At  $pH$  below 5 the reaction no longer seems to proceed, but the optimum  $pH$  depends upon the nature of the buffer. S. Morgulis

Cryolysis, diffusion and particle size. II. Studies on myosin. F. E. M. Lange and F. T. Nord. *Biochem. Z.* 281, 444-6 (1935), cf. C. A. 29, 8020<sup>2</sup>.—The prep. of myosin solns. is described. The irreversible aggregation of the myosin colloid by freezing is shown by the progressive decrease in the diffusion coeff. with decreasing temp. However, freezing to  $-79^\circ$  of a 0.1% soln. seems to result in a disaggregation. S. Morgulis

Effect of electrolytes on the synthesis of lactose. D. Miklin and T. Fetisova. *Biochem. Z.* 282, 26-31 (1935).—The enzymic formation of lactose is apparently increased under the influence of salts. The hydrating ions ( $Na$ ,  $Li$ ) increase the amt. of sugar and this is even more marked with  $Ca$ , while the  $K$  ion, with very weak hydrating power, showed the least effect on the lactose synthesis, or even caused inhibition.  $SCN^-$  inhibits the enzymic process. S. Morgulis

The micellar form of the stroma protein. A study of the structure of the red blood corpuscles. Gundo Boehm

*Biochem. Z.* 282, 32-46(1935).—The stroma protein is sepd. from hemoglobin and dissolved in neutral salt soln. From a study of the phys. behavior of this protein it is concluded that its micelles are fibers of length several thousand times their thickness. This explains why, owing to spatial relations, it is impossible to obtain solns. more concd. than 3-4%, where gels are formed. The 4% concn. of the stroma protein of the erythrocytes cannot, therefore, be limited to the cell membrane but must extend through the cell. S. Morgulis.

**Chloroform as a serum-protein precipitant.** Anders Grönwall *Biochem. Z.* 282, 85-7(1935).—The pptn. of serum proteins by  $\text{CHCl}_3$  depends upon the H-ion concn. Optimum conditions were found at  $\text{pH}$  4.3, 0.5, 8.1, 9.7 and 9.1. S. Morgulis.

**Hydrolysis of adenylypyrophosphoric acid and of arginine-phosphoric acids in the crab muscle.** K. Lohmann *Biochem. Z.* 282, 100-10(1935).—Dilysed crab muscle ext. or washed crab muscle hydrolyzes only 1 of the 2 easily hydrolyzable  $\text{H}_2\text{PO}_4$  groups in adenylypyrophosphoric acid. The resulting adenosinephosphoric acid was isolated as the Ba salt. The dephosphorylation to adenylic acid occurred only after the addn. of a Mg salt. Hydrolysis of adenylypyrophosphoric acid thus proceeds in 2 stages and by different enzymes, as is also the case in mammalian muscle. Argininephosphoric acid is hydrolyzed by dilysed crab muscle ext. only in the presence of adenylypyrophosphoric acid or adenosinephosphoric acid. It is not hydrolyzed in the presence of adenylic acid, in which respect it differs from creatinephosphoric acid. This is due to the fact that crab muscle, in the presence of argininephosphoric acid, does not change adenylic acid to adenosinephosphoric acid or to adenosine(tri)phosphoric acid, but changes readily adenosine(diphosphoric) acid to adenosine(tri)phosphoric acid. Crab muscle does not contain adenylic acid deaminase. S. Morgulis.

**Utilization of the glass electrode to determine the pressure sensitivity of the  $\text{pH}$  of whole blood.** A. Thiel and M. Gamsa. *Biochem. Z.* 282, 115-56(1935).—The measurements were made under pressure in a metal bomb. The  $\text{pH}$  of the blood was measured against a standard acetate soln. of  $\text{pH}$  4.62. On developing the exptl. pressure (up to 100 atmospheres) the potential changed very rapidly, then remained constant for hrs. On releasing the pressure the  $\text{e. m. f.}$  returned to its former value or within 1-2 mv. of this value (the difference was probably due to the unavoidable loss of  $\text{CO}_2$ ). The  $\text{e. m. f.}$  at excess pressures of 0, 50 and 100 atm. were +153, +151 and +150 mv., resp. To decide whether this is due to increased acidity of the blood or to increased alk. of the acetate soln., expts. were made on the acetate soln. against Veibel's standard (0.01 N HCl + 0.09 N KCl). The results, under the same pressure conditions, were -150, -153, -154 mv., resp., which shows that the acetate actually becomes more alk. Then, finally, the blood was compared to the Veibel standard, and the results here were +313, +311 and +314 mv., resp. From these 2 sets of expts. the  $\text{pH}$  of the whole beef blood is calcd. as 7.29 and 7.31, at 37°. Pressures up to 100 atm. are thus without any influence on the blood  $\text{pH}$ . S. M.

**Experiments on the limit of growth in tissue cultures.** Edmund Mayer. *Skand. Arch. Physiol.* 72, 210-58 (1935).—A new method is described whereby an enormous increase in the growth period and in the final size of the tissue cultures is attained. This depends on the renewal of clot structure and a thorough exchange of substances in the exptl. cultures. S. Morgulis.

**Accelerating action of mercury and copper on the decolorization of indicators in certain oxidation-reduction systems of plant seeds.** T. Thumberg. *Skand. Arch. Physiol.* 72, 243-90(1935).—Hg and Cu may accelerate rather than inhibit the decolorization of indicators used in studying oxidation-reduction systems of certain seeds. The conditions under which this reversal of effect occurs are discussed. S. Morgulis.

**The amylase of Clostridium acetobutylicum.** W. W. Johnston and A. M. Wynne. *J. Biol. Chem.* 101, 611 (1935).—The effect of the following factors on the rate of

hydrolysis of starch by the amylase of *Clostridium acetobutylicum* was studied. The optimum  $\text{pH}$  was 4.8-5.0. Na acetate, phosphate and chloride retarded the enzyme. There was linear relationship between enzyme concn. and velocity of reaction. The temp. coeff. was fairly const. with 0.1% starch but not with 1.0%. J. T. M.

**Determination of enzymes.** B. A. Rubin and L. I. Naumova. *Compt. rend. acad. sci. U. R. S. S.* 3, 83-6 (1935). cf. *C. A.* 29, 5470<sup>1</sup>.—The activity of amylase suspensions obtained from cabbage or tomato increases with increase in diln. This is probably due to a breaking up of the equal between the micro- and macroheterogeneous portions of the enzyme and is, in its effect, equiv. to successive extns. of amylase. The effect of diln. is not as great with catalase. Pctn. of enzymes by trituration with sand diminishes rather than increases the activity of catalase. Filtration of catalase soln. through a Schleicher and Shull No. 708 paper causes a sharp decrease in the activity of the enzyme. A more active enzyme is obtained in all cases in which chalk, or its equivalent, is used for the neutralization of acids. For tomato fruits, the addn. of chalk increases the activity of catalase 7 times. W. J. Peterson.

**The thickness of the wall of the red blood corpuscle.** James P. Danahy. *J. Gen. Physiol.* 19, 10-22(1935).—Pricke's assumption (*Phys. Rev.* 26, 682(1925), cf. *C. A.* 20, 1218) of a dilec. const. of 1 for the erythrocyte wall is approx. correct for a solid layer of any thickness and for a liquid layer of not more than bimol. thickness. Liquid layers of greater thickness may have dilec. const. several times greater than 1. The dilec. const. of the polar group of unimol. films of the following types of compds. are: aliphatic compd., ester 4.2, acid 5.1, alc. 6.6, ketone 6.4, nitrite 6.7, dibasic ester 7.0, sterol compd., alc. 11, ketone 2.5. C. H. Richardson.

**The escape of hemoglobin from the red cell during hemolysis.** Eric Ponder and Douglas Marsland. *J. Gen. Physiol.* 19, 35-41(1935). Small concns. of saponin cause a relatively small permeability of the cell membrane to hemoglobin. Large concns. destroy the membrane so completely that the theoretical time for loss of pigment through a completely permeable membrane is nearly attained. The dependence of permeability upon lysin concn. is explainable upon existing knowledge of the rate of transformation as it reacts with the red cell envelope. When the red cells are hemolyzed by hypotonic solns., permeability of the membranes to hemoglobin is nearly const. irrespective of the tonicity used to bring about lysis. C. H. Richardson.

**Electrophoresis of sterols.** III. Further investigations of cholesterol surfaces. Laurence S. Moyer. *J. Gen. Physiol.* 19, 87-94(1935); cf. *C. A.* 29, 7357<sup>1</sup>.—No significant differences exist between the electrophoretic mobilities of needle-shaped cholesterol crystals from acetone, the platelets from ale or the columnar crystals from fusion. The mobilities of cholesterol particles produced by grinding with ice at -10° are less than half that of the unground cholesterol particles. Equal of mobility is reached after grinding for 50 min. When the crystals ground with ice are dried *in vacuo* at room temp., the mobility reverts to that of the unground crystals. Both ground and unground crystals have an isoelec. point near  $\text{pH}$  3.0. The ground crystals probably undergo a change in wetting whereby the attainment of the steady state is accelerated. The increased adsorption of water makes by the ground crystals may lessen the no. of active places at the interface available for adsorption of ions, thus decreasing the net charge per unit area. The ratio of pos. and neg. ions on the surface would only be a function of  $\text{pH}$  and the isoelec. point would remain unchanged. C. H. Richardson.

**The molecular weight and isoelectric point of thyroglobulin.** Michael Heidelberger and Kai O. Pedersen. *J. Gen. Physiol.* 19, 95-108(1935).—The sedimentation const. of hog thyroglobulin is  $18.2 \times 10^{-13}$ , essentially the same as thyroglobulin from man. The sp. vol. is 0.72, isoelec. point (native)  $\text{pH}$  4.53 and (denatured)  $\text{pH}$  5.0. The mol. wt. is 700,000 from sedimentation and

diffusion consts., 650,000 from sedimentation equl. data. In shape the mol. deviates markedly from the spherical.

C. H. Richardson

The accumulation of electrolytes VIII. The accumulation of potassium chloride in models. W. J. V. Osterhout and S. E. Kamerling. *J. Gen. Physiol.* 19, 167-78(1935), cf. *C. A.* 26, 4841.—In models consisting of an outer and an inner aq. phase sep'd by a non-aq. phase (70% guaiacol plus 30% p-cresol), KCl enters until its chem. p. d. is much greater inside than outside. The energy for this one-sided accumulation of KCl is derived from the chem. reactions in the system and the continual supply of certain materials. The maintenance of a lower  $p_H$  inside the model by means of  $CO_2$  is important. The analogy to conditions in some living cells is pointed out. The model differs from many living cells in that Cl does not increase inside with the increase of  $K^+$ . As in *Valonia*,  $K^+$  tends to pass out of the model as KCl when the ion activity product (K)(Cl) is greater inside, at the same time it enters as KOH since the activity product (K)(OH) is greater outside. The net result of these processes is the entrance of K probably because the latter process is the more rapid.

C. H. Richardson

The color reaction and the iodometry of oxidizable substances I. M. Konishi. *Okayama-Igakka Zasshi* 47, 1043-57(1935).—The color reaction and the iodometry of the aq. ext. and pressed juice of plant and animal tissues show a parallel relation in most instances, but the degree of reaction differs among different tissues. The oxidizable substances are converted into  $EtOH$ ,  $Me_2CO$  and  $CN$  comp'd., but not into  $Et_2O$ . They can be p'd. with basic but not with neutral Pb acetate or phosphotungstic acid. These oxidizable substances are very labile toward sunlight and  $H_2O_2$ . The glutathione content of plant tissue is less than that of animal tissue and the I consumption of the glutathione is less than that of the glutathione-contg. oxidizable substances. The aq. exts. from lotus roots, pine needles, orange or oak leaves show sugar reaction. From the sugars osazone-like crystals in 204 208° can be prep'd. The oxidizable substances are adsorbed by animal charcoal, but not by clay. They are diffusible through collodion membrane, fish skin and parchment.

K. Sugura

Kinetics of an intracellular system for respiration and bioelectric potential at flux equilibrium. Gordon Marsh. *Plant Physiol.* 10, 681-97(1935).—A quant. connection is derived between the velocity of the oxidative reactions and the measured  $e.m.f.$  of a cell or tissue on the basis of Lund's theory of bioelec. currents (Lund, *C. A.* 23, 429). The properties of the system are fully described. Twenty-six references.

Walter Thomas

Some physiological and physical aspects of the surface tension of urine. P. W. Perryman and C. F. Selous. *J. Physiol.* 85, 128-44(1935), cf. *C. A.* 29, 2019.—A modification of the max. bubble pressure app. applicable to the measurement of the surface tension of body fluids is described. The app. can be used for following the changes in surface tension with time up to 2 hrs. and also for changes with temp. The occurrence of surface tension time changes in biol. fluids is confirmed and this effect in urine is investigated quantitatively. The resulting curves are of logarithmic form. This is shown not to be due to proteins or the absorption of  $CO_2$ . The theory is advanced that it is due to the slow adsorption of capillary active substances, some of which are related to the bile acids. The change of surface tension with temp. from 33° to 56° is of an approx. linear form up to 54° at which temp. the decrease becomes more rapid. In 16 normal subjects the daily variation in surface tension of urine was shown not to be due to the ingestion of food or liquid. A monthly cyclical variation was discovered in the female which corresponds closely to the menstrual cycle. The surface tension of urines cannot be related to sp. gr. or pathol. constituents such as blood, pus, albumin, bile, etc. There is no correlation between the surface tension and any sp. disease or group of diseases.

F. D. Walter

Postmortem change in the liver. Kwanji Momonoi. *Okayama-Igakka Zasshi* 47, 1480-95(1935).—In the

liver of rabbits the residual N, P and S gradually increased with the time after death. The rate of increase varied according to the season of the year and the atm. humidity. In general it was greatest in the summer, less in autumn and spring, and least in winter. In winter the S increased until the 40th day and the P until the 50th day after the death of the animals. No changes in these substances were noticeable after these periods. The max. change of the residual nitrogen was found in summer.

K. Sugura

Anomalous dispersion of elec. waves (3.8 m.) in solutions of org. amphoteric ions [elec. behavior of brain and nerve substance] (Hauser) 2.

## B—METHODS AND APPARATUS

STANLEY R. BENEDICT

Urea excretion IX. Comparison of urea clearances calculated from the excretion of urea, of urea plus ammonia and of nitrogen determination by hypobromite. Donald D. Van Slyke, Irvine H. Page, Alma Hill and Esben Kirk. *J. Clin. Investigation* 14, 901-10(1935); cf. *C. A.* 27, 331.—The routine procedure for the clinical detn. of the urea clearance is described. In human subjects when the proportion of urea in the urea +  $NH_3$  must of the urine is markedly decreased by induced acidosis (with  $NH_4Cl$ ) and by low-protein diet, the urea clearances calc'd from the excretion rate of urea alone suffered a parallel reduction. When, however, values for the excretion of urea +  $NH_3$  are substituted for urea, the clearances calc'd remain at the usual levels. These results favor the hypothesis "that the  $NH_3$  excreted in the urine of man is formed in the kidneys chiefly from urea removed from the blood, and that the work of the kidneys is excreting urea from the blood is more accurately indicated by the combined excretion of urea and  $NH_3$ , than by the excretion of urea alone." In calcg. urea clearance by means of the standard equation the value of U (urinary concn. of urea) should include both urea and  $NH_3$ .

J. B. Brown

Enzymic estimation of vitamins. Francesco Pirrone. *Ann. chim. applicata* 25, 292-309(1935).—Many attempts have been made to utilize measurements of enzymic activity ( $CO_2$  evolved) as an index of vitamin value (cf. Heaton, *C. A.* 17, 1046; Kollath, *C. A.* 20, 1645, 21, 1135). P. has measured the  $CO_2$  evolved from solns. contg. vitamins A, B, C and D. In all cases the values are roughly proportional, the vitamin value  $UV$  being approximately a linear function of enzymic activity,  $Ae$ . For vitamins A and D,  $UV = (Ae - 100) 11.36 + 23$ , for vitamin B  $UV = (Ae - 100) 0.0833 + 2.34$ , for vitamin C  $UV = (Ae - 100) 0.0025 + 0.19$ .

A. W. C.

Use of mice in the standardization of parathyroid hormone. effect of parathyroid on rachitic rats examined by the "line test." F. J. Dyer. *Quart. J. Pharm. Pharmacol.* 8, 513-24(1935); cf. *C. A.* 29, 7365.—The proposal of Simon (cf. *C. A.* 29, 5494) to use the antagonism between Ca and Mg salts for measuring parathyroid activity has been exam'd. The principle underlying the method is that the increase of blood serum Ca caused by injections of parathyroid into mice reduces the narcosis produced by injections of  $MgSO_4$ . When the method is used to compare the potency of 2 parathyroid exts. it is recommended that (1) at least 20 mice be used in each group, (2) the dose of  $MgSO_4$  be 1.7-1.8 mg. of  $MgSO_4 \cdot 7H_2O$  per g. of body wt.; (3) the max. no. of mice affected between 20 and 60 min. after the injection of Mg be recorded. The percentages of mice affected by the 2 exts. under comparison are then referred to the curve relating dose to effect and the corresponding abscissas found. The 2 potencies are then proportional to the abscissas, i. e., to the 2 doses of standard ext. used in prep'g. this curve. Until the standard powder now in course of prep'n. is ready, the potency of an ext. must be compared with that of a reliable prep'n. such as parathormone (Lilly) used as provisional standard. Parathormone was found to supplement the calcification produced by vitamin D in the "line test." At least 10 daily injections each of 20 Collip units were required to produce a measurable effect with the para-

thyroid alone. Apart from the fact that this effect is not specific for the hormone, the expense involved would not justify the use of the line test for measuring parathyroid activity. W O E

A micromethod for blood urea and an automatic urine collector for urea clearance in infants. Lee E Farr. *J. Clin. Investigation* 14, 911-13 (1935) J B B

Micromethod for the determination of phosphoric acid. Application to the determination of phosphorus in tissues. Lucien Thivolle. *Bull. soc. chim. biol.* 17, 1427-50 (1935).—The sample contg. 0.01-2.0 mg P is boiled with 2 cc. concd.  $\text{HNO}_3$  in which 2 3% Cu has previously been dissolved. Heating is continued until all the acid is evapd and the  $\text{Cu}(\text{NO}_3)_2$  is decompd to  $\text{CuO}$ . The residue is dissolved in dil.  $\text{HCl}$ , the P pptd as  $\text{NH}_4\text{phosphomolybdate}$ , and the ppt. collected on a very small asbestos pad filter which has been coated with talc to make it less porous. The washed ppt. is dissolved in dil.  $\text{NaOH}$  and then treated with Zn (free from Fe and As) and an excess of  $\text{H}_2\text{SO}_4$  to reduce the  $\text{MoO}_3$  to  $\text{MoO}_2$ . The  $\text{MoO}_2$  is reoxidized to  $\text{MoO}_3$  by titration with 0.025 M  $\text{KMnO}_4$ . Comparison with a blank detn. is imperative. Several variations of the method are discussed.

L E Gilson  
A method of destroying organic matter applicable to the benzidine micromethod of determining organic sulfur in biology. L Revol and M Ferrand. *Bull. soc. chim. biol.* 17, 1451-4 (1935).—The sample should contain less than 5 mg S. Org. matter is destroyed by boiling with concd.  $\text{HNO}_3$  in a flask equipped with a short reflux condenser. A little  $\text{H}_2\text{O}_2$  is added later to complete the oxidation. Vapors issuing from the top of the reflux condenser are passed down through another condenser for cooling, then bubbled through Br water in a special receiver to oxidize and fix volatile S compounds. The liquid in the receiver is finally added to that in the flask and the whole evapd to dryness. The sulfate in the residue is detd. by the benzidine method. The error is less than 3% of the S present. The app. is illustrated. L E Gilson

Detection of 2,4-dinitrophenol and its elimination products in urine. A Meyer and H Drutel. *Bull. soc. chim. biol.* 17, 1455-61 (1935).—To 10 cc. urine, previously defecated with  $\text{Pb}(\text{OAc})_2$ , add 2 cc. glacial  $\text{AcOH}$  and a pinch of Zn powder, allow to react 15 min. and filter. To the filtrate add 2 drops of 0.1%  $\text{K}_2\text{Cr}_2\text{O}_7$  soln. without shaking. If the urine contains 2,4-dinitrophenol a rose color develops immediately in the upper part of the soln. (Shaking, or adding too much  $\text{K}_2\text{Cr}_2\text{O}_7$  may destroy the color.) One part in 50,000 urine is readily detected. Smaller quantities can be detected by defecating 100 cc. or more of urine with Patein-Dufau acid Hg nitrate reagent, extg. with  $\text{Et}_2\text{O}$ , evapg. the  $\text{Et}_2\text{O}$  and testing the residue after dissolving in 10 cc. water. Some other tests are discussed. Nineteen references. L E Gilson

A new rapid and precise method for the determination of reducing sugars. G I Solomos. *Bull. soc. chim. biol.* 17, 1465-9 (1935).—A measured quantity of standard  $\text{K}_3\text{Fe}(\text{CN})_6$  soln. is made alk. with  $\text{NaOH}$ , then heated to boiling and the sugar soln. (blood, spinal fluid, urine or milk, suitably defecated and dild.) added dropwise from a buret until the  $\text{K}_3\text{Fe}(\text{CN})_6$  is just completely decolorized by reduction to  $\text{K}_4\text{Fe}(\text{CN})_6$ . The  $\text{K}_3\text{Fe}(\text{CN})_6$  soln. is checked against a standard soln. of pure glucose.

L E Gilson  
Determination of intracellular  $\text{pH}$  by the vital staining method. L. Lison. *Compt. rend. soc. biol.* 120, 102-4, *Protoplasma* 24, 453-65 (1935).—If substances exhibiting metachromasia (cf. C. A. 29, 4037) are present in the cells errors as great as 6  $\text{pH}$  units are possible when basic dyes are used. Thus if a buffer soln. of  $\text{pH}$  4.0 is tinted with brilliant cresol blue and 1/20,000 neutral Ca chondroitinsulfate is added the color changes to that corresponding to  $\text{pH}$  10.4. When added to a soln. of  $\text{pH}$  2.2 colored with neutral red the same comp. causes the color to change to that of a soln. of  $\text{pH}$  7.2-7.4. Sulfuric esters of polysaccharides in general have this property. L. E. G.

A method for investigating endocrine activity by serum reactions. G Richard. *Compt. rend. soc. biol.* 120,

1 444-6 (1935).—The serum from a clotted-blood sample is divided among a series of test tubes; 3 drops is placed in each. To different tubes 2, 3, 4 and 6 drops of each of various conc. isotonic exts. (Byla) of endocrine glands (thyroid, adrenal, hypophysis, gonads, etc.) are added and each mixt. is dild. to 1 cc. with physiol.  $\text{NaCl}$  soln. After incubation at 37° for 60-90 min. the hemolytic action of each mixt. on a drop of 1/40 suspension of washed sheep corpuscles is detd. Humoral reactions and endocrine dysfunction. G Richard and R Biot. *Ibid.* 446-8.—In the above method the addn. of even a very little of any of the endocrine exts. decreases the hemolytic power of normal serum. If a certain endocrine deficiency exists much more of that particular ext. must be added before the hemolytic power is decreased. L E Gilson

Determination of amylase in milk. M Manicattide, Bratescu and M Popa. *Compt. rend. soc. biol.* 120, 657-8 (1935).—A slight modification of Wolgemuth's method is described. Human colostrum contained 1000-2000 Wolgemuth units of amylase per cc. Normal human milk (80 samples) contained 128-512 units per cc. Cow milk usually contained none at all, a few samples contained 2-4 units per cc. L E Gilson

Apparatus for the microdetermination of ammonia nitrogen by distillation and aeration. I Goldberg and R F Banfi. *Rev. soc. argentina biol.* 11, 440-8 (1935).—The soln. to be analyzed is made alk. and boiled in a flask equipped with an efficient reflux condenser. A slow current of air is bubbled through the boiling soln. The air and vapors pass upward through the reflux condenser and then down through a specially designed delivery tube which dips into an acid soln. contained in a test tube or other receiver. L E Gilson

Rosenthal-Erdélyi test for vitamin A. Harry Willstaedt. *Z. Vitaminforsch.* 4, 272-6 (1935).—The Rosenthal-Erdélyi test ( $\text{SnCl}_4$  + guaiacol) for vitamin A is not specific, is neg. with carotenoids, but may be pos. with the sterols which always are present in biol. material.

Joseph S Heppburn  
Biochemical detection of carbohydrates, polysaccharides, alcohols, glucosides and other compounds. H Damm and A Lembke. *Naturwissenschaften* 23, 641 (1935).—General remarks on the possibilities of using specific enzymes derived from constitutive microbes of nonvarying species for the detection and detn. of org. substances. Use of acid-producing bacteria is suggested. At the proper temp. about 3 days is required for the reaction, it can be used as a micromethod. B. J. C. v. d. H.

Determination of chloride in biological fluids by the use of adsorption indicators. The use of dichloro-fluorescein for the volumetric microdetermination of chlorides in cerebrospinal fluids and blood serum. Abraham Saifer and Morris Kornblum. *J. Biol. Chem.* 112, 117-22 (1935).—Microdetn. of chlorides in spinal fluid and in serum which has been treated with 3:1 alc.- $\text{Et}_2\text{O}$  to remove proteins can be rapidly and accurately made by the method described in which dichloro-fluorescein is used as an adsorption indicator and standard  $\text{AgNO}_3$  (approx. 0.02 N) for titration. The entire procedure is carried out in a single tube without transfer. Quantities in the range of 1 mg. of  $\text{NaCl}$  in spinal fluid with a max. deviation of 1% and in blood serum of 2% can be detd. A P. Lothrop

A tungstic acid precipitation method for the extraction of estrogenic substance from urine. S Charles Freed, I. Arthur Mirsky and Samuel Soskin. *J. Biol. Chem.* 112, 143-7 (1935).—Extn. of small amts. of estrogenic substance from urine can be quantitatively accomplished by preliminary pptn. with  $\text{H}_2\text{WO}_4$  and extn. of the ppt. with alc. and  $\text{Et}_2\text{O}$ . Recovery of added estrogenic substance is uniformly complete within the biol. error of animal assay. A P. Lothrop

An improved apparatus for the determination of colloid osmotic pressure in small amounts of fluid. Reubenia Dubach and Robert M. Hill. *J. Biol. Chem.* 112, 313-18 (1935).—The app. described is an improvement of the Krogh-Nakazawa osmometer (C. A. 22, 600). It is made of brass so machined that the parts fit tightly and



do not require washers to prevent leakage around the membrane. A flat membrane is thus insured throughout the detn and no bulging occurs which is almost unavoidable in an app where it is held in place with a rubber washer. There is no chance of contamination of the outer fluid with the inner fluid after the detn is completed. The arrangement described by Turner (*C. A.* 26, 4094) for making several detns at the same time is also employed. The time required for the establishment of equilibrium varies generally from 3 to 6 hrs and readings are begun at the end of 3 hrs and continued at  $\frac{1}{4}$ -hourly intervals until 2 successive readings check within 10 mm.

A. P. Lothrop

A modified Rehberg buret for use with titrating solutions which react with mercury. Bernard B. Longwell and Robert M. Hill. *J. Biol. Chem.* 112, 319-21 (1935).—The Rehberg microburet (*C. A.* 19, 2148) has been so modified that Hg does not come in contact with the titration fluid. The use of the buret is as simple as is that of Rehberg and a rubber diaphragm in use with thiosulfate soln. has remained in good condition for nearly a yr.

A. P. Lothrop

A new test of cortical hormone and ascorbic acid in guinea pigs with diphtheria. W. Herbrand. *Endocrinology* 16, 236-7 (1935).—With a diphtheria toxin dose sufficient to kill guinea pigs within 40 hrs the adrenals undergo hemorrhagic destruction that cannot be prevented either by cortical hormone or by ascorbic acid administration. However, by the administration of the combination of the two substances the animals could be kept alive and the adrenals remained unaffected. The best results were obtained when the poisoned animals were given every 6 hrs for 2 days an injection of 3 cc. Pancortex C contg 150 mg. ascorbic acid. The method is suggested for the standardization of the cortical hormone.

S. Morgulis

Rapid staining with buffered Wright stain. Decostello. *Folia Hematol.* 53, 390-5 (1935).—The best dilg fluid is the ordinary phosphate mixt buffer at pH 6.2. If below 6 the erythrocytes will be too red and if above 6.4 they may be greenish, making polychromasia more difficult to detect.

John T. Myers

Studies in respirometry. IV. The use of a comparator system in refractovolumetric respirometry. Wm. R. Thompson and David M. Grayzel. *J. Gen. Physiol.* 19, 61-3 (1935); cf. *C. A.* 27, 345.—A glass buret is used as a comparator system to compensate for small changes in pressure in a respirometer (*C. A.* 27, 117, 345) adapted for the measurement of  $O_2$  and  $CO_2$  in continuous respiration of salamanders and other small animals.

C. H. R.

The estimation of estrin and of male hormone in oily solution. Ldith Bulbring and J. H. Burn. *J. Physiol.* 85, 320-33 (1935), cf. *C. A.* 27, 4290.—Seven dilns of estrin, prepd by another worker, were estd by the vaginal smear method, by making injections in oil. Lists of their strength were obtained with an error varying from 3 to 30%, with an av. error of 13%. A new method of estg estrin in oil soln by the increase in wt. of the uterus of ovariectomized rats of 40 g. body wt. is described. Over a range of dose from 0.2 to 0.8  $\gamma$  of estrin daily for 4 days, the av. increase in the wt. of the uterus is proportional to the log of the dose. The estrin is always made in comparison with a standard. The findings of Korenchewsky, Denison and Schalit (*C. A.* 27, 529) that injections of male hormone in oil cause an increase in the av. wt. of the prostate and seminal vesicles of castrated rats which is proportional to the dose, within a certain range, are confirmed. It was found unnecessary to wait 30 days after castration before making injections.

E. D. Walter

Constant-temperature device containing mercury and suitable for use in animal or plant tissue temperature determinations. Charles Sheard and Reginald Halstead (to Chemical Foundation, Inc.). U. S. 2,025,534, Dec. 24. Various details are described of a device having a thermoregulator actuated by the thermal changes in volume of the Hg and having an elec. circuit including the Hg.

## C—BACTERIOLOGY

LAWRENCE H. JAMES

The isolation and some cultural characteristics of *Clostridium dissolvens*. Jun Hanzawa and Sadahiko Yoshimura. *J. Faculty Agr. Hokkaido Imp. Univ.* 39, 1-48 (1935).—*Clostridium dissolvens*, an obligate anaerobic spore-bearing bacillus, identical with that of Khovine, has been isolated from surface soil. It decompd. none of the more common carbohydrates with the exception of cellulose. Growth on cellulose was accompanied by the production of  $H_2$ ,  $CO_2$ , pigment, HOAc, BuOH and EtOH. No indole or  $H_2S$  was detected. Vitamin B or the alc. ext. of feces was essential to the growth, probably acting as accelerators.

Rachel Brown

Action of quartz mercury lamp irradiation on various bacteriophages. C. Levaditi and J. Voet. *Compt. rend. soc. biol.* 120, 385-7 (1935).—The 3 types used (anticoxon, anti-staphylococcal and anti-dysenteric) were equally sensitive. Attenuation was produced in 5 min and complete destruction in less than 1 hr. under the conditions described.

L. E. Gilson

Influence of the composition of "peptones" on the multiplication of *Pseudomonas aeruginosa*. Jean Régner, Robert David and Jacques Morchoisne. *Compt. rend. soc. biol.* 120, 415-18 (1935).—Pancreatic hydrolyzates of muscle and casein and peptic hydrolyzates of muscle, fibrin, mucosa and casein all produced abundant growths. Growth in gelatin digested with pancreatin was scanty. Removal of diffusible compds from the hydrolyzates by dialysis did not decrease the rate of growth except in the case of the mucosa prep. Effects of glucose and mineral salts on the multiplication of *Pseudomonas aeruginosa*. *Ibid.* 418-20.—The addn of glucose or a salt mixt. to the dialyzed preps. mentioned in the above paper produced a considerable increase in the growth rate. Less NH<sub>4</sub> was formed when glucose was present.

L. E. Gilson

Polysaccharides of the gonococcus. S. Muterlich and A. Grimberg. *Compt. rend. soc. biol.* 120, 557-8 (1935).—By the method described the polysaccharide was obtained as a yellowish water-sol. powder giving neg. biuret, Millon and Fehling tests. Upon hydrolysis it yielded 35-40% of reducing sugar. It acts as a haptene capable of fixing the antigonococcus antibody.

L. E. Gilson

Deamination of alanine by bacteria. E. Aubel and T. Egami. *Compt. rend. soc. biol.* 120, 684-5 (1935); cf. *C. A.* 29, 8043a.—Discussion of the previous paper.

L. E. Gilson

The concentration and preparation of bacteriophages free from albumin. C. J. Schuurman and A. M. Schuurman-Ten Bokkel Huinink. *Geneskund. Tijdschr. Nederland. Ind. 75*, 1875-81 (1935).—The prepn of bacteriophages free from albumin according to the methods of Weiss (*C. A.* 21, 3673), and that of Asheshov (*Compt. rend. soc. biol.* 98, 770 (1928)) and Kligler-Orlitzky (*C. A.* 25, 5187) with subsequent concn. was not satisfactory as there was always a titer loss of at least  $10^{-1}$ . The concn. of bacteriophages was easily accomplished by filtration of Uchinsky lysates through glacial AcOH-colloidon-membranes of specific porosity. At a concn. of  $10^{11}$  phages per cc. the liquid becomes opalescent. A method for strengthening the membranes with Chinese paper is given.

J. C. Jurjens

Further studies of the effect of radium upon bacteria. R. R. Spencer. *U. S. Pub. Health Repts.* 50, 1642-55 (1935), cf. *C. A.* 28, 3099a.—Graphic representation of the killing effect of the  $\beta$ -rays as compared with the  $\gamma$ -rays of Ra is presented. Evidence is also presented that irradiation of bacteria over many generations may induce at times (6 out of 20 tests), but not regularly profound cultural and morphological changes. Continuous irradiation of the same culture for 30 days produced no genetic changes. Five tests in contamination-proof boxes yielded no changes. Thirteen references.

J. A. Kennedy

The cultural characteristics of *Saccharobacillus pastorianus*. J. L. Shimwell. *J. Inst. Brewing* 41, 481-7

(1935); cf. *C. A.* 30, 809<sup>4</sup>.—The morphology, culture, staining reactions, thermal death point, tolerance to hop antiseptics, etc., are given and a comparison with *Lactobacillus delbrückii* is described. Quick Landis.

Oxidations by acetic acid bacteria. III. Formation of a reducing sugar carboxylic acid (aldehyde-glucuronic acid) and of 5-ketoglucuronic acid. Konrad Bernhauer and Karl Irrgang. *Biochem. Z.* 280, 309-6 (1935).—Under the action of *B. gluconicum* on Ca gluconate, in addition to a small amt. of Ca 5-ketoglucuronate, about 50% is converted to a very easily sol. reducing Ca salt. Its intensive reaction with naphthoresazotinal HCl, as well as other considerations, leads to the assumption that this substance is Ca d-aldehyde-glucuronate. It is also produced in small amts. by the action of *Acetobacter xylinum* on glucose in the presence of CaCO<sub>3</sub> or on Ca gluconate. IV. Formation of 2 ketoglucuronic acid by *B. gluconicum*. Konrad Bernhauer and Bruno Gorlich. *Ibid.* 307-71. Under the influence of *B. gluconicum* on Ca gluconate d-2 ketoglucuronic acid is formed in addition to the main products, the d-5-ketoglucuronic and aldehyde-glucuronic acid. The substance was isolated as the well-crystallizable salt K. and was identified as the Me ester and the quinoxaline deriv. V. Comparative studies on the preparation of L-sorbose by different bacteria. *Ibid.* 376-8. *Acetobacter xylinum*, *B. xylinoides* and *gluconicum* produce L-sorbose from sorbitol, especially the last one. The yield is usually 70-80%. The best results are obtained with *B. gluconicum* in a synthetic medium in the presence of a little boiled yeast juice. By the procedure outlined practically pure sorbose is obtained. S. Morgulis.

Biochemistry of *Bacillus mesentericus hydrolyticus*, a new type of *B. mesentericus vulgaris*. Siegwart Hermann and Paul Neuschul. *Biochem. Z.* 281, 219-30 (1935).—A pure strain of an organism was obtained from carrot which morphologically resembles very closely *B. mesentericus vulgaris*, but unlike this possesses marked hydrolyzing power especially in splitting sucrose and sol. starch. This organism softens and loosens the cellular bindings of a no. of vegetables (cucumber, carrot, radish, peas, etc.), an effect which can also be produced with enzyme solns. prepd. from these organisms. S. M.

Bacterial variation. An inquiry into the underlying principles governing the cell morphology of *Bacillus megatherium*. Leo F. Rettger and Hazel B. Gillespie. *J. Bact.* 30, 213-34 (1935), cf. *C. A.* 29, 1453<sup>4</sup>.—Relatively slight changes in environment are responsible for striking changes in cell form. The factors which stimulate cellular variation are apparently unfavorable to continued normal growth, and are the result of metabolic activity. Variation is possible only when favorable and unfavorable influences are so balanced as to permit slow growth in the face of untoward circumstances. Partial O starvation is responsible for at least certain types of cell variation. There was no evidence that cellular forms develop from finely granular or apparently structureless material.

Differences in effect of phenyl mercuric chloride upon different races of bacteriophage and similarity in effect upon a phage and its homologous organism. Norman R. Goldsmith. *J. Bact.* 30, 237-42 (1935).—Phenyl mercuric chloride in dilns. of 1:20,000, 1:40,000 and 1:80,000 had no effect on the lysing ability of either staphylococcus or *S. coli* bacteriophages when in contact for 30 min. When in contact for 6 weeks at room temp., the lysing ability of the staphylococcus phage was completely destroyed while that of the colon bacillus remained as effective as the untreated controls. The toxicity of the antiseptic on the sp. phage seemed to parallel that upon the homologous organism. John T. Myers.

A bactericidal principle in excretions of surgical maggots which destroys important etiologic agents in pyogenic infections. S. W. Summons. *J. Bact.* 30, 253-67 (1935).—A potent bactericide was collected from maggots of the species *Lucilia sericata* by washing them with a spray of water during growth. It would kill pyogenic organisms in heavy suspensions in 5 to 10 min. It is non-viable and not destroyed by autoclaving 20 min. at 20 lb. pressure.

Org. material has little effect. It produces no lysis. It retains its potency when desiccated. John T. Myers.

The occurrence of a strain of *Azotobacter chroococcum* which does not ferment mannitol. Nathan R. Smith. *J. Bact.* 30, 323-8 (1935). Because of such strains, sucrose, dextrin or starch should be substituted for mannitol in Ashby's medium, unless mannitol utilizers are shown to be present in a given soil. John T. Myers.

A growth factor for rhizobia. D. W. Thorne and R. H. Walker. *J. Bact.* 30, 331-2 (1935), cf. *C. A.* 29, 1621<sup>4</sup>.—Cane sugar contains a necessary growth factor, which can be extd. with aly. alc. Yeast contains a smaller amt.

The utilization of CO<sub>2</sub> by the propionic acid bacteria in the dissimilation of glycerol. H. G. Wood and C. H. Winkman. *J. Bact.* 30, 332 (1935). John T. Myers.

A method for estimating the bacterial content of the mouth by direct count. Mary C. Crowley and U. G. Rickett. *J. Bact.* 30, 395-100 (1935). The mouth was sprayed with 0.025 N NaOH to dissolve mucin and secure more uniform counts. John F. Myers.

The denaturation of staphylococcal proteins. A. P. Kreuzer and V. C. Nichols. *J. Bact.* 30, 101-9 (1935).—Staphylococcal antigens made by mech. fragmentation and ultrafiltration contain less than 10% of the total bacterial protein in the denatured form. On exposure to heat, staphylococcal native proteins are denatured, becoming insol. at the isoelectric point, *pu* 4.6, and showing increase in SH groups. The denaturation reaction obeys the mass law and has a crit. increment averaging about 41,000. Undenatured protein should be preferable for vaccines. John F. Myers.

The effect of synthetic surface active materials on bacterial growth. I. The effect of sodium disecary butyl naphthalenesulfonate on the growth of *Mycobacterium smegmatis*. Joseph Katz and Aaron Lipsitz. *J. Bact.* 30, 419-22 (1935). The Na salt of di-secondary naphthalenesulfonic acid in dilns. up to and including 1:10,000 inhibits the growth of *Mycobacterium smegmatis*. In dilns. between 10,000 and 50,000 it produces many involution forms showing that this surface-active material is unfavorable to the growth of this organism. There was no synergistic action between neutral acrylavine and Na di-secondary naphthalenesulfonate. J. T. M.

The metabolic activity of various colon group organisms at different phases of the culture cycle. Grace Mooney and C. I. A. Winslow. *J. Bact.* 30, 127-40 (1935).—A fermentable carbohydrate (glucose) stimulates the multiplication of *S. coli* and *S. gallinarum* but inhibits *S. pullorum*. NaCl will remove the inhibitive effect. There was never any increase in the CO<sub>2</sub> per cell per hr. which is essentially independent of the organism and the medium used, hence it seems to represent a fundamental metabolic activity of the 3 species. The rate of metabolic activity varies greatly at various phases of the culture cycle. Increases can be explained only in part by increases in cell size. John T. Myers.

The role of bacteria and other microorganisms in the decomposition of cellulose in nature. Selman A. Waksman. *J. Bact.* 30, 411 (1935).—Microorganisms capable of destroying cellulose are wide spread. The mechanism of cellulose destruction varies widely. J. T. M.

Acid production and respiratory catalysts of *Escherichia acidilactici*. Kenneth C. Blanchard. *J. Bact.* 30, 412 (1935).—*E. acidilactici* contains at least 2 O-activating mechanisms which have different functions in the metabolism of the cell. John T. Myers.

The increase in bactericidal value of vapors from irradiated essential oils. W. Reimer-Deutsch and N. Molnar. *J. Bact.* 30, 411 (1935).—Irradiation of essential oils in alc. for 15 min., at a distance of 25 cm., with a Burdick air-cooled lamp increased the bactericidal power so that the vapors would kill staphylococci in 3 hrs. Such oils show a marked radiation. John T. Myers.

The influence of heat and storage on the electrophoretic migration velocities of various microorganisms. K. Pierre Dozois and Frank Hachtel. *J. Bact.* 30, 473-7 (1935).—Heat and storage did not alter the *pu* or migration rates,

below 60° Above 60° there was a slight gradual increase

John T. Myers

**Cultural characteristics of *Pasteurella tularensis***  
Corra M. Downs and Glenn C. Bond *J. Bact.* 30, 484-90 (1935) —All of 21 strains of *P. tularensis* fermented glucose and glycerol. All strains failed to ferment galactose, lactose, sucrose, raffinose, melzitose, arabinose, rhamnose, xylose, mannitol, dulcitol, sorbitol and salicin. It produces an alk. reaction in media not containing a utilizable carbohydrate, otherwise there is an initial acidity with an alk. reversion. It produces  $H_2S$  in media containing cystine, but not from peptone, blood or thiosulfate. No other S-contg. compds. stimulated growth.

John T. Myers

**The inhibition of *Bacillus subtilis* by ultraviolet-irradiated carbohydrates**  
Irvin H. Blank and Wm. Arnold *J. Bact.* 30, 507-11 (1935), cf. C. A. 29, 6269\* —Ultraviolet radiation (2537 Å) so alters agar or agar-water gels that they will not support the growth of *B. subtilis*. This results from the formation of a nonvolatile thermostable material, capable of diffusing through and from the gel. A similar inhibition results from the addition to the medium of an irradiated soln. of any of 20 different carbohydrates.

John T. Myers

**Cultural requirements of bacteria** VI The diphtheria bacillus *J. Howard Mueller J. Bact.* 30, 513-24 (1935), cf. C. A. 29, 2309\* —Animal tissue expts. apparently of whatever source contain substances essential to the growth of the strain of diphtheria bacillus studied. There is also present in most freshly prep'd tissue infusions a substance, perhaps glucose, which inhibits growth through the formation of acid. An alk. ext. of an alk. ext. of beef liver was highly effective but had no value in pernicious anemia. One or more substances essential to growth were adsorbed from soln. on wood charcoal, and recovered by elution with acid alk. Such charcoal elutes also contain growth stimulating substances, apparently inorg., perhaps K and Mg. VII Amino acid requirements for the Park Williams No. 8 strain of diphtheria *J. Howard Mueller and Israel Kapnick. Ibid.* 325-34 —A strain of Park 8 diphtheria bacillus grew heavily on a medium containing L-cystine, D-glutamic acid, D-valine, D-leucine, D-methionine, glycerol, inorg. salts and liver "elute". The medium is suitable for serial culture, the amt. of growth increasing with adaptation. Moderate variation in conditions under which the strain is routinely maintained may lead to differences in its amino acid requirements.

John T. Myers

**The accelerating effect of manganous ions on phage action** A. P. Krueger and N. S. West *J. Gen. Physiol.* 19, 75-86 (1935) —Dil. solns. of  $MnCl_2$  or  $MnSO_4$  accelerate the lytic effect of phage on *Staphylococcus aureus*. The acceleration results from the reduction of the quantity of phage/bacterium requisite for lysis.  $Mn^{++}$  reduces this ratio from 54 to about 12. Phage distribution is altered in the presence of  $Mn^{++}$ , in growing phage bacteria mixts., extra-cellular phage concn. is increased by  $Mn^{++}$  to about 4 times that in its absence, but phage formation is not enhanced nor is the rate of bacterial growth affected. As a consequence of the reduced lytic threshold produced by  $Mn^{++}$ , less phage is necessary to bring about the lytic destruction of the bacteria.

C. H. Richardson

**Cellular reactions to waxes from *Mycobacterium leprae***  
F. R. Sabin, K. C. Smithburn and R. M. Thomas. *J. Exptl. Med.* 62, 771-86 (1935) —The waxes from the *M. leprae*, like those from tubercle bacilli, are remarkable stimulants of cells. The crude wax sepd. from *M. leprae* is a mixt. of lipoids and other materials, and gives reactions that include the types of cell characteristic of the response to the tubercle polysaccharide, phosphatide and wax. The wax obtained from the purification of the lepra phosphatide shows similar cellulose reactions but with a greater proportion of foreign body giant cells. Leprosin, though a glyceride, corresponds in its phys. properties to the unsaponifiable material from the tubercle bacillus. It stimulates 2 strains of cells, fibroblasts and monocytes. The cellular reaction to the leprosinic acid and to the crystn. alks. is of 1 type only, represented by the foreign body giant cell.

C. J. West

## D—BOTANY

THOMAS G. PHILLIPS

**Advances in botany.** F. E. Denny. *Ind. Eng. Chem., News Ed.* 13, 474-5 (1935) E. J. C.

**Alkekengi.** Karl Meyer *Pharm. Ztg.* 80, 1211-12 (1935) —The results of investigations during recent years on this fruit are summarized. Of particular interest is its content of vitamin C.

W. O. E.

**Toxicity of ethyl alcohol for dormant and germinating seeds** Mine S. Lallemand and Mlle S. Lallemand *Bull. soc. chim. biol.* 17, 1509-21 (1935) —Lentils were used. When the dry seeds were soaked in a soln. contg. 7.81% EtOH by wt. 50% were killed in 2.5 hrs. and 100% in 24 hrs. Those soaked 24 hrs. contained 6.4% EtOH by wt. Soaking 24 hrs. in a soln. contg. 2.4% EtOH by wt. killed 50%. Other concns. and lengths of time were tried. If the seeds were first soaked for 5 hrs. or longer in water, to start germination, and then placed in the EtOH solns. for various periods, in every case a larger percentage, compared to the figures for dry seeds, survived and continued to germinate, although frequently they absorbed more EtOH than dry seeds under the same conditions.

L. E. Gilson

**Anatomy and chemical principles of the seeds of *Zygophyllum fabago* L.** Cornelis Teodorus *Compt. rend. soc. biol.* 120, 659-61 (1935) —The seed structure is described. The seeds contain oil, mucilage, amyloid and a saponin. Starch, reducing sugars and alkaloids are absent.

L. E. Gilson

**Changes in saponin content during germination and growth of the young plants of various species of Gramineae** T. Solacolu and L. Welles *Compt. rend. soc. biol.* 120, 662-4 (1935) —Saponin was present in the seeds of the 7 species of grass and 2 species of *Gulch* examined. It gradually disappeared after germination and was usually all gone by the time the fifth leaf appeared. It may serve as a foodstuff for the young plant.

L. E. Gilson

**Biochemical changes in phytopathology** Essential oil of *Lavandula vera* from plants infected with the parasite *Septoria lavandulae* Desm. R. Salgues *Compt. rend. soc. biol.* 120, 703-4 (1935), cf. C. A. 29, 8054\* —The diseased plants yielded less oil and the oil contained more cineole and less alcohols than that from healthy plants.

L. E. Gilson

**Biochemical changes in phytopathology** Changes in organic and mineral constituents due to parasite invasion R. Salgues *Compt. rend. soc. biol.* 120, 780-2 (1935), cf. C. A. 29, 8054\* —Analyses of healthy plants of the legume *Medicago lupulina* and plants infected with the fungus *Septoria medicaginis* Rob. are given. The diseased plants contained more protein and less ash. Differences in other constituents were small.

L. E. Gilson

**Effect of carotene on the neoformation of rootlets of *Impatiens balsamina*** O. Lazar *Compt. rend. soc. biol.* 120, 799-804 (1935) —The seeds were sprouted, the rootlets clipped, and the hypocotyls implanted in an agar emulsion. New rootlets formed more rapidly when a minute quantity of carotene was added to the emulsion.

L. E. Gilson

**Indispensable elements of plant nutrition** A. Frey-Wyssling *Naturwissenschaften* 23, 767-9 (1935) —Elements indispensable to plant life be in the periodic system on or near the line A-C-A, the "nutrient" line. Elements remote from this line are usually toxic.

B. J. C. v. d. H.

**Isolation of the toxic principle from a species of *Dimorpha*** *Dimorpha fruticosa* Claude Rimington and Doug. G. Steyn *Onderstepoort J. Vet. Sci.* 5, 79-80 (1935) —The cyanogenetic glucoside (*linamarin*) was isolated from the plant. The wilted leaves of the fresh plant contained approx. 1.5 g. HCN per 100 g. and the stems contained approx. 0.3 g. The dried powdered material contained approx. 0.3 g. HCN per 100 g.

K. D. Jacob

**Chemical investigation of *Dichapetalum cymosum* (Hook.) Engl.** I. Claude Rimington. *Onderstepoort J. Vet. Sci.* 5, 81-85 (1935) —Attempts to isolate the toxic principle of the plant in a chemically pure condition were

unsuccessful. The carboxyl, ketonic, aldehydic, hydroxyl, amino and imino groups were shown to be definitely absent from the toxin mol. The active principle almost certainly contains N but the mol as a whole does not evince basic characters. Propts., still highly toxic to rabbits, were prep'd contg no protein, amino acid or carbohydrate material. The toxic substance was thermostable, was not oxidized by  $\text{KMnO}_4$  or  $\text{H}_2\text{O}_2$ , and resisted boiling for 1 hr with 1%  $\text{H}_2\text{SO}_4$  or 1%  $\text{NaOH}$  solns. In addn. to the toxic principle the plant contains a catechol tannin, a yellow coloring matter (shown to be a methylpentoside, yielding rhamnose on hydrolysis), a histidine-like base, a base probably identical with choline and the alkaloid triconelline. K D Jacob

**Hydrocyanic acid in grasses.** A C Léemann *Understephot J Vet Sci* 5, 97-126 (1935).—A list is given of 88 grasses that have been investigated for the presence of cyanogenetic glucosides. A crit discussion is given of the literature on the following phases of the subject: (1) methods of extg. HCN from grasses, (2) relation between the HCN content of grasses and the lethal dose for animals, (3) conditions in the animal favoring or preventing toxicity, (4) external conditions leading to toxicity of the plant, (5) internal conditions of the plant leading to toxicity, (6) effect of free HCN on the plant, (7) HCN in glucoside or nonglucoside form and (8) HCN as an org. comp'd. Expts with *Eustachys paspalodes* and *Sorghum verticilliflorum* showed that HCl has an inhibiting effect on HCN production, so have marked alkyl and pepsin plus HCl. No HCN escapes during hay production although some may be transformed into other substances. Heating the grass to 59° (probable temp. of wilting) and 70° releases as much HCN as the  $\text{CHCl}_3$  test. HCN is lost when the grass is extd. with 42.5% alc but not when 95% alc is used. Pb acetate ppt's the enzyme with denaturation and also partly ppt's the glucoside. Al and Mn inhibit formation of HCN but Fe, Mg and Ca have no effect. One hundred and twenty-seven references. K D Jacob

**The influence of chlorides and sulfates on the intake of ammonia and nitrate nitrogen by plants.** A V Vladimirov. *Khimizatsiya Sotzialist. Zemledeliya* (Moscow) No. 3, 14-21 (1935).—Cl enters the plant faster than the  $\text{SO}_4$  ion and therefore is conducive to a higher absorption of K and  $\text{NH}_4$  and a lower absorption of nitrate. Univalent cations (K and Na) effect a higher absorption of  $\text{NO}_3^-$ ,  $\text{Cl}^-$  and  $\text{SO}_4^{--}$  and a lower absorption of  $\text{NH}_4^+$  than bivalent cations (Ca and Mg). The detg. moment of the absorption of  $\text{NH}_4^+$  is the ratio of the quantities of complementary cations and anions which enter the plant. A high absorption of  $\text{NO}_3^-$  from  $\text{NH}_4\text{NO}_3$  is favored either by a cation with a high entrance capacity, such as K, or an anion with the lowest capacity of entrance, such as  $\text{SO}_4^{--}$ . The reverse is true for the entrance of  $\text{NH}_4^+$ . The behavior of  $\text{NH}_4\text{NO}_3$  as a physiologically neutral or acid salt depends on the anions and cations of the accompanying salts. J S Joffe

**A physiological study of the entrance of lime and magnesia into plants.** K. P. Tulakova. *Khimizatsiya Sotzialist. Zemledeliya* (Moscow) No. 3, 22-34 (1935).—For barley the optimum concn is 182 mg. MgO per l. of soln. For flax, 263-344 mg. MgO per kg. of sand. A concn. of Ca and Mg in the ratio of 1:4 was not injurious to the plants investigated. Barley takes up from soln twice as much Ca as Mg. In barley the max. utilization of Ca takes place at the last stage of the vegetation period, of Mg, at the time of flowering. A high concn. of Ca stimulates the absorption of Mg by young barley plants. J S Joffe

**Determining the germination of seeds by detecting the embryo respiration with dinitrobenzene.** A. A. Gurevich. *Khimizatsiya Sotzialist. Zemledeliya* (Moscow) No. 4, 96-105 (1935).—The method is based on the property of living cells to reduce dinitrobenzene in the process of respiration. The reduction products, nitrophenyllidroxylamine and nitraniline, distribute themselves through the cell tissues and reacting with ammonia the nitrophenyllidroxylamine gives a characteristic color reaction which is an indication of the germination. J S Joffe

**The availability of adsorbed phosphoric acid to plants.** V. I. Shtatnov and S. V. Odintsova. *Khimizatsiya Sotzialist. Zemledeliya* (Moscow) 1935, No. 5, 37-45.—Iron hydroxide gel was treated with various quantities (100-500 cc.) of  $\text{H}_3\text{PO}_4$ , varying in concn. from 0.5 to 0.001 mols per l. The 0.001 M concn. showed complete removal of the  $\text{P}_2\text{O}_5$  with the 100 l or 500 l ratio of soln. to gel. In a second series of expts  $\text{NaH}_2\text{PO}_4$  was used in place of the  $\text{H}_3\text{PO}_4$ . The concns. used were 0.25, 0.10 and 0.001 M, the first two were used with gels of the ratio 300:1, the last one 500:1. The excess  $\text{NaH}_2\text{PO}_4$  was washed out and the gel with the adsorbed phosphate used in pot expts with oats. The results show that with gels of high adsorptive capacity the yields were high. Analyses of the plants and their appearance are given in tables and photographs. J S Joffe

**Studies of the physiological importance of mineral elements in plants.** VII. The effects of potassium and chloride ions on the diastase of broad-bean leaves. W. O. James and M. Cattle. *New Phytologist* 34, 283-95 (1934). cf *C* A 28, 3441.—The authors studied the distribution of K and Cl ions and diastatic activity in the plant and the effect of K and Cl starvation of the plant in these distributions. K as a nutrient increased the diastatic activity of leaves, although the K ion when added to the extd. enzyme does not appear to activate the enzyme. Cl ions added to nutrient have practically no effect on the diastatic activity of the leaves, but when added to the ext. increase the diastatic activity. The authors believe that the Cl ion may act as a direct activator of preformed diastase while K acts as a catalyst at some stage of the synthesis of the diastase complex. VIII. The variation in potassium content of potato leaves during the day. Norah L. Penston. *Ibid* 296-309.—The amt. of K in potato leaves increases to a max. at 3-4 p.m. and falls at night. Max. K coincides with max. dry wt.,  $\text{H}_2\text{O}$  and total ash. Fluctuations in amts. of K are less when transpiration is slow. It is concluded that the K is being continuously brought into the leaf by the transpiration stream and reexported to the phloem because a loss of K occurs when transpiration slows in the evening. H. R. Kraybill

**The peptidase system of *Aspergillus parasiticus*.** Marvin J. Johnson and W. H. Peterson. *J. Biol. Chem.* 112, 25-34 (1935); cf *C* A 29, 1460.—"The aminopolypeptidase of *Aspergillus parasiticus* requires a free  $\text{NH}_2$  group as a point of attachment for the enzyme. It hydrolyzes the peptide linkage adjacent to this free  $\text{NH}_2$  group. Dipeptides are not attacked unless the inhibiting influence of the  $\text{CO}_2\text{H}$  group is removed by decarboxylation. Peptides composed entirely of glycine are not attacked. The mold peptidase system contains enzymes capable of hydrolyzing peptides in which the free  $\text{NH}_2$  group is methylated or replaced with Cl. Benzoyldiglycine and triglycine are not hydrolyzed by the mold peptidase system. Although the aminopolypeptidase of mold does not attack triglycine and the leucylglycine-hydrolyzing dipeptidase does not attack diglycine, the crude mold peptidase complex splits both of these peptides. The apparent basic dissociation constants ( $K_p \times 10^9$ ) of leucylglycine, leucylglycine, triglycine and diglycine have been calcd. to be as follows, resp.: 10, 15, 19 and 14. A. P. Lohrhop

**Further observations on the iodine contents of Chinese marine algae.** P. S. Tang and C. S. Chang. *Chinese J. Physiol.* 9, 369-74 (1935), cf *C* A 30, 132.—The iodine contents of 12 algal species are reported. L. A. M.

**The histochemical detection of sodium in the plant.** Maximilian Steiner. *Ber. deut. botan. Ges.* 53, 720-32 (1935).—The Na is pptd. as sodium zinc uranyl acetate. Amounts of Na as low as 0.2-0.3 % can be detected. Other inorg. salts occurring in the plant do not interfere with the test. Lawrence P. Miller

**Growth substance and cell division.** Ludwig Jost. *Ber. deut. botan. Ges.* 53, 733-50 (1935).—Expts with various structures, such as pods of *Phaseolus multiflorus*, showed that a no. of chemically unrelated substances, including heteroauxin ( $\beta$ -indolylacetic acid), can cause cell division. These presumably act as chem. stimulants. L. P. M.

The apparent nitrogen assimilation of germinating peas (The applicability of Kjeldahl method in biological nitrogen assimilation experiments) Elizabeth M. Smyth and P. W. Wilson *Biochem. Z.* 282, 1-25(1935); cf. *C. A.* 30, 801<sup>1</sup>—Peas germinating either in distill. water or in media contg. salt solns or alkaloids invariably show an increase of 0.2 to 0.3% N in the dry residue. This increase is, as statistical studies corroborate, beyond the limits of the exptl. error. By the official Kjeldahl procedure only 90% of the total N according to Dumas can be recovered, and this varies with different modifications of the official procedure. The increase in the N is not the result of a symbiotic N assimilation during the germination but of the Kjeldahl method itself, owing to the fact that in the process N compds are formed which are more easily determinable. S. Morgulis

Effect of follicular hormone crystals on the growth of some cultivated plants K. Scharrer and W. Schropp *Biochem. Z.* 281, 314-28(1935)—Expts. were made with a variety of plants (wheat, rye, barley, oats, soybeans, sugar beet, etc.) grown in pots using follicular hormone crystals, free from the phytohormone auxin, in quantities ranging from 500 to 1000 mouse units. Undoubted increase was obtained in the growth of cereals in the following order: wheat > rye > barley > oats. In some of the plants the flowering occurred earlier under the influence of the hormone. The crop with broad beans was greatly increased, but this was not the case with soybeans. In the sugar beet 1000 mouse units caused a 14% increase in yield. S. Morgulis

$\alpha$ - and  $\beta$ -Amylase in ripening wheat grains P. S. Ugrumov *Biochem. Z.* 282, 74-8(1935).— $\alpha$ -Amylase rapidly dextrinizes, and  $\beta$ -amylase saccharifies, starch. The  $\alpha$ -component becomes inactivated during ripening and cannot be found in the dormant seed, but reappears during germination. S. Morgulis

The influence of humic acid on the assimilation of plants A. V. Blagoveschenskij and A. A. Prozorovskaya *Biochem. Z.* 282, 99-103(1935); cf. *C. A.* 29, 1134<sup>1</sup>—Humic acid acts as a stimulant when added to the nutritive soln. It increases the permeability of the membranes to  $\text{NH}_4\text{NO}_3$ . It has no effect on protein synthesis. S. M.

The role of glutathione in the metabolism of yeast Kenneth C. Blanchard *J. Bact.* 30, 442-3(1935).—The glucolytic and oxidative degradations of glucose are interrelated and apparently depend on the total glutathione content. John T. Myers

The formation of fatty acids by *Aspergillus niger* C. F. Schmidt, Jr. *J. Bact.* 30, 445(1935), cf. *C. A.* 29, 5478<sup>1</sup>—Fatty acid formation is stimulated by a concn of 0.001 M NaF and inhibited by 0.0035 M. J. T. M.

Inhibition of photosynthesis in *Chlorella pyrenoidosa* by the iodoacetyl radical Henry I. Kohn *J. Gen. Physiol.* 19, 23-34(1935)—Photosynthesis in this green alga is inhibited by  $\text{ICH}_2\text{COOH}$  and  $\text{ICH}_2\text{CONH}_2$ , both attacking the Blackman reaction. As  $\text{CH}_3\text{CONH}_2$  is without effect,  $\text{ICH}_2\text{CO}$  must be responsible. The ions of the acid penetrate so slowly that the results with it are without significance. The chromophore groups and probably other parts of chlorophyll are not involved in the effects of the  $\text{ICH}_2\text{CO}$  radical. As cyanide also inhibits photosynthesis by way of the Blackman reaction, this complex is probably attached at 2 different loci on the same or on different mols. The  $\text{ICH}_2\text{CO}$  radical also (1) may increase the rate of reaction with the photochem. complex if in concn. too small to inhibit photosynthesis, (2) will increase the rate of respiration in concns which inhibit photosynthesis and (3) will inhibit respiration if in concn. greater than is required to inhibit photosynthesis. C. H. R.

The reversible heat activation inducing germination and increased respiration in the ascospores of *Neurospora tetrasperma* David R. Goddard. *J. Gen. Physiol.* 19, 45-60(1935)—The ascospores of this fungus, activated by heat, may be returned to secondary dormancy by preventing respiration. From this secondary dormancy the spores may be induced to germinate by reheating. Activation brings about a large increase in respiration prior to germination, deactivation, a large decrease. Poisoning

the spores with iodoacetamide prevents germination without greatly inhibiting the increase in respiration. With the beginning of germination, a secondary rise in respiration occurs. Respiration is inhibited by HCN. The critical temp. for heat activation of the spores lies in the range of 40° to 52°; at a const. temp. within this range, the percentage of spores activated, plotted against time, gives a sigmoid population curve. C. H. R.

Physical and chemical properties of the soluble polysaccharides in sweet corn. M. W. Parker. *Plant Physiol.* 10, 713-25(1935), cf. Culpepper and Magoon, *C. A.* 18, 3632—Starch-free electroanalyzed water-sol polysaccharides of sweet-corn endosperm were found to consist of two fractions tentatively named  $\alpha$  and  $\beta$ . One fraction was deposited as a gelatinous mass around the pos. membrane, whereas the other always remained in suspension during electroanalysis. A comparison of the phys. and chem. properties of the two fractions at the milk, dough and mature stages showed that with increasing maturity many of the properties of the  $\alpha$  and  $\beta$  fractions of the sol. polysaccharides approached those of the  $\alpha$  and  $\beta$  amylose prep. from sweet-corn starch, but differed greatly from the dextrins. It is suggested that the sol. polysaccharides of sweet corn are the units for the formation of starch grains. Twenty-five references. Walter Thomas

Seasonal march of carbohydrates in *Elymus ambiguus* and *Muhlenbergia gracilis*, and their reaction under moderate grazing use. Edward C. McCarty. *Plant Physiol.* 10, 727-38(1935)—The seasonal march of the sugars plus starch of the grasses *Elymus ambiguus* and *Muhlenbergia gracilis* grown on exptl. plots on the eastern slope of Pike's Peak was in inverse ratio to the rate of growth. Clippings made periodically indicated that the amt. of herbage growth following the clipping and also the concn. of the accumulated carbohydrates were approx. proportional to the number of days between the date of the clipping and the end of the annual growth cycle. Walter Thomas

Growth and seasonal changes in composition of oak leaves Arthur W. Sampson and Rudolf Samisch. *Plant Physiol.* 10, 739-51(1935).—N and "crude fiber" accumulated at a very early stage of growth in the leaves of *Quercus gambelii* (Utah) and *Q. kelloggii* (California), but as leaf expansion continued both N and "crude fiber" decreased on a unit-area basis during the latter part of the season. Ether-sol. substances as well as Ca and  $\text{SiO}_2$  accumulated continuously throughout the season, whereas K increased rapidly in the spring and decreased on a unit-area basis later in the season. Walter Thomas

Observations on the cracking of cherries Z. I. Kertesz and B. R. Nebel. *Plant Physiol.* 10, 763-72(1935), cf. Hartman and Bulls, *C. A.* 24, 1883, Verner and Blodgett, *Idaho Agr. Exptl. Sta. Bull.* 184, 1931—The cracking of cherries is attributed to the forces of the swelling colloids of the flesh rather than to osmotic forces. Walter Thomas

Unfreezable and freezable water equilibrium in plant tissues as influenced by sub-zero temperatures Glenn A. Greathouse. *Plant Physiol.* 10, 781-88(1935)—Expressed in percentage of the total water, the unfreezable water values of cold hardened red clover roots decreased 1.40% with lowering of the temp. from -15° to -22° (cf. Jones and Gortner, *C. A.* 26, 1497; St. John, *C. A.* 26, 357) whereas the unfreezable water values of unhardened root tissue decreased 6.02% over the same temp. range and 13.04% over the temp. range -10° to -50° (cf. Robinson, *C. A.* 25, 5687, Thoenes, *C. A.* 19, 3497). The behavior of potato tubers was similar to that of unhardened red clover roots. Different unfreezable water values were obtained when the plants were grown under different conditions. Walter Thomas

Hormones in relation to root formation on stem cuttings Wm. C. Cooper. *Plant Physiol.* 10, 789-94(1935)—Girdled lemon-tree cuttings placed in sand below the girdle and treated with the plant hormone  $\beta$ -indolylacetic acid above the girdle according to the method of

Laibach (C. A. 30, 131<sup>1</sup>) showed not only increased rooting with cuttings that rooted when untreated but also the formation of roots on leafless cuttings which did not ordinarily form roots. Similar results were obtained with *Tradescantia*. The expts are being continued with other plants.

Walter Thomas  
Some chemical aspects of calcium-deficiency effects on *Psidium sativum* Dorothy Day *Plant Physiol* 10, 111-116 (1935), cf. C. A. 24, 1134.—Ca-deficient plants weighed least on both fresh-wt and dry-wt basis. The percentage on dry matter was highest in the shoots and lowest in the roots of Ca-deficient plants. At the end of the growth period of 5 weeks the plants starved of Ca had 1/3 as much Ca as those which received the highest supplement of Ca. The addn. of NaNO<sub>3</sub> to the nutrient media depressed the uptake of Ca by the plant. The expts were conducted in sand cultures in a greenhouse under controlled conditions. Walter Thomas

Some effects of fuel oil on plants Geo D Tuller and Margaret R Leadbeater *Plant Physiol* 10, 817-820 (1935)—Gas oil of 3236 Baumé gravity and 55 seconds Saybolt consisting principally of said hydrocarbons had a harmful effect when applied to tomato and peach plants in amts of 1, 2, 3, 4 and 5% by vol. of the soil content of the pots in which the plants were grown. The effect is fatal when the quantity is raised above the crit. point which varied for different species of plants. The influence of the oil on the soil is unknown. The effect, however, is not necessarily the result of penetration into the tissues of the plant. If the oil has penetrated the tissues it is found in the primary xylem, and to a less extent in the secondary xylem and the intercellular spaces of the pith and cortex. Contact does not seem to be a primary cause of death. The expts are being continued. Walter Thomas

Effect of titanous chloride on the formation of chlorophyll in *Zea mays*. O. L. Inman, Geo Barclay and Malvern Hubbard *Plant Physiol* 10, 821-2 (1935), cf. Nemec and Kas, C. A. 18, 1516.—Expts with *Zea mays*, in which the nutrient soln used by Siders (*Pineapple News* 4, 98 (1930)) was used, indicated that in the formation of chlorophyll Ti cannot be substituted for Fe. W. T.

Sublethal damage from the arsenic acid in the white smelter smoke of the As metallurgical plant (Hüsch) 9 I fluorescence spectrochemistry of the chlorophyll pigments (Dhéré, Raffy) 3

## E—NUTRITION

PHILIP D HAWK

Metabolism during an abundant protein diet I. L. D. Katshevnik, S. A. Neifakh and A. Yu. Kharit. *J. Physiol* (U. S. S. R.) 19, 508-24 (1935).—Feeding expts. lasting more than a year were carried out with men metal workers and women textile workers. The men were given daily 225-250 g. of proteins, the women, 150-215 g. All the usually accepted indices of N metabolism (N balance, ratio of urea N to general N, ratio of creatinine N to total N, residual N of blood) are the same as under normal conditions.

H. Cohen  
Feeding of sweet lupines through four generations (experiments on rats). A. Columbus Biedermanns Zeits. B. Tierernähr. 7, 543-57 (1935).—Three kinds of sweet lupines, free from the bitter substance, were fed to 4 generations of rats. The proportion of the lupines in the total food was varied during the expts. Up to 60%, the general development of the older rats was favorable; with 80% the development was arrested and with 100% it was impaired. With 60%, the litters thrived very well; with 80% some of the young died, while with 100% breeding of litters was impossible. No essential differences were observed with the several lupines, although the litters receiving the lupine thrived better than the check litters. Dissections of the rats did not indicate that there had been any injurious effects on the organs. F. L. D.

Composition and vitamin studies of green soybeans. Carey D. Miller and Ruth C. Robbins. *Hawaii Agr. Expt. Sta. Rept.* 1933, 21-5 (1934).—Cooked green soybeans are a good source of vitamins A and B. Gains in wt. in rats

on vitamin G tests of 28 g. a week resulted from feeding a daily supplement of between 1 and 2 g. of the cooked green beans daily. Green soybeans contained the following percentage compn moisture 60.1, protein 12.5, Et<sub>2</sub>O ext. 5.2, crude fiber 1.5, ash 1.8, carbohydrates 10.1, Ca 0.063, P 0.239 and Fe 0.00253. C. R. Fellers

Vitamin C in fresh pineapple juice and in guavas. Carey D. Miller and Ruth C. Robbins. *Hawaii Agr. Expt. Sta. Rept.* 1933, 25 (1934).—Guinea-pig bioassay showed fresh pineapple juice to be about 1/2 as rich in vitamin C as fresh orange juice. Fresh guava juice was equally as good as fresh orange juice, the protective level of which is stated to be 3 g. daily. One g. daily of fresh guava fruit completely protected guinea pigs from scurvy. C. R. Fellers

Relation between the vitamin A and D intake by the hen and the output in eggs. Walter C. Russell and M. W. Taylor. *J. Nutrition* 10, 613-23 (1935), cf. C. A. 30, 529<sup>1</sup>.—The output of vitamin A in eggs, calcd. as the percentage of that consumed, varied from 11 to 32%, and was det'd by the no. of units of the factor consumed, the no. of eggs produced and the potency of the yolk. The highest percentages were produced during high production. The liver vitamin A of the hens varied inversely with egg production. With vitamin D the amt which appeared in the eggs was approx 10% of that consumed. It is suggested that sunlight is more effective in increasing the antirachitic potency of egg yolk than the amt of cod-liver oil ordinarily fed. C. R. Fellers

Study of the nutritive value of mushrooms. F. W. Quackenbush, W. H. Peterson and Harry Steenbock. *J. Nutrition* 10, 625-43 (1935).—Diets contg mushrooms, *Agaricus campestris*, were consumed in subnormal quantities and growth was also subnormal. The mushrooms were relatively good sources of B and G vitamins inasmuch as levels of 10 and 5% of the diet on a dry-wt. basis gave satisfactory growth. A diet contg 10% of mushrooms as the only source of vitamin B was deficient in some factor other than B and G vitamins. Preliminary data indicate that mushroom protein is incomplete. There was no evidence of a toxic principle. The investigation of the deficiencies in mushrooms was hampered by the poor consumption by the rats. C. R. Fellers

Effect of the ingestion of saline waters upon the pH of the intestinal tract, the nitrogen balance and the coefficient of digestibility. V. G. Heller, J. R. Owen and Lucille Portwood. *J. Nutrition* 10, 645-51 (1935).—The use of drinking waters contg. considerable quantities of dissolved salts does not interfere with the N utilization of normal rats by the rat. Likewise the apparent coeffs of digestibility of the constituents of the ration are not interfered with, in fact, the trend of all detns indicates that assimilation or digestibility is aided by the presence of reasonable amts. of salts so long as the total content is kept below the concn where serious disturbances in growth and reproduction take place. The pH of the entire intestinal tract is not appreciably altered by the presence of alkali or acid salts in the drinking water, providing the concn. present does not exceed the amt. permitting a somewhat normal life. Observed changes are more apparent in the stomach than in the lower intestine. C. R. Fellers

Differential antirachitic activity of vitamin D milks. Robert W. Haman and Harry Steenbock. *J. Nutrition* 10, 653-64 (1935).—For the chick and per unit of vitamin D, cod-liver oil and irradiated cholesterol have approx. the same order of effectiveness. Yeast milk is approx. 1/10 as effective as irradiated milk. The difference was confined to the resp. butter-fat fractions and was uninfluenced by the skimmed-milk fraction. The constituents of milk as a vehicle for vitamin D do not influence its effectiveness. The data do not support the possibility that the baby chick could be used to greater effectiveness than the rat for ascertaining the degree of antirachitic effectiveness of different D vitamins for the human being. C. R. Fellers

Studies on growth III. B and G avitaminosis in cecotomized rats. Wendell H. Griffith. *J. Nutrition* 10,

667-74(1935), cf. C. A. 27, 4563.—Young rats grow normally for at least 100 days after removal of the cecum. If vitamins B, C or G are synthesized by cecal microorganisms, they are not utilized by the rat except by coprophagy. The prolonged survival of young rats on vitamin G-deficient diets is not due to a cecal supply of vitamin G. IV. Vitamin B and G content of the body tissues of normal and experimental rats. *Ibid* 675-82.—Young rats maintained on a vitamin B-deficient ration were readily depleted of most of the vitamin B originally present in the tissues. A corresponding loss of vitamin G did not occur in young rats on a G-deficient ration. The prolonged survival of rats on a G-deficient ration is related to the retention of tissue vitamin G. It is suggested that vitamin G may function in part as a tissue constituent.

C. R. Fellers

Variability of vitamin D response with temperature of environment. D. Tourtellotte and W. E. Bacon. *J. Nutrition* 10, 653-8(1935).—To a certain extent the variation in the sensitivity of the rachitic test animals is due to fluctuating lab temps during the rachitogenic and test periods. An increased severity of rachitogenesis results in vitamin D test animals when they are subjected to lab temps of 26.6° or above. By reducing the CaCO<sub>3</sub> in the McCollum diet no 3143 from 3 to 2%, good line test rickets have been produced in 21 days during periods of hot summer temps. It is fully as desirable to define a standard rachitic test animal in terms of response to a definite amt of a standard vitamin D prep as it is to prescribe the exact conditions for rachitogenesis and methods for administering the test prep.

C. R. Fellers

Supplemental value of peanuts to the laying ration (of hens). D. F. King and G. J. Cottler. *Ala. Agr. Expt. Sta., 45th Ann. Rept.* 23-4(1934).—Hens fed peanut meal as the sole protein supplement deposited a very soft fat in their bodies in comparison to hens fed a supplement of skim milk. The eggs of the peanut-fed birds also contained a very soft fat. The addition of skim milk to the peanut ration so as to supply 50% of the supplementary protein resulted in higher egg production, body wt., egg size and egg quality.

C. R. Fellers

Absorption and utilization of carbohydrates. H. B. Pierce. *J. Nutrition* 10, 693-716(1935).—A review with 87 references covers the absorption of carbohydrates from the stomach and intestine of man and animals, factors influencing the removal of carbohydrates, assimilation limits of different sugars and effect upon blood-sugar levels, relative ease of oxidation of carbohydrates, and the formation of glycogen and fat after the administration of carbohydrates to man or animals.

C. R. F.

Menhaden fish meal as a protein supplement for dairy cows. C. F. Monroe, W. E. Krauss and C. C. Hayden. *Ohio Agr. Expt. Sta., Bi-monthly Bull.* 176, 176-83(1935).—A grain mixt. contg. 8% of menhaden fish meal when compared with a check mixt. contg. the usual protein supplements of linseed and cottonseed meals produced slightly less milk and butter fat. The grain intake was lower on the fish meal ration. In general health and phys. appearance there was no difference in the 2 groups of animals. The reproductive history was somewhat better in the case of the cows fed fish meal over a 28-month period.

C. R. Fellers

Vegetable protein in turkey rations. J. E. Hunter, D. R. Marble and H. C. Kandel. *Pa. Agr. Expt. Sta., Bull.* 321, 3-13(1935).—In the mash used as a basal ration for the period 1-13 weeks, the protein from 50% dried milk powder and 50% of either the meat or fish meal was satisfactorily replaced by either soybean-oil meal or corn gluten meal, when Ca and P losses were compensated. Corn gluten meal produced a superior fleshing condition. Fourteen % soybean-oil meal or 12.5% corn gluten meal may replace equiv. amts of animal protein in the 24% turkey starter. From 33 to 66% of the total animal protein in the starting ration can be replaced without harm by soybean-oil or corn gluten meals when the Ca and P losses are compensated.

C. R. Fellers

Nutritional aspects of milk pasteurization. E. V. McCollum. *Pub. Health News, N. J. Dept. Health* 19,

1387-9(Feb. 1935); U. S. Pub. Health Eng. Abstracts 15, M1, 15(June 15, 1935).—There is no evidence that raw milk is superior to pasteurized milk in infant feeding. In fact, the latter is better digested by infants and possesses the added factor of safety.

C. R. Fellers

Effect of adrenaline on the utilization of fructose injected intravenously at a constant rate into dogs in three states of nutrition. M. Wierzechowski and H. Fiszal. *Compt. rend. soc. biol.* 120, 377-80(1935); cf. C. A. 29, 5829-30. Fructose was injected at the rate of 2 g./kg./hr. and frequent analyses of the blood and urine were made. In group 1, previously given a carbohydrate diet, the assimilation of the fructose was 94%; in group 2, meat-fed, it was 90%; in group 3, fasted 10 hrs., 83%. The injection of adrenaline with the fructose as in previous expts. caused large increases in the fructose and glucose contents of the blood and urine, i. e., fructose assimilation was decreased and glycogenolysis increased.

L. E. Gilson

Whipple's method in experimental anemias. Anemia produced by bleeding. André Arthus, M. Lourau and G. Silvestre de Sacy. *Compt. rend. soc. biol.* 120, 383-9(1935).—Rabbits were made anemic by bleeding. Feeding whole liver pulp hastened recovery; injections of hydro-alc. ext. of liver had no such effect. The active principle is probably different from that which is effective in pernicious anemia. Lead anemia. *Ibid* 553-4.—In rabbits made anemic by injections of Pb(OAc)<sub>2</sub>, the ext. mentioned above effected speedy recovery.

L. E. G.

Normal ascorbic acid content of the organism. A. Giroud, C. P. Leblonde, R. Ratsimamanga and M. Rabinowicz. *Compt. rend. soc. biol.* 120, 414-15(1935).—From an examn. of published data from various sources the following averages for common domestic animals were obtained: adrenals 1.77, liver 0.25 and muscles 0.024 mg. ascorbic acid per g. fresh tissue. For rats and cats on a diet free from vitamin C the corresponding values were 1.49, 0.19 and 0.016. Probably an ascorbic acid content of less than 1 mg./g. of adrenal tissue should be considered a pathol. condition. Attaining a normal ascorbic acid content in animals which require ascorbic acid in their diet. *Ibid* 633-5.—The ascorbic acid of the adrenals of a normal guinea pig is between 1 and 1.5 mg./g. To maintain it at the 1 mg. level the daily ration must contain an av. of 30 mg. ascorbic acid and to maintain it at a higher level much more is required. Feeding expts. are described. Reactions of animals, which require ascorbic acid, to increasing doses of the same. A. Giroud, R. Ratsimamanga, A. Baratte and P. Sylva. *Ibid* 701-3.—Guinea pigs given but 1 mg. ascorbic acid per day lived and grew. Two mg. per day was required to prevent anemia and malformation of the teeth. More than 5 mg. per day was required to prevent hemorrhagic lesions in the joints.

L. E. Gilson

Action of vitamins A, C and D on the normal thyroid and on the thyroid made hyperactive by treatment with the thyrostimulin of the anterior hypophysis. Inhibiting action of vitamins A and C in experimental hyperthyroidism. A. W. Elmer, B. Giedost and M. Schepp. *Compt. rend. soc. biol.* 120, 500-2(1935).—In moderate doses vitamins A, C and D do not have any effect on the histological structure of the thyroids of normal guinea pigs. A and C arrest the hyperactivity produced by thyrostimulin.

L. E. Gilson

Influence of diet on milk. Chemical change and sensitization in the alcohol coagulation test. Luis Echebague and Bernardo Suarez. *Compt. rend. soc. biol.* 120, 500-2(1935).—Milk from cows which had eaten large quantities of green *Xanthium caeruleum* (a weed related to the cockle bur) with other forage gave a pos. alc. coagulation test (method not described) although the acidity was normal. The effect is ascribed to an increase in the Ca content of the milk. No toxic effect on the cows was observed.

L. E. Gilson

The relation of adrenal cortical hypertrophy to muscular work and the vitamin B content of the food. Aladár B. L. Bernák and János Perjes. *Arch. ges. Physiol. (Pflügers)* 236, 181-9(1935).—See C. A. 29, 7426.

A. G.

The fertility vitamin E and its therapeutic importance

for gynecology. E. Gierhake. *Deut med Wochschr.* 61, 1674-6 (1935).—Malnutrition may lead to sterility in the human subject, presumably because of a deficiency of vitamin E. Its therapeutic use is therefore indicated. A G

Dicalcium phosphate [in prevention of dental caries] Fred Carmosin. *Dental Cosmos* 77, 1200-2 (1935).—Expts. on children over a period of 8 months demonstrated that addn. of  $\text{CaHPO}_4$  and milk to the diet was superior to the use of  $\text{CaHPO}_4$  and cod-liver oil as a preventive of dental caries. Joseph S. Hepburn

Is linoleic acid essential in the nutrition of rats? Eugene Becker. *Z. Vitaminforsch.* 4, 241-9 (1935).—Rats require small amts of linoleic acid for the maintenance of normal growth, and develop a specific deficiency disease in its absence, this disease is cured by daily doses of the acid in the form of walnut oil. Growth curves run parallel to the linoleic acid content of the oil. During its isolation from natural oils, linoleic acid may be transformed into an isomeric compd., and may also be altered with respect to this vitamin-like action. Joseph S. Hepburn

Experimental contribution to B-avitaminosis in the rat Egon Kodicek and Jiri Joachim. *Z. Vitaminforsch.* 4, 250-5 (1935).—B-avitaminosis was produced in 27 adult male rats, the majority developed the nervous type. The concn. of sugar and cholesterol in the blood and the erythrocyte count remained unaltered. The hyperglucemia, which has been reported in polyneuritic pigeons, probably was due to an increase in nonsugar-reducing compounds. Joseph S. Hepburn

Vitamin A and carotene content of human milk W. Neuweiler. *Z. Vitaminforsch.* 4, 259-71 (1935).—Human milk contains 25-300 rat units of vitamin A per 100 cc. The content is independent of the age, no. of pregnancies and daily vol of milk, is greatest in colostrum, is not essentially altered by standing for 48 hrs., increases by 100% during the day, is greater after suckling than before, and can be increased by a very high intake of vitamin A or of carotene. The carotene content of human milk is 0.005-0.4 mg per 100 cc., occurs chiefly in the colostrum and early stage of lactation, and in slight quantity or traces in the mature stage, is greater after suckling than before, and can be increased somewhat by a high intake of carotene. Joseph S. Hepburn

Dietary depigmentation of young black and pied rats, promoted by rapid growth, prevented and cured by ingestion of copper. Frederick J. Gortner. *Z. Vitaminforsch.* 4, 277-83 (1935), cf. *C. A.* 29, 7413<sup>1</sup>.—Yeast did not always prevent or cure the depigmentation, its curative effect was proportional to its Cu content. With young growing rats and  $\text{CuSO}_4$ , the preventive dose was 0.02-0.12 mg Cu per rat per diem, the curative dose 0.07-0.24 mg Cu per rat per diem. Other metals (Zn, Mn, Co, Ni, Fe) lacked curative power. Rapid growth on a ration rich in protein, sugar, vitamins A, B<sub>1</sub> and B<sub>2</sub> markedly increased the Cu requirement. Adult rats were less susceptible to complete depigmentation. At times depigmentation was promoted by yeast, egg white, fat horse flesh, milk, butter and cod-liver oil, and was not produced by bread and cereal products. The former foods promote growth; and the Cu of the latter food is more readily available. Joseph S. Hepburn

Aseptic culture of insects in vitamin research E. G. van't Hoog. *Z. Vitaminforsch.* 4, 300-24 (1935).—*Drosophila melanogaster* was reared successfully from eggs sterilized by treatment for 35 min. with a mixt. of 44 cc. 96% alc. and 6 cc. 5% soln. of chloramine. These insects required the B vitamins and the active factor in the unsaponifiable fraction of fats, but did not require vitamins A, D and E, and unsatd. fat acids. Cultures of the insects were of value as a biol. test for vitamins B<sub>1</sub> and B<sub>2</sub>. Joseph S. Hepburn

Vitamin C or the reducing power of the urine in health and disease. Theophil Baumann. *Z. Vitaminforsch.* 4, 334-6 (1935).—The iodometric titration of ascorbic acid in the urine is unsatisfactory since other urinary constituents react with I, the ascorbic acid content decreases with time even in a strongly acidified urine, and other

comps. which react with I are produced as urine acids. Joseph S. Hepburn

Vitamin C or the reducing power of the urine. Wolf v. Drigalski. *Z. Vitaminforsch.* 4, 350-7 (1935); cf. *C. A.* 29, 6627<sup>1</sup>.—The iodometric titration of ascorbic acid in the urine is approx., but of value, the amt. of other reducing comps. is so slight as to be negligible. Samples should be analyzed promptly. Joseph S. Hepburn

Comparative study of the C vitamin (ascorbic acid) content of cerebrospinal fluid and of urine during C hypovitaminosis F. Plaut and M. Bulow. *Naturwissenschaften* 23, 771 (1935), cf. *C. A.* 30, 510<sup>3</sup>.—Comparative tests showed agreement between analyses of cerebrospinal fluid (*Klin. Wochschr.* 14, 1318 (1935)) and urine (Harris-Ray, *C. A.* 29, 3005<sup>2</sup>). After doses of 600 mg. ascorbic acid (Redoxan) per os daily, patients with high ascorbic acid content in the cerebrospinal fluid (1.8 to 2.5 mg %) began to excrete it in the urine the second day, those with low ascorbic acid content (0.2-0.3 mg %) not until the 7th or 9th day. The first group excreted 44 to 53 mg. daily max., the second group 158-298 mg. Prior to the feeding of ascorbic acid there was hardly any excretion and no difference between the 2 groups. B. J. C. van der Hoeven

The effects of varying amounts of animal protein fed to White Leghorn pullets II. Factors correlated with egg-size and number of eggs C. C. Rhodes, L. H. Bartel and P. E. F. Jooste. *Empire J. Exptl. Agr.* 3, 313-19 (1935), cf. *C. A.* 29, 6628<sup>1</sup>.—Pullets receiving either low-, medium- or high-protein rations showed pos. correlations between max. body-wt. and av. annual egg-wt., but only those of the medium- and high-protein groups were significant. K. D. Jacob

Report of Wallaceville veterinary laboratory C. S. M. Hopkirk. *New Zealand Dept. Agr., Ann. Rept.* 1934-35, 25-31.—*Tympany of the rumen in dairy cows*.—There were wide variations in the CO<sub>2</sub>, O<sub>2</sub>, CH<sub>4</sub>, N<sub>2</sub> and H<sub>2</sub> contents of the rumen gases from both normal and bloated cows. The picrate paper test showed no excess of HCN in the rumen contents of bloated cows. Cows given HCN in nonlethal doses did not bloat when placed on damp early-morning pasture. There were indications that bloat may be associated with a decrease in the normal acidity of the rumen contents. *Grass staggers in dairy cows*.—Grass staggers was overcome by increasing the blood Mg of the cows through the addn. of dolomite to the feed. The Mg content of pasture grass was markedly increased by treating the pastures with  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  at the rate of 800-4000 lb/acre; dolomite was less effective. The milk and bones of affected animals contained normal amts of Mg but the urine, like the blood, was very low in Mg. Pasture grasses at Waikato tend to be lower than normal in Mg, the spring grass sometimes contg. as low as 0.3% Mg, the seasonal variation is not marked, the lowest value occurring in Dec., with increased values in the growth after Feb. rains. *Morlon Mains disease of lambs*.—The blood of affected animals contained normal amts of Ca and Mg but was slightly below normal in inorg. P and total solids, there was a slight degree of imperfect calcification of the bones. *Vitamin content of New Zealand fish oils and pasture grasses*.—The vitamin D content of eel-body, ling-liver, proper-liver and red-cod-liver oil was 47, 500, 2250 and 10 international units/g., resp. Whale-body oil contained no vitamin D. The vitamin A content of fresh grass was higher than that of hay, while the vitamin D content was the same in both cases. K. D. J.

The effect of supplementing the diet with different forms of sulfur upon the wool of Merino sheep C. M. van Wyk, M. L. Botha and J. G. Bekker. *Onderstepoort J. Vet. Sci.* 5, 177-88 (1935).—Supplements of either cystine, sulfates, KCNS or S (0.12-5.00 g.), administered daily except Sunday for 1 year, had no effect. K. D. Jacob

Mineral metabolism XXXIII. Iodine in the nutrition of sheep. Second report A. I. Mahn, P. J. Du Toit and J. W. Groenewald. *Onderstepoort J. Vet. Sci.* 5, 189-200 (1935); cf. *C. A.* 27, 5381<sup>1</sup>.—KI administered to Merino sheep at the rate of 0.002-0.06 g. daily for 30 months had no visible effects on the wts., wool production



and reproduction of the animals. It is pointed out that the ill effects ascribed to KI feeding in the earlier work were apparently aggravated by a deficiency of vitamin A in the diet. Conclusion: The addition of KI to sheep licks is unwarranted and may even produce detrimental effects under conditions of drought when a vitamin A shortage may exist. K. D. Jacob

Further studies pertaining to provitamin D of plant and animal sources. R. M. Bethke, P. R. Record and O. H. M. Wilder. *J. Biol. Chem.* 112, 231-8 (1935), cf. C. A. 28, 7311<sup>1</sup>.—Vitamin D of irradiated cholesterol is more efficacious in preventing rachitic manifestations in chicks than the rat unit equiv. in irradiated ergosterol or in calciferol. The effects of the latter 2 substances were of the same order of efficiency. The antirachitic factor of irradiated cholesterol is as potent for chicks as the vitamin D of cod liver oil. Equiv. rat units of vitamin D from irradiated animal products such as hog brains, butter fat and lard are more efficient antirachitically for chicks than vitamin D from irradiated plant products such as wheat middlings, cotton oil, alfalfa leaf meal, yeast and fungus mycelium, indicating that the provitamins of animal and plant products are not identical. There is apparently no taxonomic difference between the vitamin D efficiency of irradiated products from higher plants (cottonseed oil, wheat middlings, alfalfa leaf meal) and irradiated plants (yeast and fungus mycelium) of the lower botanical order. A. P. Lothrop

Amino acid metabolism. I. Fate of glycine, *d,l*-alanine and *d*-alanine in the normal animal. Joseph S. Butts, Max S. Dunn and Lois F. Hallman. *J. Biol. Chem.* 112, 203-74 (1935).—The ability of the amino acids to form glycogen was tested by feeding their Na salts by stomach tube to rats fasted 48 hrs. and the testing of ketolytic properties by feeding Na acetoacetate and superimposing upon this the substance whose ketolytic activity was to be studied. Glycine, *d,l*-alanine and *d*-alanine are definitely glycogenic and ketolytic, *d*-alanine being very much superior to glycine and *d*-alanine approx. twice as effective as the *d*-form as a glycogen former and definitely superior to it as a ketolytic agent. As far as glycogenic properties are concerned, it appears that only *d*-alanine is effective and that in the racemic mixt. *l*-alanine is very ineffective. A. P. Lothrop

Feeding experiments with mixtures of highly purified amino acids. VII. The dual nature of the "unknown growth essential". Madelyn Womack and Wm. C. Rose. *J. Biol. Chem.* 112, 275-82 (1935), cf. C. A. 29, 4800<sup>1</sup>.—The "unknown growth essential" referred to in earlier papers of this series is composed of 2 factors which can be separated by their soly difference in aq. BuOH. The more sol. component is isoleucine. Failure to recognize the importance of isoleucine as a constituent of the growth-stimulating fractions delayed the identification of the other component of the "unknown growth essential" for at least 2 yrs. VIII. Isolation and identification of a new essential amino acid. Richard H. McCoy, Curtis E. Meyer and Wm. C. Rose. *Ibid.* 283-302. The hitherto unknown growth essential has been isolated in crystalline form and identified as 1 of the 4 isomeric  $\alpha$ -amino- $\beta$ -hydroxybutyric acids. The spatial configuration around the  $\beta$ -carbon remains to be determined. Approx. 0.6% is the min. amt. required to induce max. growth on an otherwise adequate diet and for the first time it has been possible to rear animals on a ration containing purified amino acids as the sole source of N. Hydroxyglutamic acid and citrulline have been shown by feeding expts. to be dispensable components of the food. Expts. are under way to determine the physiological importance of the remaining amino acids:  $\alpha$ -Amino- $\beta$ -hydroxybutyric acid, in 255-7<sup>1</sup> with decanoin,  $\alpha$ -Isoleucine, 27-77<sup>1</sup> (av. of 4 preps.), *N*-benzoyl serine,  $\gamma$  151<sup>1</sup>, picric acid, in 139-5-141<sup>1</sup>. A. P. Lothrop

Magnesium deficiency in animals. VII. The effects of magnesium deprivation, with a superimposed calcium deficiency, on the animal body as revealed by symptomatology and blood changes. Harry G. Day, H. D. Kruse and I. V. McCollum. *J. Biol. Chem.* 112, 337-59 (1935), cf. C. A. 28, 6815<sup>1</sup>.—Young dogs were fed a diet deficient

in both Ca and Mg but otherwise adequate. The dietary deficiency is manifested by edema, gastrointestinal disturbance with alternate constipation and diarrhea, osteoporosis with consequent deformation of the bones, in a few cases by hyperirritability of the nervous system which led to fatal convulsions, but in the majority of cases by a marked inertness and anorexia which usually led to death from inanition. An immediate decrease in serum Mg occurs which is quantitatively similar to that which induces tetany in Mg deprivation and serum Ca likewise falls to levels usually indicative of tetany. There is a fall in alk. reserve and  $p_{H_2}$ , an increase in blood chlorides as the erythrocyte vol. falls and an increase in fibrinogen. A rise in nonprotein N and a decrease in inorg. phosphate occur terminally. There are no significant changes in serum Na and K, plasma total lipids, fatty acids, total cholesterol, cholesterol esters, lipid P, serum bile pigment and plasma albumin and globulin. The deficiency in Ca-Mg deprivation resembles more closely the low-Ca syndrome. The so-called physiologic antagonism between Ca and Mg is indicated by the prevention of symptoms of Mg tetany by the simultaneous diminution of serum Ca as serum Mg reaches abnormally low levels. Nutritive failure is more pronounced in Mg-Ca deficiency than in Mg restriction alone. Forty-six references. A. P. L.

The influence of iodides on the calcification of bones in rats in experimental rickets. Raoul Lecoq and Romuald Gallier. *Bull. sci. pharm.* 42, 528-8 (1935), cf. C. A. 29, 7412<sup>1</sup>.—Rickets was produced by a diet rich in Ca and poor in P. The addition of  $P_2O_5$  alone is not sufficient to cause improvement, but the addition of KI or  $CaI_2$  produces a prompt cure. A. E. Meyer

Sugared condensed milk in artificial alimentation. P. Rudeaux. *Presse med.* 43, 753-5 (1935).—Different formulas for diln. are developed. A. E. Meyer

Vitamin C. Ph. Pagniez. *Presse med.* 43, 801-5 (1935).—A discussion. A. E. Meyer

Vitamin C in hemorrhagic diathesis. A. Lunedei and A. Gnamoni. *Sperimentale* 89, Section Rendiconto adunanza accad. med. fis. fiorentina 20 (1935).—High doses of ascorbic acid have a favorable influence in hemorrhagic conditions. A. E. Meyer

Effect of different forms of diet on the external secretion of the pancreas. Leop. Abramson. *Acta Med. Scand.* 86, 478-85 (1935).—Results indicate that the enzyme content of the pancreatic secretion on a mixed diet or on a diet poor in protein but rich in milk and carbohydrate tends to adjust itself to the character of the diet. S. Morgulis

Studies on protein metabolism and the oxidative processes in experimental scurvy. II. Influence of ferrum hydrogenum reduction on the nitrogen balance and oxidation coefficient. L. D. Kashevin, S. A. Lidman and I. B. Friedland. *Biochem. Z.* 282, 56-61 (1935), cf. C. A. 29, 4802<sup>1</sup>.—Daily feeding of 10 mg. reduced Fe to guinea pigs during the pre-scurvy period causes an increased N metabolism and output of urea N, while the oxidation coeff. (O/N) tends to fall. On the scorbutic ration the Fe has no effect on the excretion of protein metabolic products. A neg. N balance is found at the time when the development of scurvy is complicated by the condition of partial starvation. Under the influence of the Fe there occurs an extensive deposition of fat in the then scorbutic guinea pigs. S. Morgulis

The storage of ascorbic acid in the organs of guinea pigs given crystalline ascorbic acid with a vitamin C free diet. Erik Jacobsen. *Scand. Arch. Physiol.* 72, 259-64 (1935), cf. C. A. 29, 8988<sup>1</sup>.—The vitamin C deposits of guinea pigs are emptied when the animals have been on a vitamin C free diet for 8-15 days. On a daily administration of 20 mg. ascorbic acid the exhausted deposits of such animals may be partially restored in 4-9 days but no further accumulation occurs on this dose. Parenteral administration brings about the same result more quickly than the oral feeding but the extent of the refilling of the deposits is not affected. Better results (40% greater restoration) are obtained when the 20 mg. of ascorbic acid is given in 2 doses instead of a single dose, but storage of vitamin C

in the organs is never as complete with this amt. as in normally fed animals S Morgulis

**Glaucoma and edema** Hans Schroeder. *Eye, Ear, Nose and Throat Monthly* 14, 369-73 (1935).—The relationship of glaucoma to B deficiency, NaCl, nutritional and angioneurotic edema are considered. Twenty-four references

**Recent advances in nutrition** E. V. McCollum. *Penna Med J* 39, 61-5 (1935).—The importance of vitamins, inorg. constituents and amino acids in the balanced diet is discussed. Adults need 200-300 rat units of B daily. Feeding 3 g of yeast daily to 351 underfed women and children in poor families was followed in almost all cases by increase in wt and appetite. Pellagra is associated with deficiency in dietary flavine, which is indispensable and is not the antidermatitis factor B<sub>2</sub>.

**Diagnosis and treatment of pellagra** George J. Busman. *Penna Med J* 39, 173-5 (1935).—Twenty-seven pellagrins aged 26 to 63 yrs were studied. Gastrointestinal involvements were found in 26, acilia in 21. Only 12 had been taking ale. Liver ext supplemented by an adequate diet was used in treatment. J. C. M.

**Phlyctenular disease and vitamin deficiency** Leonard G. Redding. *Penna Med J* 39, 158-61 (1935).—The decrease of phlyctenular disease in the United States and its increase in Europe are associated directly with the amt of vitamin A in the diet. Clinically, phlyctenula is developed in rats on an A-free diet. Large doses of cod liver oil have proved successful in treatment. J. C. M.

**Some clinical aspects of deficiency diseases in adults** Chester S. Keeler. *Rhode Island Med J* 18, 161-5 (1935).—Deficiency in vitamin B is associated with polyneuritis and cardiac insufficiency. The clinical aspects of avitaminosis in adults are discussed. J. C. M.

**A discussion of certain types of anemia** Geo. W. Parsons. *Tri State Med J* 8, 1556-61 (1935).—Adissoman pernicious anemia is a deficiency disease resulting from lack of the antianemic constituent of liver. It is clinically a distinct entity from idiopathic hypochromic anemia which results from deficiency in Fe in the diet. J. C. M.

**Tissue oxidation in B<sub>2</sub> avitaminosis and inanition** Hakan Rydin. *Upsala Lakareforenings Forhandl* 41, 1-182 (1935-36); cf. *C. A.* 29, 5896\*.—Critical reviews of the literature indicated division of opinion whether there is a decrease in the oxidative capacity of the tissues due to B<sub>2</sub> avitaminosis. A review of the literature is presented. The technique used in this study with statistical evaluation of results are given in 31 tables. Pigeons were kept in cages at a uniform temp and on specific diets. Adequate diets were given in the foreperiod; during the period on restricted or no B<sub>2</sub> intake they were offered polished rice ad libitum or definite amts of polished rice were given by forced feeding. In other pigeons, during the hunger period, no food was offered. From time to time biopsies were performed to obtain sections of the pectoral muscle and of the liver. Blood was obtained by heart puncture. Tests were made upon the same pigeons receiving normal diet, then a restricted or B<sub>2</sub>-free diet, or no food, followed by resumption of normal diet. Tissue oxidation was measured on the muscle, muscle press-ure and blood by the micro-respirometer method of Warburg and Barcroft, and also by the Thunberg methylene blue decolorization method. B<sub>2</sub> concentrate and crystals were made by the Kinnorsley, O'Brien and Peters method (*C. A.* 27, 4279). In some pigeons acute results were obtained. In others, chronic B<sub>2</sub> avitaminosis was produced. In some of the tests lactic acid or Na lactate and Ringer phosphate buffer were added. All tests were made in duplicate and results averaged. In some expts different portions of the brain were tested similarly. Variations in technique in testing different organs will produce great variations in quant. results. The brain, kidney, liver, muscle and blood cells of pigeons during a stage of hunger or of B<sub>2</sub> avitaminosis showed decreased O uptake by the tissue methods, as well as decreased methylene blue oxidative ability, as compared to the same animals on adequate diets. The muscle press juice showed

a decreased oxidative capacity. In the muscle of pigeons suffering from beriberi there is a decrease in one or more water-sol constituents which are present in the normal muscle. In acute beriberi the microrespirometer method showed a greater oxidative capacity than in the chronic beriberi animals. With the methylene blue method this difference was not consistently demonstrated. Inanition produced a similar decrease in oxidative capacity which paralleled results in the B<sub>2</sub> avitaminosis studies. The exptl results suggested that decreased tissue oxidative capacity in B<sub>2</sub> avitaminosis might be associated with deficient muscular nutrition. Specific decrease in oxidative capacity was found in the brain tissue. The decreased capacity for muscles did not parallel the decrease for liver tissues nor for the blood. In tests *in vitro* the crystalline vitamin B<sub>2</sub> showed no effect on methylene blue. In comparative studies microrespirometer results were often, but not always, in agreement with methylene blue data. The methylene blue action was not always proportional to oxidative change, so microrespiration methods appear most suitable. One hundred and three references.

**Recent progress in the study of vitamins** P. Karrer. *Chimie et industrie* 34, 1027-35 (1935), cf. *C. A.* 29, 4798\*.—A review. A. Papineau-Couture.

**Preparation and chemical investigation of vitamin B<sub>2</sub>** Haruo Kakufuda. *Fukuoka-Ikudaiyaku-Zasshi* 27, 1849-99 (1934), cf. *C. A.* 29, 6278\*.—Misawa's method was used to obtain vitamin B<sub>2</sub> from rice embryo ext. The following general method was used to conc. the vitamin B<sub>2</sub> and to obtain crystalline vitamin. To the Ag fraction of the embryo ext. platinum chloride or picric acid was added, gold chloride was then added. Finally with the aid of the acetone-alc. combination method needle-shaped vitamin B<sub>2</sub> crystals were obtained. The protective dose of the vitamin crystal for rats was 0.01 mg. and for pigeons 0.00258 mg. per day. Upon electrolysis the vitamin B<sub>2</sub> accumulates on the cathode. 0.1% soln of vitamin B<sub>2</sub> shows a strong diazo reaction. The mols of vitamin B<sub>2</sub> contain S but no amino N. On exposure to ultraviolet rays, it loses its activity, but Röntgen rays are ineffective. The absorption line of the spectrum of its acid salts lies at 280-265 mμ. K. Sugura.

**An effect of choline upon the weights of young rats** E. W. McHenry. *J. Physiol* 85, 343-9 (1935), cf. *C. A.* 29, 6930\*.—In feeding expts. on 120 young rats it was found that a diet low in choline and vitamin B<sub>2</sub> results in a failure to increase in wt after an initial period of about 2 weeks. The addition of either choline or vitamin B<sub>2</sub> to this diet does not cause a continued increase in wt. However, when choline and vitamin B<sub>2</sub> are supplied together the wts continue to increase. This complementary effect appears to be lessened when older rats are used. Rats which do not receive choline develop an appearance which is similar to that which has been attributed to a deficiency of vitamin B<sub>2</sub>. E. D. Walter.

Constitution of vitamin B<sub>2</sub> (Windaus, Thele) 10.

## F—PHYSIOLOGY

HOMER W. SMITH

**Artificial growth and inhibition of growth** J. Freud. *Nederl. Tijdschr. Geneeskunde* 79, IV, 4977-90 (1935).—A review discussing the influence of sex hormones on the growth of accessory organs, etc. R. Beutner.

**Phosphorus metabolism after work in healthy individuals and in cardiac patients** J. C. J. Burken. *Nederl. Tijdschr. Geneeskunde* 79, IV, 5704-9 (1935).—In the normal resting muscle phosphoric acid is tied up as a lipoidal ester. When work is done a part of it is split off. At the same time the muscle membrane becomes more permeable, which condition allows the split off phosphoric acid to diffuse into the blood stream. If the muscle subsequently returns to the resting condition, phosphoric acid again enters into chem. combination with the muscle lipoids, hence the circulating phosphate is taken up by the muscle. On the basis of these considerations B explains his exptl. findings. He observes an increase of the blood

phosphates, up to 50%, after muscular work of 3 to 4 min duration at least in patients with cardiac decompensation. (Also, the blood lactic acid is markedly raised.) Subsequently with resting muscle, the phosphate values not only return to normal but decrease materially below the normal level, later they slowly rise up to it again.

R B

Phenols in some biological fluids and their relation to phenolemia. Mariano F Castex and Alberto I Arnaudo. *Rev assoc med argentina* 49, 1063-70(1935).—Phenols from ascitic and pleural fluids were detected by Theis and Benedict's method (cf C A 18, 3398) for phenols in blood. They were found in the same concn as in blood.

E S G B

The renal elimination of bilirubin. Adolfo L. Ravces and Carlos Velasco Suarez. *Rev med quir patol femenina* 5, 559-77(1935).—No bilirubin was found in the urine of normal persons. The elimination of bilirubin in the urine depends on the total concn of bilirubin in the blood. The bilirubin giving direct van den Bergh reaction seems to increase the kidney permeability for bilirubin. No threshold for bilirubin elimination by the kidney was found.

E S G B

The renal threshold for dextrose in man. James W Sherrill and Eaton M MacKay. *Arch Internal Med* 56, 877-83(1935).—The renal threshold is dependent on many variables so that it may be misleading to give a normal av. The av for a group of persons who did not have diabetes was 128 mg glucose/100 cc, with a group of diabetics it was 149 mg/100 cc.

J B Brown

Changes in the blood and circulation with changes in posture. The effect of exercise and vasodilatation. John B Youmans, J H Akeroyd, Jr, and Helen Frank. *J Clin Investigation* 14, 739-53(1935).—"The exchange of fluid between the blood and tissues, primarily controlled by capillary and colloid osmotic pressure, is greatly influenced by a no of secondary factors, particularly by posture. The great tendency to edema in the erect quiet posture is opposed by a rising colloid osmotic pressure and by an increasing tissue pressure in the feet and legs. With muscular activity an even greater vol of filtrate than occurs on quiet standing is prevented from accumulating by a more active lymphatic drainage. Variations in these secondary factors will influence the exchange of fluid between the blood and tissues and, in the presence of even slight changes in the serum proteins and capillary pressure, may det the appearance or nonappearance of edema."

J B Brown

The nature of the substance(s) producing pain in contracting skeletal muscle. Its bearing on the problems of angina pectoris and intermittent claudication. L N Katz, E Lindner and H Landt. *J Clin Investigation* 14, 807-21(1935).—The amt of exercise required to cause pain and fatigue in the muscles of the arm of normal subjects was studied. The rate of accumulation of pain-producing substance was affected by the time allowed for recovery between contractions. The substance is a product of metabolic activity of the muscle, it diffuses in and out of the blood stream; it is nonvolatile since it persists after passing through the lungs, it is dependent not only on local production but also on the amt. transported from other regions, it is acid in nature, it is retarded by alkali, training tends to lessen its activity, possibly by altering the buffer action of the muscle concerned.

J B Brown

A clinical and experimental study of the stability of colloid osmotic pressure of serum protein. Kintaro Yanagi. *J Clin Investigation* 14, 853-62(1935).—Colloid osmotic pressures of sera have been measured by means of an osmometer which permits observations at any desired interval of time. In sera with normal protein concns, when the pressure was once developed it remained constant for 16-18 hrs. Pressures of sera with low protein were found to be unstable, rising to a max in 3-5 hrs, and then falling, this effect, however, could be produced in normal sera by dilg them with physiol saline or ultrafiltrate. Furthermore, concn of hypoproteinemic sera by

ultrafiltration produced stable osmotic pressures.

J B Brown

The Addis sediment count and blood urea clearance test in normal pregnant women. C. A. Elden and J W Cooney. *J Clin Investigation* 14, 889-91(1935).—The lower limit of normal urea clearance is somewhat less in pregnant than in non pregnant women.

J B Brown

Intubation studies of the human small intestine. IV. Chemical characteristics of the intestinal contents in the fasting state and as influenced by the administration of acids, of alkalis and of water. W. G Carr, W. Osler Abbott and Albert B Sample. *J Clin Investigation* 14, 893-900(1935).—By a new method of intubation (cf Miller and Abbott, *Am J Med Sci* 187, 595(1934)) the chem characteristics of the contents of the small intestine of man were studied. After fasting the flow of contents into any part of the small intestine is usually less than 1 cc/min, acidity is greatest in the duodenum, diminishing toward neutral or even alk in the lower ilium, bicarbonate is related to reaction; the duodenal contents, if acid or tending toward neutral, are hypotonic, but if neutral or alk, they tend toward the isotonic state of the ileac contents. After administration of acid orally, the contents of the stomach pass slowly into the duodenum where they are neutralized by bicarbonate and become isotonic. After bicarbonate ingestion (isotonic) the stomach evacuates rapidly. With hypertonic bicarbonate the stomach evacuates much more slowly. After administration of 400 cc of water the gastric contents pass into the duodenum rapidly enough to render the duodenal contents acid and to lower their osmotic pressure.

J B Brown

The metabolism of the isolated heart of dogs related to age. A E. Cohn and J M Steele. *J Clin Investigation* 14, 915-22(1935), cf. C A 30, 518.—"Decrease in the consumption of O with age was observed in heart-lung preps made in pure-bred female wire haired fox terriers living under similar environmental conditions."

J B Brown

Sodium and potassium metabolism. The effect of potassium on the sodium and water balances in normal subjects and patients with Bright's disease. Eaton M MacKay and Allan M Butler. *J Clin Investigation* 14, 923-39(1935).—The mineral and water balances of two normal subjects and of two subjects with Bright's disease were studied after ingestion of moderate amts of K and Na. Ingestion of 5-10 g. of KCl per day did not affect the excretion of Na or edema fluid, and, in a case of Bright's disease had no appreciable effect on the Na retention and development of edema. Details of the results of other mineral excretions are described under various conditions.

J B Brown

The electrodynamic theory of life. H S Burr and T S C Northrop. *Quart Rev Biol* 10, 322-33(1935).—A review.

J B Brown

The cellular transmission of substances, especially neurohumors. G H. Parker. *Quart. Rev Biol* 10, 251-71(1935).—A lecture review.

J B Brown

Dehydration in human beings. Frederick A. Collier and Walter G. Maddock. *Ann. Surgery* 102, 947-60(1935).

Rachel Brown

Follicular hormone and ovulation inhibition. Gunnar Dahlberg. *J Obstet Gynaecol Brit Empire* 42, 953-61(1935).—D postulates that the Zondek-Aschheim reaction is due to the fact that follicular hormone in the urine is resorbed and consumed more quickly than is prolactin. Therefore the effect of prolactin predominates. The percentage of high follicular hormone in the urine is not continuously high enough to prevent ovulation.

Rachel Brown

Resistance to proteolysis found in the blood serum of aborting women. Evan Shute. *J Obstet Gynaecol Brit Empire* 42, 1071-81(1935).—Seventy three % of 44 spontaneously aborting women had blood serum which showed a characteristic type of resistance to the proteolytic action of commercial trypsin. The resistance appeared to be directed against its protease fraction. Eight % or less of 128 pregnancies and self-induced abortions presented the

same phenomenon. It is suggested that many spontaneous abortions are provoked by excess of this ability of the normal blood to impede proteolytic activity. The maternal resistance is not due to serum antitrypsin, since it resists heating at 80° for 30 min. Sixty-one references. R B

Is estrin the cause of the resistance to proteolysis found in the blood serum of aborting women? Evan Shute J. *Obstet Gynaecol Brit Empire* 42, 1085-95(1935)—A substance closely resembling estrin is the factor in the maternal blood serum responsible for resistance to proteolysis in many cases of spontaneous abortion. The concn of this antiproteolytic principle is greater in the placentas of women whose pregnancies terminate prematurely than in more mature placentas. R B

Relation between the site of injection of androsterone and the comb response in the fowl. A W Greenwood and J S S Blyth *Quart J Exptl Physiol* 25, 267-77 (1935)—Injections of androsterone directly into the substance of the capon's comb produced a response greater than that elicited by the same dose injected intramuscularly in the pectoral region. Two females responding to the treatment showed less increase in the comb size than the capons. A normal male showed no response, while an incompletely castrated male gave a marked response. Rachel Brown

Effect of asphyxia on the sinus and conducting tissue of the frog heart. G Kingsepp *Quart J Exptl Physiol* 25, 291-302(1935)—Asphyxia of both the normal and of the iodoacetic acid-poisoned sinus causes alterations in the frequency which closely resemble the changes produced by asphyxia on the mechanical response of the ventricle. Asphyxia in the normal heart produces a great decrease in the rate of auriculoventricular conduction, while in the iodoacetic acid-poisoned heart asphyxia produces less interference with conduction. This difference is probably due to the production of lactic acid in the former case and its absence in the latter case. R B

The oxidation coefficient of lactic acid in the animal world. V Borsuk *J Physiol (U S S R.)* 19, 549-62 (1935)—The processes of decompn and resynthesis of glycogen in a number of invertebrates are studied. H Cohen

Metabolism during muscle work. I. Fat metabolism. A. Kharit and A Shretter *J Physiol (U S S R.)* 19, 540-8(1935)—The fat content of arterial blood during work diminishes on the av. 15.6%, compared to rest. This is an indication that fat is required during work, not by muscles, but by other organs. H Cohen

The nature of the union of the sodium and potassium in the gray matter of the human brain. L M Georgievskaya *J. Physiol. (U S S R.)* 19, 571-4(1935)—The Na and K of the gray matter of the cortex are quantitatively separated by electrodialysis. The Na and K, therefore, occur in the brain in a salt-like combination. H Cohen

Estrogenic activity of the urine of cows during pregnancy. M. M. O. Barrie, J. B. E. Patterson and S. W. F. Underhill. *Quart. J. Pharm. Pharmacol.* 8, 424-8 (1935)—The amt of estrin secreted in the urine of cows during pregnancy is less than 50 international units per l during the 1st 21 weeks of gestation. Estrin can be readily detected in the urine at the 23rd week, when the concn. is about 100 units per l. At the 30th week 700 units was obtained, at the 32nd about 9000 units, at the 34th 4000 units, and at the 37th 17,000 units per l. The variations in the amt. present in the last weeks of pregnancy are probably due to variations in the concn of the urine. W. O E

Metabolism of women during the reproductive cycle. VI. A case study of the continuous nitrogen utilization of a multipara during pregnancy, parturition, puerperium and lactation. Helen A. Hunscher, Frances Cope Hummel, Betty Nims Erickson and Icie G. Macy. *J Nutrition* 10, 574-97(1935); cf. C. A. 27, 1033—This case study for an 8-year period of child-bearing and child-rearing has been continued during another complete reproductive cycle. During the last 145 days in gestation there was an av. net storage of 3.1 g. and a maternal retention of

2.6 g. of N daily, resulting in a total observed accumulation of 446 g. at term. On the day of delivery the chemically detd. maternal loss of N in blood, placenta, amniotic fluid and vomitus amounted to 46, 20.1, 0.08 and 0.24 g., resp., the total loss from the body beyond the food consumed was 54.6 g. of N in addn to that contained in the fetus. The 9 daily balances during the lying-in period showed an av. daily loss of 5 g. of N. From the 10th to the 53d day of milk flow the gestatory reserve N had been reduced by delivery, puerperium and lactation losses of 54.6, 44.6 and 38.3 g., resp., leaving a total of 310 g. of N stored only in the last half of pregnancy. When the approx fetal content of 58.6 g. of N is deducted from the final maternal reserve, the accountable losses of the reproductive cycle by the 53rd day of lactation had left a maternal reserve of 250 g. of N for future dissipation or enrichment of the maternal body at termination of the reproductive cycle. C R Fellers

Diabetogenic action of the anterior hypophysis independent of the adrenals. B A Houssay and L F. Leloir. *Rev. soc. argentina biol.* 11, 464-73, *Compt. rend. soc. biol.* 120, 670-2(1935)—Ext of anterior hypophysis (bovine) produced the usual prolonged hyperglucemia in dogs with the adrenal medulla or the whole adrenals removed, or the adrenals denervated, and in toads after cauterization or removal of the adrenals. L E G.

Effects of large doses of follicular hormone on the structure of the ovary and anterior hypophysis of the white rat. L Desclin. *Compt. rend. soc. biol.* 120, 520-8(1935). L E Gilson

Glutathione of the tissues of hypophysectomized dogs. Further evidence of hypophyso-thyroid and hypophyso-testicular association. Léon Binet, Léon Képinov and Georges Weller. *Compt. rend. soc. biol.* 120, 589-90 (1935)—Hypophysectomy was followed by a marked decrease in reduced and total glutathione of the liver, thyroid and testicles. L E. Gilson

Occurrence of lipides in the urine of dogs after removal of the lumbar-sacral portion of the spinal cord. H Hermann, J. Dechaume and J. Vial. *Compt. rend. soc. biol.* 120, 648-9(1935)—For the first few days after the operation the urine contained 60-650 mg. ether-extractable lipid per l. Later the quantity dropped to 20-60 mg./l. An intermittent lipuria persisted for months. The lipide was principally olein. It contained no phospholipide. L E. Gilson

Concentrating action of the gall bladder. Glutathione of the bile. G. Baltaceanu and C. Vasiliu. *Compt. rend. soc. biol.* 120, 666-8(1935)—The normal liver bile of 48 dogs contained 10-200 mg. % of glutathione and the bladder bile 300-600 mg. %, usually between 5 and 15 times as much as the liver bile. The liver tissues of the same dogs contained 115-450 mg. %. L. E. Gilson

Progressive changes in the structure of the uterus and tubes of rabbits treated, from birth, with repeated injections of folliculin. A Lacassagne. *Compt. rend. soc. biol.* 120, 685-9(1935). L E. Gilson

Effect of carbon dioxide on cerebral blood vessels. J J Bouckaert and F. Jourdan. *Compt. rend. soc. biol.* 120, 788-90(1935)—In the dog head the addn. of CO<sub>2</sub> to the perfusing blood caused a marked dilation of the blood vessels of the brain. L E. Gilson

Experiments on biological test objects concerning the nature of the vagomimetic substance present in sweat. K v. Mëgay. *Arch. ges. Physiol. (Pflügers)* 236, 159-65 (1935)—The pharmacological (and menotonic) effects of sweat are due to the presence of acetylcholine and K salts. Arthur Grollman

Acetylcholine and choline in tissue extracts. F. Plattner and H. Tszdamura. *Arch. ges. Physiol. (Pflügers)* 236, 175-80(1935)—No correlation was found between the ratio of the amount of choline to the amount of acetylcholine present in various tissues of the cat. Extirpation of the adrenals, thyroid or parathyroids did not affect the tissue contents of choline or its derivatives. A. G.

The physiology of the blood glycogen. G. Golandas. *Arch. ges. Physiol. (Pflügers)* 236, 230-8(1935)—The basal glycogen contents of the blood of man, dog and

rabbit are 6.7 to 12.2, 8.6 to 15.3 and 6.4 to 14.4 mg. per 100 cc., resp. Most of the glycogen is adsorbed on the formed elements of the blood. It disappears in sterile shed blood. A. G.

Secretory phenomena in the oviduct of the fowl, including the process of shell formation examined by the microincineration technic K. C. Richardson *Phil Trans Roy Soc (London)* B225, 149-95 (1935).—The ovalbumin and glucoprotein fractions of egg white are secreted by the glands and lining epithelium of the egg-white region. The ovokeratin shell membranes have their origin in fiber-like strands of viscous material secreted by the tubular glands in the corium of the isthmus. The products of uterine activity include the org. shell matrix with its mammillae and cuticle, the thin part of the egg white and the calcification of the shell. The source of the secretory fluids contain Ca (e.g., for the calcification of the shell) has not been further localized. Thirty-three references. Joseph S. Hepburn

Continuing metabolism of nitrogen in animals H. Borsook and G. L. Keighley. *Proc Roy Soc (London)* B118, 488-521 (1935).—The continuing metabolism is the N metabolism on any 1 day which is already present in the tissues—distinguishing it from exogenous N. It is not related to the endogenous or "wear and tear" metabolism. In a man in N equil. with a daily urinary N of 10-11 g., the continuing metabolism constitutes more than half the total urinary N. The continuing metabolism is a function of the previous dietary history, and is directly proportional to the level of N intake. In an adult in N equil., extensive synthetic processes involving amino acids are continually in operation. In man,  $\text{NH}_3$  may serve as a precursor of urinary uric acid. Neither endogenous nor continuing N metabolism is stimulated by amino acids. Fifty-five references. Joseph S. Hepburn

The male sex hormones L. Ruzicka *J. Chem. Education* 13, 3-6 (1936).—A review. E. H.

The serum calcium response to ingested calcium S. Freeman, E. R. Kent and A. C. Ivy *J. Biol. Chem.* 112, 1-7 (1935).—A slight but reasonably const. elevation of serum Ca (5-6%) occurs in fasting human subjects 2 hrs. after ingestion of moderate doses daily of  $\text{CaCl}_2$  or Ca gluconate (2.3 and 10 g., resp.). In fasted normal dogs dosage of Ca influences the serum Ca response over a range of intake varying from 0.05 to 0.25 g. of Ca per kg. of body wt.  $\text{CaCl}_2$  produces a greater max. rise than a corresponding dose of Ca gluconate but the latter compound maintains a greater rise 4 and 6 hrs. after ingestion. A. P. Lothrop

Equulin prepared from pregnant mare urine Geo. F. Cartland and Roland K. Meyer *J. Biol. Chem.* 112, 9-14 (1935).—Equulin was prepd. from mare urine collected during the last 2 months of pregnancy making possible much higher yields than those previously reported. Removal of a small amt. of theelin was accomplished by repeated recrystallizations from EtOH and the purified product had a slightly higher  $[\alpha]_D^{25}$  (325°) than that reported by Girard, *et al.* (*C. A.* 26, 4090). Biol. assays in spayed rats showed that the equulin is approx. 75% as active as standard theelin when injected in aq. 10% alc. contg. 0.5%  $\text{Na}_2\text{CO}_3$ ; it is only 30% as active if injected without addn. of the  $\text{Na}_2\text{CO}_3$ . Colorimetric assay indicates an activity of only 11% so that this method is quite unsatisfactory. A. P. Lothrop

Ketosis VII. Quantitative studies on  $\beta$ -oxidation. Glycogen formation from various fatty acids Harry J. Devel, Jr., Joseph S. Butts, Lois F. Hallman and Charles H. Cutler *J. Biol. Chem.* 112, 15-23 (1935). *C. A.* 29, 5166<sup>7</sup>.—A profound difference exists in the glycolytic activity of odd- and even-chained fatty acids, significant amts. of glycogen are deposited in the liver after feeding 0.173 mole per sq. m. of body surface of the Na salts of propionic, valeric, heptic and nonylic acids and no rise above the control level is noticed after ingestion of diacetic, butyric, caproic and caprylic acids by fasting male rats. The process of  $\beta$ -oxidation applies to the acids with an odd no. of C atoms with degradation to propionic acid which serves as a building stone for glucose or glycogen.

Trivalerin is a significantly better glycolytic agent than tributyrin, the glycogen from the latter coming largely, if not entirely, from the glycerol component. Oleic acid does not bring about significant formation of glycogen, indicating that cleavage at the double bond forming 2 mols. of nonylic acid cannot be a primary change in the metabolism of oleic acid. A. P. Lothrop

Distribution of calcium and phosphorus in the diverse organs and tissues of the rat during excess and deficiency of parathyroid hormone Pietro Molinar-Tocatti *Arch. ital. biol.* 93, 140-64 (1935); *C. A.* 29, 1158<sup>7</sup>.—The parathyroids have a certain influence on the Ca and P content in organs and tissues, as shown by comparative figures. It is not possible to establish definite rules or to assert whether the action is a direct one or secondary to changes produced in the bones. A. E. Meyer

The elimination of nitrogen-containing products of metabolism in gastric and duodenal juice A. Allodi, A. Cronini and A. Robecchi *Arch. sci. med.* 60, 1-16 (1935).—The noncoagulable N in gastric and duodenal juice during rest as well as after stimulation is higher than in the blood. The contents in urea and uric acid are very low. The gastric elimination cannot act as a substitute for the function of the kidney in cases of disease of the latter. A. E. Meyer

Hypopharyng-endocrine relations VI. Hypophysis and pancreas R. Rivaire. *Presse med.* 43, 757-61 (1935), *C. A.* 30, 148<sup>7</sup>.—A review. A. E. Meyer

The skin in experimental hyperparathyroidism R. Leriche, A. Jung and C. Sureyya. *Presse med.* 43, 771-81 (1935).—Injections of large doses of parathormone cause skin lesions consisting in infiltrations, indurations and loss of hair. The H and Ca are increased. A. E. Meyer

Permanent arterial hypertension and endocrine glands Paul Halbronn and H. Pierre Klotz. *Presse med.* 43, 913-15 (1935). A. E. Meyer

Mucin and equilibrium of gastric acidity R. H. Monceaux. *Presse med.* 43, 908-9 (1935).—Mucin absorbs all acidity beyond the physiol.  $\text{pH}$ . This acid becomes available again at onset of digestion before fresh HCl is secreted. A. E. Meyer

The phosphatase elimination in normal rats and in hyper- and hypo function of the parathyroid Guido Mellini and Lylla Annesi. *Sperimentale* 89, 311-20 (1935).—The phosphatase elimination is increased in rats after thyroparathyroidectomy as well as after application of high doses of parathyroid hormone. It is concluded that in both conditions an increased phosphatase production in the bone occurs. A. E. Meyer

The functional variations of the vegetative brain centers by local application of hormones and pharmacodynamic substances following electrocardiographic changes L. Rocchetti. *Sperimentale* 89, 348-71 (1935). A. E. Meyer

Is the creatinine clearance an expression of filtration through the glomeruli? Poul Iversen and Tage Bjerring. *Acta Med. Scand.* 86, 459-68 (1935).—The glucose excretion and the mg. % of glucose in the blood above the threshold value stand in linear relation to each other. The clearance of different sugars may be taken as the rough expression of the filtration. The clearances of urea and glucose are of the same order of magnitude above the threshold value and are equiv. to the filtration corresponding to that found by the creatinine clearance. S. Morgulis

Partitioning of chloride in blood in relation to the acid-base equilibrium, and the clinical significance of the separate chloride determination in plasma and corpuscles T. N. Feldmann and R. L. Ulanovskaya. *Acta Med. Scand.* 87, 65-79 (1935).—The role of NaCl in the regulation of the acid-base equil. is discussed and it is pointed out that with a preponderance of acid the  $\text{Cl}^-$  migrates into the erythrocytes, where it combines with the proteins. The  $\text{Na}^+$  combines with, and neutralizes, the acid valences. The ratio of cell chloride to plasma chloride, which normally falls between 0.47 and 0.54, is an early sign of acidosis when it increases. The shift in the alkali reserve follows later. In acute nephritis with no manifest insufficiency there is no evidence of an acidotic condition. A change

in the ratio has been found in chronic nephroses when they become acute, but in severe affections there is actual acidosis as shown by the shift in the  $\text{Cl}^-$  ratio as well as in the alk. reserve. S Morgulis

Intermediate metabolism of glands of internal secretion IV Proteolysis and ammonia formation in endocrine organs S Epstein and S Mima *Biochem Z* 281, 229-41 (1935) —At the reaction of the organ, proteolysis is very weak in thyroid, thymus and adrenal, but is much increased in an acid reaction. The proteolysis is considerable in pancreas. In short autolysis at an alk reaction  $\text{NH}_3$  is formed in pancreas, adrenal medulla and thymus of young animals. S Morgulis

Electrokinetic potential of thrombocytes Hans D Cremer *Biochem Z* 281, 315-8 (1935) The electrokinetic potential of thrombocytes falls between 20 and 42 mv, with the majority of measurements ranging from 25 to 35 mv. No definite relationship could be found between the variations in potential and the protein content, the albumin/globulin ratio or viscosity of the serum. S Morgulis

Calcium excretion through the pancreas Gunnar Agren *Biochem Z* 281, 356-62 (1935) The blood Ca level after intravenous injections of 5-10 mg  $\text{CaCl}_2$  per kg rises higher in normal rats than in those treated with uretin. The pancreatic juice has always a higher Ca content than the serum but as the rate of secretion increases the Ca concn. tends to approach that of the serum. There is evidence that the pancreas is very permeable to Ca, and the Ca concn. of the pancreatic juice is high even when the velocity of secretion is great, if the serum Ca is much increased by injection of  $\text{CaCl}_2$ . S Morgulis

Uric acid and urea excretion through the pancreas and liver under the influence of secretin Gunnar Agren *Biochem Z* 281, 363-6 (1935) —Both the pancreas and liver are permeable to uric acid and urea. Injected intravenously in large amts these substances appear in considerably increased amts also in the liver and pancreas excretions. S Morgulis

Cyclic changes in the liver glycogen of adrenalectomized rats. Gunnar Agren *Biochem Z* 281, 367-9 (1935) —The rhythmic changes in the liver glycogen of rats disappear when the animals have been adrenalectomized. S Morgulis

Studies on acetylcholine sensitivity and the velocity of acetylcholine splitting by the human blood G Ingvarsson *Biochem Z* 281, 379-6 (1935) —In the blood from persons of widely different sensitivity to acetylcholine, both before and after an injection of acetylcholine, no variation in the speed of the acetylcholine cleavage was observed. The difference in sensitivity is, therefore, attributed not to the degree of inactivation in the blood but rather to the condition of the peripheral organ acted upon. S Morgulis

Excretion of oxalic acid by guinea pigs Stig Bergström. *Biochem Z* 281, 377-82 (1935) —Oxalic acid fed by mouth is very largely excreted through the intestine, but much larger amts. are excreted when the oxalic acid is injected subcutaneously. It is suggested that since the excretion of oxalic acid is quantitative, the substance is not oxidized in the organism. However, the complete elimination of oxalic acid given by mouth or subcutaneously requires about 14 days or 6 weeks, resp. The results obtained in expts. over brief periods of time lead to erroneous conclusions. S Morgulis

General theory of the solubility of volatile acids in blood. Ferdinand Ince *Biochem Z* 281, 383-93 (1935). S Morgulis

Occurrence of adenine nucleotide in the tissues II Heart muscle K. Lohmann and Ph. Schuster. *Biochem Z* 282, 104-8 (1935) —Calf hearts were immediately packed in crushed ice and ground 12-15 min. later in a chilled machine and at once extd. with an equal amt. of ice-cold 10%  $\text{CCl}_3\text{CO}_2\text{H}$ . The residue was pressed out and again extd. with 4% acid. The solns were filtered through kieselguhr and adjusted with NaOH to pH 8. To this was added solid  $\text{Ba}(\text{AcO})_2$  (25 g. per kg. substance) and 10% alc. and the ppt. was centrifuged off. The ppt

was dissolved in dil.  $\text{HNO}_3$  and reprecip. with a  $\text{HNO}_3$  —  $\text{Hg}(\text{NO}_3)_2$  mixt., the ppt. decompd. with  $\text{H}_2\text{S}$  and the soln. aerated, neutralized with baryta and fractionated with  $\text{Ba}$ . Two fractions are thus obtained: a difficulty sol. and an easily sol. portion. The largest part of the adenine nucleotide obtained from calf hearts is adenylpyrophosphoric acid, but small quantities of adenyridiphosphoric and adenylic acids were found also. S. M.

Cleavage, oxidation and energy metabolism in dogs III Absorption and assimilation of hexoses in organs on continuous intravenous injection of galactose, maltose and glucose, compared with hexolysis M Wierzechowski and H. Fink *Biochem Z* 282, 124-47 (1935). —When galactose and glucose are injected over a long period into a peripheral vein the liver tissue takes up the largest amt. of sugar per unit of time and per g. tissue, next in order are the organs of the portal vein system, then the head organs and last the tissues of the extremities. Only in the case of maltose does the liver seem to play a small part in the metabolism. Glucose, which in the fasting state originates in the liver, is taken up most from the blood per g. of tissue by the head organs, less by the organs of the portal vein system and least by the motor system (extremities). Simultaneously lactic acid is produced and eliminated by the organs, 40-60% of the absorbed glucose by the muscles, 14-17% by the head organs and 9% 14% by the organs of the portal vein system. From injected glucose or maltose these organs return 5-15% as lactic acid to the blood. The absorption of glucose is not const. during a protracted injection, but at first the absorption is small and associated with a considerable excretion in the urine, but becomes greater in the later phase. During the absorption of injected galactose the individual organs use up glucose at about the same rate as in the fasting condition, and the liberation of glucose from the liver ceases. About 40% of the injected galactose absorbed by the tissues is partly utilized and partly excreted by the urine when the injection is completed. The expts. were made under amylar narcosis. This by itself does not affect the total assimilation of sugar. Only after laparotomy and loss of blood does the assimilation of glucose and maltose decrease much, while the assimilation of fructose is little affected and that of galactose not at all. However, of the sugars injected under amylar narcosis fructose is best utilized. S Morgulis

Passage of hormones into milk S Konsulov *Endokrinologie* 16, 237-40 (1935). —Thyroidin administered to the lactating mother passes into the milk and causes a great increase in the  $\text{CO}_2$  production of the young. S Morgulis

Determination of the copper content of plasma and red blood cells by the Warburg cysteine-oxidation method Jannik Bjerrum and Vald. Henriques. *Skand. Arch. Physiol.* 72, 271-82 (1935) —In rabbit blood the Cu is uniformly distributed between plasma and corpuscles. On injecting Cu into the blood, this penetrates into the cells until the concn. becomes the same as in the plasma, and later falls again until the normal value for the Cu concn. is reestablished. S Morgulis

Experimental studies on the irritability of the rabbit heart muscles with variation of the carbon dioxide content of the respired air and vagus stimulation. Rune Olsson and Nils Gustav Persson *Skand. Arch. Physiol.* 72, 295-303 (1935) —A perfusion method is described whereby it has been possible to demonstrate that the irritability of the rabbit heart muscle is practically unaltered whether the animal breathes pure  $\text{O}_2$  or an  $\text{O}_2$  + 20%  $\text{CO}_2$  mixt. No evidence was found that the stimulation of the vagus directly affects the irritability of the cardiac muscle. S Morgulis

The erythrocyte and its relation to blood pressure. H. McGuire Doles *Virginia Med. Monthly* 62, 489-96 (1935) —Studies were made upon 10 normal human beings, 5 showing hypotension and 5 showing hypertension. Enlargement of erythrocytes in hypertension is due to increased  $\text{Fe}$ , possibly assoc. with kidney damage. In nephritis the actual blood pressure varies from that called from the blood findings. During treatment there was an

increased erythrocyte count, associated with decrease in cell size and in contents of hemoglobin and Fe. Plasma and urinary Fe should be determined in hypertension.

James C. Munch  
A study of the megakaryocytes in the circulating blood of rabbits inoculated with benzene and with saponin. E. M. Medlar. *Folia Haematol.* 53, 397-406(1935).—Saponin causes a much greater alteration in the blood picture than does benzene. The megakaryocytes are markedly increased after a few days.

John T. Myers  
The glutathione content of muscles following cutting of the motor nerves. Alajos Schöda. *Magyar Orvosi Arch.* 36, 327-30(1935).—Frog and rat muscles show an increase in both reduced and oxidized glutathione content of the paralyzed muscle.

Henry Tauber  
The occurrence of several kinds of hemoglobin in human blood. R. Brinkman and J. H. P. Jonxis. *J. Physiol.* 85, 117-27(1935), cf. *C A* 28, 6471<sup>1</sup>.—By measurement of the rate of alkali denaturation, 3 forms of human hemoglobin have been found. One type is present in fetal blood. This is completely replaced by the less resistant adult form at about 7 months after birth. At 3 years of age a "resistant" type of adult hemoglobin appears and remains present in adult life. The extent of surface layers of various human hemoglobins after 1 min. of spreading on *M*/300 phosphate buffers in relation to pH is summarized.

E. D. Walter  
The action of ovarian hormones on the uterine muscle measured *in vivo* and *in vitro*. J. M. Robson. *J. Physiol.* 85, 145-58(1935), cf. *C A* 29, 8101<sup>1</sup>.—In most of the experiments the reactivity to oxytocin was the same for the uterine muscle in the intact animal and for the muscle suspended in physiologic solution. The results obtained with the longitudinal muscle *in vitro* offer an accurate indication of the state of the uterus *in situ*. Estrin increases the rhythmic activity both *in vitro* and *in vivo*, but the inhibitory action of a luteal hormone on the spontaneous contractions is only observed in the intact animal.

E. D. Walter  
Separation of the pubic bones following the administration of estrogens to male mice. Harold Burrows. *J. Physiol.* 85, 159-61(1935).—Diminution in size and separation of the pubic bones was found in castrated and non-castrated male mice and in noncastrated female mice following the prolonged administration of estrin and equilin.

E. D. Walter  
The effect of food and of exhaustion on the pituitary, thyroid, adrenal and thymus glands of the rat. Dorothy H. Andersen. *J. Physiol.* 85, 162-7(1935). E. D. W.

The carbohydrate metabolism of intestine muscle. B. N. Prasad. *J. Physiol.* 85, 239-48(1935).—The colon of the cat and the ileum of the rabbit were used in this study. Isolated intestine muscle contains about 0.25% of carbohydrate available for glycolysis. In the presence of  $O_2$  the isolated muscle oxidizes about 1 mg. of carbohydrate per g. per hr. In the presence of glucose it produces considerable quantities of lactic acid both under aerobic and anaerobic conditions. Na iodacetate (1-10,000) inhibits glycolysis of glucose in Ringer solution in contact with the intestine muscle. Periodic electric stimulation increases the glycolysis about 12%. The mechanical activity of intestine muscle under anaerobic conditions. *Ibid.* 249-66.—Asphyxial arrest of the mechanical movements of intestine muscle is not due to accumulation of acid but to exhaustion of its labile carbohydrate store. The mechanical experiments confirm the biochemical findings that the intestine muscle has only a small reserve of available carbohydrate. Intestine muscle probably uses a mixed fuel of carbohydrate and noncarbohydrate material in aerobic respiration. Its activity is maintained best when it is supplied with both glucose and  $O_2$ . Iodoacetic acid-poisoned intestine muscle has a very limited activity under anaerobiosis. This suggests a small phosphagen content.

E. D. Walter  
The chemical transmitter of the sympathetic nerve to the uterus. M. A. F. Sherif. *J. Physiol.* 85, 298-308(1935), cf. *C A* 29, 4437<sup>1</sup>.—After injection of eserine, acetylcholine or some allied substance can be detected in the blood in the uterine vein of the female dog. The

concentration is increased by stimulation of the hypogastric nerve. Eserine increases the contraction of the uterus produced by stimulation of the hypogastric nerves. Conclusion. Acetylcholine acts as a chemical transmitter of this effect of the hypogastric nerve on the uterine muscle. Since the motor effect is not paralyzed by nicotine, the nerves concerned are probably postganglionic.

E. D. Walter  
The response of the chemical receptors of the carotid sinus to the tension of carbon dioxide in the arterial blood in the cat. Adli Saman and G. Stella. *J. Physiol.* 85, 309-19(1935), cf. *C A* 29, 6933<sup>1</sup>, 8149<sup>1</sup>.—At tensions of  $CO_2$  in the arterial blood at or below 32-35 mm. of Hg the receptors are at rest. Above that level they begin to discharge, and the intensity of the latter is the greater the higher the tension of  $CO_2$ . The response to variations of  $CO_2$  tension in the arterial blood is very prompt.

E. D. Walter  
The blood flow through the skeletal muscle in relation to its contraction. G. V. Anrep and E. V. Salfeld. *J. Physiol.* 85, 375-99(1935).—Potent vasodilator substances appear in the venous blood emerging from a contracting muscle. These substances are stable in blood for at least half an hour. They are produced and released from the muscle during its contraction.

E. D. Walter  
Appearance of histamine in the venous blood during muscular contraction. G. V. Anrep and G. S. Barsom. *J. Physiol.* 85, 409-20(1935), cf. preceding abstract and *C A* 29, 8042<sup>1</sup>.—No difference was found between the histamine concentration of the arterial blood and that of the venous blood emerging from a resting skeletal muscle. When the blood supply to the muscle was diminished the histamine concentration of the venous blood rose above that of the arterial blood. Muscular contraction is accompanied by an increase of the histamine concentration in the venous blood and of the total excess of histamine in the blood emerging from the active muscle. The excess histamine is greater the stronger the contraction of the muscle and the longer its duration.

E. D. Walter  
The alleged occurrence of "Krampfstoffe" in acetone extracts of mammalian brain. Eric Holmes. *J. Physiol.* 85, 400-8(1935).—The effects of the intravenous injection into animals of the material obtained from the fresh brains of animals, killed during convulsions, by acetone extract, observed by Kroll (*Z. ges. Neurol. Psychiat.* 143, 780, 146, 208, 147, 316(1933)), and attributed by him to the presence of a substance affecting the central nervous system, are not due to any such effects, but are the result of a depressant action of the material on the heart.

E. D. Walter  
Assimilation of iron in the course of embryonic development of chicken. Anna Szejman-Rozenberg. *Acta Biol. Exptl.* 8, 32-44(1933) (French summary).—The absolute increase of Fe assimilation in the total chicken embryo has two maxima in the daily curve, viz., on the 12th and 18th days, and one minimum between the 15th and 16th days of development. In the latter period assimilation of Fe is completely inhibited. This inhibition coincides with intensive increase of dry substance and albuminous substances. The ratio of Fe content of the body of the embryo to that of the membrane shifts continuously in favor of the body of the embryo. The percentage content of Fe in the liver is approximately equal to that of the total embryo. In the whole hatching period about 96% of Fe present in the egg is assimilated.

J. Wierstak

## G—PATHOLOGY

H. GIDEON WELLS

Duodothyonine and the thyroid gland. I. Snapper. *Nederl. Tijdschr. Geneeskunde* 79, IV, 5711-12(1935); cf. *C A* 29, 5161<sup>1</sup>.—The injection of iodized horse serum into rabbits leads to the formation of antibodies which precipitate iodized serum protein. This precipitation is prevented by the addition of duodothyonine and by thyroxine, but not by duodothyonine in spite of its very close chemical relationship.

R. Beutner

Role of pressor substances in arterial hypertension

R. B. Capps, E. B. Ferris, F. H. L. Taylor and Soma Weiss. *Arch. Internal Med.* 56, 864-76 (1935).—The urine of patients with hypertension does not contain increased amts. of pressor substances, nor is the response of test animals different to expts. of urine from normal and hypertensive patients. Acetone extn. of urine was not a satisfactory method of removing pressor substances, the acetone fraction contained more pressor substance and less depressor than the EtOH fraction. The pressor substance is not adrenaline or pituitary, it is a water-soluble substance which acts centrally rather than upon the peripheral nerve endings or on the vascular system. The methods of expt. the pressor substance are too inaccurate to permit of observing differences unless they are large.

J. B. Brown

Effect of low-calorie diets and resultant loss in weight on plasma cholesterol in the obese. Charles A. Poindester and Maurice Bruyer. *Arch. Internal Med.* 56, 884-90 (1935).—"The cholesterol content of the plasma in uncomplicated obesity and in obesity complicated by metabolic, arthritic or endocrine disease is not altered primarily by reduction in wt. with a low-calorie diet." In some obese patients the régime of low-calorie diets causes a definite increase in plasma cholesterol for several weeks, this effect being explained as the well recognized starvation effect, a common observation in man and animals.

J. B. Brown

Interpretation of abnormal dextrose-tolerance curves occurring in toxemia in terms of liver function. S. Soskin, M. D. Allweiss and I. A. Minsky. *Arch. Internal Med.* 56, 927-34 (1935).—The results of intravenous administration of diphtheria toxin in normal dogs caused a definitely abnormal prolongation of the dextrose-tolerance curve. In depancrectomized dogs the diabetes became more severe. The toxin affects the liver, not the pancreas, by interfering with the mechanism by which the liver decreases its supply of blood sugar in response to an influx of exogenous sugar.

J. B. Brown

Hyperglucemia and glucosuria associated with disease of the biliary tract. Herman Lande and Herbert Pollack. *Arch. Internal Med.* 56, 1097-1105 (1935).—In exptl. studies on the liver as a blood-sugar-regulating mechanism there was a direct correlation between the disturbance of carbohydrate metabolism and the degree of impairment of liver function. Restoration of normal liver function by biliary drainage resulted in disappearance of diabetic symptoms.

J. B. Brown

Idiopathic steatorrhea. James F. Weir and Mildred Adams. *Arch. Internal Med.* 56, 1109-16 (1935).—Data are presented on a metabolic study of a case of nontropical sprue with steatorrhea. Loss of fat in the stools corresponded to that reported by others, a low fat tolerance was shown. A low fat intake reduced the frequency of the stools with symptomatic relief. In spite of an abnormally high loss of N in the feces, the patient was able to store N on a high N intake with simultaneous increase in serum protein.

J. B. Brown

Cancer as a problem of metabolism. Howard H. Beard. *Arch. Internal Med.* 56, 1143-70 (1935).—A review and discussion.

J. B. Brown

Carbohydrate tolerance and intestinal flora. I. A. clinical study based on sixty cases. T. L. Althausen, J. B. Gunnison, M. S. Marshall and S. J. Shipman. *Arch. Internal Med.* 56, 1263-80 (1935).—A clinical study of cases with carbohydrate intolerance shows no correlation between this condition and the intestinal flora.

J. B. Brown

Obesity: Etiology and metabolism. C. G. Lambie. *Brit. Med. J.* 1935, II, 885-9.—An abridged report of a lecture.

J. B. Brown

Hypoglycemia in the neuroses. M. S. Jones. *Brit. Med. J.* 1935, II, 945-6.—Three cases of hypoglycemia of different origins are presented and discussed in relation to functional nervous disorders.

J. B. Brown

A study of gastric pepsin in various diseases. Clinton R. Mullins and Charles A. Flood. *J. Clin. Investigation* 14, 703-7 (1935).—The peptic activity of the gastric

contents after standard test meals was detd. by the method of Anson and Minsky (*C. A.* 27, 117). A high degree of correlation was observed between pepsin and HCl, but there were numerous exceptions. Patients with duodenal ulcer secreted more pepsin than patients without ulcer. Pepsin detn. in carcinoma of the stomach was of little value in prognosis. In pernicious anemia the pepsin secretion was diminished.

J. B. Brown

The effect of splanchnic nerve resection and sympathetic ganglionectomy in a case of paroxysmal hemoglobinuria. A. Carlton Ernstene and W. James Gardner. *J. Clin. Investigation* 14, 799-805 (1935).—In a patient with paroxysmal hemoglobinuria, hemoglobinuria could be produced at will by the application of ice packs from the feet to the level of the anterior superior spine of the ilium or ensiform cartilage, this effect disappearing with spinal anesthesia. Apparently the sympathetic nervous system played a significant role in the attacks of hemoglobinuria.

J. B. Brown

The relationship of the blood glucose to the concentration of lactose in the milk of lactating diabetic women. Edward Tolstoi. *J. Clin. Investigation* 14, 803-6 (1935).—In a study of 5 lactating diabetic women it was shown that the concn. of lactose in the milk was remarkably const., despite marked elevation or depression of the glucose level of the blood.

J. B. Brown

Dietary protein in hemorrhagic Bright's disease. II. The effect of diet on serum proteins, proteinuria and tissue proteins. B. Henry Keutmann, Samuel H. Bassett, Geraldine E. Julian, Clara H. Present and Helen E. Van Alstine. *J. Clin. Investigation* 14, 871-88 (1935), cf. *C. A.* 27, 130.—The protein balances of 3 patients with degenerative Bright's disease with proteinuria and hypoproteinemia were measured by superimposing on a basal diet different proteins or more calories as carbohydrate or fat. When the caloric and protein needs were met all patients stored large amts. of protein, indicating previous tissue protein depletion. In one patient on a protein intake of 0.8 g. per kg. daily when the caloric intake was 1½ times the basal requirement, some protein was stored, this was increased by increasing the caloric intake to twice the basal. Small supplementary feedings of egg white and serum proteins were more efficient than large. Lactalbumin and liver protein were utilized equally well on the 50- or 100-g. level. Increase of protein intake increased the albuminuria, this was due perhaps to a higher serum protein level or to increased blood flow through the kidney. In 2 patients the intensity of the renal disease remained the same with increased protein intake. In the 3rd case the lesion improved.

J. B. Brown

Studies on the anemia of pellagra. Tom D. Spies and A. B. Chunn. *J. Clin. Investigation* 14, 911-4 (1935); cf. *C. A.* 29, 1872.—The peripheral blood findings of anemia were found to occur in 63% of 30 "alcoholic" pellagrins.

J. B. Brown

The experimental production of cholesterosis of the gall bladder with observations on the cholesterol absorptive properties of the gall-bladder wall. Louis M. Rousselot and Louis Bauman. *Surgery, Gynecol. and Obstet.* 61, 585-90 (1935).—When 0.2% solns. of cholesterol in 4% bile salts are placed in the gall bladder of the dog, the ducts of which have been so ligated as to render them leakproof, about 50% of the lipid disappeared in 24 hrs. A pathol. lesion appeared which resembled human cholesterosis. Cholesterol esters increased. There was no apparent increase in the cholesterol content of the bladder wall. Under such conditions cholesterol was apparently absorbed.

J. B. Brown

Study of blood globulin with respect to its complement activity. Maurice Doladhe. *Compt. rend.* 201, 689-90 (1935).—CO<sub>2</sub> pptts. from fresh serum dild. with H<sub>2</sub>O globulins (I) contg. the midpiece of complement while the endpiece is left in soln. If a stream of CO<sub>2</sub> is passed through a physiol. salt soln. of I, proteins (II) are pptd. which are insol. in H<sub>2</sub>O or physiol. salt soln. The proteins remaining in soln. (III) contain the midpiece. When



obtained from swine serum II contains 0.2036 and III 0.0782 parts per 100 of P.

**Synovial fluid in chronic arthritis** Douglas H. Collins *J. State Med.* 43, 632-7(1935).—Pathological exam of synovial fluid in chronic arthritis should include the determination of sugar which will be lowered with bacterial contamination, total protein which is high with high cellular content, total cell count, and percentage of polymorphonuclear leukocytes which vary with the type of arthritis.

**Further evidence for the presence of a toxic factor in pernicious anemia** G. E. Wakelin and H. D. Bruner. *Science* 82, 494-5(1935).—The urines of 8 pernicious anemia patients when injected intramuscularly into pigeons in amounts of 0.1 to 1.5 cc per 100 g induced a decrease in the reticulocyte count to an av. of 2%. Normal counts were never less than 5%. These urines heated at 100° for 2 hrs lost this property. After the primary decrease in reticulocytes, most of the surviving pigeons showed a subsequent reticulocytosis. This effect was partially retained by the heated urines. The unheated urines were toxic for pigeons. Normal heated or unheated urines and the urines from 2 treated pernicious anemia patients gave no evidence of the toxic reticulocyte-decreasing substance.

**A chemical reaction characteristic of formalin treated toxin filtrates** H. Goldie. *Compt. rend. soc. biol.* 120, 313-16(1935).—The anatoxins obtained by treating filtered tetanus and diphtheria toxins with a little HCHO seem to have an oxidizing action, or at least upon addition of the Na salt of H acid (1-amino-8-hydroxynaphthalene-3,6-disulfonic acid) and boiling a dark red color develops similar to that produced by the action of H<sub>2</sub>O<sub>2</sub> on H acid. The original toxins have a reducing action and do not give the red color. Dil. NH<sub>4</sub>OH and peptone soln to which a little HCHO has been added also give the color reaction with H acid; hence the effect is probably due to nonspecific substances in the anatoxin solns.

**Liberation of histamine-like substances in intestinal infarcts** G. Ungar, X. J. Contades and R. G. Palmer. *Compt. rend. soc. biol.* 120, 326-8(1935), cf. *C. A.* 29, 6649.—Emboli were produced by injecting a suspension of lycopodium into a branch of the mesenteric artery of the chloralosed atropinized dog. A histamine-like substance was liberated in the blood which caused increased gastric secretion when the blood was transfused into another dog by the method previously described. AgNO<sub>3</sub>, injected in the same way, produced a similar effect by its irritating action without formation of emboli. Liberation of histamine-like substances by excitation of the peripheral portion of the splanchnic nerve. Hemorrhagic lesions of the intestine produced by the intraarterial injection of histamine. G. Ungar, X. J. Contades and A. Grossiord. *Ibid.* 328-30.—Elec. excitation of the nerve produced hemorrhagic lesions of the intestine (cf. Reilly, et al., *Ann. med.* 37, 339(1935)). Injection of histamine into an intestinal artery produced the same lesions, hence the nerve must have secreted histamine. That this was the case was proved by cross-circulation expts. Intervention of a neuro-humoral process, involving histamine, in the pathogenesis of pulmonary infarcts. G. Ungar, A. Grossiord and J. Bruncourt. *Ibid.* 632-3.—When pulmonary emboli were produced in dogs histamine appeared in the blood. Excitation of either the peripheral or central portion of the phrenic nerve, in dogs and guinea pigs, produced hemorrhagic lesions in the lungs and the appearance of histamine in the circulating blood.

**Regulation of serum protein imbalance by injection of blood albumin** Experiments on animals. G. Lefrou and P. Bonnet. *Compt. rend. soc. biol.* 120, 342-3(1935).—Experiments on man. *Ibid.* 424-7.—In 19 lepers, with serum albumin below normal, 10-20 twice-weekly subcutaneous injections of 10 cc of 2% blood albumin soln produced a slight increase in the albumin content of the blood serum and a slight decrease in the globulin content.

**Amino acid content of the blood of rabbits infected with**

**Yersin type tubercle bacillus** E. S. Panayotopoulou. *Compt. rend. soc. biol.* 120, 604-5(1935).—Three weeks after the beginning of the infection the blood amino acids, detd. after fasting for 18 hrs., reached 9.5-9.8 mg. % (normal is 6-7 mg. %).

**[Increase in] the tyrosine index of the serum polypeptides in rabbits infected with Yersin type tubercle bacillus** E. S. Panayotopoulou. *Compt. rend. soc. biol.* 120, 695-6(1935).

**Excessive dental calculus formation.** Joseph N. Finn and Jacques S. Gottlieb. *Dental Cosmos* 77, 1173-6(1935).—Calculus which enveloped the crowns of the lower teeth contained CaCO<sub>3</sub>, Ca oxalate and phosphate, and mucin.

**Metabolism of damaged tissue** Hermann Druckrey. *Naturwissenschaften* 23, 796-9(1935).—A review dealing especially with the metabolism of cancer tissue and its analogy to that of mechanically damaged tissue.

**Report of chemistry section.** B. J. C. van der Hoeven and C. Aston. *New Zealand Dept. Agr., Ann. Rept.* 1934-35, 60-5, cf. *C. A.* 29, 6306.—**Bush sickness in sheep**—The livers and blood of affected animals were not deficient in Cu. Healthy sheep drenched daily with 1 fluid oz. of a 1% soln of CuSO<sub>4</sub> for 3 months without access to Fe compds lost condition and became very bush-sick. In some cases bush sickness was temporarily cured by administration of As. The As content of grass from pastures in bush-sick and in healthy areas showed the same range, 0.1-0.7 p. p. m. All evidence points to Fe deficiency as the cause of bush sickness. Pampas grass contained total reducing substances (as glucose) 4-5, total hemicellulose 13-24, cellulose (Cross and Bevan) 37-41 and lignin 17-19%. The dry matter of the green leaves and the succulent bases contained 10 and 5% protein, resp. **Renal calculi**—A sheep renal calculus from Kiriopunt consisted principally of Ca phosphate, SiO<sub>2</sub>, uric acid and pigment. Renal calculi from a cow at Moerewa were composed chiefly of Mg NH<sub>4</sub> phosphate, fat and pigment; another contained SiO<sub>2</sub>, CaCO<sub>3</sub>, cystine and Ca phosphate. **Destructor ash** from Auckland contained 18% total CaO (of which about 9% was CaCO<sub>3</sub>) and about 1% each of H<sub>2</sub>O sol. K<sub>2</sub>O and total P<sub>2</sub>O<sub>5</sub>. The chem. compn. of soils from littoral lands in New Zealand is briefly discussed.

**Biochemistry of buras** III Chlorine and nitrogen in the blood. F. Rabboni. *Biochim. therap. sper.* 22, 536-46(1935); cf. *C. A.* 29, 1875.—Buras cause in rabbits a continuous decrease of the Cl in the blood, which becomes more accentuated toward death. The blood N shows a corresponding continuous increase. The administration of hypertonic NaCl soln. does not influence the decrease of blood Cl.

**Edema and imidazoles** M. Looper, E. Bloy, M. Perreault and A. Varay. *Presse med.* 43, 697-9(1935).—Edematous fluids are frequently rich in imidazoles. They are formed by breakdown of tissue, especially of the liver and the kidneys.

**Bromine in the blood** C. I. Urechia and Retezeanu. *Presse med.* 43, 701-3(1935).—The Br level in the blood has been studied in a large no. of affections. A decrease was observed only in manic-depressive psychoses.

**Iso-glucose curves in obesity** Pedro B. Landabure and José A. Pangaro. *Semana med.* (Buenos Aires) 1935, II, 1293-8.—While some obese persons show a diabetic glucose curve after administration of sugar, others develop little or no hyperglucemia followed by prompt return to normal or even lower levels. Such curves occur especially in young persons with endocrine disturbances. They represent a latent hyperinsulinism.

**The acutal reaction and the buffering power of necrotic tissue.** Giovanni Baldassi. *Sperimentale* 89, 330-47(1935).—Rabbit kidneys rendered necrotic by ligation of the artery and necrotic skin from guinea pigs and rats (by bacteria and viper toxins) have a buffer system, carbonate CO<sub>3</sub>. The alkyl can be detd. only after removal of the CO<sub>2</sub>, which shifts the p<sub>H</sub> beyond 8. The

alk. reaction of necrotic tissue explains its frequent calcification.

A. E. Meyer  
The influence of liver and spleen medication on development of far cancer in the mouse. G. I. De Gaetani and G. Panchianco. *Sperimentale* 89, 404-16 (1935).—The development of malignancy is accelerated by medication with spleen or liver either by mouth or subcutaneously.

A. E. Meyer  
Experimental studies on gastrogenic anemias in dogs. III. Review of certain experimental findings. Svend Petri, Axel S. Ohlén and David Heggild. *Acta Med. Scand.* 87, 14-32 (1935).—After surgical removal of different portions of the ventriculus and duodenum 3 different types of anemia appear: (1) hypochromatous condition with a polycythemic tendency after the extirpation of the pylorus and of the Brunner gland region in the duodenum, (2) simple but severe anemia, developing with great rapidity and generally ending in death, after the extirpation of the entire ventriculus and Brunner gland region, and (3) simple but extensive stationary anemia after the extirpation either of the fundus or of the fundus and of the entire duodenum. In no instance was a hyperchromatous condition, megalocytosis or leucopenia noted. The anemias were due to the loss of a specific antianemic function which is made up of 2 components (a hemoglobin and an erythrocyte-forming function) with definite regions of localization. In all 3 types of anemia the dogs manifested a more or less pronounced reaction to Fe but not to ventricule or liver preps. The relation of these anemia types to pernicious anemia is discussed. In one dog a condition actually developed which has a very close resemblance to human pernicious anemia and will be described in a subsequent paper.

S. Morgulis  
Effect of cations on the fermenting ability of tumor cells. V. Calcium and magnesium. 1. A. Lascutski and O. Rosenthal. *Biochem. Z.* 281, 395-401 (1935).—Neither Ca nor Mg affects anaerobic fermentation by rat implantation tumors under conditions under which K causes an increase.

S. Morgulis  
Partitioning of the phosphorus fractions in blood plasma of sarcomatous chickens. 1. Pentimalli and G. Schmidt. *Biochem. Z.* 282, 62-73 (1935).—Tumor-bearing chickens have a 50% higher total P content in plasma than normal chicks (15.2 and 11.6 mg. %). The lipid P and acid-soluble P are likewise 24 and 18% higher (8.6 and 6.9 mg. %, 5.5 and 4.5 mg. %, resp.). A protein P occurs in the plasma of the sarcomatous but not of the normal chickens. The origin of this P compd. is not known, but it may come from the tumor.

S. Morgulis  
Sex function in relation to the water content, especially in diabetes insipidus. Luigi Beltramini. *Endocrinologie* 16, 241-56 (1935).—The sex hormone exerts a definitely antidiuretic action. In a patient with an acquired diabetes insipidus of luteal origin the diuresis shrank from 12,000 to 100 cc. in 24 hrs. on the administration of folliculin. Several other similar experiences are cited. Since, however, it has been found that patients which are refractory to treatment with pituitrin are likewise refractory to the action of the sex hormone, it is suggested that the latter exerts its antidiuretic effect reflexly through its action on the hypophysis.

S. Morgulis  
Biochemical investigations on the summer encephalitis in Japan. S. Naka, N. Okumura and G. Kakihara. *Fukuoka-Ikudaiyaku-Zasshi* 27, 149-52 (1934).—Biochemical study was made upon 30 cases of summer encephalitis. In the delirious and comatous state most of the patients showed a mild or high degree of blood acidosis, but in convalescence the blood seemed to incline toward alkalosis. This acidosis was mostly compensated because the blood pH showed no distinct reduction. The  $P_{H_2O}$  of the spinal fluid seemed to increase, i.e., turned more to the alkaline side. Most patients showed slight but seldom high hyperglycemia. The albumin of the spinal fluid increased slightly, but it did not reach the pathological limit. The residual nitrogen of the fluid greatly increased.

K. Suetama  
1 in water (Rider) 14.

## H—PHARMACOLOGY

A. N. RICHARDS

"General factors" and "internal therapy" in cancer patients. G. A. K. von dem Borne. *Nederl. Tijdschr. Geneeskunde* 79, IV, 1956-61 (1935).—"General factors" include blood  $P_{H_2O}$  above 7.40, slightly higher blood sugar, markedly higher cholesterol content of the blood and failure of cancer blood to produce lysis of cancer cells (Freund and Kanner). The only therapy mentioned is that of Fischer using autolyses of spleen, lymph glands, thymus and bone marrow of young animals; the success is doubtful.

R. Reutter  
Dumenformone in large doses for the treatment of primary rheumatoid arthritis. F. C. Kuipers. *Nederl. Tijdschr. Geneeskunde* 79, IV, 5122-35 (1935).—Dumenformone, the benzox and ester of the follicular hormone, was given to two female patients with fair results.

R. Reutter  
Basic narcosis with rectidone. S. J. Gosses. *Nederl. Tijdschr. Geneeskunde* 79, IV, 5547-50 (1935).—Rectidone, the Na salt of sec-amyl- $\beta$ -methylbarbituric acid, was used as a rectal anesthetic in the place of avitin in 504 cases.

R. Reutter  
Effect of theophylline ethylenediamine on experimentally induced cardiac infarction in the dog. W. M. Fowler, H. M. Hurevitz and Fred M. Smith. *Arch. Internal Med.* 56, 1212-19 (1935).—Theophylline ethylenediamine promotes the development of the collateral circulation in cardiac infarction in the dog.

J. B. Brown  
Theophylline in the treatment of disease of the coronary arteries. Fred M. Smith, Herbert W. Ruthe and W. D. Paul. *Arch. Internal Med.* 56, 1230-42 (1935).

J. B. Brown  
Mandelic acid in the treatment of urinary infections. D. M. Lyon and D. M. Dunlop. *Brit. Med. J.* 1935, II, 1096-7.—The oral ingestion of mandelic acid as its Na salt has proved in 16 cases to be a valuable aid in producing urinary antiseptic.

J. B. Brown  
Further observations upon the changes in the electrolytes of the urine following the injection of parathyroid extract. Read Ellsworth and Wm. M. Nicholson. *J. Clin. Investigation* 14, 823-7 (1935).—After injection of parathyroid ext. to 4 human subjects, the urine became more alk., attended by increased inorg. phosphate, bicarbonate, Na and K.  $NH_4^+$  was slightly decreased. Two theories to account for these changes are proposed.

J. B. Brown  
The action of dinitrophenol and insulin on the metabolism of ethyl alcohol. Henry W. Newm and Windsor C. Cutting. *J. Clin. Investigation* 14, 945-8 (1935).—Concns. of dinitrophenol one in 5-20 million increased the rate of alc. metabolized by rat liver tissue *in vitro* about 5-10%. In the absence of the liver no increase occurred. Insulin and insulin-free pancreatic tissue affect the oxidation of EtOH in the absence of liver tissue; in therapeutic doses in man they are capable of increasing alc. metabolism 50%. Isolation of the principle responsible for this action may yield a useful accelerator of EtOH oxidation in cases of alcoholism.

J. B. Brown  
Recollections on asthma with a few suggestions as to its treatment in childhood. W. D. Allan. *Glasgow Med. J.* 6, 225-31 (1935).—Asthma in children has been successfully treated by the administration of male and female sex hormones to males and females, resp.

R. B.  
Chemotherapy. The progress of thirty years and the prospect. C. H. Browning. *Glasgow Med. J.* 6, Trans. Roy. Med.-Chir. Soc. Glasgow 30, 1-16 (1935-36).

Rachel Brown  
Treatment of diabetes mellitus. S. Vatcher and M. Douglas. *J. Trop. Med. Hyg.* 38, 278-83 (1935).—Insulin and diets in the treatment of diabetes mellitus. *Ibid.* 289-93.—A high-carbohydrate, low-fat diet is employed and insulin is given in all cases to control the blood-sugar level.

Rachel Brown  
Synergy of adrenaline and acetylcholine on the pulmonary blood vessels in the rabbit. G. Harold Ettinger and G. Edward Hall. *Quart. J. Exptl. Physiol.* 25,

259-65(1935)—Acetylcholine is capable of producing powerful constriction of the pulmonary blood vessels in the rabbit. The constriction depends upon an initial tone of the blood vessels. In the perfused blood vessels repeated injections of acetylcholine produce a condition in which the muscle fails to respond, probably because acetylcholine promotes dilatation following the constriction. The sensitivity may be restored by adrenaline, Ba or histamine. In the living animal the tone is probably mediated by the sympathetic nerves or by adrenaline. The hypothesis is offered that major changes in the caliber of the pulmonary artery and arterioles of the rabbit are brought about through parasympathetic activity.

Rachel Brown

Effect of low oxygen pressures on frog cardiac tissue. A. J. Clark and G. Kinnerep. *Quart J Exptl Physiol* 25, 279-80(1935).—The lowest O pressure which produces any measurable delay in asphyxia of the normal sinus is 5 mm. Hg and in the case of the iodoacetic acid-poisoned sinus it is 10 mm. Normal activity can be maintained in both cases by an O pressure of 20 mm. The corresponding figures for the auricle and ventricle are somewhat greater. The effect of O pressure above 0.015 atm. is in accord with Warburg's formula, but the formula does not appear to hold for lower pressures. The relative effects of asphyxia on the different functions of the heart can be correlated with differences in metabolic rates.

Rachel Brown

Pharmacology of physostigmine. Charles R. Linegar, James M. Dille and Theodore Koppanyi. *Science* 82, 497(1935).—In exptl animals in which the peripheral vagus was rendered nonresponsive to weak faradic stimulation by barbitalurates or nicotine salicylate, 3 min. after intravenous administration of physostigmine salicylate, weak stimulation of the vagus produced marked cardiac inhibition.

Rachel Brown

Cobalt salts as prophylactic and therapeutic antidotes in cyanide poisoning. V. M. Rozhkov, N. S. Stepanenko and K. M. Usova. *J Physiol (U S S R)* 19, 582-4(1935).—Co salts, like  $\text{Co}(\text{NO}_3)_2$ ,  $\text{CoCl}_2$ ,  $\text{CoSO}_4$ , produce a prophylactic and therapeutic effect on white mice poisoned by NaCN. The mortality rate sinks from 83-85% to 7-11%.

H Cohen

Methemoglobin builders as antidotes in fluoride poisoning. O. G. Vinogradova and V. M. Rozhkov. *J Physiol (U S S R)* 19, 585-6(1935).—The subcutaneous injection of  $\text{NaNO}_2$  into white mice poisoned by fluorides causes a drop in the death rate from 82% (control) to 26%. The methemoglobin produced by the  $\text{NaNO}_2$  unites with the fluoride, lowering the concn of the latter in the blood.

H Cohen

The role of fats and lipins in the blood during the absorption of some indifferent narcotics. A. I. Brusilovskaya. *J Physiol (U S S R)* 19, 587-93(1935).—The expts were performed on dogs and rabbits. The amt. of fats and lipins in the blood does not influence the absorption of benzene or benzine by the breathing organs. Contrary to the prevailing view, a diet rich in fats does not affect the concn of benzene or benzine in the blood.

H Cohen

Action of sulfurous acid on the bactericidal capacity of blood. H. Cremer. *Z. Untersuch. Leberm.* 70, 315-17(1935).—A long-continued addn of very small amts of  $\text{H}_2\text{SO}_3$  lowers strongly the bactericidal power of rabbit blood to staphylococci.

F. L. Dunlap

Fixation and elimination of ascorbic acid. A. Giroud, R. Chue, R. Ratamamanga and C. P. Leblonde. *Compt rend soc. biol.* 120, 330-3(1935).—When 50 mg. ascorbic acid (I) was injected intravenously into guinea pigs already well stocked with I much was excreted in the urine during the next 2 hrs. and very little thereafter. The I content of the kidneys rose to 10 times normal in a few min. after the injection and then declined rapidly, beginning less than a half hr. later. The I contents of the liver and adrenals showed a rapid rise for 2 hrs. then a slow decrease. No significant change in muscle I was found.

L. E. Gilson

Blood changes produced by subcutaneous injections of glucose. G. Delrue and P. Hollebeke. *Compt rend soc.*

*biol.* 120, 529-30(1935).—When rabbits received daily subcutaneous injections of 300 cc. of 10% glucose soln. they died after a few days. Changes in blood compn. observed were similar to those in nephritis with N retention and were probably the result of the anuria provoked by the glucose injections.

L. E. Gilson

Effects of repeated injections of glucose on the muscle glycogen of normal frogs. A. Moschm. *Compt. rend. soc. biol.* 120, 531-3(1935).—The injection of 1-2 cc. of 25% glucose soln. daily under the skin or into the lymph sac caused a rapid increase in muscle glycogen (max. observed 2.56%). Effects of repeated injections of glucose on the muscle glycogen of frogs under different experimental conditions. *Ibid.* 533-5.—Injections similar to the above increased muscle glycogen, though to a smaller degree, in pancreatized frogs. In the muscles of the leg, glycogen increased as usual when the sciatic nerve had been recently cut, but if the nerve had been cut long enough for tissue degeneration to take place (3-5 weeks) there was little or no increase in glycogen following the glucose injections.

L. E. Gilson

Effects of thyrostimulin on the histological appearance of the endocrine glands. B. Giedosz. *Compt. rend. soc. biol.* 120, 555-6(1935).—Guinea pigs were used. Hyperemia and other evidences of stimulation of the thyroid, hypophysis and adrenals were noted. In the ovaries ovogenesis was suppressed and numerous follicular cysts were formed. Effects of vitamins A and C on the histological appearance of the endocrine glands. *Ibid.* 557-9.—Rabbits were used. Both vitamins increased the activity of the ovaries, adrenals and anterior hypophysis. In the case of the thyroid A seemed to arrest, while C stimulated, hormone production.

L. E. Gilson

Effects of picrotoxin on the reflex excitability of the [frog] medulla. H. Schreyer and G. Perschmann. *Compt. rend. soc. biol.* 120, 625-6(1935).

L. E. G.

Effects of the continuous injection of adrenaline on blood urea, cholesterol and calcium. A. Baudouin, H. Bénard, Y. Lewin and J. Sallet. *Compt. rend. soc. biol.* 120, 629-31(1935), cf. C. A. 29, 5316\*.—Dogs were used as before. Even when the adrenaline was injected in large doses there was no significant change in blood urea, cholesterol or Ca.

L. E. Gilson

Antagonism of cryogenin and 2,4-dinitrophenol. A. Leulier and G. Béruard. *Compt. rend. soc. biol.* 120, 650-1(1935).—Pigeons were given 10 mg./kg. dinitrophenol and 170-250 mg./kg. cryogenin (phenylsemicarbazide) at the same time. The temp. rise was about 1° less than that produced by the same dose of dinitrophenol alone.

L. E. Gilson

Further experiments with lipides considered from the viewpoint of a theory of the pharmacodynamic actions of the alkali and alkaline earth metal ions. Jakob Wajner. *Compt. rend. soc. biol.* 120, 707-9(1935), cf. C. A. 29, 4229\*.—In the system  $\text{aq. } 100\text{-}\mu\text{MNH}_4\text{-peanut oil-oleic acid}$  the interfacial tension, deid. as in previous work, is 11.6 dynes. The addn of NaCl and KCl to 0.1 N concn. decreases the interfacial tension to 7.8 and 8.0 dynes, resp., while  $\text{CaCl}_2$ , 0.1 N, increases it to 14.2 dynes. Remarks on the above paper. L. Lapique. *Ibid.* 709-10.

L. E. Gilson

Hemolysis by x-rays and effect of cholesterol. B. S. Levin and C. Piffault. *Compt. rend. soc. biol.* 120, 712-14(1935).—The resistance of dil. guinea-pig blood to hemolysis by x-rays was much greater if the animal had received an intramuscular injection of 3 cc. of a 4% soln. of cholesterol in olive oil 2-10 days before the blood sample was taken.

L. E. Gilson

Pharmacological reactions of the cerebral blood vessels. J. J. Bouckaert and F. Jourdan. *Compt. rend. soc. biol.* 120, 790-2(1935).—Discussion. Twenty-three references.

L. E. Gilson

Changes in arterial pressure produced by repeated injections of streptococcus toxin in dogs. E. Dickel. *Compt. rend. soc. biol.* 120, 793-5(1935).—Daily injections caused an increase in blood pressure, an irregular increase in azotemia, and albuminuria. The effects were much like

those observed in human cases of scarlatina and infected tonsils. L. E. Gilson

Effect of Congo red on bleeding time W. de Weerd and W. van Hecke. *Compt rend soc. biol.* 120, 795-6 (1935)—In rabbits the intravenous injection of 0.5 cc./kg. of a 1% soln. of Congo red in distd. water caused a 23% decrease in bleeding time. If the Congo red was dissolved in 0.9% NaCl soln. instead of distd. water it had no effect on bleeding time. L. E. Gilson

Absorption of bilirubin, Bengal rose and tetrabromosulfonaphthalen by the liver M. Royer. *Rev. sci. argentine biol.* 11, 482-8 (1935), *Compt rend soc. biol.* 120, 809-12 (1935)—Bilirubin gives the most satisfactory results in the hepatic insufficiency test. L. E. Gilson

Adrenal cortex therapy in the toxemias of pregnancy W. Herbrand. *Deut. med. Wochschr.* 61, 1682-3 (1935)—A review of clinical reports bearing on the supposed efficacy of adrenal exs. Arthur Grollman

Pharmacology of ergometrine G. L. Brown and Henry Dale. *Proc. Roy. Soc. (London)* B118, 446-77 (1935)—Ergometrine produces central excitation with general sympathetic stimulation. It exerts merely a trace of specific paralyzing action on motor sympathetic effects, produces cyanosis of the cock's comb without gangrene, and increases body temp. in toxic doses. Its action on arterial pressure varies with the conditions of anesthesia and of the integrity of the brain, a pressor action is exerted on the spinal cat. Its most characteristic action, and sole action in small doses, is the initiation of a long-persistent rhythm of powerful contractions in a uterus normally quiescent as in the early puerperium. It apparently has a peripheral action of sympathomimetic type on several organs. It is less toxic than ergotamine, and is far more readily absorbed on oral administration. Joseph S. Hepburn

The relation between dielectric polarization and pharmacological action [of medicinal compounds] K. W. Rosenmund. *Angew. Chem.* 48, 701-5 (1935)—The following synthetic lactones were investigated for their anesthetic action: anisole lactone (I), *o*-, *m*- and *p*-cresol ether lactone (II, III, IV), phenol lactone (V), *p*-cresol lactone (VI), thymol lactone (VII) and thymol ether lactone (VIII). Their action upon leeches and ascariids compared with that of santonin was greater for I, II, V, weaker for IV, VI and no action was shown by III, VII and VIII. Their phys.-chem. behavior in relation to their medicinal action was investigated and it was found that surface tension showed no direct beneficial influence, and that surface activity is only an auxiliary property, promoting penetration to the interior of the cells. The surface tension of  $H_2O$  is lowered by equimol. solns. (concn. 0.00075 g. mol. per l.) as follows: V 1.3, I 3.3, VI 7.9, IV 8.5, III 8.8, II 9.2, VII 13.3 and VIII 15.4%. Swelling expts. with casein and gelatin in  $H_2O$  and Ringer soln., with and without addn. of lactone, also did not show any relation to medicinal action, the behavior of the lactones as excitants being the opposite to that of narcotics. The permeability of fresh and dried human ovarian membranes to methylene blue and red beet ext. was detd.; all lactones increased the permeability, particularly those contg. a free OH group. A numerical relationship was found to exist between the influence exerted upon swelling and permeability, but no relation between medicinal activity and permeability. All lactones promoted gel-sol transformation and retarded sol-gel transformation, but noticeable differences in their action could not be found. The effects of alternating polarities at the C atoms in the benzene ring, as induced by substituents, and their possible application to a theoretical explanation of the relation between constitution and medicinal action are discussed at length. They lead to the conclusion that the most active compds. are those possessing complete additive polarity, without any shielding by neighboring polar groups. The two medicinally active lactones II and IV possess the strongest polarity, as all induced charges are additive throughout the whole mol., while in the inactive III a weakening of the polarity results from superimposition of opposite charges. The most effective compd. is II,

and the somewhat lower effectiveness of IV is explained by the steric shielding which is exerted upon the strongly polar lactone group by the  $-OCH_3$  group in *o*-position to it. Steric shielding likewise occurs in III by the  $-CH_3$  group in *o*-position to the lactone group. Further examples shown are the six isomeric dihydroxybenzoic acids in their retarding action upon the yeast fermentation of dextrose as a function of polarity distribution. Five references. Karl Kammermeyer

The administration of iron G. H. W. Lucas and V. E. Henderson. *Can. Med. Assoc. J.* 34, 53 (1936)—A general review of the efficacy of the administration of Fe in large doses and a report of some expts. which showed that by cooking vegetables in heavy Fe pots, the Fe content of vegetables could be increased as much as 10 to 40 times. This Fe appears to be as available as the Fe in the vegetables themselves. G. H. W. Lucas

New investigations on the antidotism between hydrocyanic acid and sodium tetrathionate S. Sapiezna. *Arch. intern. pharmacodyn.* 51, 44-62 (1935)—A freshly prepd. soln. of equal vols. of  $Na_2S_2O_8$  +  $5H_2O$  (20 g. %) and 10% I<sub>2</sub> in 13% NaI (which results in  $Na_2S_2O_8$  5.3 g. % and NaI 12.4 g. %) injected intravenously in dogs and rabbits saves animals that have been given 5-6 times the m. l. of cyanide. The dose of the antidote is 11-13 cc./kg. body wt. P. F. Mettild

Osmotic changes in the muscle in contraction caused by veratrine and nicotine I. Gentile. *Arch. ital. biol.* 93, 190-6 (1935)—The faculty of absorbing water is reduced. A. E. Meyer

The ratio dehydroascorbic acid/ascorbic acid in tissues after administration of thyroxine Emilio Martini and Fernando Copello. *Biochim. therap.* 22, 529-35 (1935)—Repeated treatment with thyroxine produces in the guinea pig a reduction in ascorbic acid. The dehydroascorbic acid is increased in a higher degree. As a consequence, the total content of the tissues in vitamin C is increased and the quotient shows a large increase. A. E. Meyer

Treatment of coma caused by barbiturates Ch. Flaudin. *Presse med.* 43, 803-4 (1935)—High doses of strychnine, blood letting, artificial respiration and adrenaline by rectum are recommended. A. E. Meyer

Prophylaxis of accidents caused by arsenobenzenes. U. Reboud. *Presse med.* 43, 899-900 (1935)—The addn. of glycocoll, as recommended by Bénech (cf. C. A. 29, 3731<sup>9</sup>), is less reliable than the use of the aminoacids derived from liver. A. E. Meyer

The action of urine extracts on adrenals and ovaries of the rabbit Max Aron. *Presse med.* 43, 1044-6 (1935)—Urine from cancer patients causes typical histological changes of homogenization in the adrenals. The left adrenal is removed for biopsy before injections are begun and is compared with the right gland after treatment. Cancer urine gives a pos. Aschheim-Zondek reaction in 80% of the cases. A. E. Meyer

Total cinnamon of balsam of Peru and choline camphorate; a therapeutic synergism. P. Logeais. *Rev. med.* 52, 125-30 (1935)—Cinnamon consists of a mixt. of benzyl cinnamate, benzyl benzoate and some resinotannins. It is an amber-colored liquid of characteristic odor and taste. Solns. in oil are suitable for therapeutic use by injections. It causes a considerable augmentation of the leucocytes. Choline camphorate dissolved in an isotonic soln. of *d*-camphor is applied intramuscularly. It has a cardiotoxic and sympatheticoptic action, causes an increase of the erythrocytes and mononuclear cells, increases the cholesterol in the blood and neutralizes bacterial toxins. An alternating medication with both substances gave excellent results in infections, especially tuberculosis, in anemias, asthenias and during convalescence. A. E. Meyer

Action of lachesis venom Gustavo Escobar. *Semana med.* (Buenos Aires) 1935, II, 1479-84—The viper *Bothrops* (Yarará) and its habits are described. The venom is an odorless, slightly greenish and opalescent liquid of acid reaction and sp. gr. 1.03-1.05. It is stable and of little toxicity if taken by mouth. It is inactivated

at 65° and contains 65-80% H<sub>2</sub>O, besides proteins, fat, Cl, phosphate, Ca, NH<sub>4</sub> and Mg. Light is without influence on the dried product but destroys it in soln. The symptoms of poisoning, as described, involve almost the whole organism. A 2% soln of the venom dissolves many bacteria. The therapeutic dose includes dilns from the 6th decimal and following, up to 30 drops daily. It is used in cancer treatment and in appendicitis.

**Ergotone in the treatment of some mental diseases** Antonio Carelli. *Semana med* (Buenos Aires) 1935, 11, 1571-2.—1-ergotone gave favorable results in hysterical crisis, delirium, excitation and manias. A. E. Meyer.

**Intravenous animal charcoal in physiological and some pathological conditions** Giovanni Selvaggi. *Sperimentale* 89, 346-403(1935).—The charcoal is deposited in the following sequence: spleen, liver, lungs, less in the lymph glands, and the bone marrow. There is no sp. stimulation of the reticulo endothelial system. A loci of infection with staphylococcus are surrounded by a "filter" of C particles. The elimination takes place in the liver. A. E. Meyer.

**Observations on the insulin-adrenaline treatment by the Clausen method** T. Thune Andersen. *Acta Med. Scand* 86, 361-6(1935).—Diabetic patients were treated according to Clausen, by injecting 0.1 mg. adrenaline with every 5 cc. insulin. No hypoglycemic symptoms occurred in the patients so treated, in a considerable proportion of cases the glucosuria greatly decreased and the fasting blood sugar was lower than before the treatment.

**Biological assay of adrenal cortical preparations with the use of white rats and mice** G. Widström. *Acta Med. Scand* 87, 1-13(1935).—Adrenalectomized white rats, under controlled conditions and on the third postoperative day, when exposed for 4 hrs to a temp. of +3° show a fall in body temp. In about 65% of the animals this fall exceeds 9°. Treated with cortical preps during the interval after adrenalectomy the rats on exposure to cold either show no fall in temp. or the fall depends upon the dose administered. This reaction has been utilized in working out a method for assaying the cortical prep. White mice are less suitable for this assaying than rats.

**Polylyzate therapy** I. N. Kazakov. *Acta Med. Scand* 87, 33-49(1935).—The lyzates are products of artificial hydrolysis of different organs with acid or alkali under 12-15 atm. pressure. The organs are obtained from freshly killed animals. These lyzates were tested on 4000 patients. The effect of these preps has been detd. from the point of view of the dispersion state of the colloidal systems of the organism, the metabolism of the nervous tissue, the increase in blood immunity and increase of oxidative processes. Basedow patients (700) were treated with a combination of 5 lyzates (anterior hypophysis, parathyroid, adrenal cortex, ovaries and liver) to which brain lipoids and several other lyzates were added. Only in 5% of those treated was there no change, but in 17% there was complete healing, in 50% definite improvement and in 28% some improvement. Results in a number of other clinical conditions are recorded.

**Studies on the physiological chemical action of peat extract (humic substances) administered perorally or intravenously** Rudolf Schmidt. *Biochem Z* 281, 329-32(1935).—Humic substances can be fed to rabbits with impunity. A small part is absorbed. Intravenous injection of sterile aq. exts. from peat is tolerated, if the concn. is less than 1%; otherwise they cause death.

**Hormonal effects on alcohol metabolism** Erik M. P. Widmark. *Biochem Z* 282, 79-84(1935).—No definite effect on the metabolism of alc. in dogs has been found either from thyroxine, pituitrin or adrenaline. Insulin activates strongly the alc. metabolism in dogs with a sluggish metabolism, which may become doubled within 200 min. But in dogs with an initially strong alc. metabolism the insulin injection has no effect.

**Treatment of hyperthyroidism with gold salts** L. Benito Iuertes. *Indoktrinologie* 16, 257-64(1935).—

In about one-half of the cases treated with Au salts there has been improvement of the hyperthyroid condition with lowering of the basal metabolism which persisted even after the treatment. The salts are best administered by frequent small doses intramuscularly. S. M.

**A theory of the sensitization to acetylcholine, and the effect of fluoride in raising the irritability** G. Kahlson and B. Uvnäs. *Skand Arch Physiol* 72, 215-39(1935).—

Acetylcholine acting on muscles of the frog or leech previously treated with physostigmine calls forth contractions, but in untreated muscles is destroyed within a small fraction of a second. This explains the ineffectiveness of small doses of acetylcholine or the prolongation of its action in physostigminized muscles. The phenomenon, however, cannot be interpreted simply as a case of inhibition of esterase function. This would leave open the problem why the acetylcholine sensitive gastrocnemius can be sensitized like the rectus muscle by physostigmine. Furthermore, the enzyme inhibition by NaF has no effect in making the leech muscle sensitive to acetylcholine. The inhibition of the esterase activity can at best only cause a prolongation of the acetylcholine effect, but the increased sensitivity must result from other conditions. The fluoride inhibition is not complete even in very great concns and F<sup>-</sup> produces a strong increase in the acetylcholine sensitivity of the rectus muscle even in such concn.

*in vitro* does not inhibit the esterase. I. e., the sensitivity to acetylcholine may be increased either by F<sup>-</sup>, which only partly paralyzes the enzymic action, or by physostigmine, which produces complete paralysis. The pharmacological effect of the fluoride is discussed. S. M.

**Effect of phlorizin on the isolated kidney and isolated liver** Linar Lundsgaard. *Skand Arch Physiol* 72, 265-70(1935).—In a lung kidney prep. complete phlorizin poisoning was obtained by a dose of 0.5-1.0 mg. per g. kidney. The glucolytic power of a kidney poisoned with a moderate dose of phlorizin is not affected. The concn. of phlorizin which completely inhibits the glucose reabsorption in the kidney is probably less than that necessary to prevent esterification by muscle pulp or yeast. Severe phlorizin poisoning of rabbits or the addn. of a moderate amt. of phlorizin to blood perfused through the liver does not affect its carbohydrate metabolism. S. Morgulis.

**Paraldehyde idiosyncrasy** Gilbert Brown. *Brit J Anaesthesia* 13, 25-7(1935).—Prolonged, deep, unconsciousness developed in a man aged 20 and weighing 123 lbs. with a blood pressure of 120/78, after the rectal administration of 4 drachms of paraldehyde in 5 oz. of saline soln. Complete loss of all reflexes developed, assoc. with rapid respiration and very slight fall in blood pressure. Elimination methods led to recovery. James C. Munch.

**Studies in kala-azar** P. K. Guha. *Calcutta Med J* 30, 193-218(1935).—By the levulose test normal liver function was found in 15 cases of kala-azar before treatment, and 8 cases after treatment. Pos. van den Bergh tests were obtained in 4 of the 15 untreated cases. Urobilinuria was observed in all patients. Quinquevalent antimonials proved clinically effective. Sixty-eight references. James C. Munch.

**Veratrum viride in the treatment of eclampsia** R. D. Bryant. *Calcutta Med J* 30, 237-42(1935).—In treating 127 cases of eclampsia during the last 8 yrs., veratrum exts. have proved useful, death has not been observed in the last 56 patients. Veratrine, MgSO<sub>4</sub> and alkalis are given with large vols. of fluid. The blood pressure fell to 50 mm. systolic and the heart rate to 40 beats per min. Emesis was observed in many patients. Atropine or morphine was useful as an antidote. Marked variation in susceptibility was observed. James C. Munch.

**The treatment of gonorrhea with flavadin** H. O. Loos. *Dermatol. Z* 72, 149-56(1935).—In treatment of gonorrhea in females, 10 injections of a 2% aq. soln. of flavadin, an arsenical acridine deriv., proved effective. Similar effective responses were observed in involvements of the cervix and urethra. James C. Munch.

**The effects on the rabbit of repeated large intravenous doses of glucose** H. E. Harding. *Guy's Hospital Repts* 85, 372-6(1935).—The intravenous injection of 20

to 25 cc. of a 50% soln. of glucose in distd. water 4 or 5 times daily to rabbits, continued until vein damage made it impossible, produced severe loss in wt. This resulted from dehydration of the tissues and was prevented by oral administration of dil. saline soln. Similar effects were observed in connection with intravenous injection in human beings. The blood sugar of a rabbit was 141 mg. % before injection, 5 min after intravenous injection of 23 cc of 50% glucose, 955 mg. %, 110 min later, 140 mg. %. Four additional doses totaling 92 cc were given on the same day, the following day the blood sugar was 145 mg. % Of the injected glucose, 80-90% was retained.

**The use and abuse of digitalis** Arthur G Sullivan  
*J Arkansas Med Soc* 32, 107-9(1935)

**Cyclopropane** a new gas anesthetic Report of 120 cases George S Mechleng *J Oklahoma State Med Assoc.* 28, 436-9(1935)—Cyclopropane has proved as safe as any inhalation anesthetic. The explosive range for cyclopropane-oxygen extends from 25 vols % cyclopropane plus 75 vols % oxygen to 71 vols cyclopropane and 29 vols of O.

**Treatment of malaria** C D de Langen and C J Storm *S African Med J.* 9, 677-8(1935)—See *C A* 29, 6953.

**The efficiency of trypanamide in the second stage of sleeping sickness** M Bonnet *Trop Disease Bull* 32, 17(1935)—Trypanamide proved clinically effective in 150 patients.

**Urotropine intravenously associated with arsenicals in the treatment of second- and third-stage sleeping sickness** Lieurade *Trop Disease Bull* 32, 19(1935)—In studies on 12 patients, the intravenous injection of 20-30 mg of urotropine per kg at weekly intervals, associated with trypanamide treatment, proved more effective than trypanamide treatment alone.

**Sodium hyposulfite in the treatment of ocular troubles due to trypanocides** Raignard *Trop. Disease Bull* 32, 19-20(1935)—Intravenous injection of Na hyposulfite produced relief in 20 of 26 cases showing ocular involvement after atoxyl or trypanamide and also in 9 of 12 patients developing blindness.

**Action of quinine iodobismuthate on *T. gambiense*** P. Lassabriere and A. Peycelon. *Trop Disease Bull* 32, 20-1(1935)—Intramuscular and subcutaneous injection of quinine iodobismuthate to guinea pigs infected with *T. gambiense* failed to produce definite prolongation of life. This drug appears to be a valuable adjuvant, but is decidedly inferior in curative action to the arsenical compd.

**Subsequent histories of six cases of Trypanosoma rhodesiense infection treated with "Bayer 205" or "Fournneau 309."** A. J Keevill *Trop Disease Bull.* 32, 21(1935).—Six patients were found to be in normal health 2 years after treatment, and 2 of these appeared to be normal 8 years after treatment. When trypanosomes had been found in the spinal fluid, a combination of trypanamide and Bayer 205 is more effective than either drug alone.

**The dosage of moranyl in the treatment of gambiense sleeping sickness.** A. Sice and H. Mercier. *Trop. Disease Bull.* 32, 21-2(1935).—The oral or intravenous administration of 1 g. of moranyl weekly for 8 wks. produced a degree of sterilization corresponding to that produced by orsanine. However, moranyl produced marked or severe albuminuria. The combination of moranyl and trypanamide produced more favorable results.

**Mode of action of germanin in trypanosomiasis** N. von Jancso and H. von Jancso. *Trop. Disease Bull.* 32, 22-4(1935).—Failure to demonstrate trypanocidal action of Bayer 205 is attributed to difficulties in maintaining viable trypanosomes *in vitro*. A special technic is recorded for growing trypanosome on sheep serum-glucose-Ringer soln. The heart blood of infected rats, guinea pigs or mice is mixed with 1% of heparin to prevent coagulation and inoculated. No difficulty was encountered in maintaining

live cultures for 50 to 70 hrs. Bayer 205 in a concn. of 1:60,000 destroyed all parasites after a latent period of 24 hrs., acting by the production of atresis through interference with nutrition of trypanosomes. Arsenoxides act in a different manner, producing immediate effects. Germanin rendered trypanosomes fit for phagocytosis by reticulo-endothelial cells. The chemotherapeutic index on normal animals was 1:270, in animals after splenectomy and blocking the reticulo-endothelial system with colloidal Cu, the index was 1:135.

**Report of the Jamaica yaws commission for 1932-1934.** T B Turner, G M. Saunders and H. M. Johnston, Jr. *Trop Disease Bull* 32, 50-2(1935).—Consistent controlled studies have indicated that neosarsphenamine and bismuth compds are proving effective in the treatment of yaws.

**Treatment of yaws by intravenous injection of copper sulfate** A Occhino and Y Kernkamp *Trop. Disease Bull* 32, 56(1935).—Daily injection of 0.6% CuSO<sub>4</sub> for 10 to 25 days proved effective in the treatment of 200 patients.

**Detoxification of strychnine by pentobarbital sodium** Edward E Swanson *J Am Pharm Assoc* 24, 959-61(1935).—In single equiv doses, pentobarbital Na has a less effective antidotal action in strychnine poisoning than has Na amytal (cf. *C A* 27, 4304).

**Influence of various substances on the lactic acid dehydrogenase in the heart muscle.** Ichiro Yamamoto *Fukuroka-Ikudaisigoku-Zasshi* 27, 2767-72(1934).—The inhibitory effect of oxalic acid on the lactic acid dehydrogenase is independent of the Ca-ptg action of oxalic acid. NaF and Na citrate have no influence. In oxidation the formation of the macroscopic salt of lactic acid is not necessary. Lactates of Na, Li, K, NH<sub>4</sub> and Ca are readily oxidized. Ag, Hg and Cu possess a marked inhibitory effect, but alkaloid, insulin, adrenaline and nicotine have no influence on the lactic acid dehydrogenase.

**Effects of sodium citrate on the alkali reserve and coagulability of the blood.** David De Souza and F. D. M. Hocking. *J. Physiol.* 85, 168-72(1935); cf. *C. A.* 29, 5159\*.—Repeated small intramuscular injections of Na citrate have a cumulative effect in increasing the alkali reserve and the coagulability of the blood. Relatively large injections increase the alkali reserve and diminish the coagulability of the blood, after in some cases an initial increase. Changes in the coagulability of the blood produced by citric acid and some of its decomposition products. *Ibid* 173-8.—Intramuscular injections of citric acid or NH<sub>4</sub> citrate increase the alkali reserve and the coagulability of the blood. Similar injections of acetonedicarboxylic acid or acetic acid do not affect the alkali reserve, but may increase or diminish coagulability. Intramuscular injections of Na acetonedicarboxylate or Na aconitate increase the alkali reserve and the coagulability of the blood. Injections of MeCO have no effect on alkali reserve or coagulability.

**The action of adrenaline on the respiratory quotient.** Edward M Bridge and H. R. Noltie. *J. Physiol.* 85, 334-42(1935); cf. *C. A.* 24, 1425; 25, 3081\*.—Continuous intravenous injection of adrenaline into unanesthetized rabbits causes a fall of R Q to the protein-fat level. The low values of both liver and muscle glycogen, found at the end of such expts., suggest that the action of adrenaline on glycogen is a purely lytic one.

**Choline and liver respiration** O. A. Trowell. *J. Physiol.* 85, 350-74(1935); cf. *C. A.* 29, 4413\*.—Choline in concns. of 0.012% and above increases the O consumption of liver slices, as measured in differential manometers of the Barcroft-Dixon type. The greater the choline concn. the greater the effect. The effect is uninfluenced by either added fatty acid or glucose. Choline increases the O consumption of kidney slices. It decreases the O consumption of spleen and cardiac muscle. It has no effect on the O consumption of brain. Choline is oxidized by washed liver pulp with the uptake of approx. 1 atom of O per mol. of choline. It has no effect on the O uptake of an aq. ext. of liver. In concns. of 0.01% and

above choline inhibits the acetoacetic acid production of liver slices, and at 1.0% the inhibition is practically complete. In the presence of added fatty acid the inhibition is still marked. It is, therefore, probable that choline decreases the rate of oxidation in the liver. The effect of choline on the O uptake of liver slices is probably the algebraic sum of 2 step processes (1) oxidation of choline itself by the liver, (2) inhibition of the normal fat oxidation in the liver. These findings afford no explanation of the action of choline in the prevention and cure of various types of fatty liver. They do, however, rule out the explanation that choline acted by accelerating fat oxidation in the liver. These conclusions apply to the rat only.

R. D. Walter

**Influence of active substances of endocrine glands on hemolysis.** Edward Falik *Acta Biol Expil* 8, 73-9 (1933) (German summary).—The active substances of endocrine glands, viz., adrenaline, thyroxine, insulin, hypophysis anterior and hypophysis posterior raise alone or in mixts with each other the resistance of the red blood corpuscles toward osmotic influences. The expts induce the assumption that these corpuscles become permeated by hormones, and that the stroma-colloids change their state of dispersion producing a denser stroma surface, whereby the blood corpuscles become more resistant and give off their dye only in a strongly hypotonic medium.

J. Wiertelak

**The influence of lecithin upon the isolated heart of poikilothermic animals.** W. S. Holobut and Z. Bielinski *Acta Biol Expil* 8, 178-85 (1933) in French (1933).—Lecithin perfused in 0.001 to 1% solns in Ringer reagent accelerates the action of the heart of poikilothermic animals (frogs and tortoises) and increases contractions. If beforehand the parasympathetic endings in the heart are paralyzed by means of 0.0004% atropine, lecithin in the same doses as above acts in the opposite direction, slowing the action of the heart and diminishing its contractions. The phenomenon is explained by the action of lecithin as an agent paralyzing the autonomic system, the parasympathetic as well as the sympathetic of the heart, with a greater affinity toward the parasympathetic part.

J. Wiertelak

**N.C. Derivatives of barbituric acid.** E. Gryszkiewicz-Trochowski *Arch Chem Farm* 2, 1-8 (1934) (German summary).—Action of  $\text{C}_6\text{H}_5\text{CH}_2\text{NEt}_2$  (I) on N-Na derivs of C-substituted barbituric acid gives the corresponding alkylamine derivs. Thus the following compds are prepd: 5,5-diethyl-3-(diethylaminoethyl)barbituric acid  $\text{C}_{14}\text{H}_{20}\text{N}_4$ , by heating 0.1 g mol of Bayer's "Veronal-Natrium," the Na deriv of diethylbarbituric acid, in 100 cc abs EtOH and 0.1 g mol of I during 5 hrs on a water bath, HCl salt colorless, m 225-6°, easily sol in EtOH and  $\text{H}_2\text{O}$ ; 5-ethyl-5-butyl-3-(diethylaminoethyl)barbituric acid,  $\text{C}_{18}\text{H}_{26}\text{N}_4$ , prepd similarly from the Na salt of ethylbutylbarbituric acid and I, HCl salt easily sol in  $\text{H}_2\text{O}$  and alc, m 212-3°, 5-methyl-5-(1-cyclohexenyl)-3-(diethylaminoethyl)barbituric acid (II),  $\text{C}_{18}\text{H}_{24}\text{N}_4$ , from I and the Na salt of 5-methyl-5-cyclohexenyl-3-methylbarbituric acid, HCl deriv white, cryst, easily sol in EtOH and  $\text{H}_2\text{O}$ , m 150-1°, 5-ethyl-5-phenyl-3-(diethylaminoethyl)barbituric acid (III),  $\text{C}_{18}\text{H}_{20}\text{N}_4$ , from I and the Na salt of ethylphenylbarbituric acid, HCl deriv white, cryst, easily sol in  $\text{H}_2\text{O}$ , difficultly sol in EtOH, m 245-6°, III, injected intravenously into cats in an amt of 50 mg/kg of body wt of the animal, causes rapid decrease of the blood pressure to 0 mm Hg and arrests respiration, causing death of the animals. After a dose of 25 mg/kg of body wt the blood pressure sinks to 8 mm Hg and respiration becomes extremely slow. Within several min the cats return slowly to their normal state. Analgesia in cats is not observed. Peroral doses in cats and dogs have but slight narcotic effects. Subcutaneous doses of 50 mg. in cats cause death in 24 hrs., in dogs only a dose of 80 mg/kg shows considerable toxic effects. Prepn II is much less toxic. Fifty mg/kg causes a slight decrease of the blood pressure, followed by strong hypertension.

J. W

*Juniperus thurifera* L. and its essence (Revol) 17

1 Chem and physicochem analysis of the mineral waters of Valleverde [therapeutic value] (Marotta, Sorrentino) 14.

## I-ZOOLOGY

R. A. GORTNER

**Effect of temperature on the glutathione of cold blooded animals.** Léon Binet and Georges Weller. *Compt rend soc. biol* 120, 289-90 (1935); cf. C. A. 29, 6933<sup>1</sup>.—Three lots of frogs (*R. temporaria*) were kept 2 hrs at 0°, 20° and 32°, resp., then killed. The reduced glutathione contents of the livers of the 3 groups were 44, 46 and 29 mg. %, resp. No difference in muscle glutathione was found. Three lots of goldfish were kept 5 hrs at 8-10°, 18-20° and 28-30°. The reduced glutathione was found to be 10.5, 15.4 and 12.3, and total glutathione 18.2, 10.8 and 14.5 mg. % (whole fish) for the 3 lots in the order given.

L. L. Gilson

**Colloid-osmotic pressure of blood of marine fish.** Paul Meyer. *Compt rend soc. biol* 120, 303-5 (1935).—The method of Krogh and Meyer was used (*Ergebnisse Physiol* 34, 15-111 (1932)). For selachians, 4 species, the range was 3.1-6.4, and for teleostean, 5 species, 14.6-25.0 cm water. Colloid-osmotic pressure of the "blood" of marine invertebrates. *Ibid* 305-7.—Values are given in cm of water. *Sipunculus nudus* 0.7-1.2, *Pinnia nobilis* 0.8-1.2, *Murex* species 1.2-1.9, *Septa officinalis* 2.8-3.6, *Ostrea vulgaris* 3.1-3.8, 4 species decapod crustaceans 2.1-4.4 and 2 species of tunicates 1.2-2.3.

L. E. Gilson

**Ammonia contents of the internal liquid media of various invertebrates.** Mme. J. Souteret. *Compt. rend. soc. biol* 120, 453-5 (1935).—The method is described. The following values in mg. % of ammonia N were obtained: starfish 0.104-0.350, sea urchin 0.096-0.210, *Sipunculus nudus* blood 0.1-0.5, crab blood 0.3-1.4, hemolymph of various marine shellfish 0.07-0.22, *Helix pomatia* hemolymph 0.87-0.95, *Septa officinalis* blood 0.43-3.85, urine 82.3-138.9 and digestive fluid 6.8-13.9.

L. E. Gilson

**Distribution of glutathione in organs of various marine invertebrates.** A. Monier. *Compt rend soc. biol* 120, 456-8 (1935).—In echinoderms, worms, crustaceans and mollusks the hepatopancreas is richest in glutathione, contg 90-340 mg. % total glutathione. The genital organs contained 65-270, muscles 35-85 and blood 3-12 mg. %. The largest quantities were found in *Septa officinalis*. In every case nearly all the glutathione was in the reduced form.

L. E. Gilson

**Effects of removal of the mediotergal organ on the respiration of Lepidopterae (adult moths).** Anne Raffy and Gabriel Guignon. *Compt. rend soc. biol* 120, 785-6 (1935).—O consumption was greatly reduced. Nicotine caused a much smaller increase in O consumption than it did in normal moths.

L. E. Gilson

**Regulation of respiration in the flea, *Xenopsylla cheopis*, Roths (Pulicidae).** V. B. Wigglesworth. *Proc. Roy Soc (London)* B118, 397-419 (1935).—In decreased concns of O, the frequency of opening and closing of the spiracles is increased, and the closed period is shortened more than the open period. Increased concns of O prolong both periods. The spiracles remain permanently open in concns of O less than 1%, or in concns of CO<sub>2</sub> approx 2%. Low concns of CO<sub>2</sub> slightly shorten the closed period and considerably increase the open period. Introduction of lactic acid into the blood produces a rapid rhythm-like O want or permanent opening of the spiracles. The opening of the spiracles is governed chiefly by O want, the duration of the open period by the time required for diffusion away of the CO<sub>2</sub>. Reduced concn of O gives rise, in sequence, to accumulation of metabolites in the blood, increase in its osmotic pressure, extrn of H<sub>2</sub>O from the ends of the tracheoles and an increase in respiratory surface.

Joseph S. Hieburn

**The carotenoids of *Rana esculenta*.** Otto Brunner and Rosa Stein. *Biochem Z* 282, 47-50 (1935).—The liver, skin and fat body contain  $\beta$ -carotene and lutein, the latter only in the form of its esters. In the ovaries, how-

ever, lutein is present both in the free and esterified condition. The pigment content of these 4 organs has been detd. in 6 frogs. The abs amts of  $\beta$ -carotene and lutein vary greatly as does also the lutein/carotene ratio

S Morgulis

The problem of the formation of wax in the bee organism. Georg Buchner. *Fettsäure Umschau* 42, 208-9 (1935).—A brief theoretical discussion of biol syntheses of fats, fat acids and waxes from hexoses and of wax alcs from fat acids

J W Perry

Increased permeability to water of aging unfertilized eggs (*Arbacia punctulata*). A J Goldfarb. *J Gen Physiol.* 19, 149-65 (1935).—In did sea water at const temp,  $p_H$  and time, these eggs swell progressively faster with age. Swelling results from the permeability of the eggs to water. Methods are described

C H R

The chemical and energy metabolism in the course of development of insects II. The ratio of heat production to the respiratory processes in the course of postembryonic

development of insects (*Lymantria dispar* L. and *Bombyx mori* L.). N. Balzani. *Acta Biol. Exptl.* 8, 59-72 (1933) (German summary); cf. C. A. 27, 5827.—The caterpillars of *Lymantria dispar* L. and *Bombyx mori* L. show high evolution of heat during growth at comparatively low respiration dropping to a lower value during molting. In the pupal stage the heat evolved is half of that evolved in the larval stage, the lowest value being at the mid-point of the pupal stage

J Wiertelak

Duration of the acid reaction in the digestive vacuoles of *Paramecium caudatum* as a function of the  $p_H$  of the external medium. M. Chejfec. *Acta Biol. Exptl.* 8, 186-95 (1937) (in German) (1933).—The duration of an acid reaction ( $p_H$  1.6-2.0) in the digestive vacuoles is but slightly dependent on the  $p_H$  of the external medium. In the same individual no synchronization of  $p_H$  in different vacuoles is observed. The death of the individual produces instantaneous deacidifying of all vacuoles.

J. Wiertelak

## 12—FOODS

F C BLANCK AND H A LEPPER

Foodstuffs and drinks used in Brazil. A de Paula Rodrigues. *Rev quim farm* (Brazil) 1, 51 8 (1935).—A review

E S G B

Certified colors for foods. J R Hall. *Food Ind* 8, 24-5, 52 (1936). cf. C. A. 30, 165.—The uses and properties of the primary food colors and the conditions under which each will remain stable are described

C R F

Determination of salt in foodstuffs. J D Filippo. *Chem Weekblad* 32, 558 (1935).—A previous method (cf. C. A. 9, 3303) for detg. NaCl in bread by means of  $HNO_3$  was found suitable for cheese, coffee, sausage, butter and mustard. To 5 g substance in a 50-cc volumetric flask are added 35 cc of 1 to 1.5 N  $HNO_3$ , and the mixt is shaken for some time. After 5 to 10 min the flask is filled up and the contents are filtered. Cl is detd in 20 cc of filtrate by the Volhard method. Dark filtrates are cleared with  $H_2O_2$  and heating

B J C v d H

Cyanogenetic glucosides in food products. Ulas Hordh. *Anal. Assoc. quim Argentina* 23, 67 86 (1935).—Cyanogenetic glucosides (I), encountered in a no. of foods and fodders, are very toxic, and strict regulations as to permissible content of HCN allowed must be enforced, or the sale of such products prohibited if products free from I can be obtained. Plants, foods and fodders which contain I are reviewed, with data on the content of I, including oil of bitter almonds, and com substitutes. Regulations in force as to permissible contents are given, with special reference to certain prohibited materials. Methods of analysis used in the Argentine Government lab are reviewed

E M Symmes

Protein studies [on wheat and barley]. W F Geddes. Can. Dept. Trade & Commerce, Dominion Grain Research Lab., *8th Ann. Rept.* 1934, 6-15.—Data are given on the protein contents of the 1934 western Canada hard red spring wheat crop, the 1933 and 1934 western Canada Amber Durum wheat crops, the 1934 western Canada barley crop and export cargoes of contract grades of hard red spring wheat

K D Jacob

Milling and baking studies. W F. Geddes. Can. Dept. Trade & Commerce, Dominion Grain Research Lab., *8th Ann. Rept.* 1934, 15-31.—Data are given on the milling and baking characteristics of the 1934 western Canada hard red spring wheat crop and on the comparative quality of cargoes of corresponding grades of hard red spring wheat ex Atlantic and Pacific ports. The carotene contents of the flour were 1.72-2.23 p p m and increased with decrease in the grade of flour. As indicated by  $H_2O$  absorption, loaf vol., grain and texture there were no significant differences in the baking strengths of the 1st 5 or statutory grades of flour. However, the com. grades gave higher absorption, lower loaf vol., inferior crumb color and grain and texture. The protein contents of normal and weathered wheats from the Peace River

district were practically equal and weathering had no significant effect on the diastatic activity and baking strength of the flour

K D Jacob

The physical chemical characteristics of flour. F. DeRege. *Giorn. risicoltura* 25, 10-18, 132-42, 174-80, 204-13 (1935).—The "pneumodynamometer" is used for testing semolina and flour for absorption, cohesion, consistency, etc. The theoretical parts relate, e g., to the influence of temp. upon the phys. properties of doughs. Tables are given showing the amt. of water necessary for doughing 100 g semolina and the logarithm of the "doughing constant". The dough used in the tests is prepd. by adding to the semolina such an amt. of 3% NaCl soln. as will make a dough contg. 42.5 parts of  $H_2O$  (the flour is assumed to contain 14% moisture).

J A Le Clerc

Moisture determination in flour by means of dielectric constant. P. Cohen Henriquez and A. W. Renaud. *Chem. Weekblad* 32, 526 (1935).—In order to obtain good results in the direct a. detn. with flour a const. packing condition must be used with 1 cc. powder weighing at least 0.8 g. The resulting accuracy is 0.1%  $H_2O$ . The dioxane method is unsatisfactory for flour

B. J. C. v. d. H.

Detection of rice in wheat flour. Riccardo Tuffi and Elena Borghetti. *Ann. chim. applicata* 25, 351-4 (1935).—Admixts. of rice in wheat flour can be detected by dyeing with equal parts of 0.2% methylene blue and 0.1% eosin A soln. and exang. with a microscope. Rice flour is dyed a greenish blue with groups of violet-tinted granules, whereas, the wheat flour remains colorless, or is dyed a pale blue, while the accompanying proteins are dyed violet. Admix. of 1.0% rice flour can be detected by this method

A. W. Conti

Animal experimental investigation of the action of flour-treating agents II. Effect on the vitamin of flour and on the animal organism when bread is fed exclusively. István Vitéz. *Z. Untersuch. Lebensm.* 70, 258-65 (1935), cf. C. A. 29, 1890, 3738.—The results obtained in these expts confirm those previously obtained. The increase in wt. due to vitamin addn. was highest with Na perborate. This large increase, as well as the considerable retardation in development which had occurred during the feeding expts in the same group, permits the conclusion that the Na perborate injures the vitamin of the flour more than the other treating agents. Therefore, vitamin A was lacking first in the organism of the rats belonging to this group and the animals reacted to vitamin addn. with a large increase in wt. The low wt. value obtained with  $NH_4$  persulfate may possibly be explained by the fact that it liberates O more quickly than Na perborate, perhaps through the influence of the moisture of the flour, so that the flour remains in contact with the O for a shorter time, whereby the vitamin is exposed to the destructive influence in a lower degree. Although these treating agents destroy



the vitamin of the flour, that does not mean harm to man with a satisfactorily balanced diet, which supplies the vitamin requirements of the body. However, even under these circumstances, they lower the food value of the flour, not in the sense of calories, but with respect to the vitamin value, by diminishing its availability which is no longer sufficient, in the case of a one-sided diet, so that severe harm may be done. The ash content permits conclusions as to the metabolism of the bones. III. Effect of oxidizing flour treating agents on the vitamin of flour. *Ibid* 265-9.—Na perborate and benzoyl peroxide damage one vitamin of the flour to a high degree. The injurious action of the treating agents is not compensated for by special doses of vitamin B, but by vitamin A. The compensating action of vitamin A permits the conclusion that the treating agents tested destroy the vitamin A of the flour, that is, its carotene. F. L. Dunlap

Determination of sugars in flour and bread. Angela Vercillo *Ann chim applicata* 25, 379-84 (1935).—Most procedures for detg. sugar in flour or bread call for extrn. with water without making any provision for inhibiting diastase action during the extrn., which consumes some of the sugar present. By extrn. in the presence of basic Zn or Pb acetate, which is then eliminated by addn. of  $H_2SO_4$ , and then detg. the sugars, after clarifying the soln. with  $ZnFe(CN)_6$  (made by the interaction of  $K_4Fe(CN)_6$  and  $Zn(AcO)_2$ ), the above objection is eliminated. A. W. Contieri

The physicochemical properties of bread doughs. Giovanni Insoglio *Ann chim applicata* 25, 274-92 (1935), cf. C A 28, 1782a.—A tension hysteresometer is described for measuring the hysteresis of bread dough after tension has been applied. It gives a measure of the viscosity, modulus of elasticity and hysteresis of the dough. A. W. Contieri

Milk and milk products at Warsaw (Poland). Zygmunt Leyko *Wladomski Farm* 62, 503-6, 520-2, 531-4, 545-52 (550-2 in German) (1935).—The work of controlling milk and milk products in Warsaw is described. Of 28,812 samples exam'd 7.83% were adulterated and 1% contaminated or spoiled. By examg. the f. p. of the milk even less than 5% of added water can be detected, provided  $Na_2CO_3$  has been used to neutralize natural acids. However, since both ingredients are usually added the cryoscopic method does not suffice. In such a case fractionation of the serum is very helpful. Adulterants such as flour are easily recognized in cream and butter with chemicals or under the quartz lamp. Good butter shows, under the lamp, a canary-yellow color, which is changed by surface oxidation into yellow-gray. It is, therefore, necessary to exam. samples taken from the interior of the lot, to obtain the pure yellow color given by fresh butter. Rancid butter is adulterated by neutralizing with soda, washing, and flavoring with butyacet. J. Wiertelak

Inspection of milk at point of production. Jorge de la Earp *Rev. Dept. mod. produccion animal* 1, 177-98 (1934).—An outline of known methods. O. W. W.

Quantity and quality of milk from grade cows. M. H. French *Tanganyika Territory, Ann. Rept. Dept. Vet. Sci.* 1934, 73-8.—Data are given on the contents of fat and solids-not-fat in the milk of various breeds of cows in Tanganyika. K. D. Jacob

The influence of streptococcal infection of the udder on the flavor, chloride content and bacteriological quality of the milk produced. C. S. Bryan and G. M. Trout *J. Dairy Sci.* 18, 777-92 (1935).—The av. percentages of Cl in 2 streptococcus infected herds were 0.192 and 0.2263, resp., while noninfected samples from the same herds averaged 0.1374. Most samples of mastitis infected milk tasted salty. Streptococcus infection seemed to affect the quality of milk from noninfected quarters of the same cow. Philip D. Adams

Flavors of milk and their control. C. L. Roadhouse and J. L. Henderson *Calif. Agr. Expt. Sta., Bull.* No. 595, 5-30 (1935).—Full rations of green or dry alfalfa hay, or corn silage fed 1-2 hrs. before milking produced strong, undesirable feed odors in milk. If these feeds were withheld during the 5 hr. interval before milking, no

objectionable flavor was observed. In general, feed flavors were strongest in the milk about 45 min. after feeding. Improperly cured hay having a musty odor transmitted a musty flavor to the milk. Wheat bran seemed to improve the flavor of milk. Salty taste was observed in milk from cows late in lactation and also from 1 or more quarters previously affected with mastitis. Rancid milk was produced by certain cows that had been milking for longer than the usual lactation period. Lipase in the milk produces this defect. Lipase is normally present in the blood of cows but the conditions governing its presence in milk are not known. Pasteurization destroys lipase in milk. Milk which has been in contact with certain corrodible metals may develop oxidized flavors. Cu and its alloys are the most common causes of oxidized milk. Exposure of milk to sunlight may produce tallowy flavors. C. R. Fellers

Determination of the freezing point of milk. C. A. Koppejan *Chem. Weekblad* 32, 657 (1935).—The newer method of f. p. detn. of Gangl and Geschke (C A 29, 1891a) is much faster than that of Dekhuizen (C A 8, 1624, 2007, 3602) and is also very reliable. If the milk is kept at melting ice temp. for some time, there will be no noticeable difference in the f. p. of the milk whether it has previously been skimmed or not. N. Bekkedahl

The addition of mineral salts and vitamins to milk. Ir. P. Schoorl *Nederland. Tijdschr. Geneeskunde* 79, IV, 4887-91 (1935).—Mineral deficiency of cow milk is remedied by the addn. of salts to the milk rather than by the addn. of the salts to the diet of the cow. The iron and copper contents of the milk cannot be influenced by the diet. Vitamin A and D deficiency can be improved by selecting foods rich in these vitamins for the cow. To increase the vitamin B content the addn. of this vitamin to the milk is recommended. R. Beutner

Relation between the vitamin A content of the dairy ration and that of milk. Walter C. Russell, M. W. Taylor, D. F. Chichester and Logan T. Wilson *N. J. Agr. Expt. Sta., Bull.* 592, 3-11 (1935).—High-grade field-cured alfalfa, or machine-dried alfalfa, and corn silage contributed 98% of the vitamin A value of a dairy ration in which, in addn. to these constituents, there were beet pulp and a grain mixt. contg. yellow corn and corn gluten. Corn silage supplied  $1/2$  to  $1/3$  of the vitamin A content of the ration and therefore it can be classed as an important source of vitamin A in the dairy ration. When the daily vitamin A intake was of the order of 900,000 to 1,200,000 U. S. P. X 1934 units, the vitamin A potency of the milk from mixed breeds was of the order of 2500 units per qt. In another instance the feeding of 850,000 units produced a milk of 1600 units potency. In no instance did the output of vitamin A in the milk exceed 3.5% of the intake in the ration. The percentage of the factor which appears in the milk decreases as the amt. of the factor in the ration is increased and the increase in vitamin A content of the milk is not proportional to the increased consumption. C. R. Fellers

Study of the lecithin content of milk and its products. B. E. Horrall *Ind. (Purdue) Agr. Expt. Sta., Bull.* 401, 31 pp. (1935).—The Mojonnier modification of the Rose-Gottlieb method was used to ext. the org. P along with the fat from dairy products. The colorimetric method of Deniges modified by Truog and Meyer (C A 23, 4163) was found to be an accurate method for the detn. of org. P in dairy products. The lecithin content was calc'd from the P obtained by multiplying it by 25.94, which is the factor for the oleyl-stearyl type of lecithin. The lecithin contents of the milk from 3 dairy cows show that the fat contains a fairly const. percentage of lecithin after the fourth day of the lactation period. The colostrum milk fat contained a higher percentage of lecithin than did the fat of the milk later. The lecithin content increased after the calf was dropped, then decreased rapidly until the amt. was fairly const. Factory milk contained a higher percentage of lecithin in the fat than did that of the fat from milk coming from normal individual cows. The av. increase was 0.21%. Udder infections (mastitis) caused an increase in the percentage of lecithin in the fat

of the milk when compared to that coming from normal quarters of the cow. The lecithin content of skim milk was 13.91% of the fat. Raw sweet cream contained 0.428% lecithin in the fat while raw sour cream contained 0.422%. Pasteurized sweet cream butter contained an av. percentage of 0.232 lecithin in the fat while that of pasteurized neutralized sour cream contained 0.170%. Buttermilk from pasteurized sweet cream contained 19.66% lecithin in the fat while that of pasteurized, neutralized raw cream averaged 17.88. The percentage of lecithin in the fat of separator slime averaged 12.38. The results of a trial show that the lecithin content decreased materially in butter from sour cream while that of sweet-cream butter remained practically the same over a storage period of 24 days. The detn. of the lecithin content in hen eggs shows that the fat contains on the av. 26.64% lecithin and is fairly const. A method is given for the detn. of the amt. of eggs in ice-cream mix. Fifty-eight references. C. R. Fellers

Identification of vegetable lecithins as substitutes for eggs in alimentary pastes. Domenico Costa. *Ann. chim. applicata* 25, 355-79 (1935).—A detn. of the  $I_1$  value of the lecithins in alimentary pastes by itself is not enough to distinguish whether eggs or vegetable lecithins have been used, since it is possible to dissolve the lecithins in such fats that the  $I_1$  value is the same as if eggs had been used. However, the vegetable lecithins commercially known as "Plantins" can be identified by (1) the deficiency of cholesterol in proportion to the lecithin-phosphoric acid contents, and (2) by the absence of lecithin-phosphoric acid combined with albumin. A. W. C.

Substances adsorbed on the fat globules in cream and their relation to churning. IV. Factors influencing the composition of the adsorption "membrane." Charles E. Rimpla and L. S. Palmer. *J. Dairy Sci.* 18, 827-39 (1935); cf. C. A. 28, 1785.—The percentages of protein and phospholipides in the fat globule "membrane" are not const. for various samples of cream but are essentially const. for any given sample after the fourth through at least the tenth washing. The "membrane" found on butter-fat globules emulsified in sweet rennet-whey, skim milk or buttermilk differs from the natural "membrane" in its percentage and proportion of protein and phospholipides, and the Van Slyke N distributions of the "membrane" proteins are not the same. A large part of the phosphatase activity of natural cream is in the "membrane" material and is not removed by water washing. In the synthesis of milk the "membrane" is not derived from the milk plasma. The "membrane" proteins of natural cream and of artificial creams appear to contain a prosthetic group, so far not identified, which causes the N content to be abnormally low as compared with other known proteins of similar complexity. Philip D. Adams

Preparation of a nondesiccated sodium caseinate sol and its use in ice cream. E. W. Burd, H. W. Sadler and C. A. Iverson. *Ida. Agr. Expt. Sta., Research Bull.* 187, 179-208 (1935).—The body and texture of ice cream are improved by the replacement of dry skim milk by Na caseinate sols. This improvement was shown up to 2.5 to 5.0% replacement, depending on the compn. of the mix. The flavor of ice cream was progressively improved by the replacement of dry skim milk by Na caseinate sols up to 3 to 4% replacement, depending on the compn. of the mix. This flavor improvement was due to the careful pH control used in the prepn. of the Na caseinate sols. The type of melting of the ice cream was altered by the replacement of dry skim milk by Na caseinate sols. The use of Na caseinate sols increased the initial and max. overrun and decreased the whipping time of the ice creams produced. From 1.5 to 3.0% replacement of dry skim milk by the Na caseinate sols is necessary to effect sufficient improvement in whip to warrant their use. A 3% replacement would be necessary with a mix contg. 14% fat and 10% serum solids. The use of Na caseinate preps as adul. solids, i. e., in addition to the amts. of serum solids (8 to 10%) commonly used by the trade, has been suggested. The amts. of milk protein that would be required to yield sufficient improvement in whip and in body and

texture score would be large enough to make their use questionable. C. R. Fellers

Butter, fats and fatty oils. H. M. Langton. *Food* 5, 147-8 (1935).—A very brief review of recently published Brit. standard specifications, U. S. specifications for olive oil at present under consideration and recently published work on I and thiocyanogen values of butter fat, and  $Ac_1$  in butter cultures. A. Papineau-Couture

Butter, its classification on a scale of points. Beatriz G. de sa Earp. *Rev. dept. nacl. producao animal*, 1, 199-208 (1934).—A discussion of organoleptic tests. O. W. Wilcox

A new index for butter. Renato Lucentini and Elsa Drago. *Ann. chim. applicata* 25, 388-91 (1935).—A new test is proposed to replace the Reichert-Meissl test. The acids sol. in a satd. soln. of  $K_2SO_4$  are detd. in terms of cc. of 0.1 N NaOH. The test is carried out as follows: 5 g. of melted and filtered butter is saponified by heating with 2 cc. of 50% KOH soln. (carbonate free) and 20 cc. of pure glycerol for 10 min. The whole is transferred to a segg funnel, 12 cc. of  $H_2SO_4$  (conc.) added, and the fats are extrd. with  $Et_2O$ . The  $Et_2O$  is distd. off, and 100 cc. of a satd. soln. of  $K_2SO_4$  added to the residual fat. The soln. is heated for a few min., then filtered and the filter washed with the  $K_2SO_4$  soln. above. The soln. is then titrated hot with 0.1 N NaOH, with phenolphthalein as indicator. Genuine butters give acid nos. of 18 or over (18.6-20.9 reported), while suet, cacao butter and hydrogenated cacao butter, common adulterants, have values of about 3, 5 and 4, resp. A. W. Conteri

Study of the various standards adopted for the examination of Indian butter and ghee. S. D. Sunawala. *Agr. Live-stock India* 5, 480-8 (1935).—The recommended standards are  $H_2O$  content less than 0.7% (with ghee only), butyrefractometer reading 42.0-42.5 at 40°, Reichert-Meissl value not less than 20, Polenske value not more than 2.5, Kirschner value not less than 16, sapon. value not less than 218, I value 30-38 and free fat acids less than 1%. K. D. Jacob

The vitamin A activity of butter produced by cows fed alfalfa hay and soybean hay cut in different stages of maturity. J. H. Hilton, S. M. Hauge and J. W. Wilbur. *J. Dairy Sci.* 18, 795-800 (1935).—Artificially dried hay is superior to the corresponding field-cured hay in vitamin A value. Hay from young plants has greater vitamin A value than hay from older plants. Alfalfa plants have higher vitamin A value than soybean plants. Cows fed artificially dried young alfalfa hay produced butter having 45 vitamin A units per g. Philip D. Adams

Reduction of acetylmethylcarbinol and biacetyl to 2,3-butyleneglycol by the citric acid fermenting streptococci of butter cultures. B. W. Hammer, G. L. Stahly, C. H. Werlman and M. B. Michaelian. *Ida. Agr. Expt. Sta., Research Bull.* 191, 381-407 (1935).—When acetylmethylcarbinol or biacetyl was added to a tomato-bouillon culture of 1 of the citric acid-fermenting streptococci normally present in butter cultures, there was a rapid disappearance of the added reagent and an increase in 2,3-butyleneglycol. The amt. of the glycol produced accounted, in a general way, for the acetylmethylcarbinol or biacetyl that disappeared. The added reagent did not usually disappear completely. In trials with acetylmethylcarbinol the change of the carbinol to the glycol was delayed when  $H_2SO_4$  was added in amts. to yield a pH of 3.8-4.0. There was also a change of acetylmethylcarbinol or biacetyl to 2,3-butyleneglycol when 1 of these reagents was added to a milk culture of 1 of the organisms. When various amts. of  $H_2SO_4$  were added to milk cultures of the organisms, acetylmethylcarbinol was not produced at the higher pH values but was produced at the lower values, while 2,3-butyleneglycol was produced at both the higher and lower pH values. There was less of the glycol formed at the lower pH values than at the higher ones. The addn. of 0.65% citric acid to a milk culture of 1 of the organisms resulted in an increase in both acetylmethylcarbinol and 2,3 butyleneglycol. The reduction of acetylmethylcarbinol, which had been added to a milk

culture of 1 of the organisms, to 2,3-butylene glycol was not delayed by  $\text{KNO}_3$  but was delayed by the largest amt of  $\text{H}_2\text{O}_2$  employed. The increased production of acetyl methylcarbinol is accounted for by a decrease in the reduction of the carbinol to the corresponding glycol, rather than to an aldehyde condensation involving, in part, the added aldehyde. In butter cultures the decrease in acetyl methylcarbinol was accompanied by an increase in 2,3 butylene glycol, and there was commonly an increase in the total molalities of the 2 compts. When ripened butter cultures were neutralized to a low acidity there was a rapid decrease in the acetyl methylcarbinol, and in some of the trials this was followed by an increase.  $\text{H}_2\text{O}_2$ , Na fumarate and ice-water temps. delayed the reduction in either neutralized or unneutralized cultures, but the reduction was more rapid with neutralization than without.

C. R. Fellers  
Rapid casein determination in buttermilk porridge. F. Th. van Voorst. *Chem. Weekblad* 32, 478-9 (1935).—The method of Kauffmann-Havinga (*Höndelingen Genootschap Melkkunde* 1931) was modified for more rapid work. To 50 g. porridge, weighed in a porcelain dish, 25 cc.  $\text{H}_2\text{O}$  is added, 10 cc. NaOH and a few drops of phenolphthalein; the mixt. is heated to  $40^\circ$  on a water bath, cooled and again heated to  $40^\circ$ . After cooling the mixt. is washed with about 250 cc. 1% Na oxalate soln into a 500-cc. volumetric flask, left for 1 hr. after repeated shaking and filled up with Na oxalate soln. The next day 10 cc. is pipetted into a 100-cc. Kjeldahl flask, heated with 2 cc. 96%  $\text{H}_2\text{SO}_4$  until all  $\text{H}_2\text{O}$  has evaporated and then cooled, 1 cc. 30%  $\text{H}_2\text{O}_2$  added and heated until the soln. becomes colorless, and the vol. reduced to half. The contents are then diluted with  $\text{H}_2\text{O}$ , transferred to a 300-cc. Erlenmeyer flask, neutralized, first with 4 N NaOH, then with 0.1 N NaOH to exact neutrality to methyl red. Ten cc. neutralized (to phenolphthalein) formaldehyde is added and the mixt. titrated with 0.1 N NaOH to phenolphthalein. The titration figure, 2-3.5 cc. usually, multiplied by 0.715, gives the casein percentage. B. J. C. v. d. H.

The solubility-freezing point relationships of water solutions saturated with respect to sucrose and dextrose in relation to the storage of sherbet and water ice. Alan Leighton and Abraham Leviton. *J. Dairy Sci.* 18, 501-3 (1935).—The ternary eutectic temp. of the system sucrose-dextrose-water is  $-17.9^\circ$  and the proportion of sucrose to sucrose is 1 to 4.11. Philip D. Adams.

Chemical composition of duck eggs. A. K. Danilova and V. A. Nefedova. *Biedermeier Zentr. B. Tierernähr.* 7, 532-42 (1935).—A study has been made of the eggs of 2 kinds of ducks. Peking ducks and Runners. The egg wt. as a whole and the wts. of the single constituents (egg white, egg yolk and shell) changed during the laying period. The highest values were found in the eggs of the Peking duck in May, those of the Runner in June. Probably this is related to the cycle of increasing egg production. The wt. of the egg white and egg yolk of the eggs from Peking ducks changed regularly in relation to egg production, eggs of ducks with a high production contained more egg white and yolk. Such a relationship between the constituents of the eggs of the high- and medium-producing Runner was less apparent. The chem. compn. of the eggs changed during the producing period. The  $\text{H}_2\text{O}$  content in the egg white and yolk of the eggs of the Runner increased during the summer months. The protein content of the egg white increased in June and decreased in July. The ash content varied little. Protein content and ash of the egg yolk decreased in the summer months. The fat content of the egg yolk is highest in June. The contents of  $\text{H}_2\text{O}$  and fat in the eggs of Peking ducks are highest in the summer months. The influence of egg production on the change in chem. compn. of the eggs of Peking ducks is without importance. As to the Runner, the nutritive value of the egg yolk increases with increasing production. The egg white of small eggs has a smaller nutritive value. The content of  $\text{H}_2\text{O}$  is higher, while the content of protein and ash is smaller. The nutritive value of the egg yolk is higher; the  $\text{H}_2\text{O}$  content is smaller and that of the fat is higher. The shrinkage in wt. on keeping the eggs for 2

months was 19% in the case of the Runner and 12% in that of the Peking duck eggs. F. L. Dunlap.

Recent meat researches. Arthur W. Ewell. *Refrigerating Eng.* 30, 367-8 (1935).—Growth of bacteria common to meat surfaces ceased at humidities below 92% but the bacteria grew with great rapidity at the highest attainable humidity, viz., 99%. There was some evidence that with continued exposure to ozone mold became acclimated to it and growth was to a smaller degree checked. Time of storage can, however, be very appreciably increased by the use of ozone because of decrease in the spread of infection rather than on account of destruction of microorganisms. A. H. Johnson.

Lead determination in preserved meat. N. V. Shurokov and D. S. Mundina. *Z. Unters. Lebensm.* 70, 245-51 (1937).—A colorimetric sulfide method is described in which it is unnecessary to remove other metals which may be present. By this method, it is possible to detect 0.25 mg. and to det. 0.5 mg. of Pb in 1 kg. of preserved meat. The method is simple and an analysis can be finished in 2-2½ hrs. Thirty-eight references.

F. L. Dunlap  
Starch determination in sausage. N. V. Shurokov and M. K. Milovidova. *Z. Unters. Lebensm.* 70, 251-5 (1935).—The method of direct inversion gives satisfactory results. The method is not troublesome and results can be obtained in about 2 hrs. P. L. Dunlap.

Effect of method of manufacture on the composition of haddock fish meal proteins. S. R. Pottinger, Roger W. Harrison and Andrew W. Anderson. U. S. Bur. Fisheries. *Investigational Rept.* 31, 1-14 (1935).—There are 3 general conditions encountered in the manuf. of nonoil fish meal which have an effect on the compn. of their proteins. These are (1) difference in the proportion of head and backbone wastes, (2) the removal of water-sol protein material by the wet process and (3) method of drying. The backbone portion of haddock-fillet waste has a larger percentage of protein than the head portion of the waste, and the proteins of the backbone material contain higher percentages of tyrosine and tryptophan. Thus variations in the proportion of head to backbone material will not only give meals of different protein contents but proteins of different amino acid compns. Meals contg. the greater proportion of backbone waste will have a higher protein content and, because of the higher tyrosine and tryptophan percentages in the protein, should have higher biol. values. The water-sol proteins removed by the wet process are relatively devoid of tyrosine, tryptophan and cystine. The wet process yields meals of lower protein content, but the protein of such meals has a slightly higher percentage of the 3 essential amino acids being considered. The proteins of wet-process meals might be expected to show slightly higher biol. values than the proteins of dry-process meals. Drying causes a diminution of cystine if the meals are heated much over  $38^\circ$ . Tryptophan apparently is affected by drying temp. also, but to a smaller extent. Tyrosine is probably not affected at all, unless extreme temp. conditions are encountered. Because of the apparent effect on cystine and tryptophan, fish meal proteins would be expected to be of slightly lower quality than the original protein in the raw material. The relative diminutions of the 3 amino acids caused by the various methods of drying, however, indicate that appreciable difference in protein quality should not be expected unless rather extreme conditions of drying are encountered. C. R. Fellers.

Studies on drying cod and haddock waste. Andrew W. Anderson, Roger W. Harrison and S. R. Pottinger. U. S. Bur. Fisheries. *Investigational Rept.* 32, 3-41 (1935).—In the manuf. of ground-fish meal difficulty is encountered with material sticking to, and baking on, the walls of the drier. Steam pressure and vacuum cause variation in the amt. of cake formed. High steam pressures and high vacuum, both conducive to high rates of heat transfer, produce the greatest amt. of cake. Cut raw waste produces thinner cake than uncut raw waste. A charge of cut waste can be dried to a 9% moisture content more rapidly than a similar amt. of uncut material. The 5 gal. agitator produces better agitation than the scraper type

but the more complicated construction does not permit as great a charge. A spiral agitator with adjustable scraper edges probably would be more efficient. Very little cake forms with wet-process methods and none becomes detached. Raw haddock waste averages 77% moisture. Com. centrifuge cake and lab centrifuge cake average about 64% moisture. Equipment, operating and fixed costs are greater for dry-reduction plants than for wet-reduction plants of similar capacity. Dry-process methods yield 25% meal while wet-process methods yield only 20% meal from a given amt. of raw material. Providing 1500 tons or more of raw waste is utilized during the season, the 25% greater meal yield by the dry process more than counteracts the greater production cost. This advantage increases with increase in amt. of waste utilized and price of meal. Two-ton reduction plants offer the max. opportunity for profit per ton of raw waste processed, but 1-ton plants show a greater advantage for the dry process. C. R. Fellers

Comparative study of commercial pectins. G. J. van der Die. *Chem. Weekblad* 32, 557-8 (1935).—Several com. samples of citrus and apple pectins were exam'd for ash content (1.75-11.65%), furfural phloroglucide yield (Tollens, 19.5-37.1%), MeOH content (Fellenberg, 3.1-10.8%), Ca pectate yield (Harricotte, 47.9-92.4%), CO<sub>2</sub> evolution (Lefevre, 10.9-19.0%), gelling power (Tarr-Baker), viscosity of aq. 0.75% soln. at 27° (1.71-6.59),  $\eta_{sp}$  of this soln. (2.8-4.2) and acidity (0.5-1.3 cc. 0.1 N alkali). The presence of impurities in practically all samples was evident. The MeOH-CO<sub>2</sub> ratio for completely esterified carboxyl groups should be 0.727:1, it was from 0.703 to 0.233:1. The Ca pectate to CO<sub>2</sub> ratio, theoretically 4.4:1, was between 4.39 and 5.07:1. One pectin with abnormally high viscosity and poor gelling power had been acid-treated. B. J. C. v. d. H.

The cool storage of plums. First progress report. G. B. Tindale, S. A. Trout and F. H. Heulin. *J. Dept. Agr. Victoria* 33, 552-4 (1935).—As compared with the controls, the storage life of plums was reduced by 1 week in the presence of 5% CO<sub>2</sub> and by 2 weeks in the presence of 10% CO<sub>2</sub>. Serious browning of the flesh occurred in the presence of 15% CO<sub>2</sub>. K. D. Jacob

Concentrated grape juices from California. Ernst Vogt. *Z. Untersuch. Lebensm.* 70, 307-8 (1935).—Analytical data are given for 3 samples, which are designated as Riesling, Muscatel and Burgundy. F. L. Dunlap. Black-current flavor. H. Stanley. *Edgemoor Am. Perfumer* 31, 94-5 (1935).—The characteristic flavor of the black current, as also of the leaf, is either attractive or repellent, according to the taste of the resp. individual. In France the sirup known as "cassis" is made up by exg. the flavoring material from black currents with alc. & dilg. the ext. with more alc. and mixing with sugar and H<sub>2</sub>O. Sometimes flavoring materials derived from other fruits are added, or the flavor may be modified by the addn. of cloves and cinnamon. As a cold drink the sirup is used in admixt. with ice water, either alone or with the addn. of white wine, suze (a gentian ext.), etc., and is remarkably palatable and refreshing. Various formulas are presented as artificial substitutes for black current flavor, but the genuine flavor is so readily prep'd. that such substitutes, as passion fruit flavor, cannot seriously compete. W. O. E.

Preparation of and regulations concerning fruit and vegetables for export in California. Th. Frémont. *Bull. soc. sci. hyg. aliment* 23, 282-323 (1935). A. P. C.

Variations in keeping qualities of different varieties of vegetables and the reasons therefor. B. A. Rubin and V. E. Trupp. *Compt. rend. acad. sci. U. R. S. S. [N. S.]* 3, 225-8 (1935).—In this attempt to connect the variation in keeping qualities of vegetables with inner biochemical characteristics, varieties of onions and cabbages with poor and excellent keeping qualities were stored and exam'd. every 40-45 days for loss in wt., wt. of dried substance, monosaccharides, sucrose, sol. sugars, cellulose, total N, protein N, catalase and invertase. Tabulation showed that the Msterski onion, a biennial with good keeping qualities, has a high sugar loss but that, in contrast

to the poorly keeping Spanish variety, its protein complex is unchanging. Similarly, the variety of cabbage characterized by its good keeping qualities loses considerably more sugar during the period of keeping than the variety that spoils quickly. Also, the ratio of disaccharides to monosaccharides is greater in the stable varieties of both onions and cabbage. The biochem. nature of good keeping quality lies neither in weakened metabolism nor in a lowered energy expenditure of the plant substance but in the high expenditure of sugar reserves. C. R. A.

Vitamin C content of the Russet Burbank potato. Ella Woods. *Ida Agr. Expt. Sta. Bull.* No. 219, 3-29 (1935).—The raw, baked or boiled potato was usually protective at the 8-g. level. New immature potatoes contained fully twice as much vitamin C as fresh mature potatoes. Storage of 3-8 months had little effect on vitamin C content. Marked tissue changes in guinea pigs occurred before outward signs of scurvy were manifest. C. R. Fellers

Chemical determination of the quality of canned green peas. Zoltan I. Kertesz. *N. Y. Agr. Expt. Sta. Tech. Bull.* No. 233, 3-26 (1935), of C. A. 28, 6870<sup>1</sup>.—An attempt to evaluate the quality of canned peas by the detn. of total, water-insol. and alc-insol. solids in raw peas was unsuccessful. In canned peas the scores for maturity and tenderness, on 1 hand, and flavor on the other, are strictly proportional, thus conclusions regarding the flavor might be drawn from the maturity score. A study was made of the possible use of contents of total solids, alc-insol. solids, and water-insol. solids of canned peas for the detn. of maturity. Of these, the content of alc-insol. solids is best suited for the evaluation of maturity and quality of canned peas. Two possible grading systems are proposed in which the detn. of the alc-insol. solids replaces the organoleptic evaluation of maturity plus tenderness and the flavor in addn. The correlations between results obtained by the chem. methods and by the standard method of the U. S. Dept. Agr., *Circ.* No. 164 (1931), are shown and discussed. C. R. Fellers

Composition of Hungarian beans. István Szanyi. *Z. Untersuch. Lebensm.* 70, 200-74 (1935).—See C. A. 29, 2615<sup>1</sup>. F. L. Dunlap

Composition of pumpkin and squash varieties as related to the consistency of the canned product. D. M. Doty, J. H. McGilivray and H. R. Kraybill. *Ind. (Purdue) Agr. Expt. Sta., Bull.* 402, 28 pp. (1935).—The consistency of canned pumpkin and squash is markedly affected by variety, maturity and chem. compn. Of the 4 varieties studied, Boston Marrow squash, Kentucky Field pumpkin and Connecticut Field pumpkin yielded fruit producing rather low-consistency packs all 4 years of the investigation. Golden Delicious squash yielded fruit producing a very high-consistency pack the first 2 years, but a low-consistency pack the last 2 years. Fruits from all varieties yield a pack of higher consistency if canned when slightly immature or barely ripe. The contents of starch and of solids in all pumpkin and squash varieties reach a max. at about the same time that the fruit yields a pack of max. consistency. This indicates that starch and solids, especially insol. solids, affect the consistency of the canned product to a great extent. It is possible markedly to increase the consistency of the pack from low-consistency varieties by pressing or by blending with a high-consistency variety having high total solids and starch. In the com. canning of pumpkin and squash as carried out in Indiana factories, large amts. of sol. solids are lost during the wilting process and during the pressing process after wilting. During storage, pumpkins and squashes rapidly lose solids, especially starch, by respiration. Indirectly this causes a decrease in the consistency of pumpkin and squash canned after storage, so this practice should be avoided by canners as far as possible. C. R. Fellers

Analyses of corn and buckwheat. W. F. Geddes. *Can. Dept. Trade & Commerce, Dominion Grain Research Lab., 5th Ann. Rept.* 1934, 35.—Samples of the Nos. 1, 2 and 3 grades of the 1934 crop of Canadian buckwheat contained crude protein 11.4, 11.0, 10.9; crude fat 2.7, 2.8, 2.6; crude fiber 8.6, 8.9, 9.1; ash 2.0, 2.1, 1.6;

and N-free ext. 61.8, 61.7, 62.3%, resp., calcd. to the basis of 13.5% moisture. K. D. Jacob

Composition of Italian honeys Giovanni Canneri and Renato Salani *Ann. chim. applicata* 25, 397-406 (1935).—Twenty-two honeys were examd. The ratio of fructose to glucose varies from 1.011 to 1.684; thus the criterion that the ratio should be greater than 1.00 is in error. The Schiff-Sorensen test for proteins gives low results, so a mix of picric and citric acid was used to ppt. the proteins. This test gives results which agree fairly well with Lund's method. A. W. Conner

The vitamin content of honey Zofia Markuze *Arch. Chem. Farm.* 2, 175-82 (1935) (English summary).—Two samples of Polish honey, investigated for vitamins, show the presence of vitamin B<sub>1</sub>. Vitamins A, B<sub>2</sub>, C and D are not found. J. Wiertelak

Identification of cacao shells in cacao Wilhelm Flahl *Z. Unters. Lebensm.* 70, 289-90 (1935). I. L. D.

Study of the wet fermentation of coffee A. Fritz *Agron. col.* 24, 41-7, 72-81 (1935). *Chimie & Industrie* 34, 1197.—The object of the wet fermentation of coffee is to permit a perfect cleansing of the seed from the particles of mesocarp still adhering when they have passed through the depulpers. The optimum temp. is 33-40°, as pectinase possesses max. activity at 35-45°. The activity of fermentation increases with the amt. of pulp remaining on the seed, but in practice the advantage of active fermentation is offset by difficulties in the subsequent washing when there is an excessive amt. of residual pulp. Normal fermentation requires 26-36 hrs., but in practice it is limited to 12-14 hrs. to avoid the action of diastases, produced by certain molds, which act on the periphery of, and even inside, the seed to convert starch into pectin. To decrease the duration of fermentation, after depulping the seed is treated with a little lime to accelerate the conversion of pectic acid into Ca pectate and to activate the pectinase. Air is also injected into the mass up to the point at which butyric fermentation sets in. The fermenting mass is washed at the end of 12 hrs. to remove waste materials which interfere with the action of pectinase. As pectic acid is sol. in alkali carbonates and in (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, the mucilaginous tissues covering the seed after depulping can be removed without fermenting by covering the seed with water to a depth of 1-2 cm., sprinkling finely ground Na<sub>2</sub>CO<sub>3</sub> and washing after allowing to stand for 15 min. Stirring by means of mech. agitators or compressed air is advisable. It is preferable first to soak the seed in a weak milk of lime. The Na<sub>2</sub>CO<sub>3</sub> treatment can also be applied after a 12 hr. fermentation. Fermentation may be preceded by a 30-60 min. treatment with papain, which decomps. proteins with formation of amino acids.

Tea leaves III. Chemical constitution of tannin in tea leaves Yasuyoshi Oshima *J. Agr. Chem. Soc. Japan* 11, 750-6 (1935); cf. C. A. 28, 1115<sup>1</sup>.—Gallic catechol was isolated from tea leaves. Pentacetyl-gallic catechol was converted by Me<sub>2</sub>SO<sub>4</sub> and 50% KOH into gallocheatechol pentamethyl ether (I) colorless prisms, m. 184°, [α]<sub>D</sub><sup>20</sup> = -15.2°. Reduction of I by Na and methylation by Me<sub>2</sub>SO<sub>4</sub> and KOH gave white plates, m. 91°, of α-2,4,6-trimethoxyphenyl-α-3,4,5-trimethoxyphenylpropane (II). 3-Hydroxy-5,7,3',4',5'-pentamethoxyflavylum chloride, violet needles, m. 204-6°, was prepd. from II and phloroglucinolaldehyde dimethyl ether. Reduction with Pt black and AcOH gave 5'-hydroxy-catechol pentamethyl ether, which was identified with I. Thus the cryst. tannin isolated from tea leaves is 5'-hydroxycatechol. IV. Enzyme chemistry of manufacture of black tea. Yasuyoshi Oshima and Kaneo Hayashi *Ibid.* 757-9.—When the ext. of the bud was added to colorless gallocheatechol with McIlvaine standard buffer soln. in the presence of O<sub>2</sub>, the color of the soln. changed to reddish brown. When the ext. was boiled or the soln. was treated in a N<sub>2</sub> atm., the coloration was very faint. A reddish brown powder obtained from d-catechol and gallocheatechol by the enzyme of the tea bud was proved to be their oxidation products. Y. Kihara

Short method for determination of sodium chloride in

mustard. F. Th. van Voorst. *Chem. Weekblad* 32, 498 (1935).—Five g. mustard (liquid) is transferred with 100 cc. warm H<sub>2</sub>O into a 200-cc. volumetric flask. After cooling 5 cc. N.K.<sub>2</sub>Fe(CN)<sub>6</sub> and 5 cc. 2 N Zn(OAc)<sub>2</sub> are added, the flask is filled up to the mark, the contents are filtered, 25 cc. 0.1 N AgNO<sub>3</sub> is added to 150 cc. filtrate and this is titrated back with KCNS. The percentage of NaCl is 0.156 times the no. of cc. AgNO<sub>3</sub> used.

B. J. C. van der Hoeven  
Treatment of molasses for the production of foods, feeding stuffs and fertilizers T. Gaspar y Arnal *Agricoltura* (reprint) 1934, 8 pp. (Aug.-Sept.); *Chimie & Industrie* 34, 931.—Beet molasses keeps indefinitely when undiluted; dilute to a concn. of about 20% it rapidly acquires a repugnant odor and taste. To prevent this decomposition, the soln. from the diffusers is defecated with K<sub>2</sub>Fe(CN)<sub>6</sub>, which removes NH<sub>3</sub>, amines, Rb, Cs, etc. The excess reagent is removed with Mn sulfate, the ppt. is thrown down by centrifuging and the Mn is recovered by treating with CaO.

A. Papineau-Couture  
Simple graphic aid in the practical feeding of cattle II. The calculation of feed mixtures with the desired starch value and content in digestible protein by means of nomograms E. Brouwer and A. M. Frens *Biedermanns Zentr. B. Tierernähr.* 7, 496-505 (1935); cf. C. A. 29, 6027<sup>2</sup>.—In practical feeding it is often necessary to prep. with the aid of 2 feeding stuffs, a mixt. with a definite quantity of starch equiv. and digestible crude or pure protein. Usually the solution is found with the aid of 2 equations and with 2 unknown quantities. By means of the principle worked out by B. and F., the desired amt. of both feeding stuffs can be detd. without any computation. Some further applications are also indicated. III. A clear comparison of price values of feed-concentrates without the work of calculation. A. M. Frens and E. Brouwer. *Ibid.* 506-16 (1935).—The relation between price and feed value of concentrates is usually estd. in Holland with reference to starch values and protein content. By the application of a system of double scales on a diagram, corresponding to Neubauer's "Feeding Price Table," a nomogram can be constructed, by means of which and without calcs., it is possible to survey the prices of an arbitrary no. of feeding stuffs, in relation to their starch values and protein contents. Some practical applications of nomograms are discussed. F. L. D.

Acidified potato tops as a feed for milch cows J. Schultz, H. Augustin and H. Finzenhagen *Biedermanns Zentr. B. Tierernähr.* 7, 517-31 (1935).—Cold fermentation of chopped potato tops proved to be favorable, but the addn. of some mineral acid or food sugar is to be recommended. A utilization trial with 2 wethers indicated that the ensilaged potato tops had the following digestion values: org. matter 49, crude protein 48, pure protein 33, amides 97, crude fat 58, crude fiber 41 and N-free ext. 52%. The ensilaged potato tops were fed to dairy cows and after a certain period, they ate, on an av., 8 kg. per day. No unfavorable influence was noticed on the state of health, live wt. or milk yield. F. L. Dunlap

Effect of manufacture on the quality of nonolly fish meals Roger W. Harrison, Andrew W. Anderson and Samuel R. Pottinger *U. S. Bur. Fisheries, Investigational Rept.* 30, 1-30 (1935).—The nutritive value of fish meal produced by haddock and cod wastes is influenced by the process of manuf., the method of drying and the portion of the waste used. The removal of the water-sol protein by the wet process had little effect on the ultimate efficiency of the protein, but decreased the vitamin G value. High drying temps. are detrimental to the quality of the protein and vitamin G, particularly the latter. The head portion of the waste is richer in vitamin G than the backbone portion, but the latter is of higher quality. Flame-dried nonolly meals are of inferior quality and should be replaced by meals dried at low temps. The mech. difficulties encountered in drying nonolly fish wastes without removal of the water-sol extractives can be reduced materially by careful control of steam pressure and vacuum. Under optimum conditions of drying, the cost of rendering 1 ton of waste is greater for the dry than for the

wet process. In the prepn. of dry-process nonoil fish meals, the conditions required for most efficient operation are most conducive to improvement in nutritive value. Vacuum-drying possesses no appreciable advantage over steam drying, either from the standpoint of quality of meal or cost of production.

C. R. Fellers

**Maize silage** M. H. French, Tanganyika Territory, *Ann. Rept. Dept. Vet. Sci.* 1934, 86-90.—Silage made in Tanganyika from maize cut 73 days after planting contained crude protein 6.20-7.45,  $\text{Et}_2\text{O}$  ext. 1.93-3.20, N-free ext. 52.71-53.26, crude fiber 31.43-31.90 and ash 5.03-6.85% on the  $\text{SiO}_2$ -free dry basis. As detd. by expts with oxen, the digestibility coeffs. were for crude protein 23.53-36.06,  $\text{Et}_2\text{O}$  ext. 69.37-78.44, N-free ext. 61.78-74.70, crude fiber 60.42-71.74, org. matter 61.06-71.89 and dry matter 60.99-71.31%. The aq. exts. of the crop and of the silage contained volatile org. acids (calcd. as  $\text{AcOH}$ ) 0.107-0.955, 0.165-1.070, nonvolatile org. acids (calcd. as lactic acid) 1.23-1.38, 5.27-7.38, amino acids (calcd. as crude protein) 0.83-1.45, 1.73-4.10, and volatile bases (calcd. as crude protein) 0.080-0.095, 0.16-0.74%, resp., calcd. on the basis of 100 g. dry matter.

K. D. Jacob

**The chemical composition of grass veld.** M. H. French, *Farming S. Africa* 10, 346, 348 (1935).—P is deficient in the grass pastures of most of the drier parts of S. Africa, and in those of many of the parts which have a higher rainfall. The young grass is generally not in itself deficient in P, but the old and mature grass even when green, from Nov. onward, contains less than 0.3%  $\text{P}_2\text{O}_5$  and often less than 0.2%. After the first frosts the  $\text{P}_2\text{O}_5$  content falls to 0.03-0.07%. Ca is on the low side without being actually deficient throughout the S. African veld. A few species contain more than the usual 0.5% CaO. Grasses on granite soils are generally very poor in Ca. The Ca content increases in grasses with the advancing season. Na is decidedly deficient over wide areas.

K. D. Jacob

**The nutritive value of groundnut cake made by primitive methods.** M. H. French, Tanganyika Territory, *Ann. Rept. Dept. Vet. Sci.* 1934, 83-5.—On the dry basis, samples of groundnut cake made in Tanganyika by primitive methods and modern methods contained crude protein 53.19, 46.24-51.31; true protein 51.36, 44.50-50.51, amides 1.83, 0.80-1.74;  $\text{Et}_2\text{O}$  ext. 9.89, 4.71-4.76; N-free ext. 27.86, 33.31-36.75, crude fiber 4.76, 4.77-5.64; total ash 5.30, 5.85-6.66, and  $\text{SiO}_2$  0.68, 1.51-2.24%, resp. As detd. by expts with sheep, the digestibility coeffs. of the samples were for crude protein 91.08, 92.20-93.62;  $\text{Et}_2\text{O}$  ext. 82.88, 83.08-94.39, N-free ext. 96.36, 95.62-99.58; crude fiber 80.74, 82.08-90.77; org. matter 91.36, 92.57-95.83; and dry matter 87.26, 89.54-94.09%, resp.

K. D. Jacob

**Some so-called "salt licks" of Tanganyika Territory.** M. H. French, Tanganyika Territory, *Ann. Rept. Dept. Vet. Sci.* 1934, 69-72.—Samples of "edible earth" from 16 localities contained  $\text{H}_2\text{O}$  1.27-19.26, loss on ignition 1.02-15.53,  $\text{H}_2\text{O}$ -sol. material 0.02-60.46, material sol. in dil.  $\text{HCl}$  0.21-62.46, CaO sol. in dil.  $\text{HCl}$  0.03-1.03,  $\text{P}_2\text{O}_5$  sol. in dil.  $\text{HCl}$  trace-0.94,  $\text{H}_2\text{O}$ -sol. sulfate (expressed as  $\text{SO}_4$ ) 0.07-10.55, sulfate sol. in dil.  $\text{HCl}$  0.22-11.93,  $\text{H}_2\text{O}$ -sol. Cl 0.003-9.87,  $\text{H}_2\text{O}$ -sol.  $\text{Na}_2\text{O}$  0.03-20.17,  $\text{HCl}$ -sol.  $\text{Na}_2\text{O}$  0.05-22.61,  $\text{H}_2\text{O}$ -sol.  $\text{K}_2\text{O}$  0.004-0.83,  $\text{HCl}$ -sol.  $\text{K}_2\text{O}$  0.008-0.83 and  $\text{H}_2\text{O}$ -sol. carbonate (expressed as  $\text{CO}_2$ ) trace-14.18. Na was the chief  $\text{H}_2\text{O}$ -sol. cation in all the earths. Chlorides, carbonates and sulfates were the most important anions.

K. D. Jacob

**The nutritive value of the pods of *Acacia arabica* and *Dichrostachys glomerata*.** M. H. French, Tanganyika Territory, *Ann. Rept. Dept. Vet. Sci.* 1934, 79-82.—On the dry basis, the pods of *A. arabica*, *A. spirocarpa* and *A. albidia* contained crude protein 12.86, 12.54, 8.82; true protein 11.62, 11.54, 8.19; amides 1.24, 1.04, 0.63;  $\text{Et}_2\text{O}$  ext. 2.51, 1.82, 1.44; N-free ext. 63.82, 57.90, 61.73; crude fiber 15.21, 22.37, 24.40; total ash 5.60, 5.63, 3.60;  $\text{SiO}_2$  0.39, 0.50, 0.34; CaO 7.2, 1.37, 0.91; and  $\text{P}_2\text{O}_5$  0.33, 0.56, 0.52%, resp. As detd. by expts with sheep the digestibility coeffs. of the 3 species were for crude protein 51.72, 46.23, 51.04;  $\text{Et}_2\text{O}$  ext.

1 88.17, 73.93, 71.42; N-free ext. 79.01, 70.65, 74.84, crude fiber 21.21, 42.03, 16.47; org. matter 66.22, 64.44, 56.21, and dry matter 64.50, 62.87, 56.72%, resp. On the dry basis, the pods of *D. glomerata* contained crude protein 10.82, true protein 8.82, amides 2.00,  $\text{Et}_2\text{O}$  ext. 1.42, N-free ext. 55.76, crude fiber 26.64, total ash 5.36,  $\text{SiO}_2$  0.41, CaO 0.71 and  $\text{P}_2\text{O}_5$  0.45%. The digestibility coeffs. were for dry matter 59.21, org. matter 61.15, crude protein 37.24,  $\text{Et}_2\text{O}$  ext. 68.96, N-free ext. 67.32, crude fiber 55.31 and ash 32.60%, resp. K. D. Jacob

**Detn. of the water contents of [foodstuffs] (Richter) 7**  
HCN in grasses (Lémann) 11D Use of cleaners in the dairy plant (Barnum, et al.) 13 Sizing and sepg. fines from crude materials such as flour (U. S. pat. 2,026,318)  
1 Clarifying beverages (Ger. pat. 558,369) 16 App. for treating fish to recover oil from them and to prepare 3 them for canning (U. S. pat. 2,026,022) 27.

**Preserving food** Halvor S. Egeberg (Dortheus Andreas Hansen, inventor) Ger. 621,350, Nov. 6, 1935 (cl. 53c 3 03) Addn. to 593,054 (C. A. 28, 3148\*) See Brit. 382,644 (C. A. 27, 3261)

**Metal-vulcanized vacuum chamber or container suitable for holding foods, etc.** Eugene L. Schellens (to Shellwood-Johnson Co.) U. S. 2,024,065, Dec. 10 A wall metal such as iron is used with a sealing metal, such as Cu, of lower m. p. than, and alloying affinity, for the wall metal and sealing is effected by heating in a nonoxidizing atm. Various mfg. details are described.

**Cereal foods** Edmund L. Griffith U. S. 2,026,034, Dec. 31 Cereal material such as bran is mixed with a flavoring sirup and with a small proportion of paraffin and mineral oil to coat the particles of the product and 5 act as an intestinal lubricant.

**Milled products from seeds of *Ceratonia siliqua*, etc., for food purposes.** Montz Bienenstock, Ladislav Csaki, Julius Pless, Adalbert Sági and Eugen Sági U. S. 2,025,705, Dec. 31 Milled germinal substance of the seeds of *Ceratonia siliqua* or the like is mixed with a material such as citric acid which reduces the water-binding power of the germinal substance and forms a mixt. which is suitable for use in macaroni and various 6 baked foods.

**Bread** Herbert Schou Tr. 786,088, Aug. 26, 1935 The particles of dough are greased and sepd. by the addn. of a very viscous emulsion of water or aq. liquid in an oily constituent, the ratio of aq. liquid to oily constituent being above 1/5.

**Apparatus for preparing alimentary pastes.** Mario Braibanti and Giuseppe Braibanti U. S. 2,026,667, Jan. 7 Various structural, mech. and operative details.  
7 **Baking yeast** Károly Dinich Brit. 434,249, Aug. 28, 1935 Bakers' yeast is made from brewers' yeast by using it to ferment dil. mash first at 4-7°R., then in stages at 7-10, 10-13, 13-16, 16-19, 19-22 and 22-25°R. and finally at 25-28°R., the aged and weak cells being at the same time artificially destroyed and the yeast freed from these dead cells which would cause it to spoil. Cf. C. A. 29, 2638\*

8 **Deodorizing milk, cream, etc.** Fritz G. Cornell, Jr. U. S. 2,025,196, Dec. 24. A preheated stream of the liquid is flowed and expanded into a sub-atm. pressure zone, the liquid is then collected into a confined stream within this zone, steam is injected into the stream and discharged into the zone, and evolved vapors and gases are withdrawn. App. is described.

**Treatment of cream** W. & H. Kühle G. m. b. H. Ger. 621,378, Nov. 6, 1935 (Cl. 53c 5). Churned cream 9 for use in making confectionery is stabilized and prevented from sepg. into layers by adding to the cream a small proportion of a flour prepd. by baking and grinding a dough not contg. sugar, salt and leaven. The flour or a part of it may be rebaked before it is used.

**Emulsifying apparatus suitable for making reconstructed cream.** Torris H. Alfréd (to Herbert J. Taylor) U. S. 2,026,604, Jan. 7 Various structural and mech. details.

**Margarine** Benjamin R Harris U S 2,024,355, Dec 17. A triglyceride such as hydrogenated cottonseed oil or oleostearin is partially saponified with an alkali such as NaOH and the soap thus formed is in part at least decomposed as by treatment with water and HCl or other suitable acid, and the resulting pasty product is incorporated in oleomargarine to improve its texture and spreading properties, etc.

**Margarine** Advin D Joyce (to Durkee Famous Foods, Inc.) U S 2,024,647, Dec 17. In making margarine from a margarine base selected from edible oils and fats substantially free from milk solids, the base is congealed to a supercooled uncrystallized form and incorporated with a milk component.

**Margarine** Herbert Schou Fr 787,373, Sept. 21, 1935. A product more closely resembling butter in taste is obtained by going to the final product in the melted state a viscosity of at least 36 centipoises at 50°. This is attained when the water used for the water-oil emulsion is finely distributed, and in a stable manner at 50° and higher, in the exterior phase.

**Margarine** Gerrit Westervink Ger 621,326, Nov 5, 1935 (Cl 53A 102) Addn to 582,141 (C A 27, 5122). The churn used in the process of Ger 582,141 is replaced by a mixing and kneading vessel capable of winging about a hollow horizontal axis, the latter being connected to a suction device.

**Margarine** Nohlee & Thorl G m b H Ger 621,327, Nov 5, 1935 (Cl 53A 103). In the manufacture of margarine containing a small proportion of lecithin, the latter is added as lecithin-oil-glycerol mixt obtainable as described in Ger 599,639 (C A 28, 7574) or 602,935 (C A 29, 9504). The tendency of the lecithin to decompose is thus restrained.

**Rindless cheese** Franz Zwick (trading as Schachtel-Käsefabrik Franz Zwick) Brit 434,321, Aug 29, 1935 See C A 29, 8168.

**Preserving liquid egg material** Goran A Moller Brit 435,242, Sept 17, 1935 See Fr 780,903 (C A 30, 7844).

**Frozen egg products** Benjamin R Harris and Marvin C Reynolds U S 2,026,631, Jan 7. Egg material which when frozen and subsequently thawed has increased viscosity and improved emulsifying value in cake batters, etc., is prep'd by adding hydrophilic lipins such as fat acid esters of a polyglycerol, glycerol, sugars, mannitol and other alk sugars and hydroxy carboxylic acids. Several examples with details are given.

**Lecithin from vegetable raw materials** Samuel O Sorensen and Geo I Beal (to American Lecithin Co.) U S 2,024,398, Dec 17. For obtaining lecithin free from bitter impurities and suitable for use in foods, raw material such as crushed soybeans is ext'd with a volatile hydrocarbon solvent such as hexane, preferably with a b p not over about 60°, the solvent is driven off from the ext and water vapor is passed into the residual oil to cause coagulation of the lecithin and the latter is sep'd.

**Preserving fruit** Alexander Gerner-Rasmussen, Brit 435,587, Sept 24, 1935 See Fr 778,633 (C A 29, 4851).

**Food jellies** Speas Mfg. Co Brit 435,033, Sept. 9, 1935 See Fr 770,163 (C A 29, 5194).

**Gelatin food preparations** Royal Baking Powder Co Brit 435,258, Sept. 17, 1935 This corresponds to U.S. 1,993,269 (C A 29, 24109). The moisture need not be removed.

**Cake-batter ingredients** Benjamin R Harris U S 2,024,357, Dec. 17. With a batter for making baked products such as cake, there is incorporated a hydrophilic lipid such as a sulfate or phosphate deriv. of fat acid materials or corresponding alcs, or esters or ethers or salts of such derivs, which gives a smoother batter and permits use of an increased proportion of water. Numerous examples are given.

**Confections** Benjamin R Harris U S 2,025,985, Dec 31. In making confections such as those containing sugar, cocoa powder and a fat, the viscosity of the mixt is reduced by adding a small proportion of a nonnitrogenous phosphoric acid ester of glycerol in which at least one OH group of the glycerol is esterified with a higher unsat'd. fat acid radical (various details and examples being given). U. S. 2,025,986 relates to confections of generally similar character.

**Confections such as icings and candy** Benjamin R Harris U S 2,024,356, Dec. 17. Materials such as sugar, cocoa powder and milk powder are dispersed in fatty material as cacao butter and there is incorporated in the product a viscosity reducing ingredient comprising a higher fat acid ester of polyglycerol. Various examples are given.

**Cacao** Richard Dierbach Brit 434,750, Sept. 9, 1935 See Fr 772,566 (C A 29, 15314).

**Flavorings** Royal Baking Powder Co Brit 435,272, Sept 18, 1935. A flavoring prep'n, e.g., for use in prep'g gelatin deserts, is obtained by forming an emulsion of a flavor with the inclusion of a substantially nontoxic H<sub>2</sub>O-sol org. comp'd of higher b. p. than the flavor for reducing the volatility of the flavor, e.g., octyl or nonyl alc, dibutylcarbinol, phenylbutyl alc, l-tartrate, benzyl benzoate, diamyl ether, dibutyl ketone, benzophenone, p-methoxybenzaldehyde. The emulsion may be made in H<sub>2</sub>O with addn of an emulsifying agent, e.g., gelatin, albumin, an oleate, a gum. An edible hygroscopic agent, e.g., invert sugar, glucose, glycerol, may also be added.

**Removing objectionable flavor from soybeans** Lowell O Gill (to A. E. Staley Mfg Co.) U S 2,026,676, Jan 7. Whole or broken soybeans are moistened without ext'g their solubles, then heated to above the b. p. of water (suitably to about 160°) to remove the natural flavor but without scorching and for a sufficient time for the stated purpose, and the temp for final drying is reduced before the increased moisture content is eliminated.

**Apparatus for treating grain with conditioned air or gas** Marcel Mallet. Fr 787,827, Sept 30, 1935.

**Fodder** Hans Fettingner. Austrian 143,318, Nov. 11, 1935 (Cl 53c). Sludge from the sats of beet-sugar juices is mixed with bran, subdivided hay, ground oil cake, fish meal or like materials, and the mixt is worked up into granules, flakes or like products containing about 10% of water.

## 15—CHEMICAL INDUSTRY AND MISCELLANEOUS INDUSTRIAL PRODUCTS (PLASTICS, RESINOIDS, INSULATORS, ADHESIVES, ETC.)

HARLAN S. MINER

**The Italian chemical industry in 1934** Grete Blumen-thal *Chimie & Industrie* 34, 909-73 (1935) A P-C

**Some aspects of the Polish chemical industry** Constantin de Kownacki *Chimie & Industrie* 34, 974-6 (1935) —A brief discussion bringing out the general tendency toward increased production A P-C.

**Acetylene in the chemical industry** A. F. G. Cadenehead *Can Chem Met* 19, No 12, 325 (1935) —C<sub>2</sub>H<sub>2</sub>, black and AcOH and Me<sub>2</sub>CO are the principal products at the present time from C<sub>2</sub>H<sub>2</sub>. Biacetyl, acetylacetone,

dimethylfuran, dihydro-p-tolualdehyde and 1,3 butylene-glycol-diacetate are by-products for which uses must be sought W. H. Boynton

**Reactions with oxygen** A. H. Behnke *Chem. Weekblad* 32, 611-15 (1935). —A theoretical discussion of various systems comprising O alone, O in conjunction with 1 and 2 oxidizable, 1 and 2 nonoxidizable substances and mixts of oxidizable and nonoxidizable substances O. W. Wilcox

Further progress in production and use of high grade

zinc; oxide situation interesting. Frank G. Breyer. *Mining Met* 17, 24-6(1936).—The following are the major developments in the Zn field in 1935 in the order of their importance: (1) construction of further continuous vertical-retort smelting capacity in the U. S. and Germany, (2) expansion of fractional distill capacity in the U. S., (3) great expansion in the Zn-base die-casting industry in the U. S. and abroad, (4) wide acceptance of heavy galvanizing by the steel industry, (5) construction of first commercial wire electroplating plant, and (6) expansion of use of Zn dust as a reducing agent in org. chem. industry. In the field of Zn compds., the following were the major developments: (1) further decrease in the use of ZnO in rubber compounding, (2) wider appreciation of the value of leaded ZnO in paint, (3) threat of Ti-inert combinations to lithopone in the bulk whitening field, and (4) threat to TiO<sub>2</sub> of ZnS for concd. whitening. Alden H. Emery.

Industrial minerals record progress over a wide front. Oliver Bowles. *Mining Met* 17, 28-33(1936).—Recent developments in the following fields are reviewed: barite, heat insulation, glass, mortars, synthetic stone, cement, clay, feldspar, fluor spar, fuller's earth, gypsum, cyanite, lime, nephelite, novaculite, olivine, potash, pyrophyllite, Na<sub>2</sub>SO<sub>4</sub>, S and talc.

Asbestos. Hans Diegmann. *Tek Tid, Uppl. C, Bergenskaps* 65, 86-8, 89-94(1935).—A description of the applications of asbestos in the electrochemical and the chem. industry, as heat-insulating material, floor- and wall-covering, brake lining, etc. Its origin, mineralogy, mining and main occurrences on the earth are reviewed. D. Thuesen.

Asbopekohlite, a new high-quality lamellar material. E. I. Barg and K. M. Bekreneva. *Plasticheskie Massy* 1935, No. 3, 30-1.—This asbestos-pitch material surpasses textolite for many uses. H. M. Leicester.

New views for seeking substances useful in chemical warfare. M. Procházka. *Chem. Listy* 29, 271-2(1935).—P. presents and extends the views of Hackman (C. A. 29, 519<sup>4</sup>). For destroying the effectiveness of the filters of gas masks P. shows that a high concn of gases (arsine) effected by a large no. of gas cylinders can exhaust the absorption capacity of the filters very quickly and that the unstable Fe(CO)<sub>5</sub> yields CO against which the filters are ineffective. Following Nekrasov, P. shows the poor relation between the structure and the physiol. properties of org. compds.: an increase in the no. of halogens decreases the irritability of a compd. but increases its suffocating power; sym. halogens are more irritable than unsym. ones; compds. with a halogen at the end are more active physiologically than those with a halogen in any other position, an introduction of a NO<sub>2</sub>, —NOH, or —C≡N group into the org. nucleus reinforces the physiol. action of such a nucleus; unsatd. compd. are more effective than satd. compds. The action of dichloroformoxime and derivs. upon the eyes and the respiratory organs is presented; the stinging sensation produced by dichloroacetone and trichloroacetophenone is described, the changes produced by an NH<sub>2</sub> group substituted into adamante are analyzed. In an attempt to produce a yperite deriv. which would remain active on the terrain for several hrs., P. shows that in order to have a physiol. active prepn. the halogens must be in the β-position, that the α-H atoms cannot be replaced or substituted, that the S must be bi- or quadri-valent, and that the EtCl group is the most potent group in its present position.

Detection of poison gases. André Kling and Maurice Rouilly. *Compt. rend.* 201, 1373-5(1935).—Vesicant and suffocating gases (e. g., COCl<sub>2</sub>, yperite, lewisite, etc.) are all distinguished by a very labile halogen. In contact with H<sub>2</sub>O they hydrolyze, producing a marked diminution in *pH*, readily detected by bromothymol blue. Accordingly to test a suspected atm. 8-10 l. is aspirated through H<sub>2</sub>O to which this indicator has been added. If the atm. contains more than 1% CO<sub>2</sub>, the H<sub>2</sub>O must be warmed. C. A. Suberrad.

The two-piece protective suit. Pepp Seidl. *Gasmasken* 7, 95-8(1935).—The suit is designed to protect its wearer

against mustard gas. A description of the suit is combined with detailed directions for putting it on and taking it off. A series of photographs illustrate these directions.

A. L. Kibler. Escape apparatus. Karl Ed. Pütter. *Gasmasken* 7, 89-92(1935).—Escape apparatus is designed primarily for the use of miners who are caught in mine air containing insufficient O and dangerous amounts of CO<sub>2</sub>, such as result from mine explosions. The requirements of such an apparatus are discussed and various types are described.

A. L. Kibler. Silicosis. Wm. D. McNally and W. L. Bergman. *Ind. Med.* 4, 61-5(Feb., 1935). *U. S. Pub. Health Eng. Abstracts* 15, IHS, 10(Nov. 2, 1935).—A new theory is advanced that the fibrosis in silicosis is produced by chem. action of NaF in blood upon the SiO<sub>2</sub>. Si dusts become more harmful as the amt. of SiF<sub>4</sub> is increased in the dust. A chem. examn. of normal and silicotic lungs is given.

C. R. Fellers. The chemical stability of phenol plastics. E. Tievich, B. Polyatizkma and M. Glikina. *Plasticheskie Massy* 1935, No. 3, 22-6.—Numerous Russian lacquers were found to resist the action of H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> much better than that of org. acids. Monolith and carbolite swell more in weak than in strong acids. H. M. Leicester.

The acid stability of plastic compounds for accumulators based on coal tar pitch and hydroxynaphthenic acids. V. M. Shalfeev and M. M. Katzin. *Plasticheskie Massy* 1935, No. 3, 26-9.—A good material is obtained from 48% coal-tar pitch, 12% hydroxynaphthenic acids free from H<sub>2</sub>SO<sub>4</sub>, 10% cotton waste and 30% kieselsol. H. M. L.

Transparent plastics for aircraft windows. Gordon M. Kline. *Ind. Eng. Chem., News Ed.* 13, 479-80(1935).—Light weight and flexibility of transparent plastics have made them desirable for aircraft windows, but since their surface hardness is not great laminated glass is still used where clear vision is essential. Com. plastics, trade names, compn., Army and Navy specifications and ideal properties to be attained are listed, cooperative efforts on the part of government agencies and private manufacturers are in progress to improve the plastics for aircraft. W. C. Ebaugh.

The application of furfural and its derivatives to the manufacture of plastics. Paul F. Bruns. *Iowa State Coll. J. Sci.* 10, 51-3(1935).—S. derivs. of C<sub>4</sub>H<sub>3</sub>OCHO are accelerators for vulcanization of rubber. Polythiofurfural is formed by bubbling H<sub>2</sub>S into dil. aq. solns. of C<sub>4</sub>H<sub>3</sub>OCHO and coagulating the suspension by means of HCl. It is an ultra-accelerator producing high tensile strengths in rubber at 104.5° but was not effective at 100°. Diethyl ethylene secured as a residue from the vacuum distn. of polythiofurfural is not an accelerator. Then the accelerating action must be due to the C and S grouping. Furfural was prepd. by passing NH<sub>3</sub> into C<sub>4</sub>H<sub>3</sub>OCHO. When 19.3% of the C<sub>4</sub>H<sub>3</sub>OCHO was converted to furfural, the resulting mixt. was set to a gel, in a few min., by the addn. of HCl, and in an hr. had become firm, hard, black and shiny without heating. Fillers such as corncstalk, cob flour, wood flour, asbestos and oat hull residues were added and molded articles produced from the mixts. The resultant material had a density of 1.44, a tensile strength of 500 lb./sq. in., a compressive strength of 3500 lb./sq. in. and a resistivity of 2 × 10<sup>10</sup> ohms per cc. It does not burn with a flame and resists the action of all ordinary acids and alkalis. The acid has not been successfully removed from the finished product. Slow drying or baking gives the best results. F. E. B.

The use of wood tar from gas generators for the preparation of plastic compounds. I. P. Losev, G. S. Petrov, K. A. Andrianov and P. I. Panasyuk. *J. Chem. Ind. (Moscow)* 12, 1171-6(1935).—The phenol fraction extd. from such tars consists mostly of xylenols and can be condensed with CH<sub>3</sub>O or MeCHO to give good resins if 25% of coal-tar PhOH is added to the mixt. The pitch from the tars can replace asphalt pitch in making battery plates.

H. M. Leicester. A new plasticizer, benzyl dihydroxyacetate. S. N. Ushakov and A. A. Rozenfel'd. *Plasticheskie Massy*



1935, No 3, 4-8.—Ground rosin is treated with  $\text{Na}_2\text{CO}_3$  soln. and chlorinated. The  $\text{aq}$  reaction mixt is then heated in an autoclave for 6 hrs at  $150^\circ$ . The resulting Na salt of dihydroxyabietic acid is heated with  $\text{PhCH}_2\text{Cl}$  for 16-18 hrs to yield 80-7% of benzyl dihydroxyabietate, a viscous oil. It is a good plasticizer with cellulose esters, but is insol in cellulose ethers. H M Leicester

The use of hydroxy-naphthene acids in the production of plastic compounds from phenol-aldehyde resins. G S Petrov and N B Kruglaya. *Plasticheskie Massy* 1935, No 3, 16-20.—The acids obtained by oxidizing paraffin oil, sp gr 0.860, can be condensed with  $\text{PhOH}$  and  $\text{CH}_2\text{O}$  or furfural. H M Leicester

A method for determining the fluidity of celluloid and other thermoplastics. L F. Aksberg and A V. Shelton. *Plasticheskie Massy* 1935, No 3, 20-2.—The celluloid is pressed under standard conditions in a Raschig press, and the fluidity is measured by the length of the column obtained. H M Leicester

Thermoplastic resins from aniline condensation products. Karl Frey. *Kunststoffe* 25, 305-8 (1935).—A brief review (1) of the chemistry of the conversion of  $\text{PhNH}_2$  and  $\text{CH}_2\text{O}$  into thermoplastic resins and (2) of the mech and elec properties of the latter and of products made from them, e g, impregnated paper. J W Perry

Synthetic resins from vinyl chloride. H I Waterman, J J. Leendertse and P J G Colthoff. *Chem Weekblad* 32, 550-3 (1935).—The process of working up ethylene to resins is analyzed.  $\text{C}_2\text{H}_4\text{Cl}_2$  is made from  $\text{C}_2\text{H}_4$  by passing cooled  $\text{C}_2\text{H}_4$  into liquid  $\text{Cl}_2$  at  $-78^\circ$  until sat'd, keeping the temp rise down to  $3-5^\circ$ . The product is washed with dil.  $\text{KOH}$  and with water, and then dist'd; the yield is 93%, it b  $84.9-85.5^\circ$  and has  $n_D^{20} = 1.4444$ . The  $\text{C}_2\text{H}_4\text{Cl}_2$  with  $\text{AlCl}_3$  (10%) at  $45-55^\circ$  gives  $\text{HCl}$  and a resin mass which, after washing and drying and extrn by  $\text{C}_2\text{H}_4\text{Cl}_2$ , yields 40% of a brown transparent resin, decomp at  $250^\circ$ , it is brittle and impure. If vinyl chloride is made from  $\text{C}_2\text{H}_4\text{Cl}_2$  (Kogerman method, C A 25, 487) a 91-93% yield is obtained. The  $\text{C}_2\text{H}_4\text{Cl}_2$  is then polymerized either under pressure in sunlight, (one month required, good yield) or by  $\text{AlCl}_3$  in different solvents. In  $\text{C}_2\text{H}_4\text{Cl}_2 + \text{C}_2\text{H}_4$  soln little polymer is formed up to  $18^\circ$ . If  $\text{C}_2\text{H}_4$  is used as solvent a  $\text{HCl}$  evolution takes place (Friedel Crafts reaction) between  $0^\circ$  and  $18^\circ$ , a soft resin is obtained and also other reaction products distg up to  $115^\circ$  with 2 or 3 rings. Hydrogenation of the latter compds gives, among other compds, diphenylethane. With toluene as solvent at  $-78^\circ$  the  $\text{AlCl}_3$  reaction gives similarly a mixt of 50% resin and 50% double-ring by-products. B J C van der Hoeven

Light-colored coumarone resins. A Roustchinsky. *Chem & Industrie* 34, 766-72 (1935).—Ordinary com solvent naphtha obtained in the distn of crude benzene gives chiefly dark-colored coumarone resins. A much higher proportion of light-colored resin can be obtained by prep't of the solvent by stear distn of the crude benzene. A Papineau-Couture

The use of artificial resins in various branches of technology. Ludvík Šprk. *Chem Listy* 29, 273-6 (1935).—Reviews formaldehyde resins and discusses their use as mordants in tanning and textile industries, as protective films in ceramics, as fillers or matrixes in articles, as substitutes for rubber, as catalysts for vulcanizing, as insulating materials and as a replacement for nonbreakable glass. Frank Mareš

Establishing types and standards for pressed materials of artificial resin. R Houwink. *Kunststoffe* 25, 313-14 (1935).—The desirability of detg and standardizing mech and elec properties is emphasized. J W Perry

Improving pressed objects from artificial resins by additional heating. V T Renne and V V. Pasunkov. *Plasticheskie Massy* 1935, No 3, 31-2.—The elec resistance of monolith objects is increased and their hygroscopicity is decreased by heating them to  $120^\circ$  for 16 hrs after pressing them. H M Leicester

Preparation of mixtures of phenol resins and rubber. H I Waterman, C van Vlodrop and A R Veldman,

1 *Chem. Weekblad* 32, 622-4 (1935).—When an alk. mixt. of a phenolic resin and latex (with fillers if desired) is acidified with dil.  $\text{AcOH}$ , a mixt of resin and rubber is pptd. as a finely divided powder; after filtering, washing and drying it can be used as a molding powder. A flow sheet for a factory process is given. O. W. Wilcox

Use of the dielectric constant in the chemical industry. B van Steenberghe. *Chem. Weekblad* 32, 466-78 (1935).—The Ebert app for rapid detn of the dielec const is described and various applications of the  $\epsilon$  detns are discussed, such as moisture detns on active charcoal, dextrin, tobacco, cement, lime, marmalade, etc., detns of permeability of lacquers for water, absorption of  $\text{H}_2\text{O}$  by surfaces, distn control and sedimentation analysis. B J C van der Hoeven

Wetting and spreading properties of aqueous solutions—oleic acid-sodium carbonate mixtures. H L Cupples. 3 *Ind Eng Chem* 28, 60-2 (1936); cf. C. A. 29, 8173.—Surface tensions, interfacial tensions and spreading coeffs on a refined paraffin oil were detd for aq.  $\text{Na}_2\text{CO}_3$ -oleic acid mixts contg 1.0%, 0.3%, and 0.1% oleic acid and with varying alkali-fatty acid ratios. These mixts are much less sensitive to variation of this ratio than are the corresponding  $\text{NaOH}$ -oleic acid mixts. The spreading coeff,  $S$ , of a soap soln over the standard oil is defined by the equation  $S = T_B - T_A - T_{AB}$ , where  $T_B$  = surface tension of oil (30.5 dynes/cm at  $25^\circ$ ),  $T_A$  = surface tension of soap soln, and  $T_{AB}$  = interfacial tension. At a given concn the wetting properties of the carbonate mixts are much less sensitive to variations in the alkali-fatty acid ratio, which should make it easier to maintain a consistently high value of the spreading coeff in the prep'n of the carbonate solns. There should also be less uncontrolled variation in the wetting properties because of acidity or alkali of the water used in dilg the mixt. W H Boynton

Analysis of the causes of variations in the results of tests of properties of (molded) articles. A I Goldshtein. *Zavodskaya Lab* 4, 918-23 (1935).—Attempts were made with the aid of statistical data and their interpretation to relate the frequent variations in the results of tests of elec properties of phenolic moldings to the methods of manuf and controllable and uncontrollable factors of examn. No pos results could be obtained at this stage of investigation. Chas Blanc

Basalts from Dorges Bobriza, Ukraine from the viewpoint of their suitability for casting. A Gunzberg. *Trav Inst petrog Acad. Sci U S S R* 4, 83-91 (1933), *Neues Jahrb Mineral Geol*, Referate II, 1934, 864.—Chem and microscopic data on andesite basalts show their usefulness as telephone insulators. J F Schauer

7 Detection of the break point in the determination of retentivity of activated charcoal. Franz Krczal. *Z. ges. Schiess-Sprengstoffw* 30, 354-6 (1935), cf. C. A. 30, 1878.—Another app is described in which the break point of the charcoal filter is detected by means of the pptn of the gas in liquid or solid form on a 60-cm brass rod immersed in a mixt of ether and solid  $\text{CO}_2$  at one end and in a mixt of water and ice at the other end. The app. must first be calibrated empirically. Other methods are described for detecting the break point by means of indicators, either in soln or deposited on test papers. These are adapted to specific gases. Methods of prep't the test papers are given and the sensitivity of the tests is indicated. A L Kibler

Comparison of various methods of determining the humidity of air. O R. Monod. *Ann. serv. tech. hyg. Paris* 15, 61-82 (1934), *Chimie & Industrie* 34, 1082.—

9 Absorption method. The air is passed over pumice impregnated with  $\text{H}_2\text{SO}_4$ , the increase in wt of which is detd. With pumice 2-3 mm in diam packed in a straight tube 12 cm long and 10 mm in diam, absorption is complete in the 1st tube when the air is passed rapidly (2 l per min), when the air is passed slowly the  $\text{H}_2\text{O}$  seems to follow the air stream and is absorbed only in the 2nd and 3rd tubes. The method has an accuracy of 1-1.5 mg Dew-point hygrometer.—The air passing through a volatile

liquid ( $\text{Et}_2\text{O}$ ) cools a polished metal surface, and the temp. of the liquid is noted on the metal. The moisture content is obtained from Regnault's tables. The various instruments tried out by M. gave satisfactory results, provided the plates were wiped carefully and observations were effected at a certain distance to avoid interference by the observer's breath, etc. These 2 methods (the only ones which give directly the moisture content) furnish comparable results. *Psychrometers*—The readings given by these instruments (both stationary and sling types) are affected by the velocity of the air current, and insufficient wetting of the bulb introduces a further error. In the Assmann suction psychrometer the velocity of the air is constant and all thermal effects are eliminated by means of polished metal nuts, nevertheless, it gives very irregular results as compared with the Alluard dew-point hygrometer. *Hair hygrometers*—These instruments indicate relative humidity, not moisture content. The hairs should at intervals be placed in an atm. sat'd with  $\text{H}_2\text{O}$  vapor. They give reliable results only over a rather narrow range in the neighborhood of the point at which they were standardized (generally around 40%). At 80% the readings of these instruments are discordant, the error easily reaching up to 20%.

**A. Papineau-Couture**  
Coal dust and rock dust as constituents of air. M. Stipanac. *Berg- und Hütten-männisches Jahrbuch*, 83, 77-92 (1935).—Three groups of dust are distinguished: (1) coarse dispersion, particles larger than 0.1 micron, (2) colloids, 0.1 micron to 1 millimicron, (3) mol dispersoids, particles smaller than 1 millimicron. The existence of dust, its nature and detn. in different mines are described, sizes, fineness, wt and structure of the particles were detd. The capacity to fly is detd. by the ratio of vol. to surface. The danger of explosion increases also with increasing surface ratio. The behavior of colloidal dusts in the ventilation of mines and the distribution of dust over the section were detd. The nature of coal-dust explosions is discussed as a colloid-chem and colloid-phys phenomenon; the effect of rock-dusting as preventive means for explosions is described. M. H.

Use of cleaners in the dairy plant. H. J. Barnum, P. S. Lucas and Bruce Hartsuch. *Mich. Agr. Expt. Sta., Special Bull. No. 262*, 24 pp (1935).—On the basis of their compn. cleaners are classified into modified or neutral soda, soda ash, special alkalis,  $\text{Na}_2\text{PO}_4$  and colloidal materials. Some of the cleaners were highly buffered and therefore resistant to reduction in their cleansing ability. The modified sodas were most stable in buffer action; next were soda ash and colloidal materials. The special alkalis were most caustic,  $\text{Na}_2\text{PO}_4$  was second, soda ash and colloidal cleaners were equal, and modified sodas least caustic. The emulsifying power was detd. with butter fat. In order of decreasing emulsifying power the cleaners ranked special alkalis, soda ash, colloidal cleaners, modified sodas and  $\text{Na}_2\text{PO}_4$ . Soda ash was most efficient as a water softener; next were colloidal cleaners, modified sodas, special alkalis and  $\text{Na}_2\text{PO}_4$ . Al, Cu and tinned steel were severely scratched by the volcanic ash content of some of the cleaners. Chrome-Ni steel was not affected. Special alkalis were most severe in their tarnishing action and corrosiveness; next were  $\text{Na}_2\text{PO}_4$ , soda ash and colloidal cleaner. Tinned steel and Al were corroded most severely. A table of properties of the cleaners is given. C. R. Fellers

Fumigation of ships by Clayton gas in Egypt. J. Gil-mour. *Bull. mens offic. intern. hyg. pub.* 26, 271-82 (1934), U. S. Pub. Health Eng. Abstracts 15, P. 2 (Aug. 10, 1935).—Expts. on ship fumigation showed that with a concn. of 1% Clayton gas for 3 hrs all rats were killed. At a concn. of 2% with exposure for 2 hrs. all rats were dead within 1 hr. It was found impossible to obtain theoretical calcd. concns. of gas in ship compartments, even if all dead spaces were opened. It is believed that for complete elimination of the rats the pumping of the  $\text{SO}_2$  must be continued into the ship until a concn. of 2% is attained in the upper parts of the compartments for at least 2 hrs. A new buret for the estn. of gas concns. correct to approx. 0.1% is described. C. R. Fellers

Catalytic hydrogenation of phenolic oil of low-temp. tar [manuf. of synthetic resins] (Andó) 21. Varnishes and plastic compns. (Fr. pat. 787,690) 26. Purifying oils, waxes and synthetic resins (Brit. pat. 434,672) 22. Recovery of  $\text{H}_2\text{SO}_4$  and resins from acid tar produced in purifying benzene hydrocarbons with  $\text{H}_2\text{SO}_4$  (U. S. pat. 2,026,450) 18. Oxygenated org. compds. from hydrocarbons [use as plastics] (U. S. pat. 2,024,954) 10. Hydrogenation of tertiary alkyl-phenols to form hydroaromatic alics [use as plasticizers for coating and molding compns.] (U. S. pat. 2,026,668) 10. Thiocetraphosphates [as a detergent] (U. S. pat. 2,025,503) 18

Chemical and physicochemical treatments. I. G. Far-benaid. A-G. Fr. 786,128, Aug. 27, 1935. The reaction vessels are submitted to very rapid mech. oscillations whereby increased speed of reaction is obtained.

Plastic masses. Studien- und Verwertungs-G. m. b. H. (Franz Fischer and Otto Horn, inventors). Ger. 618,500, Sept. 16, 1935 (Cl. 12a 26 02). Coal-like fuel is submitted to the action of  $\text{PhOH}$  or phenolic mixts. at temps. below the decompn. temp. of coal to give a plastic mass. The reaction may take place under pressure and in the presence of a condensing agent. Thus, lignite is heated to  $180^\circ$  with cresol to give a plastic mass useful as a filling, softening or flowing agent. Cl. C. A. 30, 185.

Plastic material. Ange. Lamguasco. Fr. 787,142, Sept. 17, 1935. A transparent and noninflammable material is prep'd by dissolving a metal perchloride, chloride or oxide (1-10 g.) in pure white acetone (500-1000 cc.), adding cellulose (5-10 g.), mixing in the cold and adding carefully cellulose tetracetate (5-50 g.) and continuing to stir until complete soln. The product may be used as a substitute for glass or for making cinematographic films.

Plastic compositions, lacquers, etc. Deutsche Hydrierwerke A-G. Brit. 435,058, Sept. 13, 1935. Varnishes, filaments, films, etc., from cellulose esters and ethers and natural and synthetic resins contain as solvent or softening agent a monoether of glycerol or polyglycerol with an aliphatic alc. contg. not less than 8 C atoms or a cycloaliphatic alc. or an ester obtained from such an ether by esterifying free OH groups with org. acids. Among examples, (1) cellulose nitrate moistened with methylated spirit is dissolved in  $\text{AcOBU}$  contg. dodecyl glycerol ether acetate, Albertol resin and  $\text{PhMe}$  to form a lacquer and (2) nitrocellulose, diglycerol octadecyl ether and  $\text{Ph}_3\text{PO}$  are worked in a kneading machine and the mass is rolled into sheets.

Plastic composition for doll faces, etc. John O. Baker (to Sweets Laboratories, Inc.). U. S. 2,024,124, Dec. 10, 1935. Roll-milled rubber which has been broken down to a considerable degree is used with a resin of high stability such as coumarone resin, an oil which is nonvolatile at room temps. and of high chem. stability such as a mineral oil, and a natural chloride, blended together to a substantially homogeneous mass.

Plastic composition suitable for coating walls. Michael J. Batelja. U. S. 2,025,369, Dec. 24, 1935. Ground oats are mixed with a fibrous filler such as wood, paper or rag pulp, water, a substance such as a cement having the property of setting after being wetted,  $\text{PbO}$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$  and a waterproofing substance such as linseed or China wood oil or varnish.

Sheets or films from plastic composition containing cellulose derivatives. Du Pont Viscoid Co. Ger. 621,048, Oct. 31, 1935 (Cl. 39a. 16). See Brit. 393,957 (C. A. 28, 321).

Plastic rubber derivative suitable for molding. Turey F. Ford (to B. F. Goodrich Co.). U. S. 2,024,987, Dec. 17, 1935. A mixt. such as may be formed of rubber, an aldehyde such as polymerized formaldehyde and a phenol such as  $\text{PhOH}$ , is heated (suitably to about  $135^\circ$ ) in the presence of a small proportion of an aromatic sulfonic acid such as *p*-phenolsulfonic acid.

Synthetic resin. Roy H. Kienle and Paul F. Schlingman (to General Elec. Co.). U. S. 2,025,538, Dec. 24, 1935.

phenol is treated with an excess of paraformaldehyde at a temp not exceeding 130° (suitably about 120°) in the presence of a relatively small proportion of sol alkyl resin catalyst of an acid number between about 140 and 210, until the free  $\text{CH}_2\text{O}$  content is less than about 6% and a liquid resinous product is produced and the product is cast and cured at a temp of 65-125°

**Synthetic resin** Roy H Kienle (to General Elec Co). U S 2,025,539, Dec 24. In forming articles such as gaskets, washers, etc., a totally cured resin formed from initial materials such as glycerol, phthalic anhydride, etc., is compounded with a partially cured resin which has a cure point of 20-25 sec at 150° and which is formed by the reaction of a phenol and an excess of aldehyde in the presence of a fusible, sol., acidic alkyl resin of an acid number about 200, and the compounded mixt is cured at 150-200° to a homogeneous, flexible product.

**Synthetic resins** Sydney L M Saunders Brit 434,850, Sept 10, 1935. These are prep'd by heating a phenol with a phenol di-alc in the presence of an acid catalyst and in the presence or absence of a vegetable oil, natural resins or their esters and, if desired, in the presence of an inert gas. The products may be used in the manuf of lacquers and varnishes. Among examples, (1) *p*-cresol diol (1) and cresylic acid, (2) PhOH and the di-alc's derived from cresylic acid and (3) I, diphenylethane and China wood oil, are heated in the presence of HCl. Cf. C A 30, 189<sup>1,2</sup>.

**Synthetic resins** Compagnie française pour l'exploitation des procédés Thomson-Houston Fr 787,562, Sept 25, 1935. Resins of the alkyl type are prep'd from polyhydric alcs and homologous derivs of phthalic acid or anhydride prep'd by synthesis from maleic acid or anhydride and comp'ds of diolefinic structure. Thus, a comp'd called euprene prep'd by passing  $\text{C}_2\text{H}_4$  over Cu powder at 200-60° under the influence of elec discharges is heated with maleic anhydride and the product is caused to react with glycerol. In the same way  $\text{C}_2\text{H}_4$ , isoprene and their partly polymerized comp'ds, China wood oil, and  $\alpha$ - and  $\beta$ -oleostearic acid are caused to react with maleic acid or anhydride and then condensed with glycerol, glycol, pentaerythritol, mannitol, polyglycerols, etc.

**Synthetic resins** I G Farbenand A-G 787,374, Sept 21, 1935. Solid, pulverizable, fusible and transparent resins are obtained by submitting to the polymerizing action of catalysts, of the "Friedel-Crafts" type, liquid hydrocarbons obtained by cracking in the vaporized state the products rich in H from the destructive hydrogenation of carbonaceous materials.

**Synthetic resin compositions** Irvin W Humphrey (to Hercules Powder Co) U S 2,025,947, Dec 31. An ester of abietic acid, such as the ethyl, methyl or glycerol ester is treated with maleic anhydride.

**Resinous compositions suitable for use in lacquers, etc** Ernest A Rodman (to E I du Pont de Nemours & Co) U S 2,025,612, Dec 24. See Can 351,668 (C A 29, 666<sup>9</sup>).

**Synthesizing resins with aluminum chloride** Stewart C Fulton and John Kunc (to Standard Oil Development Co) U S 2,025,738, Dec 31. A cracked petroleum tar or distillate from such a tar is agitated with a polymerizing agent such as  $\text{AlCl}_3$  at a suitable polymerizing temp (which may be about 120-180°) and the metal halide sludge is allowed to settle and is drawn off, the sludge free reaction product is treated with  $\text{H}_2\text{SO}_4$ , the acid sludge is removed and oily constituents are dist'd from the remaining product to obtain a resin as dist'n residue. App is described.

**Paracoumarone resins** Karl H Engel (to Barrett Co) U S 2,024,568, Dec 17. Light-colored resins are produced from oil contg polymerizable constituents of the styrene-coumarone indene type by effecting polymerization with a mixt of  $\text{H}_2\text{BO}_3$  and  $\text{H}_2\text{SO}_4$ .

**Arylated esters and synthetic resinous compositions** Merlin M Brubaker (to E I du Pont de Nemours & Co) U S 2,025,642, Dec 24. Oxidation-resistant products suitable for use in coating comp's and for other purposes are formed by dissolving materials such as abietic

acid, ester gum, alkali-refined linseed oil, or cottonseed oil in a solvent such as  $\text{C}_6\text{H}_6$ , toluene or xylol and heating the soln with anhyd  $\text{AlCl}_3$  which is gradually added. A product thus formed may be further treated with a polyhydric alc. and a polycarboxylic acid. Numerous details and examples are given.

**Resinous condensation products** Ernst Elbel and Fritz Seebach (to Bakelite Corp) U S 2,024,212, Dec 17. A heat hardenable resinous phenol-aldehyde condensation product in a state of advancement characterized by soly. in acetone and infusibility but softening on heating is formed and then is ppt'd from soln in a solvent such as cyclohexanol by adding a diluent such as ligroin. The product is suitable for various uses.

**Pressing fibrous masses impregnated with synthetic resins** Allgemeine Electricitäts Gesellschaft, Ger 618,708, Sept 13, 1935 (Cl 39a 19 06).

**Electrical insulating tubes** Gordon R Langley (to General Elec Co) U S 2,025,540, Dec 24. Paper having an inner metallic coating is wound on a mandrel and over this there is wound paper coated with fused phenolic condensation product, and the materials are heat cured.

**Electrical insulating material** Guard T. Kohman (to Bell Telephone Laboratories, Inc) U S 2,026,316, Dec 31. A cellulosic material to be used as insulation, such as paper, is exposed to water or alc vapor at temps of 50-150° and then immediately immersed in a bath of chlorinated naphthalene and the moisture is removed from the material at an elevated temp.

**Electric conductors** Henry Dreyfus Fr 787,456, Sept. 23, 1935. The conductors are insulated by a covering of thread made from a cellulose deriv (acetate) contg staple fibers.

**Heat insulation suitable for use on underground steam conduits** Earle R. Williams (to Johns-Manville Corp) U S 2,023,986, Dec 10. Juxtaposed sheets are used of water-permeable, water-resistant insulation such as asbestos paper and a water-permeable, wrapping and retaining material of good wet strength and which includes a wire reinforced woven asbestos fabric.

**Heat- and sound-insulating material** Soc anon française Eternit, Fr 787,396, Sept. 21, 1935. A binder such as cement is emulsified to give it a cellular texture and fibrous material, preferably asbestos, in a floccular state is incorporated therewith by appropriate mech. treatment.

**Adhesive** Frank H Shoals (to Modern Panels, Inc) U S 2,025,180, Dec 24. An adhesive suitable for use with veneers is prep'd by hydrolyzing starch, controlling the viscosity of the starch during the hydrolyzing stage by adding a mixt of borax and soda, dilg with water, adding an alk latex-coagulating-control agent and allowing the mass to cool to room temp., adding latex soln and gently agitating during the addn of the latex.

**Wetting agents** etc Imperial Chemical Industries Ltd, Alfred W Baldwin and Henry A Pigott Brit 435,039, Sept 12, 1935. Sulfuric esters having wetting, cleansing, foaming and emulsifying properties are prep'd by treating with an addn product of  $\text{SO}_2$  and a tertiary amine the aliphatic hydroxysulfides obtained by causing alkyl mercaptans having 10-30 C atoms to react with halohydrins. Among examples, cetyl mercaptan is caused to react with ethylenechlorohydrin in the presence of MeONa and the 2-hydroxyethylcetyl sulfide produced is sulfonated by means of Na pyrosulfate and pyridine.

**Wetting agents, etc** Deutsche Hydrierwerke A-G Brit 435,290, Sept 18, 1935. Products having wetting, washing, emulsifying, foaming and dispersing properties are prep'd by (a) treating glycols having at least 6 C atoms with H halide, converting 1 of the ester groups so introduced into a sulfonic group by means of a sulfite or a sulfide followed by oxidation, and causing the other ester group to react with  $\text{NH}_3$  or primary or secondary amines, (b) treating glycols having at least 6 C atoms with polybasic O-contg mineral acids and causing 1 of the ester groups so introduced to react with  $\text{NH}_3$  or primary or secondary amines including heterocyclic bases, e.g., piperidine, (c) esterifying only 1 OH of the higher glycols

by means of H halide or polybasic O-contg. mineral acids, causing the ester group to react with  $\text{NH}_3$  or primary or secondary amines and esterifying the remaining OH with a polybasic O-contg. mineral acid, (d) treating the higher glycols as in (c), but instead of esterifying with polybasic O-contg. mineral acids in the final step, esterifying with H halide and treating with sulfites or with sulfides followed by oxidation. Among examples, 1,6-hexanediol or triolein alk. is esterified with  $\text{HCl}$ , treated with  $\text{FeNH}$  and the resulting amuro-alk. converted into the sulfonic ester. Cf. C. A. 29, 1179<sup>1</sup>.

**Wetting agents.** I. G. Farbenindustrie A.-G. Brit. 433,885, Sept. 10, 1935. Products having wetting, washing and dispersing properties are obtained by sulfonating, neutralizing and extracting with organic solvents, comprising fatty acids and alcohols having a carbonyl value of 10 or below, that are derived from nonaromatic hydrocarbons containing more than 12 C atoms by (a) oxidation of said hydrocarbons in the liquid phase and subsequent hydrogenation until the carbonyl value is reduced to 10 or below, or (b) oxidation in the liquid phase in the presence of  $\text{H}_2\text{O}_2$ ,  $\text{AcOH}$ ,  $\text{EtOH}$  or their anhydrides. Among examples, a middle oil obtained by the hydrogenation of lignite is oxidized in the presence of Mn stearate and the carbonyl value is reduced to 8 by hydrogenation in the presence of a Co catalyst, the product is dissolved in benzene, treated with  $\text{ClHSO}_4$  in  $\text{Li}_2\text{O}$ , neutralized with aq.  $\text{Na}_2\text{CO}_3$ , the benzene soln. sepd. and the soap soln. extrd. with benzene. Cf. C. A. 29, 697<sup>1</sup>.

**Wetting and other agents.** I. G. Farbenindustrie A.-G. Fr. 786,334, Aug. 31, 1935. Auxiliary agents for the textile and allied industries consist of compounds the mol. of which contains at least 1 N atom, 2 or more radicals fixed directly to the N and in which the N exists in a state of oxidation more complete than in "free" derivatives of  $\text{NH}_3$ , and which contains at least one aliphatic radical of at least 8 C atoms. Such compounds include methyldecylhydroxylamine (by heating dodecylmonooxethylamine in  $\text{EtOH}$  with  $\text{H}_2\text{O}_2$ ) and compounds obtained by oxidizing dimethyldecylamine, cetyldecylamine, octadecylamine, dodecylamine, dodecylamine of  $\beta$ -methylaminoacetic acid and  $\alpha$ -diethylthioethylenediamine in the same way.

**Wetting and other agents.** Moritz Treiberer. Fr. 786,391, Sept. 2, 1935. Auxiliary products for the textile industry are obtained by disaggregating albuminous and albuminoid substances to a degree beyond that of protalbumin and hyaline acid but not reaching that of amino acids.

**Wetting and other agents.** I. G. Farbenindustrie A.-G. Fr. 787,305, Sept. 24, 1935. Primary or secondary unsatd. aliphatic amines of high mol. wt. are treated with acylating and sulfonating agents in any order. Examples are given in the prepn. of sulfonated benzoyldecylamine and butyric methyldecylamide.

**Esters of hydroxy carboxylic acids (wetting, foaming, emulsifying and detergent agents).** Benjamin R. Harris. U. S. 2,023,984, Dec. 31, 1935. Esters such as mono- and palmate, the stearic acid ester of tartaric acid, etc., are prepd. by the reaction of an acid such as succinic acid or tartaric acid with an acid halide such as palmityl chloride or stearyl chloride in a vehicle such as dry pyridine. Various examples are given.

**Molding composition suitable for making various articles.** Carlo Stresino (to A. O. Smith Corp.). U. S. 2,028,107, Dec. 31, 1935. Wood which may be in the form of chips or shavings is impregnated with sulfite liquor and the impregnated material is heated with phenyl chloride to effect chem. reaction.

**Molding materials.** Edward Weatherford (to The Borden Co.). Brit. 434,573, Sept. 4, 1935. In molding a foundry core or other article from sand or the like mixed with a binding material, the latter comprises milk, buttermilk or whey, which may be used with other binders, e. g., core oil. The milk, etc., may be in condensed or powder form and used with  $\text{H}_2\text{O}$ .

**Molding objects from polystyrene.** Siemens-Schuckertwerke A.-G. Fr. 787,318, Sept. 20, 1935. Rough-shaped hollow objects are pressed to their final shape by fluid under

pressure at the same time as the walls are stretched so that their fragility is reduced and their flexibility increased.

**Inlaid designs on molded articles such as molded table tops.** Carlo Stresino (to A. O. Smith Corp.). U. S. 2,028,105, Dec. 31, 1935. A varnish carrying a pigment is applied to a sheet or resin-impregnated material, the varnished sheet is placed in contact with a pattern formed of resin-impregnated material of different color than the varnish to block out all portions of the designs in which the pigment is not to show, and the varnished sheet and pattern of resin-impregnated material are united by the action of heat and pressure, and are used so as to provide an inlaid surface.

**Artificial leather.** Vereinigte Schmirrenwerke Vosseler & Bonger. Fr. 786,049, Aug. 24, 1935. Fibrous material is impregnated with a liquid containing rubber latex, the material being alternately compressed and allowed to expand until completely and uniformly impregnated. App. is described.

**Impregnated fibrous articles or sheets such as leather substitutes.** George A. Richter and Milton O. Schur (to Brown Co.). U. S. 2,024,600, Dec. 17, 1935. A material such as paper has its pores and interstices partially impregnated with regenerated cellulose and is further impregnated with a solid, fluxed water-repellent material such as an asphalt or rubber compound, etc.

**Apparatus for making leather substitutes by treating fabrics with suitable compositions.** James G. Grower. U. S. 2,024,284, Dec. 17, 1935. Various structural, mech. and operative details.

**Condensation products of ureas, aldehydes, etc.** Leon Lilienfeld. U. S. 2,024,972, Dec. 17, 1935. Condensation products which are suitable for LMs, etc., are produced by heating to 40-60° a mixt. comprising urea, thiourea or dicyanamide, and aldehyde such as  $\text{CH}_3\text{CHO}$  and a halogen deriv. of a polyhydric alk. such as  $\alpha$ -dichlorohydrin. Numerous examples are given.

**Cleansing composition.** Gilbert F. Lavery. Fr. 786,241, Aug. 29, 1935. The compn. contains river tuff (35, white soap (72% of oil) 25 and  $\text{H}_2\text{O}$  10%.

**Cleansing composition for internal-combustion engines.** International Hydrocarbon Patents Co. Ltd. Fr. 787,321, Sept. 24, 1935. Asphalt and resinous materials are removed by destructively hydrogenated solvent naphthas. These are prepd. by treating gas-oil or kerosene, or preferably a strongly cracked fraction of gas-oil or kerosene or an ext. therefrom by means of liquid  $\text{SO}_2$ ,  $\text{PhOH}$ ,  $\text{PhNH}_2$  or 2-furaldehyde, with H at above 480° in the vapor phase. The products, called "hydrocarbons," b. between 215° and 258° may be mixed with other solvent oil, preferably a lubricating oil of relatively high b. p.

**Detergents.** Johann Bertram and Erich Schieweck. Brit. 434,846, Sept. 10, 1935. See Ger. 616,443 (C. A. 29, 73319).

**Washing agents.** I. G. Farbenindustrie A.-G. Fr. 787,819, Sept. 20, 1935. Washing agents contain water-sol. salts of aliphatic carboxylic acids of high mol. wt. in which at least 1 atom of N is situated between the carboxylic group and the aliphatic radical and which are rendered neutral or acid by adding compounds of acid action, e. g., borax, citric, tartaric or oxalic acid or  $\text{NaHSO}_4$ . The acids contg. N include octadecylaminoacetic acids, oleylsuccinate and the saponates of coco-oil fat acid.

**Washing and rinsing liquids.** Rutgerswerke A.-G. Fr. 787,546, Sept. 24, 1935. The liquids contain alkylated naphthalene sulfonic acids or sulfonated fat alcohols, or their salts in admixt. with acid salts or having an acid reaction, particularly F compounds, e. g.,  $\text{NaHF}_2$ ,  $\text{KHF}_2$ ,  $\text{NH}_4\text{HF}_2$ , and  $(\text{NH}_4)_2\text{S}_2\text{F}_8$ .

**Cleaning and polishing composition.** Alfred Herrmann. Fr. 787,223, Sept. 27, 1935. The compn. contains, e. g., beeswax 1, paraffin 2.5, turpentine oil 6, sawdust 3, sand 10, yellow ochre 0.5 and vinegar 0.02 kg.

**Foaming agents.** I. G. Farbenindustrie A.-G. Brit. 435,331, Sept. 19, 1935. Finely distributed dust, especially rock dust in mines, is prepd. by means of foam prepd. by introducing air or other gases in fine division into aq. dispersions containing wetting agents and solvents having the

formula  $R'OCH_2(CH_2OCH_2)_nCH_2OR$ , in which  $R'$  is H or alkyl,  $R$  is H, alkyl, aralkyl or aryl and  $n$  is a whole number, if  $R'$  and  $R$  are H, zero or a whole number. Urea, thiourea, degraded albuminous compounds, etc., favoring foam-formation, may be present. In an example, oleic acid-*N*-methylaurine, contg. about 30% of inorg. salts, diethyleneglycolmonoethyl ether and urea or thiourea are used for the prepn of foam.

**Electromagnetic metal** I G Farbenind. A.-G. Fr. 787,781, Sept. 28, 1935. Magnetic cores, etc., are made from the powder obtained by reducing the powd. metal oxide coming from the calcination of the corresponding carbonyls.

**Printing plate** Edgar J Wright. U. S. 2,024,840, Dec. 17. A matrix of a printing surface is rotated in an inclined plane and during rotation its surface is sprayed with liquid material such as molten metal capable of hardening until a substantial body of type material is built up, and a backing is subsequently applied to the deposited material.

**Polychromatic printing plate** Serge Tchechounin. U. S. 2,025,559, Dec. 24. A printing plate for polychromatic reproduction comprises an ale-sol. base such as gum-lac, soap, a fatty material such as linseed oil, naphthalene, HOAc and a pigment, etc. Cf. C. A. 29, 4107<sup>3</sup>.

**Etching metal printing forms** Robert A. Zarse. U. S. 2,026,603, Jan. 7. A surface to be etched such as one of Cu carrying an enamel is etched by immersion in an aq. soln. contg. NaCl and Fe chloride.

**Magnetic cores** I G Farbenind. A.-G. Fr. 787,557, Sept. 25, 1935. The metal powders, particularly those obtained by pyrolytic decomposition of the carbonyls, are coated by an insulator comprising polystyrene, either alone or in conjunction with other insulators, particularly gum-lac or mixed polymerization products of vinyl chloride and acrylic esters.

**Protection of pipe joints such as those of pipe lines in the ground** Robert G Roberts (to Barrett Co.). U. S. 2,024,602, Dec. 17. A preformed coupler housing of substantially waterproof satd fibrous material such as pitch-satd felt is mounted around a pipe coupling and serves to retain molten protective material such as pitch during its application and until hardened.

**Porous products from liquid slags** Carl H. Schol. U. S. 2,024,308, Dec. 17. A porous product suitable for insulation, etc., is obtained by applying a thin layer of a swelling liquid such as water to a wide smooth even supporting surface and spreading out the hot fluid melt such as a liquid slag in a thin layer on the layer of swelling liquid. App. is described.

**Transparent sheets or coatings** Kraft-Phoenix Cheese Corp. Brit. 434,993, Sept. 12, 1935. Films are prepd. by bringing casein into aq. soln. of such high concn. e. g., 15%, and viscosity that the mineral salts and other impurities become insol. therein, filtering, treating with a deficiency of a setting agent, e. g.,  $CH_3O$ ,  $(CH_3)_2N$ , trioxymethylene, aldehydes, Cr salts, tannic acid,  $Me_2CO$ , coating a base with the soln. and drying the coating. The film produced may be rendered moisture proof by coating with a cellulose lacquer.

**Corrosion-preventing coatings** Chemische Fabrik R. Baumbach A.-G. Brit. 435,003, Sept. 12, 1935. See Fr. 779,878 (C. A. 29, 5549<sup>9</sup>).

**Carrier belt for casting transparent sheet material such as gelatin or casein sheets** Edouard M. Kratz (to Marbo Products Corp.). U. S. 2,024,826, Dec. 17. A laminated fibrous web carries a smooth flexible nitrocellulose coating on its top surface and a pigmented cellulose ester coating on its back to prevent curling.

**Metal cement** Nelson W. Larmore (to Clifford W. Frederick). U. S. 2,026,475, Dec. 31. Al powder 2, is mixed with hydraulic cement 12, whitening 24, iron filings 80 and lacquer 28 parts.

**Oil-proofing materials such as paper containers** James K. Delano. U. S. 2,025,729, Dec. 31. As an oil-proofing compound there is used a mixt. of salts of glyceric acid and salts of homologous acids derived from ext. of glycer-

rhiza, such as the K, Na, NH<sub>4</sub> and Ca salts, in admixt. with an albuminoid material such as a glue.

**Luminescent paper** Michel A. A. Binetti. Fr. 786,474, Sept. 2, 1935. Paper is made impermeable by a soln. of rubber, and then given a coat of luminous paint obtained by mixing a luminous sulfide (of Sr, Zn or Cd) in a soln. of rubber. The luminous layer is protected by a thin film of rubber.

**Hectograph copying pads** Wm B. Whitmore (to Ditto, Inc.) U. S. 2,024,408, Dec. 17. A gelatinous mass formed of a gelatinous substance such as gelatin together with water and glycerol (with a major proportion of glycerol) is treated with a minute proportion of a light-sensitive tanning agent such as  $(NH_4)_2Cr_2O_7$  and subjected to the action of concd. ultraviolet light to effect tanning.

**Artificial sponge** I G Farbenindustrie A.-G. Brit. 435,128, Sept. 16, 1935. A viscose sponge mass contg. a fusible or sol. pore-forming substance is applied to a support consisting of or contg. cellulose, e. g., paper, fabrics, wood, so as to become united to the cellulose and is then treated to produce the viscose sponge. In this manner wooden articles may be enwrapped in, or lined with, the sponge. The process may be applied to the production of polishing cloths, window rubbers, mops with wooden handles, spongy filler cloths, floor coverings, in-soles, etc.

**Vinyl derivatives** Georges E. Zelger. Fr. 780,495, Sept. 3, 1935. In condensing polyvinyl alc. with  $CH_3O$  in the presence of a catalyst to obtain products which are entirely sol. in appropriate org. solvents and given solns. of a high degree of viscosity, the max. dose of catalyst giving a product still insol. in the solvent chosen is detd. experimentally, and this amt. is used with a slight excess. The excess is below 1% if HCl is the catalyst and if ordinary temp. is used. Cf. C. A. 29, 539<sup>9</sup>.

**Hardening gelatin and other plates** Telefunken Ges. für drahtlose Telegraphie m. b. H. Ger. 618,822, Sept. 16, 1935 (Cl. 42g 15 01). Gelatin, casein and similar hardening plates of albumin compounds, used for gramophone records, etc., are hardened by enclosing them in a container with a water-sizing reagent and  $CH_3O$ -generating reagent, the latter being glowing Pt and MeOH.

**Molded friction material such as brake lining** Joseph N. Kuzmick (to Manhattan Rubber Mfg. Division of Raybestos-Manhattan, Inc.). U. S. 2,025,951, Dec. 31. Dry fluffy asbestos fibers are mixed with a potentially reactive synthetic resin and a plasticizer such as toluene-sulfonamide to produce a relatively dry mixt. which is then subjected to heat and pressure to render the synthetic resin infusible and produce a molded product of the desired shape.

**Braided material suitable for brake lining** Philip D. Cannon (to Johns-Manville Corp.). U. S. 2,025,038, Dec. 24. Strands of reinforced asbestos yarn are provided with a continuous coating of viscous impregnating material such as a rubber compn. and are formed into a braid (various details of app. and operation being described). U. S. 2,025,039 relates to an automobile brake lining material or the like formed of strands of wire reinforced asbestos yarn and other material such as a rubber compn.

**Friction material suitable for brake lining** Fred L. Hess (to Johns-Manville Corp.). U. S. 2,025,052, Dec. 24. Reinforced asbestos yarns are braided into a tube of loose fabrication; a semiplastic friction binder of hardenable character such as a described rubber compn. is placed within the tube, the tube and binder are pressed into a flat band, and the binder is hardened *in situ*. App. is described.

**Gritty material for treating ice-coated roads** Wm R. Collings (to Dow Chemical Co.). U. S. 2,026,121, Dec. 31. Moist sand at least 80% of which will pass through a No. 10 sieve is mixed with about 6-36% of comminuted partially dehydrated  $CaCl_2$  contg. not more than 23% of water and which will pass through a No. 40 sieve.

**Artificial wood** Théophile Gerber. Fr. 786,282, Aug. 30, 1935. Glue,  $NH_4Cl$ , resorcinol, salicylic acid,  $NH_4OAc$  or other mineral or vegetable tanning agents are mixed with sawdust, wood fibers or cork waste and plaster. The

whole is mixed and submitted in an autoclave to the action of  $\text{Cl}_2\text{O}$  gas

**Pitch composition.** Max Singher Tr. 786,216, Aug. 29, 1935 Bitumen or pitch from petroleum or coal is mixed with oocerite to form a paste very suitable for fixing wood parquets to cement and which may be mixed with cork grains, sawdust, peat or fibers for various uses

**Composition for cleaning the hands** Roman Kornmann Austrian 143,657, Nov. 25, 1935 (Cl. 23b) A mixt. of calcined soda, soft soap, turpentine and water is heated to below the b. p. mixed with sawdust, cooled, dried and mixed with pumice powder

**Composite dental plates** Stefan Loos U. S. 2,025,750, Dec. 31 Various operative details are described for making composite plates of a plurality of layers of metals such as Cu, Ag and Au

**Cellulose acetate projection screens** David F. Newman (to Trans-Lux Daylight Picture Screen Corp.) U. S. 2,024,382, Dec. 17 A sheet formed mainly of cellulose acetate material contains an agent such as triphenyl phosphate for imparting flexibility and a light-diffusing agent such as ZnO and cobalt blue proportioned so that a substantial amt. of light traverses the screen when it is included in a rear projection system Cl. C. A. 29, 258<sup>7</sup>

**Shoe-bottom filler composition** Harry H. Beckwith (to Beckwith Mfg. Co.) U. S. 2,025,432, Dec. 24 A compn. which is plastic and spreadable at room temp. comprises an aq. dispersion of rubber contg. rosin mixed with granulated cork particles which are coated with waterproofing material such as bitumen Cl. C. A. 29, 1544<sup>4</sup>

**Insecticide suitable for use on poultry perches** Arthur H. Teigen (to Pratt Food Co.) U. S. 2,024,897, Dec. 17. A nonvolatile, sticky, adherent fluid contains free nicotine and corn oil

**Material for tobacco-pipe bowls** Michael A. Dorian and Leo Roon (to Roxahin Flexible Lacquer Co.) U. S. 2,025,811, Dec. 31. A moisture-permeable tobacco-pipe bowl such as one formed of wood carries on its exterior surface a coating of a heat-resisting, nonthermoplastic compn., permeable to water vapor, which may be formed from nitrocellulose, ethylcellulose and specified solvents.

**Colloidal compositions.** Hubert Degude Fr. 787,267, Sept. 19, 1935. Animal and vegetable waxes such as beeswax, Carnauba wax and candelilla wax are treated with  $\text{Ba}(\text{OH})_2$  to obtain compds. which when heated with turpentine, white spirit or other mineral oil form colloidal sols. which may advantageously be used for making polishes, etc. The colloidal gel obtained by heating Ba soaps with  $\text{CCl}_4$  is used as a fire extinguisher Ba stearate, obtained by the action of  $\text{Ba}(\text{OH})_2$  or stearins, is used in making varnishes and is added to stearins, paraffins, waxes, etc., to increase their m. p.

**Fire extinguishers** H. Th. Böhme A.-G. Brit. 434,856, Sept. 10, 1935 See Fr. 779,096 (C. A. 29, 4558<sup>9</sup>)

**Fire-resisting hydrocarbonaceous material suitable for impregnating fibrous materials** James H. Young (to H. H. Robertson Co.) U. S. 2,025,929, Dec. 31. A normally nonfluid hydrocarbonaceous material such as asphalt, Montan wax, etc., is mixed with a sufficient proportion of chlorinated biphenyl to render the compn. fire-resisting

## 14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

**Operation of the Athens water supply.** R. W. Gausmann. *J. Am. Water Works Assoc.* 27, 1458-76(1935).—The bacteriological quality is influenced by rainfall. Treatment at the plant includes aeration, coagulation, sedimentation, filtration and chlorination. The water is not corrosive. D. K. French

**Water supply of the city Schleswig for 400 years.** E. Peter. *Gas u. Wasserfach* 78, 849-55(1935).—History, geology and present status are presented R. W. R.

**Chemical character of ground water in Pennsylvania.** W. D. Collins. *J. Pa. Water Works Assoc.* 6, 81-8(1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 74(July 13, 1935).—The analyses of 500 Pa. ground waters are discussed. Ca and Mg are responsible for the hardness. The Ca exceeds in brines assoc. with oil but the opposite is true in sea water. The Fe content is av., while the hardness of the waters as a whole is less than av. for other states C. R. Fellers

**Municipal ground water supplies of Ontario.** W. B. Redfern. *J. Am. Water Works Assoc.* 27, 1533-46(1935).—See C. A. 29, 5057<sup>4</sup>. D. K. French

**Improved method for rating and sampling wells.** Paul F. Howard. *Water Works and Sewerage* 82, 422-3(1935).—App. and method are described for detg. the quantity of water delivered at the normal operating rate of the well. Samples for water analysis are taken at the pt. of discharge. G. H. Young

**Some first-year experiences at Easton, Pa.** R. W. Haywood, Jr. *J. Pa. Water Works Assoc.* 6, 68-77(1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 74(July 13, 1935).—A summer musty taste was not removed by 6 p. m. of C, but prechlorination at 4 p. m. was entirely effective. Changes in C dosage did not affect the Cl. conen. reaching the filter but did affect that in the effluent. No treatment eliminated taste at the time the ice broke up, but filtration through a granular C filter was effective on a lab. scale. With no change in the alk., the pH may rise in the summer months from 7.4 at day-break to more than 9.0 in midafternoon owing to algal attack on carbonates and bicarbonates. This action is reversed during the night. C. R. Fellers

**Coagulating the water of the Delaware River at Easton, Pennsylvania.** R. W. Haywood, Jr. *J. Am. Water Works Assoc.* 27, 1572-80(1935).—Reactions and formulas governing coagulation are accepted. Flocculation conformed with generally accepted rules controlling coagulation. Wide variations in pH of the river water had no measurable effect on coagulation requirements. Alky., turbidity and org. load were the 3 main factors in coagulation, and are stated in order of importance. D. K. French

**Chemical and physicochemical analysis of the mineral waters of Valleverde (Abruzzo).** Domenico Marotta and Ermenegildo Sorrentino. *Ann. chim. applicata* 25, 324-50(1935).—Complete analyses of these waters are given. They have a therapeutic value in treatment of gout and uremia, probably on account of the small amts. of Ag present. A. W. Conti

**Detection of heavy water in French and some other (Norway, Sweden) mineral waters.** Klaus Hansen, Erling Rustung and Johan Hvedung. *J. pharm. chim.* 21, 538-41(1935); cf. C. A. 29, 5594<sup>4</sup>; Bonhoeffer, C. A. 28, 967<sup>4</sup>, Richards and Shipley, C. A. 6, 2219.—The  $\text{D}_2\text{O}$  concn. of 7 samples examd. did not differ from that of ordinary  $\text{H}_2\text{O}$ . S. Waldbott

**Milwaukee's water-purification plant.** L. R. Howson. *Water Works and Sewerage* 82, 403-10(1935).—A description of plant lay-out and equipment. G. H. Y.

**Decalcification of drinking water by filtration processes.** P. Hirsch. *Wasser* 17, 243, 255(1933); *Gesundh. Ing.* 57, 171(1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 32(Apr. 6, 1935).—The use of  $\text{MgCO}_3$  (magnesite calcined at about 1000°) for the decalcification of water is superior to that of marble, which can only affect decalcification up to the lime- $\text{CO}_2$  equil. Magnesite by treatment with water contg. lime acquires the ability of decalcifying water beyond this equil. C. R. Fellers

**Lime demand of acid water.** Lewis V. Carpenter and Geo. R. Lyle. *J. Pa. Water Works Assoc.* 6, 47-8(1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 64(June 29, 1935).—The simplest method for detg. lime demands of water is to run the methyl orange detn. for free mineral

acids and the Fe content by titration with  $\text{KMnO}_4$ . In making the Fe detn., 20 cc of 1 to 3  $\text{H}_2\text{SO}_4$  is added to all waters to clear up the soln., e. g., sufficient lime is added to ppt the Fe in the  $\text{Fe}^{+++}$  state and neutralize the mineral acids to methylorange. The av. of 14 waters with acidities of 40-865 p. m. required an av. of 0.000562 g. of  $\text{CaO}$  per l. of water for each p. m. of acidity, which is equal to 4.67 lb. per million gal. C. R. Fellers

The practical determination of the hydrogen ion concentration of water. E. Naumann and Käte Naumann. *Gas u. Wasserfach* 78, 901-3 (1935).—Colorimetric and potentiometric methods of detg the  $\text{pH}$  of water are reviewed, and a new portable potentiometric indicator is described. A new non fluid type of bridge is used between the 2 cells. A  $\text{KCl}$ ,  $\text{HCl}$ , quinhydrone electrode is used for comparison. R. W. Ryan

Preparation of water for the textile industry. removal of algae and sterilization. G. Ornstern. *Melland Textilber* 16, 760 (1935).—Addn of a soln of a  $\text{Cu}$  salt, or over-chlorination followed by the removal of the excess  $\text{Cl}$  with active charcoal is the method used to remove algae from textile industrial waters. F. H. Moser

A new method of chemical determination of small amounts of silver in water. J. Just and A. Szmolis. *Arch. Chem. Form.* 2, 170-5 (1935) (English summary); cf. J. Just and A. Szmolis, *C. A.* 29, 7538<sup>1</sup>.—Acidity 100 ml. or more of clear or filtered water to be tested with 5-10 drops of  $\text{HCl}$  (1:1) and add 2 drops of  $\text{KI}$  (1:10). Filter, after mixing, through a membrane filter of 1- $\mu$  porosity and 2.5 cm. diam. placed in a Kolkwitz filter app. for detn. of plankton. Moistened the ppt on the filter with 1.2 ml. of freshly prep'd  $\text{H}_2\text{S}$  water, suck dry the colored disk, dry it in air and compare with standard disks prep'd. of known  $\text{Ag}$  salt soln., e. g., 0, 5, 10, 15 and 20  $\gamma$   $\text{Ag}$  per l. Thus  $\text{Ag}$  contents down to 2.5  $\gamma$   $\text{Ag}$  in the sample can be det'd. By prep'g standards nearer each other and decreasing the area of the membrane greater accuracy can be obtained. The soly of  $\text{AgI}$  is about 0.0011 p. m. and is greatly reduced by the presence of  $\text{I}^-$  ions. The natural color of  $\text{H}_2\text{O}$  up to 40 p. m. Pt has no visible influence. J. Wiertelak

Base exchange in the subterranean vadose waters of Tunisia. M. Schoeller. *Bull. soc. géol. France* [5], 4, 389-420 (1934).—The base-exchange theory as applicable to the case under consideration is sketched, and by comparison of numerous analyses of the waters with those of the strata from which the waters are derived and through which they pass, is shown to explain the comp'n of the waters. Full details are given for 3 areas the vadose waters of which are characterized, resp., by their content of (1) alkaline earth chlorides, (2)  $\text{Na}_2\text{SO}_4$ , and (3)  $\text{NaHCO}_3$ . Lastly it is shown that the predominance of waters contg. much  $\text{Na}_2\text{SO}_4$  in the south, and of waters contg. much alkaline earth chloride in the north of Tunisia is explicable by the smaller rainfall and greater evap'n in the south as compared with the north. C. A. Silbert

Solid matter in boiler water foaming. III. Effect of calcium carbonate and magnesium hydroxide precipitated inside the boiler. C. W. Foulk and H. C. Brill. *Ind. Eng. Chem.* 27, 1430-5 (1935); cf. *C. A.* 28, 2814<sup>1</sup>.—Expts were conducted at a pressure of 17.6 kg./sq. cm. (250 lb./sq. in.) and at an evap'n rate of 450-500 cc./min. in an exptl. boiler identical with that described by Christman, Holmes and Thompson (cf. *C. A.* 25, 3748). Coarse granular  $\text{CaCO}_3$  pptd. by decomp'n of  $\text{Ca}(\text{HCO}_3)_2$  within the boiler reduced priming (i. e., the amt. of liquid carried over into the steam line). The fluffy ppt. of  $\text{CaCO}_3$  formed by pumping  $\text{CaCl}_2$  soln. into  $\text{Na}_2\text{CO}_3$  soln. increased priming, while the  $\text{CaCO}_3$  ppt. formed by pumping  $\text{Na}_2\text{CO}_3$  soln. into  $\text{CaCl}_2$  soln. had little effect. After contact for several hrs. with the hot boiler water, even the fluffy  $\text{CaCO}_3$  lost its ability to increase priming and became more granular.  $\text{Mg}(\text{OH})_2$  pptd. by pumping  $\text{MgCl}_2$  soln. into  $\text{NaOH}$  soln. reduced priming. A similar effect was noticed when  $\text{Mg}(\text{OH})_2$  was pptd. along with fluffy  $\text{CaCO}_3$  by  $\text{Na}_2\text{CO}_3$ . Oscar T. Qumby

Removal of silica from solution at boiler temperatures. Frederick G. Straub. *Ind. Eng. Chem.* 28, 36-7 (1936);

cf. *C. A.* 26, 5245; 27, 1426.—Removal of  $\text{SiO}_2$  from soln. at 182-232° was investigated. Heating 0.00376 molar  $\text{SiO}_2$  and 0.015-0.050 molar  $\text{NaOH}$  did not ppt.  $\text{SiO}_2$ . Addn. of  $\text{Ca}_3(\text{PO}_4)_2$  did not ppt.  $\text{SiO}_2$ .  $\text{MgO}$  reduced  $\text{SiO}_2$  to 0.001 molar.  $\text{NaAlO}_2$  reduced  $\text{SiO}_2$  to 0.0003-0.0005 molar. Addn. of excess  $\text{NaOH}$  or small amts. of  $\text{NaCl}$  or  $\text{Na}_2\text{SO}_4$  did not increase the soly of  $\text{SiO}_2$ . The  $\text{Al}_2\text{O}_3$  content did not exceed 0.0005 molar until  $\text{SiO}_2$  had been pptd. to that level. Foster Dee Snell

2. Analcite. Preparation and solubility between 182° and 282°. Frederick G. Straub. *Ind. Eng. Chem.* 28, 113-14 (1936), cf. preceding abstr.—By heating suitable solns. of  $\text{Na}_2\text{O}$ ,  $\text{SiO}_2$ , and  $\text{Al}_2\text{O}_3$  rounded crystals of analcite,  $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ , were obtained of a comp'n similar to the natural analcite. The solubilities in  $\text{H}_2\text{O}$  up to 0.00625 molar with  $\text{NaOH}$  are given and indicate greater soly. with increase of temp. and slightly greater with increase of  $\text{NaOH}$ . The  $\text{SiO}_2/\text{Al}_2\text{O}_3$  ratio of the dissolved material is not the same as in analcite. Results indicate that with  $\text{SiO}_2$  below 0.0022 molar, 120 p. m. of analcite scale will be prevented. Foster Dee Snell

The chlorine-ammonia treatment at Houston, Texas. L. P. White. *Southwest Water Works J.* 17, No. 9, 13-15 (1935).—Contrary to the usual practice  $\text{Cl}$  added before the  $\text{NH}_3$  gave the best results. The distribution of  $\text{N}$  in  $\text{H}_2\text{O}$  with no residual  $\text{Cl}$  followed 1 of 3 changes: The free  $\text{NH}_3$  and albuminoid  $\text{N}$  increased; free  $\text{NH}_3$  decreased, albuminoid  $\text{N}$  increased and sometimes nitrates occurred, free  $\text{NH}_3$  decreased with formation of nitrates and nitrites. Reducing the ratio of  $\text{NH}_3$  to  $\text{Cl}$  from 1.45 to 1.60 increased residuals and decreased  $\text{H}_2\text{S}$  odors. O. M. S.

Ammonia dosage in ammonia-chlorine treatment of water. M. L. Koschik. *J. Am. Water Works Assoc.* 27, 1477-91 (1935).— $\text{Cl}$  absorption drops as the  $\text{NH}_3$  dosage rises. A higher content of org. matter increases the amt. of  $\text{NH}_3$  required. Increasing the  $\text{Cl}$  dosage necessitates an increase in  $\text{NH}_3$ . Slightly acid water ( $\text{pH}$  6.7) demands more  $\text{NH}_3$  than when reaction is slightly alk. ( $\text{pH}$  7.2). Bactericidal effect of  $\text{Cl}$  is retarded by  $\text{NH}_3$ . Dosing  $\text{NH}_3$  according to the amt. of  $\text{Cl}$  cannot guarantee success in all instances. D. K. French

Effect of organic load on chloramine sterilization. A. C. Beyer. *Western Construction News* 9, 334 (1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 66 (June 29, 1935).—A higher velocity sterilization was obtained with both  $\text{Cl}$  and chloramine at  $\text{pH}$  6.6 than at either  $\text{pH}$  7.4 or 8.2. With org. loads above 60 p. m., chloramines showed a marked superiority over  $\text{Cl}$  in the sterilization efficiency. C. R. Fellers

Ferric sulfate coagulation. Philip J. Horton, Jr., and Elwood L. Bean. *Waterworks & Sewerage* 81, 229-32 (1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 64-5 (June 29, 1935).—Dry, com. "Ferrisul" now used contains 94%  $\text{FeSO}_4$ . If tanks and feed lines are lined with rubber, disintegration from chem. action is largely prevented. The coagulant is introduced into the raw water at Providence, R. I., aerated, treated with  $\text{Ca}(\text{OH})_2$ , to the point of slight causticity, mixed, settled and filtered. The  $\text{pH}$  of the filtered water is maintained at 9.2-9.6 to stop corrosion in the distribution system. C. R. F.

Experience with fluorine in water. Jane H. Rider. *J. Am. Water Works Assoc.* 27, 1510-24 (1935).—A brief review of the published data relative to  $\text{F}$  in drinking water supplies, and its physiological effects. In children of susceptible age, 0.9 to 1.0 p. m.  $\text{F}$  will cause mottled enamel. No satisfactory, economical methods of removing  $\text{F}$  have been developed. D. K. French

Removal of iron and manganese from humus-containing water in a closed system at high filtration rates. R. Marschner. *Gas u. Wasserfach* 78, 833-5 (1935).—Open filtration plants for purifying this water have previously operated at rather low filtration rates. The raw water contained 2.7 mg. of  $\text{Fe}_2\text{O}_3$  and 0.2 mg.  $\text{Mn}$  per l. and was highly colored. The exptl. filter consisted of 2 units, each provided with a 1.5-m. depth of filter sand. The sand in the coarse filter was 2 to 3 mm. and reduced the iron content to 0.35 mg.  $\text{Fe}_2\text{O}_3$  per l., while the fine filter contained 0.5-0.8 mm. sand and reduced the iron

content to less than 0.1 mg. Fe O<sub>2</sub> per l. When Mn-coated sand was used in the fine filter, the Mn content was reduced to traces, at a max velocity of 15 m per hr with an operating period of 60-70 hrs R. W. Ryan

A modern turbidimeter Grant Laughlin *Water Works and Sewerage* 82, 423(1935)—Brief description of a portable turbidimeter using a photocell cell hook-up, and calibrated to read p. p. m. directly from a galvanometer deflection Comparisons are made against distd. H<sub>2</sub>O as having zero turbidity G. H. Young

Filter-washing experiments at the Chicago experimental filtration plant Herbert E. Hudson *J. Am. Water Works Assoc.* 27, 1547-63(1935)—Descriptions are given of many experiments on filter washing. All available information indicates the desirability of keeping filter sand as clean as possible Backwashing failed to keep filters clean though the higher the rate the greater the success Surface washing in addn kept filters in very good condition Much detailed information is given of installations in various cities D. K. French

Operation of filter plant at Pottstown before and after new sedimentation facilities were installed E. K. Grubb *J. Pa. Water Works Assoc.* 6, 33-8(1934); *U. S. Pub. Health Eng. Abstracts* 15, 72(July 13, 1935)—The turbidity varies from 10 to 1000 p. p. m. As the water is pumped into the sedimentation basin it first passes through the screen chamber and then to the rapid sand filters. The filters are washed every 10 hrs. The plant capacity is 6 million gal per day. The mixing chamber has a retention period of 20 min and the sedimentation basin one of 5 hrs. Since the new installation the turbidity on the filter has been reduced from 40 p. p. m. to 15, the coagulant has been reduced by 30%, the wash water by 80%, and the power consumption by 25% Sedimentation vastly improved the quality of the effluent C. R. Fellers

Pipe cleaning Charles Haydock *J. Am. Water Works Assoc.* 27, 1566-71(1935)—Soft waters, those low in total solids and those most desirable for domestic and industrial purposes are the most corrosive in pipe lines. Ordinarily pipe cleaning is to remove tuberculation. Mech. processes have been most generally used, although chem. processes have been used in certain cases. Pipe cleaning is not expensive when the benefits derived are considered D. K. French

Lime reclamation at Winston-Salem sewage plant R. M. Rogers *Mun. San. S.* 408(1934); *U. S. Pub. Health Eng. Abstracts* 15, 5, 30(July 6, 1935)—A dosage of 800 p. p. m. of lime gives a quick-settling floc and excellent clarification. It is suggested that the CaO be reclaimed by using CO<sub>2</sub> generated from the calcining sludge to ppt. the excess CaO. The pptd. CaO would be re-calcined and then fed back to the incoming raw sewage C. R. Fellers

Operation of the North Toronto sewage-treatment works Geo. Phelps *Can. Engr.* 69, No. 16, 33-4, 40(1935)—Industries are urged to pretreat their trade waste to make it similar to domestic waste. Operating data showing the extra cost of high-degree treatment and photographs are given Ann Nicholson Hird

Danger of gasoline in sewerage system J. R. Donald *Can. Engr.* 67, No. 21, 13(1934); *U. S. Pub. Health Eng. Abstracts* 15, 5, 33(July 6, 1935)—As a result of a series of explosions in a 2-mile length of sewer in Ottawa, it was called that 150 gal of vaporized gasoline would create an explosive mixt. in the 6-ft. sewer. In order to secure an explosive mixt. of coal gas, approx. 826,000 cu ft., or 1/3 of the amt. manuf. in the city daily, would be required C. R. Fellers

Operation of Guelph's new activated-sludge sewage plant H. S. Nicklin *Can. Engr.* 69, No. 16, 29-32(1935)—In the summer the sludge is treated with powder lime the day after pumping, to avoid odors. After 8 days the dried chips are raked off. Photographs are shown Ann Nicholson Hird

The Menomonee Falls (Wis.) activated-sludge sewage-treatment plant Robert Craner, Jr. *Water Works and Sewerage* 82, 418-21(1935)—Lay-out and equipment are

described and data obtained on test runs given

G. H. Young  
Industrial wastes and pollution of inland waters. J. Ricardo Alves Gumaes, Arcanio Faria and Francisco Bergamin. *Rev. dept. nac. producao animal* 1, 71-104(1931).—Distillery slop from cane molasses distilleries in Brazil was added to aquarium H<sub>2</sub>O in the proportions of 50, 40, 30, 20, 10, 5 and 2 1/4%. The 1st 4 dilns quickly killed all fish of several species placed therein. Only the females of 1 species (*Phallopterus januaris* Hensel) survived the 10% diln for 24 hrs. *P. januaris* (agam females only), *Geophagus brasiliensis* Q. and G. and *Fundulus sensuensis* Agassiz survived the 5% diln. The 2 1/4% diln was practically harmless to all species, this also appears to be the limit of tolerance for water insects, tadpoles, microcrustaceans and worms. The limit for subaqueous plants is about 5%, floating plants 30%, protozoans 50%. Neutralization greatly diminishes the toxicity. When subjected to biol. purification (Calmette) the slop is innocuous in the proportion of 20%. Chem. analyses are given O. W. Wilcox

Clarification of polluted water with particular reference to colliery waste and sewage R. D. Gifford *J. Inst. Engrs.* July 1934, *Sewage Works J.* 6, 1187-7(1934); *U. S. Pub. Health Eng. Abstracts* 15, 5, 26(Mar. 16, 1935)—The paper describes a process for the rapid settlement and dewatering of sludge developed by R. A. Henry of Liege, Belgium, and adopted for the treatment of the sewage of Micheroux, a city of 100,000. Basically the process consists of the use of starch and NaOH as a coagulant after establishment of a condition of alkali approximating pH 11.0. It is highly advantageous to use frozen starch and imperative that the lime be thoroughly incorporated with the water. It is preferable to grind the lime under water so as to subdivide it into minute particles and thus present a relatively large surface to the water. The Micheroux sewage works have a series of lime crushers in the sewage channels. The lime causes the sludge to settle at once and the sludge and scum are removed without delay to a conical settling tank where rapid conn. is effected by the use of frozen starch and NaOH, and the dewatering of the sludge by vacuum filtration C. R. Fellers

Preliminary report on the disposal of oil field brines in the Ritz-Canton oil field, McPherson County, Kansas C. J. Wilhelm and Ludwig Schmidt. *Bur. Mines, Rept. of Investigations* No. 3297, 20 pp (1935)—Pos. disposal of brine is accomplished by return of brine to subsurface formations. Pptn. of Fe compds and salts in soln. may seal the formation. Controlled diln. of the brine by pumping into surface streams is possible. Alden H. Emery

Distillery wastes. Chemical and filtration studies C. R. Hoover and F. K. Burr *Ind. Eng. Chem.* 28, 39-41(1936)—Chem. pptn. of distillery wastes by coagulation is not economically satisfactory on the basis of reported results with CaO and Fe or Al salts. Filtration at 1,000,000 gal. per acre per day on a 10-ft. deep trickling filter built of laths gives 90% reduction of B. O. D. with clarified grain waste, or relatively clear apple brandy or molasses ale wastes. For treatment these are dtd. below 2000 B. O. D. The result is 98-100% complete in 6 ft. Intermittent sand filters are inefficient because of clogging and low capacity. Coke breeze of 1-mm. effective size is more satisfactory than sand. A 4-ft. depth gave 98% reduction of the above wastes at 200,000 gal. per acre per day. Temps. of 18-30° did not affect the results obtained Foster Dee Snell

Garbage reduction and incineration combined in Philadelphia plant Joseph E. Gill. *Am. City* 50, No. 12, 57-8(1935)—Stored garbage, instead of awaiting rubbish for combustion, is digested and the tankage used as fuel G. H. Young

Gas cleaning and sludge-recovery system (Wille, Mohr) 21 Waste products of city dumps as fertilizer (Schecklein) 15 Use of Zn alloys for water pipes (Cazaud, Petot) 9 Rotary-drum app. for filtering and dewatering slimes such as those from sewage (U. S. pat. 2,024,358) 1. Exhaust



steam condenser and feed water heater and purifier 1  
U. S. Pat. 2,025,043 1 Thionitratophosphates [for water softening] U. S. Pat. 2,025,503 18

Purifying water Douwe N. Kuipers Brit 434,015, Aug. 23, 1935 H<sub>2</sub>O is purified by aerating and filtering through a series of filters the 1st and last of which have a slower speed of filtration than the others

Clarifying tanks for water purifying apparatus Karel L. Hagemans and N. V. Machiniefabriek Reneveld Brit 434,030, Sept. 10, 1935 H<sub>2</sub>O to which reagent has been added is supplied to a settling vessel through a trough fitted with baffles a central tube, lateral passages and annular chambers

Apparatus for clarifying water Karel L. Hagemans and N. V. Machiniefabriek Reneveld Fr 787,518, Sept. 24, 1935

Apparatus for filtering water through granular material such as gravel Willis H. Jones U. S. 2,024,646, Dec. 1, 1935 Structural and operative details

Filtering water by granular layers with means for clearing grains of sediment Norton Co Fr 786,310, Aug. 11, 1935

Softening water Christopher G. Marshall Brit 434,012 Aug. 21, 1935 A H<sub>2</sub>O softening plant is provided with an app. for indicating the correct flow of H<sub>2</sub>O during backwashing

Apparatus for softening water Bengt Rybo and Aktiebolaget Filtrum Brit 434,535, Sept. 4, 1935 Brine for regenerating base-exchange material in a main container is

formed from salt in an inner container through which H<sub>2</sub>O is forced as an upwardly directed rotating stream

Treating waste waters, sewage, etc. René A. Henry Fr 787,831, Sept. 30, 1935 A suspension of clay in an aq. soln. of lime is added, the amt. of clay being detd. by the amt. which can be adsorbed by the org. material, colloids, etc., present The amt. of lime is sufficient to give a pH value of 11 and this value is maintained sufficiently long to destroy living organisms. App. is described

Apparatus for treating sewage Wm. A. Dundas and Philip Harrington Jr. 787,813, Sept. 30, 1935 The residues are freed from their moisture content and incinerated and the heat of combustion is used for driving a further patch

Apparatus for sewage purification by aeration, settling, etc. Henry Elrod U. S. 2,024,345, Dec. 17, 1935 Structural and operative details

Apparatus for treatment of sewage by aeration and sedimentation Augustus C. Durdin, Jr. (to Chicago Pump Co.) U. S. 2,024,986, Dec. 17, 1935 Various structural and operative details

Sewage sludge treatment Henry J. Stebb U. S. 2,020,306, Dec. 31, 1935 Sewage sludge is sep. into fragments which are surface-coated with dry material or surface-dried to prevent them from adhering to one another, the fragments are dried, the upper portions of the dried fragments are heated (suitably on a perforate-bottom carrier) and heated gases from such portion are passed through the lower portion of the fragments without burning the latter to distill noxious gases and the latter are burned. App. is described

## 15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

M. S. ANDERSON AND K. D. JACOB

Summary of work done under the agricultural chemist and soil physicist, Agricultural Research Station, Sakrand, for the year 1933 34. V. A. Tamhane Ann. Rept. Dept. Agr. Serv. (India) 1933 34, 111-20—Soils—Ca is the predominant exchangeable base in the solonchets group of Sind soils. In the kalar (salt) soils the Ca is as high as 0.0, as compared with 7.5-8.0 in the normal soils. The N-fixing power of the normal soils in the natural condition is low but is greatly increased by addn. of org. matter in the form of green manure. The N-fixing power is not enhanced by addn. of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> without org. matter. The N-fixing power of kalar soils is almost negligible and is not enhanced by addn. of org. matter. In general, the amts. of total and of injurious soil salts in kalar soils were significantly decreased by leaching the soil with 32 acre-inches of water applied at the rate of 8 acre inches per week. The treatment markedly reduced the NaCl content of the soil and permitted the growth of a good crop of cotton. Good results were also obtained by scraping off the incrustation of salts prior to flooding the soil. Very bad kalar soil was not improved in 1 season by treating with alum at the rate of 800 lb./acre and flooding with water. CaCl<sub>2</sub> was more effective than either gypsum or superphosphate in improving kalar soil. Capillary rise of soil salts in soils that had been leached previously with water was checked by the addn. of green manure to the soil. Effect of soil salts on wheat—In pot tests no growth of wheat was obtained in the presence of 0.5% of either NaCl, KCl, MgCl<sub>2</sub>, CaCl<sub>2</sub>, Na<sub>2</sub>CO<sub>3</sub>, K<sub>2</sub>CO<sub>3</sub>, or (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. The toxicities of other salts decreased in the order NaNO<sub>3</sub>, KNO<sub>3</sub>, K<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>SO<sub>4</sub>, MgSO<sub>4</sub>, the last having comparatively little toxic effect. In the earlier stages of growth wheat was adversely affected by the presence of 0.3% NaCl, but addn. of NaCl during and after the flowering stage seemed to have no adverse effect on the crop. K. D. Jacob

The proton in soils and soil degradation I. D. Sedletskii Khimicheskii Sotsialist. Zemledelie (Moscow) 1935, No. 5, 20-27—A theoretical discussion on the mechanism of the entrance of the H<sup>+</sup> ion into the crystal lattice of the micelle, as contrasted to surface adsorption

of other ions, this causes the disruption of the micellar nucleus and brings about degradation. J. S. Joffe

Fundamental amelioration of solonchets N. I. Belkin Khimicheskii Sotsialist. Zemledelie (Moscow) 1935, No. 6, 45-55—Pot. exps. were conducted with soil taken from the columnar B horizon of a solonchets. After addn. of gypsum or org. matter plants were grown 3 years in succession, analyses being made on the soil for change in reaction, nitrification and structure. It is concluded that about 14 tons of gypsum or 240 tons of manure per hectare changes the soil fundamentally and adapts it for cropping. A hay crop of sweet clover is suggested for the first 2 years after ameliorating the solonchets condition. J. S. Joffe

Experimental results on the chemuration of marsh soils in the far north P. A. Turnas and A. F. Karatnikova Khimicheskii Sotsialist. Zemledelie (Moscow) 1935, No. 6, 15-22—Apatite and nephelite wastes can be used for the neutralization of acidity in high-moor peat soils just as efficiently as lime. A mixt. of these 2 materials also supplies Pand K. Burning virgin moors brings enough haves into circulation to obviate the necessity of adding neutralizing agents. Equiv. applications of apatite and acid phosphate give the latter the preference for the first year. In the years following the differences are obliterated. A double portion of apatite gave just as good results as a single portion of acid phosphate. The K in nephelite was found to be as effective as the standard K salts. The low moors did not respond to K applications. J. S. Joffe

Observations in connection with atmospheric additions of sulfur to arable soils Gabriel Bertrand. Bull. soc. chim. [3], 2, 2102-5 (1935)—See C. A. 29, 6098a.

G. G. The influence of phosphates on the cation-exchange capacity of the fundamental soil types of the U. S. S. R. K. Kh. Aidiyan Khimicheskii Sotsialist. Zemledelie (Moscow) 1935, No. 4, 13-22—Soils of the chernozem, gray forest steppe, podzole, solchets, chestnut and red earth zones were said with Na, treated with NaH<sub>2</sub>PO<sub>4</sub> or NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, the absorbed P was detd., the soils were electrodyalysed and the exchange capacity was detd.

The red soil absorbed appreciable quantities of P and increased the exchange capacity. Treatment of chernozem with  $\text{NH}_4\text{H}_2\text{PO}_4$  decreased the exchange capacity from 28.8 milliequiv., as detd by  $\text{NH}_4\text{Cl}$  treatment, to 20.9 milliequiv. J. S. Joffe

Relations among the exchangeable acidity and the exchangeable substances in the soils. Chikabumi Ichikawa. *J. Agr. Chem. Soc. Japan* 11, 817-24 (1935).—When the acidity of the soils by the KCl method increased, the quantity of  $\text{Al}_2\text{O}_3$  in the KCl ext increased, while that of  $\text{CaO}$  decreased. There is a reciprocal relation between the exchangeable acidity, the exchangeable  $\text{Al}_2\text{O}_3$  and the exchangeable  $\text{CaO}$ . Y. Kihara

Determination of hydrolytic acidity by various methods on soils of the Sverdlov region. V. A. Serdyukov and A. D. Kazantsev. *Khimicheskaya Setzislust Zemledel'ya* (Moscow) 1935, No. 4, 68-71.—Podzol soils of different degrees of podzolization and several varieties of chernozem were tested by the Csiky (C. A. 25, 3756) and Kappen methods for hydrolytic acidity to det their lime requirements. The Csiky method gave results which were closer to the actual condition, whereas with the Kappen method the factor 1.75 had to be applied to compare with the results by the Csiky method. J. S. Joffe

The present position of the mechanical analysis of soils. A. Srenivasan. *Madras Agr. J.* 23, 443-51 (1935).—A review with 68 references. K. D. Jacob

The methods of determining potassium in soils for agro-chemical purposes. Ya. V. Peise. *Khimicheskaya Setzislust Zemledel'ya* (Moscow) 1935, No. 6, 71-83.—A modification of the  $\text{K}_2\text{NaCo}(\text{NO}_2)_6$  method is presented. The K is extd from a 1:2 soil- $\text{NaCl}$  mixt., and absorbed  $\text{NH}_3$  in the soil does not interfere with the detn. J. S. Joffe

The Gedroz potash method for mass analyses of the absorption capacity of carbonate soils. B. V. Ostromov. *Khimicheskaya Setzislust Zemledel'ya* (Moscow) 1935, No. 6, 61-71.—The Gedroz method (see the 3rd edition of his book "Chemical Analyses of Soil," C. A. 20, 2223) of using  $\text{K}_2\text{CO}_3$  on soils contg carbonates for the detn of the exchange capacity by titrating the carbonates formed in the exchange reactions has been modified by using potentiometric titration. In this manner it was found possible to use this method on solonchic soils. J. S. Joffe

A new method of determining the adsorbed potassium in carbonate soils. D. Guseinov. *Khimicheskaya Setzislust Zemledel'ya* (Moscow) 1935, No. 5, 53-7.—The efficiencies of  $\text{AcONH}_4$ ,  $\text{CaCl}_2$ ,  $\text{AcONa}$ ,  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{SO}_4$  were compared in extg K from soils rich in carbonates. It is shown that the  $(\text{NH}_4)_2\text{SO}_4$  is the most efficient reagent and may be used as an index of the available K in soils. J. S. Joffe

Soil, field-crop, pasture and vegetable-crop management for Eustafson. New York. I. Soils and field crops. A. F. Gustafson. N. Y. (Cornell) Agr. Expt. Sta., Bull. No. 639, 5-55 (1935); cf. C. A. 29, 853.—Characteristics of the 12 soil types present are given in detail and their value for crop production is outlined. As a result of examing 201 soils, the use of lime on most soils is recommended. In general, moderate application of superphosphate to most soils and crops was profitable. II. Pasture improvement and management in Erie County. D. B. Johnstone-Wallace. *Ibid.* 56-57.—Pasture improvement is best accomplished by the application of 500-800 lb. of superphosphate per acre once in 3-5 years. III. Vegetable crops. F. O. Underwood. *Ibid.* 57-115.—The soils yield good crops of potatoes, peas, sweet corn, beans, cauliflower, cucumbers and tomatoes. IV. Soil map and soil-type descriptions. C. S. Pearson, F. B. Howe and A. F. Gustafson. *Ibid.* 115-20. C. R. Fellers

Some bacteriological and chemical effects of calcium and magnesium limestones on certain acid lowa soils. Harold L. Dean. *Iowa State Coll. J. Sci.* 10, 69-71 (1935).—In greenhouse and lab. expts., conducted to compare the effects of high-Ca and high-Mg limestones and pure carbonates of Ca and Mg, the results obtained indicate: (1) the early effects of Ca limestone are superior,

(2) after a long period there is no difference, (3) the time order of effect is pure carbonates, fine limestone, coarse limestone, (4) the soil contained enough of either Ca or Mg so that the addn. of large amts. of either did not produce deleterious effects. F. E. Brown

Fixation of atmospheric nitrogen in the soil and the utilization of molasses as a fertilizer. N. R. Dhar and S. K. Mukerji. *Proc. Acad. Sci. United Provinces Agr. Oudh, India* 5, 61-70 (1935); cf. C. A. 30, 555, 556.—Sterile mixts of soil and sucrose illuminated in sunlight under sterile conditions in quartz vessels showed an increase in  $\text{NH}_3$  and total N. Appreciable amts. of  $\text{NH}_3$  were formed when sterile air free from N oxide was passed through solns of glucose and sucrose in the presence of freshly pptd  $\text{Fe}(\text{OH})_3$ . This indicates that fixation of atm. N can take place without bacterial action provided energy is supplied by photochem. or induced oxidation of sugars. The exposure of sterile molasses soil mixt. to sunlight leads to an increase in  $\text{NH}_3$  up to a limiting value. This is true of soil in the field, and expts. show that molasses applied as fertilizer increased the yield of sugar cane up to 36% and exerted a beneficial effect upon rice culture. C. E. P. Jeffreys

Metabolism of soil fungi. Unto Vartiavaara. *Suomen Maataloustieteellinen Seuran Julkaisuja Acta Agraria Fennica*, 32, 107 pp. (in English 108-12) (1935) (reprint).

Various fungi were grown on synthetic media with cellulose and other substances as C sources. *Sporotrichum carnis* produced a max. yield of mycelium and effected a max. decompn of cellulose at 24°, while with *Monospora daleae* the temp. was 20°. If the mycelium is first grown at an optimum temp., cellulose decompn. proceeds at 5° at only a slightly reduced rate. Both species produced the highest yield of dry matter between pH 4 and 7. Good aeration was essential for good growth. Wide, shallow culture vessels were the best. Both species grew well on dihydroxyacetone, pentoses, hexoses, disaccharides, dextrins, higher alks., certain org. acids, and particularly on fructose, maltose and sol starch. Technically purified cellulose was decompd. effectively by both species. Maltose at 2-5% of the cellulose promoted the decompn. of the latter, while higher amts. of maltose inhibited it. Both species readily decompd. straw, roots, wood, leaves and needles, but lignum was only slightly attacked. The use by the fungi of inorg. N compds. greatly changed the reaction of the medium,  $\text{NH}_3$  making it acid, and nitrates alk. The amt. and soly. of N compds. in the mycelium decreased markedly with the age of the mycelium. When dried fungus material was used as N fertilizer for oats, the amt. of N taken up by the plants corresponded to the degree of mineralization in parallel decompn. expts. in sand. J. J. Williams

The influence of varying the time interval of applying sylvinit to the agro-chemical properties of the soil and the yield of potatoes. E. A. Zhorikov. *Khimicheskaya Setzislust Zemledel'ya* (Moscow) 1935, No. 4, 32-6.—Application of sylvinit to a podzolic sandy soil increased the active acidity by replacing the H ions, decreased the Ca and Mg in the soil complex of the upper horizon, decreased the nitrification energy, and prevented the absorption of Ca and Mg by the potato, thereby decreasing the yield. The injurious effects of this KCl bearing mineral could be avoided by applying it in the fall. J. S. J.

The causes of the mobilization of phosphoric acid upon liming podzolic soils. S. S. Yarusov and I. S. Treilikh. *Khimicheskaya Setzislust Zemledel'ya* (Moscow) 1935, No. 5, 28-37.—Iron phosphates with different ratios of basoid to acidoid were prepd. by washing the Merck prepa. with water, a higher basoid content being thus obtained, and by pptg. salts of Fe and phosphate at various pH values. The phosphates were used in culture expts. by applying the method of isolated feeding, with oats as the exptl. plant. The pH of the inner pots was kept const. by adding  $\text{Ca}(\text{OH})_2$ ,  $\text{NaOH}$  or  $\text{H}_2\text{SO}_4$ . From the series of expts. run during 1928-1931 it is indicated that the phosphates with a high basoid content are less sol. and available than the ones with a low basoid content. The soly. of phosphates of low basoid content does not change up to a pH

4.0 when acid is added and down to a pH 7.0 when alkali is added. In the pot expts the high-basoid phosphate became more available upon the addn of NaOH. The low basoid phosphate did not become more available upon the addn of NaOH. Addns of Ca(OH)<sub>2</sub> decreased the availability of the low-basoid phosphates, but increased the availability of the high-basoid phosphate. The genesis of podzols is conducive to the formation of high-basoid phosphates, hence the addn of lime makes the P<sub>2</sub>O<sub>5</sub> available. J. S. Joffe

The effect of liming on the liberation of potassium in some Iowa Soils. Hartzell C. Dean. *Iowa State Coll. J. Sci.* 10, 73 (1935), cf. *C. A.* 29, 3760<sup>2</sup>.—The expts were conducted in both lab and greenhouse. Analysis of 12 high Ca soils revealed large amts of CO<sub>3</sub>, N and K, but 11 of the 12 were deficient in available K. An acid Tama silt loam had a smaller exchange capacity than a high Ca soil, was unsatd with respect to bases, released more K on liming, and acquired the K-exchange characteristics of a high Ca soil within 6 months after the addn of 6 tons of lime per acre. Sterile un inoculated soils contained more available K than sterile inoculated soils under similar treatment. The action of microorganisms contributes to the unavailability of K, but depressed hydrolysis and reversion of K to insol forms are also factors. F. E. Brown

The significance of potassium in the system of fertilizing sugar-beet rotations. F. A. Lut. *Khimskaya Soiznitsa Zemledeliya* (Moscow) 1935, No 6, 33-40.—Exptl results over a period of 12 years on the effect of K on sugar beets in the rotation prompted the following conclusions. In the chernozem zone K and P are first in order for sugar beets. The need for N is not clearly marked in the rotation with clean fallow, grain or legumes. The more often sugar beets appear in the rotation, the more often it is important to add K. The beneficial effects of K on sugar beets are especially marked when grasses are in the rotation. The other crops in the rotation, especially winter wheat, benefit from the residual effects of the K added to the sugar beets. J. S. Joffe

The effect of reaction and lime content of soil on the yield and composition of several leguminous green manure crops and green corn. Rokuo Kawashima. *J. Sci. Soil and Manure* (Japan) 9, 389-410 (1935).—Varieties of leguminous green-manure crop were found to be indifferent to the relation between the growth of the crop and the reaction as well as the Ca content of the soils. With several manure crops and green corn (soy bean, lupine, common vetch, hairy vetch and serradella) the optimum ranges of Ca content and reaction of the soils to the plant-growth were different. Y. Kamoshita

The problem of fertilizing eroded slopes. Ya. V. Korney. *Khimskaya Soiznitsa Zemledeliya* (Moscow) 1935, No 5, 12-19.—Erosion removes more N and less P from the lower portion of the slope than from the upper, hence the lower portion responds more to N and less to P fertilizers. The reverse is true for the upper portion of the slope. J. S. Joffe

A general summary of experiments with sugar cane. W. G. Taggart, C. B. Gouaux, F. C. Simon, C. W. Edgerton, L. C. Tims, P. J. Mills, W. E. Hinds and B. A. Osterberger. *La Agr. Expt. Sta., Bull. No. 267*, 2-16 (1935).—To avoid serious deterioration (loss in sucrose content) cane should be delivered to the sugar mill without delay after cutting. An application of 100 lb of NaNO<sub>3</sub> per acre was found most profitable for most La. cane soils. The resistance of the various canes to mosaic and other diseases is discussed. Among the controls proposed for the sugar cane borer, *Diatraea saccharalis*, is dusting in July with high-grade light Na<sub>2</sub>SiF<sub>6</sub>. C. R. Iellers

The methods and time of introducing fertilizers. A. S. Koruzhev. *Khimskaya Soiznitsa Zemledeliya* (Moscow) 1935, No 4, 23-8.—Exptl results are presented showing that acid phosphate gives better results when ground to a fineness of less than 2 mm. Nitrophoska of less than 1 mm gave better results than larger particles. Local application was usually superior to broadcasting. Physiologically acid fertilizers gave poorer results with local

application on podzol soils, whereas Nitrophoska gave better results. On a limed podzol soil raw phosphates when applied locally gave good results in combination with (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. J. S. Joffe

Artificial fertilizer inspections—report for 1935. W. C. Robertson. *J. Dept. Agr. Victoria* 33, 608-13 (1935), cf. *C. A.* 29, 2040<sup>2</sup>.—Analyses of fertilizers and fertilizer materials sold in Victoria are tabulated and discussed. K. D. Jacob

The waste products of city dumps as fertilizer. S. L. Shebekin. *Khimskaya Soiznitsa Zemledeliya* (Moscow) 1935, No 4, 62-7.—Analyses of a no. of dump materials and their composts are given. J. S. Joffe

Sulfates and chlorides as fertilizers. VII. Influence of both salts on the physical constitution of paddy-field soil fertilized annually for five years with the same fertilizers. Isenosuke Onodera and Hiroshi Hasegawa. *J. Sci. Soil and Manure* (Japan) 9, 357-64 (1935), cf. *C. A.* 28, 2452<sup>1</sup>.—Fertilizing the soils (paddy field soil of alluvial loam) with several combinations of sulfates (NH<sub>4</sub> and K), chlorides (NH<sub>4</sub> and K), Na phosphate, Ca superphosphate, farmyard manure, etc., caused an increase in the amts of fine and coarse sands in the soils. The amt of clay was observed to be large in the soils of chloride plots but not in those of sulfate plots, the cases were reversed for the amts of fine sand and silt. The minute soil particles seemed to be attacked by farmyard manure and dissolved by chloride applied as fertilizer. Y. Kamoshita

Nephelite as a fertilizer. N. D. Smirnov. *Khimskaya Soiznitsa Zemledeliya* (Moscow) 1935, No 6, 23-32.—Vegetation and field expts. with a no. of crops on moss, peat, sandy loam and loam podzol soils show that nephelite can serve as a neutralizing agent and as a source of K and P. J. S. Joffe

Potassium from Solikamsk and the use of mixed fertilizers. B. S. Rozenberg. *Kalif* (U. S. S. R.) 1935, No 5, 26-31.—A discussion of economic advantages of the use of mixed fertilizers (N, P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O). Tables are given of the proportions and amt of different formulas of fertilizers for different districts of U. S. S. R. A. P.

Potassium fertilizers for flax and hemp. V. M. Filippenya. *Kalif* (U. S. S. R.) 1935, No 4, 50-2.—K fertilizers increase the yield of flax and improve its quality, 45-60 kg of K<sub>2</sub>O per hectare is required. For hemp planted on peat soil (reclaimed from marshes) the best amt was 90-120 kg K<sub>2</sub>O per hectare. A. Pestoff

Physicochemical basis of obtaining complex potassium fertilizers. A. G. Bergman and L. B. Polyakova. *Kalif* (U. S. S. R.) 1935, No 8, 24-8.—The polytherm of the diagonal cross section KNO<sub>3</sub>-NH<sub>4</sub>Cl-H<sub>2</sub>O of the mutual system NH<sub>4</sub>NO<sub>3</sub> + KCl = KNO<sub>3</sub> + NH<sub>4</sub>Cl was studied in the range of +30° to the freezing of soln, -16.4°. The eutectic at -16.4° contains 6.1% KNO<sub>3</sub>, 18.7% NH<sub>4</sub>Cl and 75.2% H<sub>2</sub>O (by wt). The triple nonvariable transition point at +23.4° contains 21.9% NH<sub>4</sub>Cl, 25.6% KNO<sub>3</sub> and 52.5% H<sub>2</sub>O (by wt). Four references. A. Pestoff

Cementation in the storage of fertilizing salts. N. E. Pestov. *Kalif* (U. S. S. R.) 1935, No 7, 28-31.—The causes of cementation of fertilizer salts in storage are hygroscopicity of the salt, climatic conditions, humidity, time of storage; form of the particles of the salt and the character of the surface. The smooth sphere is the best form. Other important factors are size of particles; homogeneity of the size of particles, size of the pile and pressure developed on a part of the mass. Isolation from the air and admixt with different materials often inhibits cementation. The prevention of cementation is accomplished by the manuf. of salts in the form of large crystals (finely cryst salts must be granulated). Addn of oils, CaO, MgO, CaSO<sub>4</sub>, and other materials in small quantities is often beneficial. A. Pestoff

The utilization of by products containing copper and ores of a low copper content as fertilizers for marsh soils. A. V. Zenyuk. *Khimskaya Soiznitsa Zemledeliya* (Moscow) 1935, No 5, 45-53.—Expts with CuSO<sub>4</sub> at the rate of 25-30 kg per hectare on marsh soils during 1939-1932 have proved the stimulating effects on wheat,

barley, flax and oats. In 1933-34 other Cu compds. were tested: nitrate, oxide, chloride, acetate, carbonate, malachite and a prepn. AB the compn of which is not stated. In all cases there were large increases. Upon the addn. of a complete fertilizer the yield did not increase unless an extra dose of Cu was added. Chalcedony flux contd. about 0.4% CuO at the rate of 300 to 1200 kg. per hectare and 2 or 3 contg. 1 to 1.5% CuO at the rate of 300 kg. per hectare were used in pot and plot expts. In both cases the results compared favorably with those of CuSO<sub>4</sub>. The chalcedony flux was especially effective in increasing the yield of grain. In general, Cu was more effective in marsh soils rich in P. J. S. Joffe.

Insecticide tests to control the codling moth at the Vincennes, Indiana, laboratory during 1934. L. F. Steiner, R. F. Szama and J. E. Fahey. *Trans. Indiana Hort. Soc., 74th Ann. Meeting 1935*, 66-71.—None of the materials tested gave satisfactory control, but those appearing equal to Pb arsenate without leaving objectionable residue or causing injury were 1% summer oil with nicotine sulfate (1800) in the 2nd-brood sprays and nicotine benzoate (1.25 lb./50 gal.) with summer oil during 2nd-brood spraying or throughout the season. Washing was necessary wherever Pb arsenate or cryolite was used after the 1st cover spray. Natural and synthetic cryolite gave as good results as Pb arsenate but caused some reduction in size of fruit and left a residue load that could not be removed by ordinary washing methods. CuCN and oil gave the best control of both worms and stings but caused serious russetting of the fruit. Lab. tests showed that larvae were able to enter fruit from the tops of the trees much more readily than fruit taken from the lower half, the Pb and As residues were nearly twice as heavy on fruit from the lower half of the tree as on fruit from the top. All materials, particularly Pb arsenate, had extremely low efficiencies against 1st-brood larvae, apparently because of the rapid growth of the fruit and the difficulty of immediately building up an effective deposit as the eggs start to hatch. K. D. Jacob.

The significance of supplementing controls in combating the codling moth. J. J. Davis. *Trans. Indiana Hort. Soc., 74th Ann. Meeting 1935*, 72-82, cf. C. A. 29, 2648.—A material suitable for treating cracks and cavities in trees for the purpose of killing overwintering codling-moth larvae is prep'd by heating together  $\alpha$ -naphthylamine 1 and Parawax 2 parts and adding hydrated lime, with const. stirring, until the mixt. has the consistency of very thick batter. The mixt. is applied, hot if necessary, to the cracks and cavities, taking care to avoid covering very much live bark. K. D. Jacob.

Environmental and control studies of the common scab disease of potatoes caused by *Actinomyces scabies* (Thaxter). Guss B. J. Dippenaar. *Univ. S. Africa Dept. Agr., Sci. Bull. 136*, 78 pp. (1933).—In greenhouse expts., soil pH values of 5.0 and lower either controlled or reduced the disease in severely scab-infested soils but did not eliminate scab. However, pH values of 4.78 and lower also had an adverse effect on the potato plant. The amt. of scab occurring in soils of pH 5.3 and lower was negligible under field conditions. The max. rate of increase of the scab organism under field conditions occurred in soils of pH 5.9 and above. The limiting acid reaction of the S. African strain of the scab organism was found to be approx. pH 5.7. Flowers of S. alone or in combination with green manure, applied at the rate of 300-1200 lb./acre, failed to control scab in field expts. during 3 seasons. Green pea vines, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, applied at the rate of 120-360 lb./acre, and Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, at the rate of 360-1080 lb./acre, also failed to control the disease. Treatment of severely scabbed tubers with 0.2% HgCl<sub>2</sub> plus 1% HCl for 5-10 min. gave as good control of scab as did treatments with 0.1% HgCl<sub>2</sub> for 1.5 hrs. or cold formalin (1:240) for 2 hrs. In 1 expt. the first treatment proved to be far superior for the control of *Rhizoctonia*. Forty-one references.

Relative effects of calcium and acidity of the soil on the occurrence of potato scab. F. M. Blodgett and E. K. Cowan. *Am. Potato J.* 12, 265-74 (1935).—When they

were added in varying amts. to a sterilized acid soil (pH 4.48) that was then inoculated with the scab organism, Ca compds. (CaSO<sub>4</sub>, CaCO<sub>3</sub> and Ca(OH)<sub>2</sub>) had no significant effect on potato scab other than through their effect on the acidity of the soil. In greenhouse expts. with a soil treated with either H<sub>2</sub>SO<sub>4</sub> or Na<sub>2</sub>CO<sub>3</sub> to give initial pH values ranging from approx. 4.0 to 8.5, there was a pos. correlation between the pH of the soil and the percentage of scabby tubers. The pH values of the soils, 2 det'd by the quinhydrone electrode, at the time the first 2 crops were dug ranged from 4.71 to 7.39, this indicates that the scab organism tends to change the soil toward a neutral reaction which is more favorable for its growth. When a heavily infected soil (original pH of 7.2) which produced only scabby potatoes was limed with Ca(OH)<sub>2</sub> to pH 8.5, 47% of the tubers were clean, when it was limed to a pH of 9.0, 91% of the tubers were clean. Growth of potatoes did not occur in soil limed to pH 9.3.

K. D. Jacob.  
The eradication of gublaar (*Dichapetalum cymosum*). A. C. Lémann. *Farming S. Africa* 10, 233-6 (1935).—The plant can be controlled by ringbarking the main stem below the surface of the ground, moistening the ringbarked portion with CaCl<sub>2</sub> soln. (0.5 lb./gal. H<sub>2</sub>O), enclosing the ringbarked portion with small stones and packing into the enclosure a mixt. of finely powdered CaCl<sub>2</sub>, 2, crude com. CuSO<sub>4</sub>, 1 and soil 2 parts applied at the rate of 1 lb./1-in. diam. of stem. The hole is then filled with soil to the surface of the ground. K. D. Jacob.

Fruit fly in citrus results with sodium fluosulfate bait. L. B. Ripley and G. A. Hepburn. *Univ. S. Africa Dept. Agr., Sci. Bull. 143*, 8 pp. (1935).—Promising control of the insect (*Pterandrus rosa*) was obtained by spraying the trees with a soln. of Na<sub>2</sub>SiF<sub>6</sub> (0.25 oz.) and white sugar (0.5 lb.) in water (1 gal.), applied at the rate of 4-12 oz. per tree according to size. The spray is applied in the form of comparatively large droplets to areas on each side of the tree. Complete coverage of the tree is not necessary because the insects are attracted to the sprayed portions. Terpinyl acetate was a powerful attractant for male flies but not for females, and there were strong indications that only unmated males are attracted by it. K. D. Jacob.

Spraying, pruning and die-back (of roses). H. R. Rosen. *Am. Rose Ann. 1935*, 43-7.—Bordeaux mixt. (4.4 50), Cu acetate (1-2 lb./50 gal. H<sub>2</sub>O) and Palustris sulfonate B (2 tablespoonfuls/gal. H<sub>2</sub>O) were approx. equally effective in controlling powdery mildew and black-spot when Aresco was added as a wetting agent at the rate of 1 teaspoonful/gal. of spray. At the rate of 2 lb./50 gal., Cu acetate gave no observable spray deposit on the leaves, but it burned the foliage badly and caused much leaf shedding, no injury occurred when it was used at the rate of 1 lb./50 gal. With Palustris sulfonate B serious burning of the leaves occurred on bushes sprayed with the last of the material remaining in the tank, the material also had a marked tendency to gum up the spray equipment. K. D. Jacob.

Chlorosis of *Hydrangea hortensis*. G. H. Poesch. *Ohio Agr. Expt. Sta., Bimonthly Bull.* 175, 142-3 (1935).—Plants growing in soil with neutral reaction responded to applications of FeSO<sub>4</sub> and Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>. The Fe<sup>++</sup> gave quicker response and returned chlorotic plants to a normal green color within a short time. MnSO<sub>4</sub> and ZnSO<sub>4</sub> were not effective in controlling chlorosis. C. R. Fellers.

The cause of chlorosis, or yellowing (of roses). K. N. Clapp. *Am. Rose Ann.* 1935, 41-2.—Chlorosis of roses growing in alk. soil (pH 8.5) was temporarily overcome by spraying the plants at 4-6-week intervals with a soln. of FeSO<sub>4</sub>·7H<sub>2</sub>O (0.5 oz./gal. H<sub>2</sub>O). The condition was permanently overcome by neutralizing the soil with S applied at the rate of 1 lb./sq. yd. and dug into the soil. With soils having pH values below 8.5 satisfactory results were obtained by the application of Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> (1 lb./sq. yd.) with repetition at 10-day intervals if necessary.

K. D. Jacob.  
Injury (to roses) from spray materials. L. M. Massey. *Am. Rose Ann.* 1935, 35-40.—Finely divided S, either from sprays or dusts, is likely to cause burning at atm.

temperatures of 60° or higher. High temps and humidities favor the formation of H<sub>2</sub>O-sol. As on the leaves of plants sprayed or dusted with mixts of Pb arsenate and S, foliage is injured by the sol. As. K D Jacob

**Heart and dry rot of beets.** L Kraus *Deut Landw Presse* 62, 297 (1935). *Rev Applied Mycol* 14, 733—Good results in treating beets for heart and dry rot were obtained by using 15 or 30 kg of borax per hectare. The yield from one series of plots increased from 244 to 470 S and 528 S kg, respectively, and from another series from 430 to 458 and 463 S kg. Oden E Sheppard

**Physiological diseases of beets.** III Further field tests in the study of the effect of borax on sugar and fodder beets. K de Haan *Mededel Inst Suikerbietenelt* 4 92-102 (1934). *Rev Applied Mycol* 14, 733—Treatment with borax at the rate of 40 kg per hectare decreased heart rot of beets from 66 to 0.1%. At 10 kg per hectare borax increased the sugar yield and content by 34 and 10%, resp. and reduced incidence of heart rot to 2%. Leaf yield was increased 25 and 50% by 10 and 40 kg borax, resp., while corresponding figures for roots were 22 and 26%. Fodder beets showed an increased sugar yield and content of 65 and 10%, resp., with application of 20 kg borax per hectare. NaNO<sub>3</sub> (contg 0.6 kg borax per 1000 kg) showed good results in mild cases of heart rot. Oden E Sheppard

**Successful combating of heart and dry rot of sugar beets with borax.** K Meyer-Hermann *Deut Zuckerrind* 60, 1019 (1935). *cf C A* 29, 4509—After the symptoms and the known causes of these diseases are enumerated, data are given of the increase in beet yield after treating the suspected fields with 10-15 kg of borax per acre. F R Bachler

**The woolly aphid parasite.** Effect of orchard sprays on *Aphis mali*. N S Noble *Agr Gas N S Wales* 46 23 (1935)—In lab expts, emergence of the wasps was not significantly affected by spraying parasitized woolly aphids (*Eriosoma lanigerum*) with either miscible white oil (1 40), nicotine sulfate (1 600-800) plus soap (1 lb / 25 gal), nicotine sulfate (1 600) plus miscible white oil (1 200) or lime-S (1 35). K D Jacob

**"Kelsey spot" on plums.** B J Dippenaar *Farming Africa* 10, 333-6 (1935)—Applications of N, P and K fertilizers had no effect on the incidence of the disease. K D Jacob

**Efficiency of commercial sodium cyanide and sulfonic acid in liberating hydrocyanic acid gas for fumigation.** M Shaikh and A Amer *Ministry Agr Egypt, Tech Sci Service Bull* 160, 5 pp (1935)—Satisfactory evolution of HCN was obtained by treating 1 part NaCN with a soln of 0.9 part com H<sub>2</sub>SO<sub>4</sub> in 1.6 parts ordinary tap water. K D Jacob

**Lead arsenates—analyses of samples, 1935 36.** W C Robertson *J Dept Agr Victoria* 33, 538-41 (1935). *cf C A* 28, 7410—Calcd on the dry basis, 14 samples of pastes and powders contained As<sub>2</sub>O<sub>3</sub> 30-32%, PbO 12-20-67% and H<sub>2</sub>O-sol As 0.17-0.43%. The percentage of As remaining in suspension after standing 5 min was 71.2-98.4 and after 30 min 13.92-87.00. The amt of As<sub>2</sub>O<sub>3</sub> in suspension 5 min after agitation was 2.41-3.54 g/gal. The acidity of the preps was 1.0-42.0 cc 0.01 N NaOH per 2.5 g, the acidity exceeded 5.3 cc in only 1 sample. K D Jacob

**Studies on contact insecticides.** VIII Technique for tracing penetration of petroleum oil in insect eggs. Some determinations of oil penetration into insect eggs. W C O'Kane and W C Baker *N H Agr Expt Sta, Tech Bull* 60, 1-12 (1934). *cf C A* 28, 6921—Two procedures were followed to trace the penetration of a petroleum oil into grasshopper eggs. (1) An unstained oil is applied to the egg, and sections are cut and stained in such a way as to make visible any globules of petroleum oil present within the sections. (2) A stained oil is used, after fixation with Bouin's soln, sections are cut and given each subsequent treatment as will differentiate any petroleum oil present. The stains used were osmic acid-red O, the Rohrbach stain of Nile blue sulfate-red O (*C A* 28, 1373), and osmic acid Nile blue sulfate-red O

<sup>1</sup> The petroleum oils used for sprays definitely penetrate the eggs of both the grasshopper, *Chortophaga* sp., and cockroach, *Periplaneta americana* IX Further determinations of oil penetrations into insect eggs *Ibid* 62, 3-8 (1935).—Further staining studies showed penetration of petroleum spray oils into the eggs of the snowy tree cricket, *Oecanthus nivicus*, the Mexican bean beetle, *Epilachna corrupta*, the squash bug, *Anasa tristis*, the pine leaf aphid, *Delicatus pini*, the lacewing, *Chrysopa oculata* and the cecropia moth, *Platysamia cecropia*. A predominance of oil was found in the micropylar region of the eggs. X Penetration of arsenic into insects. W C O'Kane and L C Glover. *Ibid*, 63, 1-S (1935)—Careful lab tests showed definite penetration of As in all organs of the cockroach, *Periplaneta americana*, after contact with As<sub>2</sub>O<sub>3</sub>. The As content of the different parts of the insect was detd by a micro Gutzeit test. Neither paralytic nor corrosive action was observed. The mechanism of penetration is unknown. C R Fellers

**Treatment of molasses for the production of fertilizers.** (Gaspary A Arnal) 12. Prepn of a high percentage Ca cyanamide by the action of NH<sub>3</sub> and CO on Ca oxide and Ca carbonate (Postnikov, *et al*) 18. Rept. of Wallaceville veterinary laboratory (Hopkirk) 11E. Rept. of chemistry section [on soils from littoral lands] (Aston) 11G

**Fertilizer.** Heinrich Tramm (to Rubchemie A-G). U S 2,025,915, Dec 31. An ammoniacal soln of NH<sub>3</sub>-U<sub>2</sub> is added to a mixt. of acids and other substances such as H<sub>3</sub>PO<sub>4</sub>, KCl, etc., until substantial neutralization of the NH<sub>3</sub> is effected, and the temp of the reaction is controlled by passing a strong current of NH<sub>3</sub> over the reacting mixt to produce a mixed fertilizer. U S 2,025,916 relates to a process in which HNO<sub>3</sub>, H<sub>3</sub>PO<sub>4</sub>, a fertilizing K salt and NH<sub>3</sub> are simultaneously introduced into a melt comprising a reaction product of HNO<sub>3</sub>, H<sub>3</sub>PO<sub>4</sub> and NH<sub>3</sub> in such proportions as to maintain continuously a slightly acid or neutral reaction in the melt, to form a fertilizer. App is described.

**Fertilizers.** Zjednoczone Fabryki Zmazków Azotowych w Moszczach i w Chorzowie *Austrian* 143,305, Nov 11, 1935 (Cl 16). In the manuf. of granular fertilizers by spraying a melt contg NH<sub>4</sub>NO<sub>3</sub>, a phosphate or carbonate of Ca, and not more than 20% of a flux, e.g., Ca(NO<sub>3</sub>)<sub>2</sub> or Mg(NO<sub>3</sub>)<sub>2</sub>, nonhygroscopic products are obtained by adding to the melt, immediately before it is sprayed, a substance which reacts with the flux to form a water-insol compd., e.g., (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> or (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>.

**Fertilizer.** Hubert Kappen *Ger*, 618,644, Sept 12, 1935 (Cl 16 G). Mg salts with water of crystn are treated with CO<sub>2</sub> and NH<sub>3</sub> to give a fertilizer contg MgCO<sub>3</sub>. In examples, MgCl<sub>2</sub>·6H<sub>2</sub>O and MgSO<sub>4</sub>·7H<sub>2</sub>O are used, giving a mass contg MgCO<sub>3</sub> together with NH<sub>4</sub>Cl and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, resp. *cf C A* 30, 563.

**Phosphates and fertilizers.** Soc d'études pour la fabrication et l'emploi des engrais chimiques (Robert Flatt, inventor) *Fr* 787,201, Sept. 19, 1935. Natural phosphates are treated with HNO<sub>3</sub> of such a concn., and in such amt. that the Ca(NO<sub>3</sub>)<sub>2</sub> and H<sub>3</sub>PO<sub>4</sub> formed remain dissolved. The unattacked muds are sepd and the clear liquor is neutralized by the addn. of substances more basic than Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> (CaO, CaCO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, NH<sub>3</sub>, CaH<sub>2</sub>PO<sub>4</sub>, or Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>) which causes the Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> to ppt, and the nitrates remaining dissolved are transformed in known manner. In a variation the amt of HNO<sub>3</sub> used is sufficiently small or (and) sufficiently strong to cause the Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> to ppt during the attack of the crude phosphate, thus Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> is sepd or not sepd from the liquor with the muds before continuing the process as above.

**Continuous apparatus for making superphosphates.** René Moritz *Fr* 787,620, Sept. 20, 1935.

**Insecticide.** Leon C Heckert (to Rohm & Haas Co.). U. S. 2,024,098, Dec 10. An aromatic thiocyanate the org residue of which contains a neg substituent comprising a carbonyl group, an aryl radical, an unsubstituted org radical, a nitro group, a halogen, a cyanide group, an alkyl group

or an org. radical bound to O as in groups of the type of 1 alkoxy and ester groups, such as benzyl thiocyanate, *p*-thiocyanodimethylaniline or the like is used as a vapor or spray ingredient

Insecticides Herbert Schotte and Karl Görmütz (to Schering-Kahlbaum A.-G.) U. S. 2,024,392, Dec. 17. Rotenone and veratrine or drugs containing them are used with talc, etc.

Insecticides Erik G. A. Wikström Brit. 434,660, Sept. 6, 1935. Horticultural, etc., insecticides consist of 2 cymol, or sulfonated, chlorinated, nitrated or other deriv. thereof, together with the active constituent of pyrethrum, derris or quassia, or an equiv. vermin-destroying agent of vegetable origin. Etheral oils, soap and emulsifying agents may also be added. In 434,661, Sept. 6, 1935, the cymol or deriv. is mixed with an etheral oil, e. g., eucalyptus, cedar wood or citronella, that has been chlorinated

or sulfurated by treatment with aqua regia, liquid Cl<sub>2</sub>, aq. Cl<sub>2</sub> soln., H<sub>2</sub>SO<sub>4</sub>, S chloride or an alkali sulfide or polysulfide. Soap and an emulsifying agent may also be added.

Insecticidal and fungicidal solutions James W. Van Meter (one-half to G. Edward Fetter). U. S. 2,025,365, Dec. 24. Reactive agents such as S and Sb in a container are immersed in an absorbent liquid such as water and heat to promote desired reaction is induced by local heating such as use of a fuse and Mg primer. App. is described.

Fumigant which can be used on seeds, bulbs, foods, etc. Richard T. Cotton and Harry D. Young (to the Government and People of the U. S.) U. S. 2,024,027, Dec. 10. For combating insects such as *Tribolium confusum*, etc., solid CO<sub>2</sub> is used with ethylene oxide, CS<sub>2</sub>, or other fumigants. Various examples and operative details are given.

## 16—THE FERMENTATION INDUSTRIES

C. N. FREY

Theories of fermentation. A critical study and review S. S. Epstein and S. Laufer. *Am. Brewer* 68, No. 1, 14-15, 31, No. 2, 22-3, No. 3, 22-5, No. 4, 22-5, No. 5, 24, 26-7 (1935).—The old and new theories of fermentation are reviewed with particular reference to brewing practice. S. Laufer.

The production of solvents by fermentation I. S. Logotkin and B. N. Cheboksarov. *J. Chem. Ind. (Moscow)* 12, 828-37 (1935).—The operation of the Gromenskii acetone-butyl alcohol factory is described.

H. M. Leicester. Fermentation method for production of *d*-lactic acid E. L. Tatum and W. H. Peterson. *Ind. Eng. Chem.* 27, 1493-4 (1935).—*d*-Lactic acid is prep'd by fermenting a medium containing 3% cereose and 3% malt sprouts, with a *d*-lactic acid culture (mostly *L. delbrückii*) at 30-37°, depending on the organism, after 24 hrs. an excess of sterile CaCO<sub>3</sub> is added and fermentation continued 6 days with frequent shaking. The yield of pure *d*-acid was 90-95% of sugar used. Malt sprouts with glucose in concns up to 10% may also be used. O. W. Wilcox.

Influence of vitamin C on yeast fermentation and lactic acid formation in *Aerobacter aerogenes* cultures and in muscle pulp K. Zipf and M. Thurnau. *Biochem. Z.* 282, 51-5 (1935).—Ald. yeast fermentation is not affected by vitamin C which, however, inhibits lactic acid formation by *Aerobacter aerogenes* (in concn of 1/250 or more). The lactic acid formation in muscle pulp is likewise inhibited by vitamin C (concn. 0.02 M or more) but this effect does not materialize on the addition of glycogen.

S. Morgulis. The reesterification reaction of phosphoryluric acid in alcoholic fermentation of sugar. O. Meyerhof and W. Kiessling. *Biochem. Z.* 281, 240-70 (1935).—The velocity of cleavage of phosphoryluric acid depends upon the fermentation velocity of the sugar which is esterified with the liberated H<sub>2</sub>PO<sub>4</sub>. When hexose is no longer fermented in the presence of fluoride, 2 mols. each of CO<sub>2</sub>, alc., hexosediphosphate and phosphoglyceric acid arise and 2 mols. of phosphoryluric, hexose and H<sub>2</sub>PO<sub>4</sub> disappear. In the presence of CH<sub>3</sub>CO-H the velocity of fermentation is the same but only 1 mol. hexosediphosphate is formed and the acetaldehyde remains unchanged. A revised scheme is offered for the fermentation reaction. S. M.

Enzymic synthesis of some glucosides. I. Vantileen, C. N. Ionescu and A. Klyzk. *Bul. soc. chim. Roum.* 17, 131-6 (1935).—To prep. trimethylglycol- $\alpha$ -glucoside (I), 50 g. glucose, 100 cc. H<sub>2</sub>O, 250 cc. trimethylglycol (II) and 100 cc. of a 10% yeast suspension are added to a vol. of 500 cc. and fermented for 6 weeks. Distn. of the filtrate at 15 mm. pressure removes II at a temp. of 135-140°. Unbound glucose is removed by dissolving the residue in 500 cc. H<sub>2</sub>O and after the addn. of 2-3 g. fresh yeast allowing fermentation to proceed further for 8-10 days. Subsequent decolorization with charcoal

and further distn. under reduced pressure leave a residue (III) which fails to crystallize. Acetylation of III with Ac<sub>2</sub>O in C<sub>6</sub>H<sub>6</sub>N gives the pentaacetyl deriv. (IV) of I, m. 84°, with [ $\alpha$ ]<sub>D</sub><sup>20</sup> 112.62° (EtOH). Sapon of IV with Ba(OH)<sub>2</sub> gives I, m. 96°, with [ $\alpha$ ]<sub>D</sub><sup>20</sup> 137.48° (AcOEt). Mannose- $\alpha$ -monoglucoside, m. 140°, with [ $\alpha$ ]<sub>D</sub><sup>20</sup> 83.41°, is obtained by a similar fermentation method, but in this case the unbound reactants are sep'd. by fractional crystaln. in alc., this obviates the necessity of acetylation.

W. J. Peterson. Calcium gluconate from juice of cull and surplus apples. Clifford Frost, J. L. St. John and H. W. Gerritz. *Ind. Eng. Chem.* 28, 75-9 (1936).—A study has been made of the relative fermentative capacity of apple juice when subjected to the action of gluconic acid forming organisms as *B. xylinus*, *B. aridus*, *P. diacetylum*, *P. purpureum* and *B. citrinum*. The first 3 grow poorly, the last 2 grow well in apple juice. The best yields were obtained with *P. citrinum*. Calcd. in terms of glucose employed, good yields of gluconic acid resulted through fermentation by *P. citrinum*. Based on total sugar used, however, the yield was low. Fermentation by *P. citrinum* produced gluconic acid from sucrose and glucose solns. but not from fructose soln. This probably explains the low yield of gluconic acid based on the total apple-juice sugars destroyed. W. O. E.

Study on hops F. M. Dupont. *Am. Brewer* 68, No. 11, 21-3 (1935).—Problems of improving the quality of American hops are discussed, and recent investigations on the bitter principles are reviewed. S. L.

Pa and its importance in brewing. Max Wallerstein. *Am. Brewer* 68, No. 11, 23-8, No. 12, 52-4 (1935).

S. Laufer. The "break" of unstable albuminoids (in brewing). P. Petit. *Brasserie & malterie* 25, 273-7 (1935).—A brief discussion of the mechanism of and principal factors which affect the "break" of albuminoids in brewing.

A. Papneau-Couture. Hydrogenation of freshly distilled spirits. Carroll A. Hochwalt, Charles A. Thomas and Ernest C. Dyball. *Ind. Eng. Chem.* 27, 1404-7 (1935).—In the aging of whisky 2 distinct processes are recognized: (1) removal of unpleasant odor and taste (greenness) which is here shown not to depend on oxidation; and (2) maturation, involving chem. esterification and polymerization, imparting body and mellow taste. The rates of 1 and 2 are different, 2 being the faster. It is found that 1 can be accelerated by hydrogenation (reduction) in vapor phase (Ni catalyst) or in liquid phase (Pt black); 3 lb. Ni is sufficient for 1500 gals. freshly distd. spirits. After hydrogenation the whisky can be mellowed in much less than the usual time. O. W. Wilcox.

A simple micro-method for the determination of arsenic in must and wine. Joseph Burkard and Bernhard Wallerstein. *Z. Unters. Lössm.* 70, 308-15 (1935).—This

method depends on the distn of the As as  $AsCl_3$  and the conversion of the latter into  $H_2AsO_4$ . This is detd by the Zinzadse colorimetric method (C A 24, 5255). The method described can be used in series detns for As in foodstuffs and other materials. The accuracy of the colorimetric method of Zinzadse can be considerably increased so as to det 0.01-0.05 mg. of As. P. L. D.

Changes in whisky stored for four years. Peter Valaer and W. H. Frazier. *Ind Eng Chem* 28, 92-103 (1936).—The largest increase of acids, esters, solids and color is during the 1st 6 months of storage. There was no fixed relationship between the acids and esters as found by Crampton and Tolman (C A 2, 683) at the end of 4 years. The acid-ester ratio did not reach a ratio of 1:1. There was an actual gain in acids over the 4-yr. period of 24.9-56 g. per 100 l. (av 40 l.) when calcd to original vol. Similarly, there was an actual gain in esters of 7.4-21.3 g. per 100 l. (av 15.5). There was an actual loss of fusel oil during the aging period which varied from 6.9 to 58.4 g. per l. (av loss 28.6), when calcd to the original vol. Quick-aging by a heat treatment increased the color, solids and acids of whisky. Storage of whisky in glass usually caused a decrease in acids and a tendency for esters and color to increase. Practically all the slop taste and odor disappeared after aging for 4 years in glass. The constituents of whisky are constantly undergoing changes during the aging process. The increase in acids in charred bbl is due partly to fixed acids extd. from the wood but mostly to the formation of volatile acids during the aging process. A total of 198 samples drawn from 11 different distilleries was used in this investigation. Approx 1584 detns. were made on these samples.

Carcinæ (in beer). P. Petit. *Brasserie & Malterie* 25, 290-4 (1935).—A brief discussion of the cause of the presence and development of carcinæ in beer.

Has beer a temperature of maximum density? Philip P. Gray. *Am Brewer* 68, No. 6, 16-18 (1935).—The temp. of max. density was detd for a no. of samples of market beer with results indicating a range of 0-2° for this value. The amt. of real ext. affected the density, while the alc. content was without influence. S. L.

Areometric determination of the yeast content of wort and its application to the separation of yeast in yeast factories. Witah Heller. *Przemysł Chem* 19, 3-9 (1935).—The yeast content can be detd areometrically, with a mean error of 0.25-0.5%, according to the d. of the wort. A no. of formulas are given, connecting the ds. of solns. with their yeast contents, they are applicable to the control of the various stages of yeast production.

B. C. A.

Ability of yeast to assimilate higher- and lower-molecu-

lar nitrogen compounds. Niels Niel-en. *Compt rend trav lab Carlsberg, Ser Physiol* 21, 139-50 (1945), (C. A. 30, 232).—The N compds. of beer wort were sep'd by dialysis through collodion and then used in nutrient media. Yeast could assimilate the lower-mol. fractions far better than the higher. Probably not more than half the N in wort is assimilable by yeast. J. J. W.

Alcoholic fermentation of hydrol. David A. Legg (to Commercial Solvents Corp.). U. S. 2,226,237, Dec 31 Hydrol. at a sugar concn. suitable for yeast fermentation, is subjected to acid hydrolysis, and a mash prep'd from the product is subjected to fermentation with yeast (suitable after adding  $NH_3$  to produce a suitable pH and with admixt. of relatively small proportions of ground corn products or the like which may be hydrolyzed with the hydrol). Various details and modifications of procedure are described.

Lining ferroconcrete fermentation vessels and like containers with composition sheets containing asphalt, wax, pitch, etc. Borsari & Comp. Ger. 621,288, Nov 4, 1935 (Cl 6b 17). A method of procedure is described.

Apparatus for aerating fermentation vats. Strauch & Schmidt. Ger. 621,350, Nov. 6 1935 (Cl 6a 15 03).

Aerating and deaerating means for rotary drying or malting drums. J. A. Topf & Söhne. Ger. 621,449, Nov 7, 1935 (Cl 6a 4).

Fermentation of worts. Joseph Revelin and Jean Mercier. Fr. 787,666, Sept. 26, 1935. Means is described for supplying  $SO_2$  to the vats to moderate the fermentation.

Wort-clarifying vat. Friedrich Schmatz. Ger. 618,560, Sept. 11, 1935 (Cl 6b 11). Details of regulating the running off are given.

Beverage. Felix Werner. Ger. 621,289, Nov 4, 1935 (Cl 6c 3). A foaming alc. beverage is prep'd by adding sugar to a decoction of unmalged grain, preferably rye, wheat or maize, and fermenting the product.

Clarifying beverages. Chemische Werke Marienfelde A.-G. Ger. 558,369, Oct. 4, 1935 (Cl 6d, 3). See U. S. 2,002,145 (C. A. 29, 4219).

Beer. Heinrich van de Sandt. Ger. 618,890, Sept. 18, 1935 (Cl 6b 8 02). Wort or beer with a considerable yeast content is given an addn. of liquid contg. vitamins and heated. The vitamin liquid is adulterated with nonheated normal wort or beer. The resulting wort or beer mixt. is fermented with fresh yeast. Examples are given. Cf. C. A. 29, 52191.

Yeast. Soren Sal. Ger. 620,093, Oct. 31, 1935 (Cl 6a 17 02). Addn. to 607,234 (C. A. 29, 1936). This corresponds to Brit. 308,324 (C. A. 24, 201), but includes a sp. example.

## 17—PHARMACEUTICALS, COSMETICS AND PERFUMES

### W O E M E R Y

Improved deodorants. S. P. Jannaway. *Perfumery Essent. Oil Record* 26, 375-7 (1935).—A short discussion with formulas of liquid, cream, powder and stick deodorants. Philip D. Adams.

The use and abuse of fixatives. II. A problem of present-day perfumery. Frank Atkins. *Perfumery Essent. Oil Record* 26, 378-81 (1935).—A discussion.

Manufacture of cachous. A. G. Arend. *Perfumery Essent. Oil Record* 26, 380-8 (1935).—A discussion of the problems and prep'n of mouth- and breath-perfuming materials. Philip D. Adams.

A brief study of cinchonas. Luis Paria. *Rev quim farm (Brazil)* 1, 73 (1935).—Historical. E. S. G. B. Essential oil of *Cinchthonium maritimum* L. G. A. Pevtsov. *J. Gen. Chem.* (U. S. S. R.) 5, 1185-91 (1935).—The essential oil obtained by steam distn. of the ripe fruit of *Cinchthonium maritimum* L. grown in Crimea is composed of 90% hydrocarbons, of which limonene,  $\beta$ -cymene and

sabinene were identified. A tertiary alc.,  $C_{15}H_{27}OH$ , b. about 205°, was isolated. *Cislimene* found in the French and Italian variety is not present in the Crimean plant. Twenty references. Chas. Blanc.

Nature of the crystalline substance in the essential oil of *Lachnophyllum gossypinum* Bge. V. V. Williams, V. S. Smirnov and V. P. Gol'mov. *J. Gen. Chem.* (U. S. S. R.) 5, 1195-1204 (1935).—*Lachnophyllum gossypinum* Bge., an annual plant growing wild in Central Asia, gave an essential oil, d<sub>4</sub> 0.8944, n<sub>D</sub> 1.526, solidifying to a cryst. mass at -3° and carbonizing with a flash of flame at 175°. It contains 58% of hydrocarbons, among which were identified  $\beta$ -pinene and camphene, and cryst.  $C_{15}H_{27}O$  (I). The oil on being cooled to -15° sep'd 30% I, m. 32.6-2.8° (dil. alc.), d<sub>4</sub> 0.989, d<sub>20</sub> 0.932, n<sub>D</sub> 1.5255, n<sub>D</sub> 0, M. R. 50.82 (calcd.), M. R. 57.2 (obs.), exaltation 6.65, insol. in  $H_2O$ , sol. in alc.,  $Et_2O$ ,  $CH_2Cl_2$ , and 40%  $CO_2H$ . I on exposure to the daylight is decomp'd. with discoloration from yellow to orange. In the air I de

composes with formation of a product irritating to the nose and eyes. Attempts to distil I under atm. pressure and in *vacuo* resulted in a rapid decompn with a flash and carbonization. The most probable structure of I,  $\text{PrC} - \text{CC} \cdot \text{CCH} \cdot \text{CHCO}_2\text{Me}$ , is based on analysis and identification by degradation. Thus, the action of aq. KOH caused besides the usual sapon the addn of  $\text{H}_2\text{O}$  at the triple bonds with the subsequent decompn of the  $\beta$ -diketone and formation of methyl butyl ketone and butyric acid on the one hand and valeric and acetylacrylic acids on the other hand (cf. Wolff, *Ann.* 264, 253 (1891)). Oxidation with 50%  $\text{HNO}_3$  gave butyric acid and considerable oxalic acid. I reduced by catalytic hydrogenation was converted into methyl caprate. With HI it was partially saponified with the formation of MeI. The parachors, detd. and calcd. by the method of Mumford and Phillips (*C. A.* 24, 5258), agree well  $P_{\text{calcd.}} = 440$ ,  $P_{\text{obs.}} = 450$ . The presence of a compd. with a triple bond in plants was previously observed only in 2 other cases (*Compt. rend.* 134, 842, *Bull. soc. chim.* [3], 27, 484 (1902), *C. A.* 27, 479b). I strongly affects the sympathetic nervous system even in small doses.

**Tobacco products.** I. Constantin Prikri and Hans Dittmar *Z. Unters. Lebensm.* 70, 297-306 (1935).—Results are given of the nicotine, total reduction of the carbonyl groups, the polyphenols and the calcd. polyphenol coeff. found in cigars, cigars and pipe tobacco purchased on the Dresden market. F. L. Dunlap.

**Heracleum lehmannianum.** I. Vasserman *Am. Perfumer* 31, 65-7 (1935).—This new source of anethole yields at the rate of 80% from the leaves. The plant grows naturally and luxuriantly in Central Asia, and is susceptible of lucrative cultivation. The essential oil is contained in the leaves and petioles, its rate varying from 0.25 to 0.40% of the wt. of freshly cut leaves. In the plant the oil is represented by glucosides, so that the rate of liberation depends on the enzymes causing their degradation into glucose and essential oil. The sources of anethole, anise and fennel, heretofore employed are from an agricultural standpoint inferior to *Heracleum*, in that it is apparently not subject to disease, and furthermore furnishes a good fodder for cattle. The ash of this plant may be utilized for prep. a black soap of excellent quality.

**Oil of celery.** Ernest S. Guenther *Am. Perfumer* 31, No. 3, 75-8 (1935).—The celery-oil industry in the Rhone valley and adjacent territory from the earliest (Phoenician, Roman and Saracen) down to the present period is described.

**Simple volumetric estimation of mustard oil in spiritus sinapis.** H. Kaiser and E. Furst *Apoth. Ztg.* 50, 1734-5 (1935).—An app. is shown and described whereby it is possible to det. mustard oil volumetrically in 15-20 min. The app. consists essentially of an inverted 150-cc. pear-shaped vessel having a side tube through which the sample 10 g. and about 150 cc. satd.  $(\text{NH}_4)_2\text{SO}_4$  soln. are introduced. The inverted upper end extends to a narrow tube graduated in  $\frac{1}{10}$  cc. The lower end connects with a rubber tube carrying a pinchcock, said tube connecting by means of a glass tube with cock with a long rubber tube attached at the other end to a globular 150-cc. buret, contg. further satd.  $(\text{NH}_4)_2\text{SO}_4$  soln. After careful agitation of the pear-shaped vessel, whereby the oil is salted out, the globular buret is raised to force the pptd. oil into the graduated tube, the vol. of which may then be noted.

**Camomile and peppermint of the 1935 vintage.** Hanns Will. *Apoth. Ztg.* 50, 1757-8 (1935); cf. *C. A.* 29, 883<sup>2</sup>.—Oil contents are reported for 30 samples of camomile and 26 samples of peppermint.

**Investigation of drugs.** Kurt Handke. *Apoth. Ztg.* 50, 1778-9 (1935).—H. is undertaking to show from the results of his analytical experience some of the shortcomings in pharmaceutical control. The present paper covers the official preps. calcium lactum and magnesium peroxidum.

**Homeopathic preparations. IX. Essences and tinctures containing arbutin.** A. Kuhn and G. Schäfer.

*Apoth. Ztg.* 50, 1800-3 (1935); cf. *C. A.* 30, 569<sup>1</sup>.—As characteristic constituents of the homeopathic essences and tinctures of *Chimaphila umb.*, *Psoralea unifl.* and *rolundif.*, *Epigaea rep.*, *Gaultheria proc.*, *Kalmia latif.*, *Ledum palustre*, *Rhododendron chrys.* and *ferrug.* and *Uva ursi* arbutin, hydroquinone, ercolin, ursone, rhododendrin, monotropiside, ascebin and romedotoxin are briefly characterized. In the case of tinctures arbutin, hydroquinone and ercolin are characteristic ingredients.

A peculiarity in the capillary picture of *Chimaphila umbellata* is described, namely the yellow crystals occurring in the middle zone of the capillary picture are ursone. Erroneous literature data on the fluorescence of the capillary pictures and of arbutin are rectified. These tinctures were evaluated by estn. of their arbutin and hydroquinone contents. The ratio of the split to unchanged glucoside (hydroquinone-arbutin) in tincture and dry plant has been detd. Among the tinctures examd. were those belonging to the *Pyrolaceae* and *Ericaceae*. The constituents and pharmacological action of members of these families are reported. The detection of arbutin, hydroquinone, ercolin, gaultherin, rhododendrin, tannin reactions, ursone and ercolin is described. X. Analysis of coumaric acid preparations. *Pharm. Ztg.* 80, 1253-5 (1935).—In certain homeopathic preps. contg. derivs. of coumaric acid are coumarin, umbelliferone, daphnin, fraxin and scopolin. These substances are briefly characterized chemically. A characteristic test for this group is described. Likewise are described for the pure substances and their tinctures characteristic fluorescence phenomena under filtered ultraviolet light. Capillary pictures of such tinctures are described and their special characters discussed. The detection limits of their fluorescence are further characterized through special chem. tests.

**Alkaloids of European Lycopodium species.** J. Muszynski. *Arch. Pharm.* 273, 452-7 (1935).—From the results obtained in the examn. of a series of *Lycopodium* species it appears that alkaloids are characteristic constituents thereof. Some of these alkaloids are volatile and some fixed. They are highly toxic and of comine-like action. The fixed bases are sol. in  $\text{H}_2\text{O}$ , extractable with  $\text{CHCl}_3$  and yield yellow, amorphous, very bitter salts. The volatile bases are likewise water-sol. and yield hygroscopic salts.

**Step photometric measurements of tinctures.** P. W. Danckwort. *Arch. Pharm.* 273, 467-75 (1935); cf. *C. A.* 28, 5397<sup>1</sup>.—The examn. of tinctures by means of the Pulfrich step photometer appears to promise gratifying results. Many tinctures yield such characteristic curves that recognition becomes possible. The changes in tinctures on standing and in absence of light are not great, so that the typical characteristics of the curves (the measuring ranges and break points) show distinctly after 1 yr. Furthermore, the graded photometric study gives a good insight into the aging process of tinctures.

**New and improved methods for the synthesis of pharmacologically important amines X. Catalytic hydrogenation of  $\alpha$ -mrostyrenes to  $\beta$ -arylethylamines.** Karl Kinder and Eduard Brandt. *Arch. Pharm.* 273, 478-83 (1935).—Mainly a review.

**Polymorphism of phenobarbital.** A. Kofler and R. Fischer. *Arch. Pharm.* 273, 483-7 (1935).—Phenobarbital crystallizes in 3 modifications belonging apparently to the monoclinic system and characterized by their refractive indices,  $n_{\text{D}}$ , and in part by their crystal form. The stable crystals  $m$  174°, the unstable ones  $m$  156-7° and 166-7°, resp. Photographic illustrations of the 3 types are shown.

**Leucithin and hydroquinone as antioxidants for vitamin A.** Harry N. Holmes, Ruth E. Corbet and Eva R. Hartzler. *Ind. Eng. Chem.* 28, 133-5 (1936).—Hydroquinone and leucithin were studied as antioxidants for vitamin A in halibut-liver and cod-liver oils at both room and higher temps. Each affords protection for the vitamin in varying degree, depending on the concn. of the antioxidant. A combination of the 2 affords a remarkable protection,



much greater indeed than would be expected from additive effects. W O E.

Solid pharmaceutical hydrogen peroxide compounds and their stability. G. Schneider and R. Folger. *Pharm. Zentralbl.* 76, 737-7 (1935).—A comparative study has been made of the stabilization of H<sub>2</sub>O<sub>2</sub> when combined with various substances, notable with urea and Na<sub>2</sub>P<sub>2</sub>O<sub>7</sub>. Combinations with anhyd. Na<sub>2</sub>P<sub>2</sub>O<sub>7</sub> are almost as stable as are those with urea. W O E.

Salt diet. F. Hoffmann. *Pharm. Ztg.* 80, 1313-14 (1935).—The various special brands of table salt are discussed from a dietary standpoint in connection with S. references. W O E.

Economy in the sterilization of surgical dressings. R. Hahn. *Hygien. Staatsanstalt (Hamburg)*. *Pharm. Ztg.* 80, 1313-14 (1935).—10 references.

Rehmann and the homeopathic pharmacopoeia. W. Peyer (von Breslau). *Pharm. Ztg.* 80, 1320-1331 (1935).—A series of 32 homeopathic preps. are enumerated comparatively with those taken from Hahnemann's early papers. W O E.

Rotatory dispersion of essential oils. Bernard Angier. *Ann. chim. anal. chim. appl.* 17, 341-7 (1935).—To illustrate the application of the rotatory dispersion for the establishment of the purity of an oil, the results actually obtained in the exam. of geranium oil from Algeria are described. W. T. H.

Chemical examination of the bark of *Terminalia arjuna* Bedd. I. Isolation of arjunin. Radha Ramani Agarwal and Subhadrachari Datt. *Proc. Acad. Sci. United Provinces. Agra Coll.*, India 5, 50-4 (1935).—The ext. of the bark of *Terminalia arjuna* was investigated for the active principle responsible for its long use as a powerful astringent and diuretic. A colorless, cryst. compd., arjunin, C<sub>22</sub>H<sub>22</sub>O<sub>10</sub>, was obtained from a benzene ext. of the plant. It is an acid in 10%. It is probably an aglycone of a phloeoide present in the plant. The Pb, Ag and Na salts, the dacteryl diglycerol and pentaerythritol derivs. were prepd. Its physiol. properties are being studied. C. E. P. J.

Emulsifying agents in the pharmaceutical and cosmetic industries. Fr. Heemann. *Arznei. Chem.* 43, 773-6 (1935).—A discussion with five references. K. K.

An interesting method of adulterating rad. valentia. G. Oehm and M. Psova Kardova. *Časopis Československých Lékařů* 15, 225-8 (1935).—The adulteration of this drug by means of chavory root (*Cichorium intybus*) is described. It was so clever that it escaped the observation of a specialist. The adulteration can be easily ascertained by placing a small amt. of the drug in a vessel filled with water—some pieces for some time float on the surface while others begin sinking at once, being surrounded by clouds of dark-brown powder falling at the same time to the bottom. These yellowish or whitish pieces are the pieces of chavory root which were in a certain way surrounded by dark brown and completely useless powder. The chavory can be proved macroscopically and chemically by means of molin. V. D. K.

The bactericidal and photochemical properties of irradiated petrolatum and mineral oil. Franklin A. Stevens. *J. Lab. Clin. Med.* 21, 20-30 (1935).—The photoactivity of petrolatum or paraffin oil irradiated with ultraviolet light in the presence of O<sub>2</sub> is due to the evolution of gaseous org. peroxides and aldehydes which diffuse readily through a film of cellulose acetate. Neither the irradiated oils nor the gaseous oxidation products are bactericidal *in vitro*. E. R. Mann.

Iodured oil. A practical method of preparation. Rav M. Balvart, Everett Seiler and Virgil Outbier. *J. Lab. Clin. Med.* 21, 187-91 (1935).—Prepare ICl<sub>3</sub> by the action of Cl<sub>2</sub> on 55 g. of I crystals at 15° and dissolve in 150 cc. of water, keeping the temp. at 5-15°. Add the product with agitation to 100 cc. of poppy-seed oil. Pour into 100 cc. of CHCl<sub>3</sub>, shake and discard the aq. layer. Remove the CHCl<sub>3</sub> by warming to 80° and filter. The product has d<sub>4</sub><sup>20</sup> 1.241-28, I 22-25%, Cl 6-8%, and should contain no free I or free acid. E. R. Mann.

Tincture of iodine. L. Vigor. *Bull. sci. pharm.* 42, 191-8 (1935).—Tincture of I kept in bottles not sealed hermetically becomes more concd. by evapn. of EtOH. Metallic caps with paraffin-covered inside liver prevent such changes. A. E. Meyer.

*Juniperus thurifera* L. and its essence. Louis Revol. *Bull. sci. pharm.* 42, 577-89 (1935).—A description of the plant and account of its distribution is given. The leaves and fixed ext. comes in guinea pigs and dogs an intense congestion in the intestines and the genito-urinary organs. Essence has been obtained by distn. with steam. It has a sp. gr. slightly above 0.88, is d-rotatory and contains only a small quantity of esterified alc.; 70% dissolves below 170°. Inhalation of the vapors kills mice, but the tincture in guinea pigs, dogs and cats is comparatively low; it did not cause abortions at doses at which saline oil would do so. Conclusion: The toxicity of the plant is not caused by the essence alone. The inadvisability of its official use is discussed. A. E. Meyer.

The acetyl index (by acetylation in pyridine) of the essences of the French Pharmacopoeia. R. Delaby and V. Brignon. *Bull. sci. pharm.* 42, 589-97 (1935).—The index is given as g. AcOH fixed by 100 g. of essence. The pyridine acetylation data, only primary and secondary alcs. It is of value for the following essences: neroli (orange blossom) 5.4, mentha 32.5; rose 38.5, saral 78.1-79.6. It is necessary to consider the initial alc. and a possible EtOH content. The data of esters and of free alcs. permits the calcul. of total alcs.; this avoids the long and often inaccurate detn. by acetylation. A. E. Meyer.

The detection of small quantities of hashish (*Cannabis indica*) in different drugs by the use of filtered ultraviolet rays of Wood. Joseph Khouri. *Bull. sci. pharm.* 42, 593-612 (1935).—While cannabis gives an intense fluorescence many other drugs show no fluorescence at all. The use of exts. obtained with different solvents supplies a further means of differentiation. A. E. Meyer.

Experiments to isolate the autanemic principle of the liver. Burger Strandell. *Acta Med. Scand., Suppl. LXXI*, 52 (1935).—A preliminary report of the prepn. of a liver ext. of which 0.2 mg. corresponds to 100 g. liver, and which is highly effective against pernicious anemia when an aq. soln. of 2 mg. is injected intragastrically. S. Morfals.

Therapeutic and dietetic properties of "karkade" (*Rhusus sabbardana* L.), a new colonial pink tea. P. Roreux. *Formules Ind. 3*, No. 1, 13-16 (1935). *Chim. & Industrie* 34, 1150.—The product contains 2 coloring matters, *karkasin* and *purpurin*, it is used, in combination with a natural base, for coloring syrups and liquors. It contains no active principle and can be used as a substitute for tea and coffee for persons who are sensitive to excitants. The therapeutic properties of karkade are due to its citric acid content and to the presence of a large amt. of an emollient and sedative mucilage. Its effects on the organism are: abundant diuresis accompanied by slightly diaphoretic action, activation and neutralization of hepatic secretion, activation of gastric secretion and intestinal contractions which permit of rapid digestion, decrease in hyperviscosity of the blood and in arterial pressure, whence its efficacy in arteriosclerosis; its opioric action has a favorable effect on the functions of the stomach; it possesses a high intestinal antiseptic action and can be used to combat various infectious intestinal diseases; it gives a euphoric impression and acts as a reconstituent in spite of the fact that it possesses weight-reducing properties. A. Papenau-Couture.

The saponins. A. Wall. *Rev. sci. med. nat.* 39, 437-8 (1935), of Teichsche, C. A. 29, 7338.—The chemistry and applications of saponins are described. Mention is made that the saponins of marrows and quills root are most commonly used. Halser E. Sillman.

The preparation and stability of Dakin solution. Svend A. Schou. *Dansk Tids. Farm.* 9, 215-14 (1935) (summary in French).—The prepn. of a soln. of NaOCl contg. 0.5% active Cl (Dakin soln. (B)) sufficiently alk. to be stable and yet nonirritating to the tissues was investigated. Conversion of Ca(OCl)<sub>2</sub>, triturated in water,

with  $\text{Na}_2\text{CO}_3$  is inapplicable on account of the uncontrollable amts. of Ca compds. introduced. In using a filtered, concd. soln. of  $\text{Ca}(\text{OCl})_2$ , it may be assumed that the content of  $\text{Ca}(\text{OH})_2$  will be within narrow limits. To det.  $\text{Ca}(\text{OH})_2$ , add 20 cc. 3%  $\text{H}_2\text{O}_2$  to 20 cc. soln. and heat until the evolution of gas ceases. Titrate with 0.1 N  $\text{HCl}$  (methyl red indicator). The amt. of  $\text{Ca}(\text{OH})_2$  in freshly prep'd, filtered solns. of  $\text{Ca}(\text{OCl})_2$  (19) was almost const. and corresponded to 7.5 cc. 0.1 N  $\text{HCl}$  per 20 cc. The amt. of  $\text{NaOH}$  formed by the conversion with  $\text{Na}_2\text{CO}_3$  can therefore be considered const. and be eliminated with a const. amt. of  $\text{NaHCO}_3$ . Very basic solns. contg. excess of  $\text{Na}_2\text{CO}_3$  are very stable, only 4% of the content of active Cl being lost after 11 months at 25°. A calcn. of the variation in the final reaction of I prep'd by adding a const. amt. of  $\text{NaHCO}_3$  to  $\text{NaOCl}$  solns. contg. 2 to 7 g.  $\text{Na}_2\text{CO}_3$  per kg. shows that the resulting reaction in all cases will be between  $pH$  9.2 and 10.0 and thus suitable for therapeutic purposes. These solns. were, however, unstable and the more so the lower the original content of  $\text{Na}_2\text{CO}_3$ . I should therefore be prep'd as follows: Add 30 g.  $\text{Na}_2\text{CO}_3$  dissolved in 270 g. water to 200 g. filtered, concd.  $\text{Ca}(\text{OCl})_2$  soln. contg. 2.4–2.6% active Cl (prep'd from standard  $\text{Ca}(\text{OCl})_2$ ), contg. 25–35% active Cl. Stir and let stand until the ppt. is cryst., filter and wash with sufficient water to make 500 g. filtrate. This strongly basic  $\text{NaOCl}$  soln. is stable for 1 year and can be kept as stock in a cool place protected from light. When it is dispensed, add 10 g.  $\text{NaHCO}_3$  dissolved in 400 g. water. The final prep'n. thus contains 0.5% active Cl, has a suitably low  $pH$  and may be considered stable for 2–3 weeks. D. Thuesen

**Investigations on methylatropine bromide.** The stability of solutions of methylatropine bromide and the determination of methylatropine. F. Reimers *Dansk Tids. Farm.* 9, 215–27 (1935) (summary in English).—In view of the observations of Schou and Bjerregaard (*C. A.* 27, 5145) that  $\alpha$ -solns. of atropine (I), buffered to  $pH$  7.3, hydrolyze strongly when heated, the stability of  $\alpha$ -solns. of methylatropine bromide (II), which are neutral, were investigated under similar conditions. S. and B.'s method for the detn. of I + tropine and tropic acid (III) in solns. of I could not be employed for solns. of II because the strongly basic methylatropine (IV) and methyltropine were not liberated by  $\text{Na}_2\text{CO}_3$ , even  $\text{NaOH}$  liberated only about 20% of the theoretical amt. of IV. The method is modified as follows: To 10.00 cc. soln. of II add 10 drops dil.  $\text{HCl}$ , or, if the soln. contains buffer, sufficient  $\text{HCl}$  to give a red color with 1 drop methyl red indicator, followed by 10 drops dil.  $\text{HCl}$ . Shake 3 times with 15 cc. of a 3:1 mixt. of  $\text{CHCl}_3$  and iso- $\text{PrOH}$ , filter the combined exts., distil off the extn. liquid on a steam bath and dissolve the residue in a small amt. of hot water. Titrate when cold with 0.1 N  $\text{NaOH}$  (phenolphthalein). One cc. 0.1 N  $\text{NaOH}$  corresponds to 0.0341 g. decomp'd II. Add 0.5 g.  $\text{NaHCO}_3$  to the titrated soln., shake as above with a mixt. of  $\text{CHCl}_3$  and iso- $\text{PrOH}$ , filter into a separatory funnel and shake the combined exts. with 5.00 cc. 0.1 N  $\text{HCl}$ . Let stand, drain off the extn. liquid and titrate the excess of  $\text{HCl}$  with 0.1 N  $\text{NaOH}$  (methyl red) (I and tropine). Solns. of II undergo very little change on heating because III, which is formed by a slight hydrolysis, increases the acidity of the soln. and prevents further decomp'n. Expts. with very weak solns. showed, however, that the amt. of III which must form before the hydrolysis is checked is the same in all cases; thus in weak solns. a relatively larger percentage of II decomposes. If a small amt. of  $\text{HCl}$  is added (0.1–1.0 cc. 0.1 N  $\text{HCl}$  per 100 cc.) the soln. becomes stable and will stand autoclaving for 20 min. at 120° and remain unchanged for half a year or more. If a buffer is added so as to maintain the  $pH$  above the value at which the soln. reaches the point of stability ( $pH$  about 4), or in strongly acid soln., hydrolysis takes place. Formation of I or tropine could not be detected either in neutral or in acid solns. Solns. of *eumydrin* (IV-nitrate) behaved similarly, the hydrolysis being slightly higher. If an excess of  $\text{NaOH}$  is added to a soln. of II complete hydrolysis takes place in a few min. Based on this, 2 analytical

methods were developed: (1) For the detn. of II in mixts. with acids or other substances not interfering with the extn., add 2 cc.  $\text{NaOH}$  soln. to a soln. of 0.1–0.2 g. II, if necessary obtained by concg. a weaker soln. Let stand for 15 min., add 1 drop methyl red indicator and dil.  $\text{HCl}$  to change of color, followed by 10 drops of dil.  $\text{HCl}$ . Ext. 3 times with 20 cc. of a 3:1 mixt. of  $\text{CHCl}_3$  and iso- $\text{PrOH}$ , filter the combined exts. through a small, firmly packed cotton filter and wash with a few cc. of the solvent. (2) Evap. the filtrate on a steam bath and dissolve the residue in 5–10 cc. hot water. When cold add 3 drops phenolphthalein indicator and titrate with 0.1 N  $\text{NaOH}$ . One cc. 0.1 N  $\text{NaOH}$  corresponds to 0.03841 g. II. (2) For the examn. of pure II, dissolve 0.2 g. II in 5 cc. water in a flask and add 10 cc. 0.1 N  $\text{NaOH}$ . Stopper the flask and let stand for one half to one hr. Add 3 drops phenolphthalein indicator and titrate the excess of  $\text{NaOH}$  with 0.1 N  $\text{HCl}$ . D. Thuesen

**The determination of morphine.** V. The value of some methods for the determination of the absolute content of morphine in opium. H. Baggesgaard-Rasmussen and F. Reimers *Dansk Tids. Farm.* 9, 229–43 (1935) (in German), cf. *C. A.* 29, 3459<sup>4</sup>.—In various methods of detg. morphine (I) the material which finally is titrated or weighed, when subjected to a preliminary purification by extn. with benzene, contains one or more by-alkaloids of a phenolic nature. The methods of Mannich (*C. A.* 29, 3462<sup>4</sup>), Eder and Stucki (*C. A.* 27, 2760), Szeghő (cf. *C. A.* 29, 3775<sup>4</sup>) and Eder (*XII Congrès International de Pharm.*, Bruxelles 1935) give values that are too high. When I is shaken out, dissolved and crystd. after addn. of  $\text{NH}_4\text{Cl}$  the loss is larger than for pure solns. of I treated in the same manner. This loss is practically of the same magnitude as the decrease in the amt. of I on benzene purification, carried out as follows. Dissolve I as completely as possible in 5 cc. benzene on the steam bath and evap. to dryness so as to render it cryst. If necessary repeat this operation. Add 10 cc. cold benzene and let the flask stand overnight or at least 2–3 hrs. with frequent shaking. Decant the benzene through a small filter and wash I twice with 5 cc. benzene. Dissolve crys'tals of I on the filter in warm  $\text{MeOH}$ , transfer to the flask and evap. to complete dryness. A method of detg. the abs. content of I in a lime ext. of opium would consist of (1) shaking out with a 3:1 mixt. of  $\text{CHCl}_3$  and iso- $\text{PrOH}$  at  $pH$  about 9, (2) tetrapon purification of the alkaloid shaken out (cf. *C. A.* 27, 2760), (3) benzene purification of the alkaloids again shaken out, and (4) titration of I. A single shaking out, followed by benzene purification and pptn. cannot be employed because I in some cases is then so impure that the benzene purification fails. D. Thuesen

**Cascara adulteration.** H. W. Blair *Pharm. J.* 135, 682; *Chemist and Druggist* 123, 737 (1935).—In sampling a consignment about 2% of a deliberate admixt. of large smooth stones and bags contg. chips of *Pseudotsuga douglasii* was brought to light. S. Waldbott

**Preservation of chloroform to be used in anesthesia.** Adele Lussievici-Draganescu. *J. pharm. chim.* 21, 533–8 (1935).—Addn. of 1% alc. and keeping the  $\text{CHCl}_3$  in the dark in small colored vials will protect it from deterioration for months, even a yr. S. Waldbott

**Activity of different salts of cocaine.** J. Régner and R. David. *J. pharm. chim.* 22, 16–22 (1935).—A summary of previous work (cf. *C. A.* 28, 4175<sup>4</sup>; 29, 293<sup>4</sup>, 551<sup>4</sup>, 4833<sup>4</sup>). S. Waldbott

**Isolation and some properties of an alkaloid from *Crotalaria spectabilis*.** Roth, W. M. Neal, L. L. Rusoff and C. P. Ahmann. *J. Am. Chem. Soc.* 57, 2560–1 (1935).—Extn. of the seed, leaves and stems, resp., of *Crotalaria spectabilis* Roth with 5%  $\text{NH}_4\text{OH}$  for 48 hrs. and extn. of this filtrate with  $\text{HCl}$ , gives 0.4, 0.07 and 0.015% of crude extn., from which was isolated *monocrotaline*, m. 190–73°; it slowly decolorizes  $\text{KMnO}_4$  and shows characteristic alkaloidal reactions; it lowers the blood pressure in dogs and decreases the rate and amplitude of terrapin heart *in situ*; the 24-hr. lethal dose for chickens is 65 mg./kg. live wt. C. J. West

**Heart glucosides of *Digitalis purpurea* L. and *Digitalis***

*lanata* Erh. Artur Stoll. *Arch. Chem. Farm.* 2, 129-38 (1938 in German) (1935), cf. *C. A.* 30, 240<sup>4</sup>.—The application of a mild procedure for extg leaves of *Digitalis purpurea* and *D. lanata* led to the prepn of a series of new genuine glucosides, whose mutual relations could be definitely established. The total cryst. prepn obtained from *D. lanata*, called diglanide, has been sepd into 3 components, viz. diglanide A (I), B (II) and C (III). These could be converted into previously known glucosides in 2 ways, i. e. by chem. sapon and subsequent enzymic cleavage of 1 mol of glucose or a reverse sequence of the above treatments. Thus I gave digitoxin, II gitoxin, and III digoxin, all of which differ from the resp. diglanides by a lack of 1 Ac group and a mol of glucose. Processing of *D. purpurea* by the same scheme was seriously hampered by a comparatively larger amt of inactive accompanying substances and the amorphous nature of the genuine glucosides. Nevertheless genuine and pure but amorphous *purpurea* glucosides A (IV) and B (V) could be prep'd differing from I and II by a lack of 1 Ac group, and being identical with desacetyldiglanide A or B, resp. IV is transformed by the *purpurea* leaf substance enzymically into digitoxin, V into gitoxin. For prep'n pure genuine glucoside prep'n *D. lanata* is better suited than *D. purpurea*, since IV and V, being amorphous, are isolated with difficulty and with a poor yield. A det'n of the components of the total prep'n of genuine *purpurea* glucosides does not exist, while with *D. lanata* the total diglanide content as well as the ratio of the resp. components I and II and III is readily det'd., and the prep'n can be obtained in a good yield and by a technically easy method. J. Wiertelak.

**Comparative investigations on the adsorptive power of charcoals for medicinal purposes.** Aleksey Rausch. *Arch. Chem. Farm.* 2, 182-96 (1935) (German summary).—R. stresses the necessity of a more exact definition of the term *medicinal charcoal* (I) from the medical point of view. The raw material does not seem to play any role in prep'n. I. It is also desirable to decide definitely which chem. substances should be chosen to be adsorbed in the evaluation of I. Requirements for a high adsorption of iodine and HgCl<sub>2</sub> should be barred since in the products of food metabolism substances of such a strong chem. reactivity are absent. On the other hand, too little attention has been paid hitherto to gases, as H<sub>2</sub>S, and to decompn. products of albuminous substances. A new peptone method for exam'g. I is proposed. Shake 100 cc. of a 0.15% aq. soln. of Merck's peptone for 0.5 hr. with 0.2 g. of I, filter the suspension, and det. the non-adsorbed peptone colorimetrically (cf. M. Dominikiewicz, *Chem. Polski* 1909, 297). For this purpose add to 50 cc. of the filtrate 5 cc. of a 30% NaOH soln. and 5 cc. of a 1% CuSO<sub>4</sub> soln. and make up to 100 cc. After shaking, filter the mixt. through dry paper, and compare the amethyst-colored soln. with a standard. The method is most practical for work in pharmacies. In add'n. adsorption of R-salt is introduced, of which the non-adsorbed portion can be easily det'd. iodometrically. Thirteen different charcoal samples are tested in respect to the adsorption of iodine, HgCl<sub>2</sub>, CCl<sub>4</sub>, H<sub>2</sub>S, methylene blue, R-salt and peptone. J. Wiertelak.

**Conductometric analysis and its application in pharmacology.** Konstanty Hrynakowski and Feliks Modrzejewski. *Wiadomosci Farm.* 62, 427-9, 441-3, 457-60 (1935) (German summary).—The application of conductometric titrations to det'n. of weak acids and bases gives generally good results. Extending this applicability to pharmaceutical products, H. and M. det. with an av. error of 0.2-0.5% derivs. of barbituric acid (barbital and phenobarbital) as well as salicylic acid, coumarin, urotropine and papaverine. Substances insol. in water but sol. in EtOH can be titrated in alc. solns. Better results are obtained, however, if the weak acids, after soln. in excess alkali, are titrated back with HCl, or weak bases, correspondingly, after soln. in HCl, with NaOH. The dissociation const. (*K*) of all substances, mentioned above, is greater than 10<sup>-10</sup>. The det'n. of substances which have *K* equal to, or smaller than 10<sup>-10</sup>, is hindered by incipient hydrolysis, e. g., urea

(*K* = 1.5 × 10<sup>-10</sup>) in aq. soln. does not interact at all with HCl. The same is true of antipyrine. For the same reason errors occur with biacidic alkaloids, as cinchonine (*K*<sub>1</sub> = 1.4 × 10<sup>-11</sup>, *K*<sub>2</sub> = 1.2 × 10<sup>-10</sup>). The latter gives good results on titration with HCl to a 0.02 M soln., while at a lower concn. too low results are obtained because of hydrolysis. Brucine and strychnine, with *K*<sub>2</sub> of the order 10<sup>-11</sup>, have in a 0.01 M soln. the second group to a major part hydrolyzed. In less concd. solns. the hydrolysis is total and these alkaloids can then be det'd. as monoacidic bases. In alc. solns. hydrolysis proceeds farther than in aq. HCl solns. of equal strength already in 0.01 M alc. solns. Strychnine and brucine behave as monoacidic bases. J. Wiertelak.

**Essential oils of eucalyptus.** Antonio da Costa Cabral. *Anais inst. super. agron., Univ. tech. Lisboa* 6, No. 2, 78-81 (1934).—Samples of leaves of fruiting and non fruiting eucalyptus trees (*E. globulus*) growing in Portugal were taken fortnightly between Dec. and Oct. and the cinole content was det'd. The essential oil obtained has the same cinole content as the Australian and California eucalyptus oils but the yield from these Portuguese trees is much smaller than is found in any other country, being 0.41-0.501%. The oil contains no phellandrene. O. W. Willcox.

**Eighteenth-century English medicine (Comrie) 2.** Catalysis of hydrogenation. IV. Hydrogen no. a new const. for essential oils (Ginzberg, Evdokimova) 27. Derivs. of carbamylcholine chloride (Ercoli) 10. Detection of heavy water in mineral water (Hansen, et al.) 14. Hydrogenation of tertiary alkyl-phenols to form hydro-aromatic ales [for use as ingredients for cosmetics and soaps] (U. S. pat. 2,026,668) 10.

**Physiologically active preparations.** I. G. Farbenund. A.-G. (Carl Ludwig, Willy Ludwig and Fritz Lindner, inventors). Ger. 618,789, Sept. 14, 1935 (Cl. 12p. 17.10). Addn. to 614,861 (C. A. 29, 8238<sup>9</sup>). The method of 614,861 for obtaining active prep'n. from hypophysis lobes is modified by subjecting the lobes to autolysis or digestion after ext'n. with the org. solvent, but before ext'n. with water.

**Antithyroid concentrate.** Ferdinand Blum (to Sachsches Serumwerk A.-G.). U. S. 2,024,676, Dec. 17. Material such as blood, liver or spleen is treated (suitably with acetone or alc.) to ppt. protein and after filtering the pptd. proteinous material is repeatedly ext'd. with water to produce an aq. ext., the filtrate and aq. ext. derived from the ppt. are conc'd. and then are dialyzed and may be evap'd. to dryness. A product thus prep'd. may be used as a therapeutic agent.

**Alkaloids.** Jean B. A. Lafon. Gr. 787,611, Sept. 26, 1935. Alkaloids are ext'd. from tea, coffee, tobacco, etc., by means of SO<sub>2</sub>, which is afterward removed by simple expansion. The harmful alkaloids may first be removed from the SO<sub>2</sub> by an adsorbent such as silicotungstic acid.

**Separating cinchona alkaloids.** C. F. Boehringer & Soehne G. m. b. H. (Heinrich Thron and Wilhelm Dirscherl, inventors). Ger. 621,456, Nov. 7, 1935 (Cl. 8p. 7). Cinchona alkaloids having unsat'd. side chains are sepd. from those having sat'd. side chains by taking advantage of the fact that the alkaloids with unsat'd. side chains combined with Hg salts to form compds. sol. in aq. NH<sub>3</sub> but insol. in ether. These compds. are decomp'd. after the sepn., preferably with H<sub>3</sub>PO<sub>4</sub>. Sp. processes are described. The method may be used in analyzing mixts. of the alkaloids.

**Nicotine.** Constantin de Gendre and Paul Bary. Fr. 787,194, Sept. 18, 1935. Tobacco juice prep'd. from waste tobacco is treated with an alkali then with HCOOH, AcOH or other acid forming with nicotine a salt insol. in petroleum, and the salt is sepd. by decantation.

**Solutions of alkoxy phenols suitable for medicinal uses as for injection into body cavities.** Walter Kropp (to Winthrop Chemical Co.). U. S. 2,025,869, Dec. 31. A water-sol. 1-phenyl-2,3-dimethyl-5-pyrazolone-4-

aminomethane-sulfonate or -sulfinate is used in prep. aq. solns. of alkoxy phenols such as guaiacol or the like

**Vuzine derivative.** Vereinigte Chminfabriken Zmmmer & Co G. m. b. H. Zweigniederlassung Mannheim (Iritz Johannesohn and Heinrich Thron, inventors) Ger 621,198, Nov. 2, 1935 (Cl 12p 12) The vuzine salt of camphoric acid is prep. by any of the usual salt-forming processes. Sp processes are described. The salt has a stronger bactericidal action than the known vuzine salts

**Chloromethylene derivatives** Marcel Sommet and Israel Marsak Ir 787,655, Sept. 20, 1935 An aldehyde is caused to react in strong HCl medium with aromatic compds such as  $C_6H_5$ ,  $PhMe$ ,  $PhI$ , xylene, anisole, phenetole and butoxybenzene, preferably in the presence of a solvent or diluent. Examples are given of the prep. of *p*-chloromethylanisole, *b*, 100-3°, *p*-chloromethylphenetole, *b*, 105-7°, monochloromethyl deriv. of butoxybenzene, *b*, 135°, monochloromethyl deriv. of methoxybenzene, unstable,  $\alpha$ -chloroisobutyl deriv. of methoxybenzene and  $\alpha$ -chloroethyl deriv. of *m*-xylene, *b* 98-115° The products are intermediates for perfumes, etc

**Salts of methylaminomethylheptene** Knoll A-G chemische Fabriken (Wilfried Klavohn, inventor) Ger 618,973, Sept. 19, 1935 (Cl 12p 8-10) Salts of 6-methylamino-2-methyl-2-heptene, readily sol in water are prep. by treating salts or salt-like compds of the above with 4-dimethylamino-1-phenyl-2,3-dimethyl-5-pyrazolone, in optional presence of water. Instead of the salts of methylaminomethylheptene, the base and acid constituents may be treated with the above pyrazole deriv. The substances are used in medicine. 1 examples are given Cf C A 30, 810<sup>4</sup>

**Salts of organic arsenic acids** I G Farbenind A-G. (Karl Streitwolf and Hubert Oesterlin, inventors) Ger 618,567, Sept. 11, 1935 (Cl 12p 1-30) Acridine bases are treated with org. arsenic acids which are not exclusively substituted by primary  $NH_2$  groups and which contain complex-bound metals, or these acids are treated with salts of basic substituted acridine compds. Thus, the Na salt of 2-argemontmercaptopbenzoxazole-5-arsonic acid is dissolved in water and treated with a warm soln. of 2,3-dimethoxy-6-nitro-9-( $\gamma$ -diethylamino-8-hydroxypropylamino)acridine-di-HCl to give a salt. Other examples are given. The salts are used in therapy and are sol. with difficulty in water. Cf C A 30, 730<sup>4</sup>

**Alkali and alkaline earth salts of sugar phosphoric acid esters.** Seigo Funakura U S 2,021,030, Dec. 10 Sugars phosphoric acid esters are synthesized by the interaction of sugar dissolved in aq. pyridine contg about 30% of water with  $POCl_3$  dissolved in  $CHCl_3$  at -20°, removing excess of pyridine from the reaction mixt (suitably by vacuum distn.), neutralizing the residual syrupy soln. with the carbonate and hydroxide of a metal such as  $CaCO_3$  and  $Ca(OH)_2$ , removing the remaining pyridine by passing a current of warm air at 35-40° through the soln. and by vacuum distn., removing various impurities by dialysis, removing traces of chloride with Ag carbonate, and finally pptg the ester salts with  $EtOH$ . The products thus obtained are suitable for medicinal use

**Solutions of rare earth oxalates.** Chemische Fabrik von Heyden A-G. (Richard Moller, inventor) Ger 621,309, Nov. 5, 1935 (Cl 30k 2) Water-insol or sparingly sol. rare earth oxalates are brought into aq. soln. with the aid of salts of polybasic hydroxy acids, e. g., citric, malic or tartaric acid. The process is preferably effected in an alk. medium, and protective colloids may be added to the solns. The products are of therapeutic value. Sp processes are described.

**Vitamin concentrate from oils such as cod-liver oil.** Horatio P. Loomis (to Salmo Chemical Co.) U S 2,020,395, Dec. 31. Part of the saponifiable portion of the oil is saponified with an alkali hydroxide and the acid soaps and neutral soaps formed are dissolved in aq. alc.; the unsaponified portion of the oil contg. vitamins is then sep'd by gravity or by centrifuging. Various details and modifications of procedure are described.

**Hormones.** I. G. Farbenindustrie A-G. Brit. 435,545, Sept. 19, 1935. Preps. of the hormone of the

suprarenal cortex are obtained free from the toxic action of 1-(3,4-dihydroxyphenyl)-2-methylaminoethanol by treating the cortex or the whole suprarenal capsule, or any preps. from these, with  $CH_3O$  or a compd. that splits off  $CH_3O$ . Excess  $CH_3O$  may be eliminated by adding  $NH_3$ .

**Hormone preparations** I G Farbenindustrie A-G. Brit 435,195, Sept. 17, 1935 Stable solid mixts. of hormones with diluents are prep'd by dissolving the hormone together with an albumin or albumin decompn. product of high mol. wt. in a solvent for both constituents and then adding a precipitant for the 2 substances. Among examples, (1) posterior hypophysis hormone and Na protalbin are dissolved in glacial  $AcOH$  and pptd. with a mixt. of  $Me_2CO$  and  $Li_2O$ , and (2) the sexual hormone of the anterior hypophysis obtained from urine is dissolved in  $H_2O$ , mixed with normal serum and pptd. with alc and  $Li_2O$  Cf C A 29, 5900<sup>1</sup>

**Follicle hormone preparations** F Hoffmann-La Roche & Co A G Brit 431,106, Aug. 30, 1935 Concd. aq. preps. for injection or oral administration are obtained by dissolving the hormone in aq. resorcinol solns. Other phenols, e. g., trisecol, guaiacol, may be added to the resorcinol soln. to increase its capacity to dissolve the hormone

**Suspensions of histological animal cells** I G. Farbenind A-G Ir 780,057, Aug. 26, 1935 Stable suspensions which do not agglutinate are obtained dispersing the fresh cellular material in the presence of substances which lower the surface tension or prevent coagulation of blood while not appreciably modifying the cells, preferably in conjunction with agents having an antiseptic action. The above substances may be administered to a living animal from which the cellular material for the prep. is to be obtained. Thus, to a cellular suspension is added an aq. soln. of hirudin or *m*-aminobenzoic- or *m*-amino-*p*-methylbenzoic-1-naphthylamine-4,6,8-trisulfonic acid, or a mixt. of *sec*-octyl alc.,  $LiOH$  and water.

**Radioactive compositions of therapeutic value** Gottfried Erhard and Gerhurd Schaefer Ger 650,477, Nov. 16, 1935 (Cl 30k 2) Ra emanation is absorbed in solid alimentary fats

**Local anesthetic.** Sverre Quisling. U. S. 2,025,399, Dec. 24. An aq. or dil. alc. soln. of guanicol is prep'd. with use of Na salicylate or benzoate or both as a solubilizing agent.

**Solution suitable for sterilizing aluminum articles** Lamont J. Benson (to Aluminum Co. of America). U S 2,021,755, Dec. 17. A soln. comprising a water-sol alkali metal disulfate and available Cl is prep'd., e. g., from Na disulfate and NaOCl

**Deodorant suitable for use as a liquid spray** Max Cline (to International Paper Co.) U S 2,024,145, Dec. 17. Zn sulfocarbolate, starch and glycerol are used in an aq. vehicle

**Cosmetic grease paint** Frank Factor (to Max Factor & Co.) U S 2,025,913, Dec. 31. A freely flowing liquid compn. contains a nonvolatile oil such as olive and mineral oils in sufficient proportion that after application of the compn. to the skin a flexible film is formed which remains moist and resistant to cracking, together with a pulverulent "texture ingredient" such as Zn and Ti oxides and a suspended pigment and an ingredient for reducing surface tension such as triethanolamine and stearic acid which renders the paint readily removable by the use of soap and water.

**Skin creams** The Chrs. H. Phillips Chemical Co. Fr 787,532, Sept. 24, 1935. The cream contains  $Mg(OH)_2$ , an oil, an alc. of high mol. wt. contg. at least 16 C atoms, a wax and water. An example contains  $Mg(OH)_2$ , 2-4, cholesterol 2.7, petroleum 30.3, ceresin wax 1, water 60.4, ethylene glycol 3 and perfume 0.2%.

**Dentifrice.** Herl L. Crowther. U S 2,023,148, Dec. 17. A dry powd. material such as a mixed dentifrice is impregnated with a wax such as ceresin wax for leaving a thin protective coating on the teeth when brushed with the compn.

**Dentifrice.** Benjamin R. Faunce. U. S. 2,025,655,

Dec 24 A sterilized glyceride of lute freed of pathogenic bacterial matter is mixed with about an equal amt. of glycerol, an excipient such as surup and a cleansing and polishing agent such as pptd.  $\text{CaCO}_3$ , etc

## 18—ACIDS, ALKALIES, SALTS AND OTHER HEAVY CHEMICALS

### E M SYMMES

The production of boric acid in the U. S. S. R. L. E. Berlin *J. Chem. Ind. (Moscow)* 12, 821-8(1935).—The app and methods used for decompn of datolite by  $\text{H}_2\text{SO}_4$  and extn of  $\text{H}_2\text{BO}_3$  from the residue are discussed

H. M. Leicester  
Manufacture of hydrofluoric acid. Pedro Roca. *Quim. Ind. Ind.* 12, 236-8(1935).—Com methods employing  $\text{CaF}_2$  and  $\text{H}_2\text{SO}_4$  are described

L. E. Gilson  
A rapid method for the determination of available phosphoric acid in freshly prepared superphosphate N. V. Il'in and V. F. Chapuign *J. Chem. Ind. (Moscow)* 12, 819-20(1935).—The standard methods are used, except that for extn of the citrate-sol  $\text{P}_2\text{O}_5$  the sample is heated 45 min at  $70^\circ$  with the Peterman reagent. The method is accurate to 0.2%. Unless fresh superphosphate is used, the extn takes much longer.

H. M. L.  
Rapid method of determining nitrogen oxides in tower gases of sulfuric acid production. M. N. Merlis and O. D. Petrova *Zavodskaya Lab.* 4, 938-9(1935).—For the detn of  $\text{NO}$  and  $\text{NO}_2$  in the tower gases of  $\text{H}_2\text{SO}_4$  manuf.,  $\text{NO}$  is oxidized to  $\text{NO}_2$  with  $\text{H}_2\text{O}$  satd. with  $\text{O}_2$  in the presence of  $\alpha\text{-C}_6\text{H}_5\text{NH}_2$  (I) and sulfamic acid (II). The color intensity of the soln of the azo dye formed is compared with that of an aq. mixt. of I and II of the same concns titrated with a standard soln of  $\text{NaNO}_2$ . Since only half the combined  $\text{NO}_2$  reacts in the diazotization ( $2\text{NO}_2 + \text{H}_2\text{O} = \text{HNO}_2 + \text{HNO}_3$ ), the results of detn. must be multiplied by 2. Fill a 10-bulb Lunge tube with 75 cc  $\text{H}_2\text{O}$  satd. with  $\text{O}_2$  and introduce 1 cc. of the I soln (0.1 g. I, 6 cc of 80%  $\text{AcOH}$  and 94 cc  $\text{H}_2\text{O}$ ), 1 cc of 1% II and 10 drops of 80%  $\text{AcOH}$ . Pass into the tube 500 cc. of the gas sample from an aspirator. Discharge the soln. with the dye into a flask, wash the tube, heat the united soln to  $70-80^\circ$ , and dil the soln to 300 cc. Mix 280 cc. of hot  $\text{H}_2\text{O}$ , 1 cc of the I soln, 1 cc of II soln and 10 drops of  $\text{AcOH}$ , titrate the soln. at  $70-80^\circ$  with  $\text{NaNO}_2$  to the same color intensity as the first soln, and calc.

Chas. Blanc  
Determination of arsenic in flue gases in sulfuric acid production. G. B. Zil'berman and E. N. Polikarpova. *Zavodskaya Lab.* 4, 760-2(1935).—More reliable results were obtained by the colorimetric method of Sanger and Black (C. A. 2, 975) by using  $\text{H}_2$  generated from  $\text{H}_2\text{SO}_4$  and Al activated by etching with 3%  $\text{HgCl}_2$  for 2-3 min.

Chas. Blanc  
Methods of preparing fluorides. A. I. Selezneva. *J. Chem. Ind. (Moscow)* 12, 1041-7(1935).—Methods for converting fluorapatite into cryolite,  $\text{AlF}_3$ , and  $\text{NaF}$  are reviewed

H. M. Leicester  
The preparation of high-quality chloride of lime. M. E. Pozin *J. Chem. Ind. (Moscow)* 12, 810(1935); cf. C. A. 30, 822<sup>2</sup>.—When  $\text{Ca(OH)}_2$  is suspended in  $\text{CCl}_4$  and chlorinated for 5 hrs. at  $0^\circ$ , the product contains 42-49% active  $\text{Cl}_2$ . If an aq. suspension of yellow  $\text{HgO}$  is chlorinated,  $\text{HgCl}_2$  and  $\text{HOCl}$  are formed. The soln is filtered and neutralized with lime. It gives a product contg 45% active  $\text{Cl}_2$ . Chlorination of a suspension of  $\text{CaCO}_3$  in  $\text{H}_2\text{O}$  at  $0$  to  $-5^\circ$  gives  $\text{CaCl}_2$  and  $\text{HOCl}$ . The latter is extd with  $\text{CCl}_4$  and neutralized with finely ground lime. The resulting  $\text{Ca(OCl)}_2$  contains 51.5% active  $\text{Cl}_2$ .

H. M. Leicester  
The preparation of a high percentage calcium cyanamide by the action of ammonia and carbon monoxide on calcium oxide and calcium carbonate. V. F. Postnikov, T. I. Kunin and N. A. Ereemeeva. *J. Chem. Ind. (Moscow)* 12, 795-802(1935).—The optimum conditions for the reaction are to pass a 6-fold excess of  $\text{NH}_3$  and 4 times thus amt. of  $\text{CO}$  over  $\text{CaO}$  at  $750-800^\circ$ . Decompn. of the  $\text{NH}_3$  is favored by the presence of Fe, which should therefore be avoided, and hindered if the gas stream con-

tains 50%  $\text{N}_2$ . Addn of 3%  $\text{Al}_2\text{O}_3$  and 10% C catalyzes the reaction.  $\text{CaCN}_2$  contg 26.81%  $\text{N}_2$  is obtained thus in 2 hrs. The reaction goes more easily if  $\text{CaCO}_3$  or natural limestone is used. In this case, the optimum temp is  $800-50^\circ$ , the ratio of  $\text{NH}_3$  to  $\text{CO}$  is 1:3, the catalyst contains 1.5%  $\text{Al}_2\text{O}_3$  and 17% C, and other conditions are unchanged.  $\text{CaCN}_2$  contg 28%  $\text{N}_2$  is thus obtained. A yield of 5%  $\text{HCN}$  is obtained as a by-product. Attempts to convert the  $\text{CaO}$  in technical  $\text{CaCN}_2$  into  $\text{CaCN}_2$  by this process were not successful.

H. M. Leicester  
Sulfate formation from the action of chlorine on mixtures of cupric sulfate, cupric oxide, cuprous sulfide and ferric oxide. D. M. Chizhikov and G. S. Babikhina. *J. Chem. Ind. (Moscow)* 12, 1038-41(1935).—When  $\text{Cl}_2$  reacts with a mixt of  $\text{CuSO}_4$  and  $\text{CuO}$ , a small amt. of  $\text{CuSO}_4$  is formed. Addn. of  $\text{Fe}_2\text{O}_3$  to the mixt. greatly increases the amt. of  $\text{CuSO}_4$ , especially at higher temps. Addn. of  $\text{CuSO}_4$  contg.  $\text{H}_2\text{O}$  of crystn decreases the formation of more  $\text{CuSO}_4$  at high temp. Anhyd.  $\text{CuSO}_4$  has little effect.  $\text{CuSO}_4$  and  $\text{Cu}_2\text{S}$  react to give S, which also oxidizes  $\text{CuO}$ , but not as easily as  $\text{Cu}_2\text{S}$  does. When a mixt. of  $\text{CuSO}_4$  ( $\text{H}_2\text{O}$ ),  $\text{CuO}$ ,  $\text{Cu}_2\text{S}$  and  $\text{Fe}_2\text{O}_3$ , approximating the compn. of Cu pyrites slag, is chlorinated, the amt of  $\text{CuSO}_4$  formed rises to 85% of the possible amt.

H. M. Leicester  
The production of magnesium and barium fluorides. N. S. Nikolaev and S. M. Kamorgorodskii. *J. Chem. Ind. (Moscow)* 12, 1047-50(1935).—Solns of  $\text{MgCl}_2$  and  $\text{MgSO}_4$  give colloidal ppts with  $\text{NaF}$ . The resulting  $\text{MgF}_2$  is very impure. If these salts are treated with  $\text{HF}$ , gels are formed. However,  $\text{MgO}$  and  $\text{MgCO}_3$  react with  $\text{HF}$  to give good yields of rapidly filterable  $\text{MgF}_2$ .  $\text{BaCl}_2$ ,  $\text{Ba(NO}_3)_2$ , and  $\text{BaS}$  all give good yields of  $\text{BaF}_2$  when they are treated with  $\text{NaF}$ . When  $\text{BaS}$  is used, one of the reaction products is  $\text{NaOH}$ , whose alkyl, permits the use of  $\text{NaF}$  contg much  $\text{SiO}_2$ . Details of large-scale applications of these methods are given.

H. M. L.  
Microchemical investigation of the potassium chloride crystals obtained on treatment of sylvinite. Ya. E. Vil'nyanskii and Z. S. Bannukh. *Kaif* (U. S. S. R.) 1935, No. 8, 19-21.—Photomicrographs of different intermediate products (vacuum salt, lower salt and dry salt) are reproduced. The manuf. of vacuum salt is recommended as a means to decrease the caking of  $\text{KCl}$  in storage.

A. Pestoff  
The use of oxygen in soda manufacture. A. D. Kron. *J. Chem. Ind. (Moscow)* 12, 784-8(1935).—Economic considerations are discussed.

H. M. Leicester  
Obtaining bromine from the sylvanite alkaline mother liquors. V. I. Nikolaev and E. M. Petrova. *Kaif* (U. S. S. R.) 1935, No. 8, 18-19.—A table shows the distribution of Br between the liquid and solid phases in the tertiary system  $\text{KCl-KBr-H}_2\text{O}$  for Br contents of 0.5-0.3% over a temp. range of  $0^\circ$  to  $100^\circ$ . At  $100^\circ$  the liquid contains 2.4 times more Br than the solid. Max. content of Br in Solikamsk sylvanite liquor was 0.18%.

A. Pestoff  
Some solvents for the extraction of iodine and bromine from dilute solutions. A. G. Balchikov. *J. Chem. Ind. (Moscow)* 12, 1062-6(1935); cf. C. A. 29, 2313<sup>2</sup>.—Benzene and kerosene are easily brominated, and so cannot be used for  $\text{Br}_2$  extn, even if they have been treated with oxidizing agents.  $\text{CCl}_4$  exts  $\text{Br}_2$  and  $\text{I}_2$  effectively from  $\text{H}_2\text{O}$ , but less effectively from  $\text{NaCl}$  solns. Addn of up to 30%  $\text{CaH}_2$  to the  $\text{CCl}_4$  improves the extn. of the halogens from both  $\text{H}_2\text{O}$  and brine, but the sp. gr. of the solvent should be kept well above that of the brine to promote good sepn. of the layers.

H. M. Leicester  
Obtaining hydrogen and hydrogen nitrogen mixtures by the explosive oxidation of methane. N. I. Kobozov,

Ya. S. Kazarnovskii and L. I. Kashtanov. *J. Chem. Ind. (Moscow)* 12, 1030-6 (1935).—The explosion of equimol. amts of  $\text{CH}_4$  and  $\text{O}_2$  gives a yield of 37.8%  $\text{CO}$  and 49.4%  $\text{H}_2$ , independent of pressure from 0.72 to 3.7 atm. The walls of the reaction vessel had no effect, but reduction of the diam. of the vessel hinders the reaction without changing the final products. Addn. of  $\text{N}_2$  to the mixt. lowers the yield of  $\text{CO}$  and  $\text{H}_2$ , somewhat, but this effect can be counteracted by the presence of  $\text{CO}_2$ . Addn. of  $\text{H}_2\text{O}$  to the mixt. causes incomplete reaction. Calcs. show that the temp. conditions of the reaction probably det. purely thermodynamically the direction of the explosion. H. M. Leicester

The use of waste nitrous gases to produce nitrogen and nitrogen-hydrogen mixtures. I. Ivanovskii, M. Korsh and I. Krushal. *J. Chem. Ind. (Moscow)* 12, 803-9 (1935).—The  $\text{NO}$  and  $\text{O}_2$  which are present in small amts in waste  $\text{N}_2$  can be removed by passing the gas at 300-500° with  $\text{H}_2$  over a catalyst composed of 2%  $\text{Cu}$  and 3%  $\text{Ni}$  deposited on chamotte. This catalyst can also be used to remove small amts of  $\text{O}_2$  from electrolytic  $\text{H}_2$ . H. M. Leicester

Controlling and operating devices in the sulfur plant at Rönnskär. Per Agren. *Ira* 1935, 83-8.—The instruments and control board for continuously recording and controlling the operations in the Rönnskär plant for the manuf. of  $\text{S}$  from  $\text{SO}_2$  and producer gas are described. E. M. Symmes

Mining of carnallites in the First Potassium mine A. N. Andreichev. *Kali* (U. S. S. R.) 1935, No. 4, 27-36, No. 5, 14-25.—A discussion of the location of the mine and proposed methods of the mining of carnallite. The carnallite contains  $\text{KCl}$  19.90,  $\text{MgCl}_2$  25.18,  $\text{NaCl}$  27.44,  $\text{H}_2\text{O}$  25.63,  $\text{CaSO}_4$  0.956, insol. matter 0.48%. A. Pestoff

The Carnallite-Magnesium Combine. Ya. M. Khelfetz. *Kali* (U. S. S. R.) 1935, No. 7, 8-15.—A description of the manuf.  $\text{Mg}$  is prepd from carnallite, with  $\text{KClO}_4$  as a by-product. A. Pestoff

The preparation of cryolite from a solution of sodium fluoride and aluminum fluoride. V. S. Yatlov and L. M. Korzon. *J. Chem. Ind. (Moscow)* 12, 1030-4 (1935).— $\text{Na}_2\text{SiF}_6$  is treated with a slight excess of 15-20%  $\text{Na}_2\text{CO}_3$  soln. at 90-100°. The  $\text{SiO}_2$  ppts. and settles rapidly. It is well washed. The filtrate contains 96-98% of the  $\text{NaF}$   $\text{AlF}_3$  prepd. from  $\text{Al}(\text{OH})_3$  and  $\text{HF}$ , must be crystd rapidly at low temp., since large crystals react slowly. The  $\text{AlF}_3$  suspension is treated at not too high a temp. with  $\text{NaF}$  soln., and the pptd. cryolite is washed with  $\text{H}_2\text{O}$  at 95° to remove adsorbed sulfate ions. H. M. L.

The cooling of syrilite solutions in vacuo. Ya. E. Vil'yanski and V. Zelyanski. *Kali* (U. S. S. R.) 1935, No. 7, 16-22.—A description of different app. and their operations. A. Pestoff

Salt plant design and construction. Clinton S. Robinson. *Can. Chem. Met.* 19, No. 12, 324-5 (1935).—A brief description of the plant of the Goderich Salt Co. at Goderich, Ont., for the production of very pure, kindred salt by a continuous process. A filter seps the excess brine from the salt slurry, as the slurry is delivered from the evap. pans. The drum employed acts as a continuous filter and drier. The slurry of brine and salt of a predet. d. is received through an ingenious top-feed mechanism. The filter drum is fully encased in a housing having ducts connected to a series of heating units. A const. pressure is maintained on the filter housing by means of a blower. The machine is constructed of cast "Ni-resist" with housing and screen of monel metal. Noncorrosive metals are used throughout. W. H. B.

The production of lampblack from anthracene and naphthalene. P. Zmit. *J. Rubber Ind. (U. S. S. R.)* 12, 851-3 (1935).—A discussion. A. Pestoff

Surface activity of silica gel treated with calcium hydrazide. Aldo Maffei and Arnaldo Battaglia. *Ann. chim. applicata* 25, 309-18 (1935).— $\text{SiO}_2$  gel which has been heated and then cooled, whether slowly or rapidly, adsorbs  $\text{Ca}(\text{OH})_2$  from its soln. less readily than does fresh  $\text{SiO}_2$  gel. Gel was satd. with  $\text{Ca}(\text{OH})_2$  by passing a satd.

$\text{Ca}(\text{OH})_2$  soln. through the gel.  $\text{Ca}(\text{OH})_2$  adsorbed was detd. by treating the product with  $\text{HCl}$  to remove the  $\text{SiO}_2$ . Extn. of the mixt. with  $\text{HCl}$  does not remove all the  $\text{Ca}(\text{OH})_2$ . The activity and adsorptive powers of  $\text{SiO}_2$  gel are increased by treatment with  $\text{Ca}(\text{OH})_2$  soln. A. W. Conteri

The use of siderite as a source for catalysts for the synthesis of ammonia. Ya. A. Fed'man, K. N. Ivanov and A. I. Naumova. *J. Chem. Ind. (Moscow)* 12, 1036-8 (1935).—When Bakal siderite is fused without removing any impurities, most of the  $\text{Fe}_2\text{O}_3$  becomes  $\text{Fe}_3\text{O}_4$ , and a good catalyst for  $\text{NH}_3$  synthesis is obtained. Addn. of  $\text{Al}_2\text{O}_3$  or  $\text{K}_2\text{O}$  does not improve it. H. M. Leicester

Conversion of  $\text{H}_2\text{S}$  from coal gas into concd.  $\text{H}_2\text{SO}_4$  (Hoelzer) 21

Hydrochloric acid. Hermann Fischer. *Ger.* 621,056, Nov. 1, 1935 (Cl. 12: 5). For manuf., storing or cooling concd.  $\text{HCl}$ , use is made of app. made of or lined with  $\text{Sb}$  or  $\text{Bi}$  or their alloys, and the  $\text{HCl}$ , before it comes into contact with such app., is treated with a reducing agent, particularly  $\text{H}_2\text{S}$ . Corrosion is thus avoided.

Apparatus for phosphoric acid manufacture. Harry A. Curtis (to Tennessee Valley Authority). U. S. 2,026,519, Jan. 7. Various structural and operative details are described of an app. for combined oxidation and hydration of elemental P.

Recovering sulfuric acid from acid tar, etc. Ludwig Harbort and Fritz Keil. *Ger.* 621,123, Nov. 1, 1935 (Cl. 12: 25). Acid tar or like waste acid, e. g., spent  $\text{H}_2\text{SO}_4$  from the refining of benzene, is halogenated without diln., if necessary at a raised pressure and slightly raised temp. The resinous impurities sep. out, and the clear acid layer is withdrawn and blown with air or other gas to remove halogen and hydrogen halide.

Recovery of sulfuric acid and resins from acid tar produced in purifying benzene hydrocarbons with sulfuric acid. Geo. W. J. Bradley. U. S. 2,026,450, Dec. 31. The acid tar is passed in a thin stream into a body of boiling water equal to 0.5-1.0 the volume of the tar while the water is maintained in a state of ebullition by blowing steam through it, thus producing sepn. into a dil. acid and a resinous layer, and solidification of resin is effected by steaming. App. is described.

Alkali perborates. N. V. Industriele Maatschappij Voorheen Noury & van der Lande. *Brit.* 434,991, Sept. 12, 1935. This corresponds to *Fr.* 776,485 (C. 29, 3472).

Noncaking alkali metal metaphosphate. Geo. W. Smith (to Hall Laboratories, Inc.). U. S. 2,024,543 Dec. 17. A water softener or detergent which sequesters  $\text{Ca}$  and  $\text{Mg}$  in a but slightly ionized condition comprises an alkali metal metaphosphate mixed with about 3-20% of an intumescent alkali metal borate, such as borax.

Alkali sulphydrates. I. G. Farbenund A.-G. *Fr.* 786,441, Sept. 3, 1935. Stable, strongly concd., and dry preps. of alkali sulphydrates are obtained by evap. solns. thereof contg. a small amt. of alkali sulfide. Thus, a soln. contg.  $\text{NaSH}$  55,  $\text{Na}_2\text{S}$  3 and  $\text{Na}_2\text{S}_2\text{O}_3$  2.5% can be evapd. to dryness under vacuum without decompn.

Solid alkali silicate mixture suitable for dissolving in water. Franz Albertshäuser (to Philadelphia Quartz Co.). U. S. 2,026,451, Dec. 31. A solid pourable mixt. comprises a cryst. sol alkali silicate such as that of  $\text{Na}$  and an amorphous alkali silicate in which the ratio of silica to alkali is greater than in the cryst. silicate and which is interspersed with and protected from the atm. by the cryst. silicate.

Thiotetraphosphates. Augustus H. Fiske and Charles S. Bryan (to Rumford Chemical Works). U. S. 2,025,503, Dec. 24. A product having water softening and detergent properties is obtained, e. g., by heating  $\text{NaPO}_3$  4 with  $\text{Na}_2\text{S}$  1 mol. proportion (suitably at about 150°). *Cl.* C. A. 30, 578.

Apparatus for ammonium chloride manufacture. Elam C. Curtis (to Mathieson Alkali Works). U. S. 2,024,680,

Dec 17 App is described with a saturator comprising a closed chamber, a vessel within and opening at its upper end into said chamber, a connection for supplying a liquid reaction medium such as  $\text{NH}_4\text{Cl}$  soln. to the vessel and a connection for discharging the liquid reaction medium from the lower end of the chamber, sep means for introducing 2 gaseous reactants such as  $\text{NH}_3$  and  $\text{HCl}$  into the vessel and for vaporizing liquid  $\text{NH}_3$  in heat exchange with the contents of the vessel, and a vent from the upper end of the chamber

Ammonium nitrate Bamag-Megum A-G Fr 786,080, Aug 26, 1935  $\text{NH}_4\text{NO}_3$  poor in water and solidifying wholly on cooling is obtained from dil  $\text{HNO}_3$  and gaseous  $\text{NH}_3$ , by using the heat of neutralization and soln to conc the soln of  $\text{NH}_4\text{NO}_3$  formed. A soln of  $\text{HNO}_3$  of medium concn and  $\text{NH}_3$  are introduced into the lower part of a reservoir filled with a molten mass of  $\text{NH}_4\text{NO}_3$  already formed and b at atm pressure or lower. The untransformed vapors are submitted to a washing by hot molten masses already formed, if desired under reduced pressure App is described

Ammonium sulfate Frederic M Pyzel and Jan D Ruys (to Shell Development Co) U S 2,026,280, Dec 31 Acid sludge, such as that from refining hydrocarbons with  $\text{H}_2\text{SO}_4$ , is mixed with  $(\text{NH}_4)_2\text{SO}_4$  soln and the mixt is neutralized with  $\text{NH}_4\text{OH}$  soln in a reaction system. Liquid products of the reaction are passed into a separator maintained in a quiescent condition, so that oily products of reaction sep from the aq products of reaction, and the aq products of reaction are passed through a tube bundle, whereby further amounts of oily products sep from the aq products. An arrangement of app is described

Ammonium sulfate I G Farbenindustrie A-G. Brit 435,007, Sept 12, 1935 See Fr 781,750 (C. A. 29, 6374)

Ammonium sulfate I G Farbenind A-G Fr. 786,321, Aug 31, 1935 Large crystals are obtained by crystallizing from a neutral or alk. soln contg a small amt. of salts of alkali or alk. earth metals, Mg, Cr, Mn or Zn, or org sulfonic acids. Examples are given of the use of sulfates of Mg, Mn, Zn and Cr and sulfamic acid

Antimony trioxide Thomas Midgley, Jr., Albert L Henne and Robert R McNary (to General Motors Corp) U S 2,024,008, Dec 10 SbCl<sub>3</sub> is treated with HF (suitably in a steam-heated Pb receptacle)

Calcium sulfate Montan- und Industrialwerke vorm Joh D Starek Ger 620,327, Oct 19, 1935 (Cl. 12: 21) See Austrian 142,563 (C. A. 29, 8255)

Horizontal rotary kiln and associated apparatus suitable for calcining calcium and magnesium carbonates, etc Ruland R Shafter and Richard Bernhard (to Traylor Engineering & Mfg Co) U S 2,026,441, Dec 31 Structural, mech and operative details

Basic lead sulfate Fred E Gregory, John I McClaren and Paul R Hamilton (to Eagle Picher Lead Co) U S 2,026,033, Dec 31 Fumes from an ore hearth are introduced into a setting system, burned Pb sulfide particles are removed from the setting system, ground and introduced into a flash furnace, and the fumes escaping from the flash furnace are passed into the setting system and mixed with the fumes from the ore hearth. App is described

Magnesium sulfate Metallgesellschaft A-G Fr. 787,558, Sept 25, 1935 Solid monohydrated  $\text{MgSO}_4$  and similar salts are obtained in a form having a reduced content of water of crystn, particularly sulfates the soly of which in water increases first as the temp. increases, but afterward decreases, and which have less water of crystn at raised temp. The concd solns are heated by successive stages to a temp which is situated on the descending branch of the soly. curve, but at which no extn of solid salt takes place, and these solns are afterward cooled by auto-evapn, preferably by successive stages to temps which are situated on the rising branch of the soly. curve, but which are still within the limit of the formation of salt with the desired content of water of crystn. The solns circulate in closed circuit through

heaters and evaporators, the salt extd being sepd. between the evaporators and the heaters

Manganese phosphate I G Farbenind A-G (Alois Maier, inventor). Ger 618,787, Sept. 14, 1935 (Cl. 12: 31) See Fr 771,978 (C. A. 29, 7027)

Potassium nitrate Oskar F. Kaselitz U S 2,024,370, Dec 17 A soln. satd at an elevated temp with respect to  $\text{KNO}_3$ , and substantially also with respect to  $\text{K}_2\text{SO}_4$  and K-Mg sulfate is cooled to ppt.  $\text{KNO}_3$ , which is sepd,  $\text{K}_2\text{SO}_4$  and  $\text{Mg}(\text{NO}_3)_2$  are incorporated with the mother liquor, this mixt. is heated to evap part of the water from it and is cooled to a limited extent, so that K-Mg sulfate seps and the resulting liquor is reused in cycle with regulation of the percentage of water in the liquor to give it a quantity and concn such as those of the starting soln

Potassium sulfate Imperial Chemical Industries Ltd. Fr. 787,713, Sept. 27, 1935  $\text{K}_2\text{SO}_4$  and (or) a double salt  $\text{K}_2\text{SO}_4 \cdot \text{CaSO}_4 \cdot \text{H}_2\text{O}$  are made by causing KCl to react with  $\text{CaSO}_4$  in aq medium contg.  $\text{NH}_3$  and using an excess of KCl.  $\text{K}_2\text{SO}_4$  is obtained when the concn of KCl and (or)  $\text{NH}_3$  is relatively high and the double salt when the concns are relatively low. A limited amt. of NaCl as impurity in the KCl is not harmful. The  $\text{CaSO}_4$  may be added in steps, with or without sepn of the solid reaction product after each step

Sodium carbonate decahydrate Geo L Cunningham (to Matheson Alkali Works) U S 2,024,679, Dec 17.  $\text{NaHCO}_3$  or  $\text{Na}_2\text{CO}_3$  is subjected to the action of an ammoniacal soln of NaCl at a temp. below about  $15^\circ$  to ppt.  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  while the ratio of  $\text{CO}_2$  to free base in the soln is maintained at not exceeding about 0.5. An arrangement of app is described Cl. C. A. 29, 304

Sodium sulfate from calcium sulfate Markus Larsson U S 2,025,756, Dec 31.  $\text{CaSO}_4$  is treated with a soln. of  $\text{Na}_2\text{CO}_3$  in the presence of  $\text{Na}_2\text{SO}_4$  of about 30% concn and in which an excess of 1-4%  $\text{Na}_2\text{CO}_3$  is maintained during the reaction, after sepn of the  $\text{Na}_2\text{SO}_4$  and its crystn, the mother liquor contg.  $\text{Na}_2\text{CO}_3$  is returned to the initial reaction. An arrangement of app. and various operative details are described.

Zinc oxide American Zinc, Lead & Smelting Co. Brit 435,003, Sept 12, 1935 ZnO of acicular form is obtained by vaporizing Zn in a reducing atm. at approx.  $1200^\circ$  and passing the vapors into an oxidizing zone where the air supply is regulated to promote the growth of the oxide particles, e. g., by slowly mixing the vapors with air to burn them with a lazy flame while maintaining a temp. of substantially  $1100^\circ$ . App is described, in which the Zn is obtained by reduction of Zn ore

Nitric oxide and hydrogen chloride from nitrosyl chloride Oskar F Kaselitz U. S. 2,025,391, Dec. 24 A mixt. of  $\text{NOCl}$  and H is heated to  $150-500^\circ$  (suitably  $150-500^\circ$  in the presence of activated C or exposed to light rays).

Sulfur dioxide Metallgesellschaft A-G Brit. 435,116, Sept 16, 1935 See Ger. 606,447 (C. A. 29, 1594)

Fluorine compounds I G Farbenind. A-G Fr 786,123, Aug 27, 1935 F is introduced into org compds using a substantially pure F and carrying out the reaction in a chamber subdivided into small compartments by means of a substantially inert solid filling material, e. g.,  $\text{CaF}_2$ . Examples are given and app is described

Recovery of lithium values from amblygonite, etc. John H Coleman and Ned E Jaffa (to Warner Chemical Co.) U. S. 2,024,026, Dec 10. A mineral such as amblygonite or a similar Al phosphate mineral contg. lithia and siliceous gang is digested in a  $\text{NaOH}$  soln. so as to dissolve Al phosphate and to concentrate the lithia in insol. form in a solid residue, and the residue is subjected to an acid treatment, as with 35%  $\text{H}_2\text{SO}_4$ , to ext the lithia and leave a final residue of silica and silicates.

Stable water-soluble colloidal molybdenum Perry Vesie U. S. 2,025,403, Dec. 24 An aq suspension of Mo oxides contg. a protective colloid such as gum arabic is treated with sodium formaldehydesulfonate and the product is sepd in a solid state from the resulting soln (suitably by treating with  $\text{MeOH}$  and ether, cooling and filtering).

**Smelting salt.** R. Englert & F. Becker Ger 618,788, Sept 14, 1935 (Cl.121 3). Smelting salt, free from gypsum, is prep'd by fusing crude rock salt, adding alkali equiv. to the gypsum to convert this to  $\text{Na}_2\text{SO}_4$ , and an insol. Ca salt which can be sep'd by settling. The alkali may be alkali phosphate, borate or hydroxide. Examples are given.

**Table salt.** Chem Werke vorm H & E Althert (Hans Mengele, inventor) Ger 621,230, Nov 4, 1935 (Cl. 53k 1 01). In the manuf. of a table-salt mixt containing cations and anions in about the same proportions as the blood, the sparingly sol salts required for the mixt are formed in the presence of the necessary alkali chlorides under such conditions that they are ppt'd in a finely subdivided state on the chlorides. A product completely sol in water is then obtained. An example is given of the prep'n of a mixt contg K 31.5, Na 320, Ca 12.8, Mg 2.96, Fe 1.7, Cu 0.02, Cl 520.9,  $\text{P}_2\text{O}_5$  36.6, SO<sub>2</sub> 16.7 and lactic acid 8.3 parts.

**Storage of soluble salts such as soda ash.** Wm R. McCann (to Atmospheric Nitrogen Corp.) U S 2,024,530, Dec 17. For the storage of a sol salt such as  $\text{Na}_2\text{CO}_3$  which with water forms a mass of smaller apparent vol. than the solid salt prior to mixing with water, the solid salt is mixed with water in sufficient amt. to form a soln of the salt filling the interstices between the particles of an integral mass of undissolved salt comprising the major proportion of the salt and to give a mass of solid and soln having a smaller apparent volume than the solid salt prior to adding the water, and large quantities of the resulting mixt. are stored in a container in which the integral mass is formed. App is described.

**Decomposing beryllium minerals.** Deutsche Gold- und Silber-scheideanstalt vorm Roessler Brit 435,092, Sept 13, 1935. Beryl, etc., is decompd by heating with caustic alkalis in the initial presence of, e. g., 5-25% of  $\text{H}_2\text{O}$  to not exceeding 500°, e. g., 400°. About 16 mols of NaOH per mol of beryl may be used and the alkali may be replaced by substances yielding it under the conditions of operation, e. g., trialkali phosphate. In a modification, the decompn may be conducted in an autoclave, preferably with stirring, at 150-200°. After extn. of the product with  $\text{H}_2\text{O}$  or aq. alkali, the residue may be worked up by treatment with concd.  $\text{H}_2\text{SO}_4$ .  $\text{SiO}_2$  may be removed by evapn., the Al in the filtrate may be ppt'd as alum, and Be may be ppt'd as hydroxide by  $\text{NH}_3$ . Alternatively, the Al, Fe and Be may all be ppt'd. as hydroxides and the Be recovered from the ppt. by treatment with  $(\text{NH}_4)_2\text{CO}_3$ .

**Carbon from carbon monoxide.** Theodor W. Pfirrmann and Georg Gros Ger 621,599, Nov 9, 1935 (Cl. 22f.14). Addn to 565,053 (C A 27, 1110). The method of Ger. 565,053 is modified by adding a pulverulent catalyst to the CO. When the catalyst is a compd. of a carbonyl-forming metal, e. g.,  $\text{Co}(\text{CN})_3$ , it may be removed from the product by treating the latter with CO at a temp. and pressure suitable for the production of the carbonyl.

**Powdered carbon.** Georges Boname Fr 786,380, Sept 2, 1935. In making powd. C by catalytic decompn of a gas rich in CO, a part of the reaction gas is directed as a jet on to the catalyst so as to form a cloud of the catalyst which assures continuity of the reaction. More than one jet may be used at different levels of the reaction chamber and the catalyst may be allowed to fall as grams through the rising current of gas.

**Activated carbon.** Frank H. Cone and Clive B. Houlder (to Activities Ltd.) U S 2,026,355, Dec 31, Fr 787,769, Sept 28, 1935. Finely divided carbonaceous material such as powd. coal is injected into a zone of incandescent gas in which the material is instantaneously raised to a temp. of about 1400° to decompose the material into carbon and hydrocarbons in a fraction of a sec. and before oxidation of the carbonaceous material can take place. App is described.

**Active carbon from peat.** Victor Weerts U S 2,025,367, Dec 24. See Fr 776,328 (C A 29, 3474).

**Catalysts.** Paul Lafitte and Pierre Grandadam. Fr 787,837, Sept. 30, 1935.  $\text{PtO}$  and  $\text{PtO}_2$  (Fr. 785,082, C A 30, 5811) are used as they are or after reduction instead of the usual Pt black as catalysts in the prep'n of  $\text{NH}_3$  from N and H and in the hydrogenation of C,  $\text{CH}_4$ , oxides of C, water gas and camphor, and in the oxidation of  $\text{SO}_2$  to  $\text{SO}_3$  and  $\text{NH}_3$  to oxides of N.

## 19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. E. BARTON AND C. H. KLAR

The glass, ceramics and cement industries in Poland in 1934. Constantin de Kownacki. *Chimie & Industrie* 34, 1217-20(1935). A. Papineau-Couture.

**Determination of the rate of formation of glass.** A. P. Zak. *Mon. Phys. Phys.-Chem. Glass* (Moscow) 30 (1933), *Sprechsaal* 68, 730-7(1935).—The time necessary to complete the formation of glass when melting pure  $\text{Na}_2\text{O} + 2\text{SiO}_2$  at various temps was 1300°, 34 min.; 1250°, 42 min.; 1200°, 54 min.; 1150°, 113 min.; 1100°, 248 min.; 1050°, 490 min.; 1000°, 1088 min. Pure raw materials powdered to a grain size of the 100 to 576 screen sieve were used, the charge was 21.7 g. and was melted in a Pt crucible in an elec. furnace. The rate of formation-temp. curve is approx. hyperbolic for Na silicate.

M. V. Kondoidy. The effect of rocks on the process of glass melting and on some physico-chemical properties of glasses. A. P. Zak. *Mon. Phys. Phys.-Chem. Glass* (Moscow) 37 (1933); *Sprechsaal* 68, 737.—Three kinds of glass of the following compn. were studied:

1	$\text{SiO}_2$ 69%	$\text{R}_2\text{O}_3$ 5.0%	RO 11%	$\text{R}_2\text{O}$ 15%
2	$\text{SiO}_2$ 64%	$\text{R}_2\text{O}_3$ 10.0%	RO 11%	$\text{R}_2\text{O}$ 15%
3	$\text{SiO}_2$ 59%	$\text{R}_2\text{O}_3$ 15.0%	RO 11%	$\text{R}_2\text{O}$ 15%

These glasses were melted from pure raw materials and also from rocks. Twenty, 40 and 60% nephelite or volcanic ash were added. The rate of the formation of glass was det'd. at 1300° for all glasses. The time necessary for the completion of the formation of glass was less than half the time required for mfg. glass from pure raw materials. Volcanic ash accelerated the formation of glass also, al-

though not to such an extent as nephelite. Batches were simultaneously heated to 1400° to det. the time necessary to obtain melts free from bubbles, this temp. was kept for 2 hrs. after which the melts were cooled at the same rate and the no. of bubbles was determined by projection on a screen. The refining time was prolonged by rocks: glass contg. 20% nephelite contained 2853 bubbles, the same glass without nephelite contained only 1033. The tendency of the glass to devitrify is not changed when rocks are introduced. Light absorption is lowered much more by nephelite than by volcanic ash. M. V. Kondoidy.

The ultraviolet test as an aid in the glass industry. Julius Grant. *Glass* 12, 520-2(1935).—Bibliography.

Herbert S. Willson. Setting and antissettling properties of clays. F. H. Clews. *Foundry Trade J.* 53, 422-4(1935). D. S.

Some characteristics of fire clay with special reference to manufacturing difficulties. C. A. G. Thomas and J. R. Adderley. *Brit. Clayworker* 44, 275-76(1935). See C. A. 29, 76001. E. C. Petrie.

Discoloration of wall tile by plant organisms. K. Lameke and R. Kühn. *Keram. Rundschau* 43, 567 (1935).—The green coloration observed in cracks in the glaze of dairy wall tile was due to algae. A moist atm. favored their growth. Minerals in the tile served as nutrient material. P. S. Roller.

Dry working of ceramic mixes. A. Primakovskii. *Novosti Tekhniki* No. 15, 15-16(1934). *Referatkovii Silikatliteratur* No. 593(1934). M. V. Kondoidy.

Germany's pottery industry during 1934. Rolland Welch. *Ceram. Age* 26, 242-3, 246(1935). H. S. W.



A study of some auxiliary fluxes in combination with different feldspars in an earthenware body Ralston Russell, Jr., and Arthur S. Watts *Ceram. Age* 26, 219-21 (1935) Herbert S. Wilson

**Fluorescence VII** The relation of soluble salt content to fluorescence B. Butterworth *Brit. Clayworker* 44, 271-5 (1935), cf. C. A. 27, 5166—The term fluorescence includes damage to plaster by crystals as well as damage to brick.  $\text{CaSO}_4$  is sparingly sol and will not cause defects in brick. The Mg content should not be over 0.65% or serious damage will result. E. C. Petrie

**Fluidity, lubrication and thixotropy of bentonite suspension** Osamu Kumura *J. Chem. Soc. Japan* 56, 1349-52 (1935)—The yield value of Korea bentonite suspension (1.25%) is max. when its NaCl content is 100-200 millimol. The suspension then shows a mm. static coeff. of friction T. Katsurai

**Determination of mullite in ceramic refractories** V. B. Kraft and T. A. Gurvich *Zerodskaya Lab.* 4, 844-90 (1935)—Satisfactory results were obtained in the detn. of mullite in clay refractories by the method of Raacke and Schade (*C. A.* 25, 5742) Chas. Blanc

**Performance of refractories used in basic alloy steel production** Gilbert Solar *Brick and Clay Record* 87, 2-6, 298 (1935)—Nonmetallic inclusions in finished steel are traced to unsatisfactory refractories. The interlocking crystal structure of silica brick makes them ideal for roofs and sidewalls of open-hearth furnaces. Although acid chemically, silica brick resists basic slag because of the formation of immiscible liquids with ferrous, Ca, Mn and Mg oxides. Also in *Bull. Am. Ceram. Soc.* 14, 283-8 (1935) E. C. Petrie

**Grinding** G. Ullmann *Ferroelektrik & Präzision* 43, 193-6 (1935)—A brief historical sketch is given of the development of the art of grinding M. Hartenbaum

**Radiant-tube heating offers many advantages to enamellers** E. F. Gehrig *Industrial Gas* 14, No. 6, 18-20, *Ceram. Age* 26, 223-4, 247 (1935)—The combination of gas within radiant tubes within the oven permits better control of temp. and atm. than with muffles. Max temp. inside the tube wall does not exceed 1850°F. Also in *Natural Gas* 16, No. 12, 20-1 (1935) R. W. Ryan

**Photometric app. for measuring the hiding power of opal glasses** (Brit. pat. 434,126) 26 Rollers for displacing articles at high temps., particularly glass plates or sheets (Brit. pat. 434,315) 1 Concrete [producing bricks or tiles] (U. S. pat. 2,020,257) 20

**Glass, etc.** Sendlinger Opusche Glaswerk G. m. b. H. (Georg Jaekel, inventor) *Ger.* 618,931, Sept. 13, 1935 (cl. 45 15 04)—To produce satisfactory illumination of living and work rooms by combined day- and artificial light when the former is of insufficient intensity, the daylight is passed through a yellowish brown filter to alter its spectral compn. to cause complementary blending with the artificial light. The filter consists of glass of the compn.  $\text{SiO}_2$  71.5,  $\text{Na}_2\text{O}$  15.0,  $\text{CaO}$  11.0,  $\text{Fe}$  1.0 and  $\text{Fe}_2\text{O}_3$  0.2%. Filters of cellulose, dextrin, or solid org. condensation products tinted with suitable dyes may be used.

**Fusion of raw materials to make glass** Walter O. Amler (to Hartford-Empire Co.) U. S. reissue 19,788, Dec. 17, A reissue of 1,869,510 (C. A. 27, 1720)

**Furnace for fusing glass, etc.** Paul Rachalet. *Fr.* 786,123, Aug. 27, 1935

**Electric furnace for fusing glass** Osa participations industrielles Soc. anon. *Fr.* 786,166, Aug. 28, 1935

**Glass furnace with a continuous closed circuit for liquid glass to be delivered to a casting machine** Etienne Fechez. *I. S.* 2,025,581, Dec. 24 Various structural, mech., and operative details

**Supplying molten glass to forming machines** Thomas Semhouse (to Hazel-Atlas Glass Co.). U. S. 2,026,267, Dec. 31 Various details of app. and operation

**Apparatus for feeding molten glass and forming hollow glassware such as bottles** Fritz Eckert (to Hartford

Empire Co.). U. S. 2,026,225, Dec. 31 Various structural, mech., and operative details.

**Device for feeding charges of glass to the molds of a forming machine** The United Glass Bottle Manufacturers Ltd. and Thomas Courtney Mosehead. *Brit.* 434,639, Sept. 5, 1935

**Plungers for glass molds for producing lenses** Richard W. Luce (to Eastman Kodak Co.). U. S. 2,024,653, Dec. 17. Various mfg. and structural details.

**Glass-shaping machines of the kind in which a number of mold units rotate about one or more stationary columns, the various movements of the molds being effected by cams that are fixed to the columns** Europäischer Verband der Flaschenfabriken G. m. b. H. *Brit.* 433,829, Aug. 21, 1935

**Sheet glass** Luke C. Mambourg (to Libbey-Owens-Ford Glass Co.). U. S. 2,026,186, Dec. 31. An app. and operation are described in which a pair of revolving rolls exert a pressure on the glass only sufficient to form a sheet of the desired thickness

**Apparatus for making sheets of glass** Pilkington Bros. Ltd. *Fr.* 787,423, Sept. 23, 1935

**Heat-treating sheets of glass** American Security Co. *Fr.* 786,370, Sept. 2, 1935 Supporting means for the sheets is described

**Case-hardening glass sheets** David H. Goodwille (to Libbey-Owens-Ford Glass Co.). U. S. 2,026,163, Dec. 31 After the sheets are passed through a furnace to heat them to approx. their softening point, they are passed into an envelope which is cooled from both sides so that the opposite surfaces can be chilled. App. is described

**Glass-rolling apparatus for forming glass sheets** Alfred L. Harrington (to Pittsburgh Plate Glass Co.) U. S. 2,025,102, Dec. 24 Rolls are driven by a motor the speed of which is regulated, by a thermostatic control device, in accord with temp. changes in the glass.

**Apparatus for making glass plates** Compagnie réunies des glaces et verres speciaux du nord de la France *Ger.* 618,701, Sept. 16, 1935 (Cl. 23a. 18)

**Apparatus for holding glass plates during case-hardening** Lloyd V. Black (to Pittsburgh Plate Glass Co.) U. S. 2,025,686, Dec. 24 Structural details

**Apparatus for tempering glass plates** Soc. Assurex *Fr.* 787,132, Sept. 17, 1935

**Tempering glass** Assurex le "Roi des Verres de Sécurité" Magnien, Monnier & Coe. *Brit.* 433,748, Aug. 29, 1935 Glass plates are hardened by means of air currents produced by the combined effect of air jets of small dimensions arranged along the sides of adjacent regular polygons and large air-suction openings arranged at the centers of the polygons

**Tempering glass** Soc. anon. des manufactures des glaces et produits chimiques de St-Gobain, Channy & Cuy. *Brit.* 424,231, Aug. 29, 1935 In app. for tempering sheet glass, the sheet remains stationary while being heated and cooled and the heating and cooling devices are so arranged that the cooling period follows the heating period without any time interval.

**Blow-glass containers** Gilbert Schaffer and Guy F. Jardine (to Hazel-Atlas Glass Co.) U. S. 2,024,748, Dec. 17. Various details of app. and operation.

**Marking glass** Theodore B. Drescher (to Bausch & Lomb Optical Co.) U. S. 2,024,277, Dec. 17. A compn. for marking glass surfaces is formed from  $\text{HF}$ ,  $\text{AmOAc}$ ,  $\text{SeCl}_4$  and petrolatum.

**Gazing glassware** Cortland W. Davis (to Mantle Lamp Co. of America). U. S. 2,024,818, Dec. 17. An article to be glazed such as a glass chimney is subjected to a hot medium such as combustion gases at a temp. just short of that at which the article softens and sags, until the temp. of the article is substantially equal to that of the heating medium, and the part of the article to be glazed is then heated to the fusion point. App. is described.

**Glass-polishing tool** Theodore E. Oborg (to Gall & Lenthke, Inc.) U. S. 2,024,293, Dec. 17. A polishing tool carries a layer of carnauba wax shaped to the same form as the tool, for polishing lenses, etc.

**Glass objects** Compagnie internationale pour la fab.

uration mécanique du verre (Procédé Libbey-Owens) Soc. anon. Fr. 787,833, Sept. 30, 1935. Parts of the app and articles which come in contact with the molten glass are made in fused quartz instead of chamotte.

**Mirrors** Soc. anon. des manufactures des glaces et produits chimiques de Saint-Gobain, Chauxy & Cirey Fr. 786,226, Aug. 29, 1935. Glass used for mirrors contains  $\text{NiO}$  oxide, whereby a warmer color is given to objects reflected in it.

**Internal frosting of electric lamp bulbs** Gustav Herzberg and Wm. Ambrun (to General Importing Co.) U. S. 2,025,598, Dec. 24. The inner surface of the bulb is intermittently brought into contact with a heated frosting mixt. such as one contg.  $\text{Hf}$  and acid salts during an interval of the order of 5 sec., and after such contact heat and moisture are applied to the surface during a similar interval intermittently, followed by treatment with cold water for about the same time.

**Colored coating compositions** Colloidal Colours, Ltd. Fr. 787,787, Sept. 28, 1935. See Brit. 421,919 (C. A. 29, 5248).

**Manufacture of glass corner tiles** Gustave P. Mattman (to Cincinnati Advertising Products Co.) U. S. 2,021,775, Dec. 17. App. and various mfg. details are described.

**X-ray absorption glass** Frederick Gelsharp (to Pittsburgh Plate Glass Co.) U. S. 2,025,999, Dec. 24. Glass is formed from a batch substantially free from alkali and contg. approx. 30-65% of  $\text{Pb}$  oxide and 4-15% of  $\text{BaO}$ .

**Luminescent glass** Jules A. Mauler, Paul de Cagny and Jean Purson Fr. 786,147, Aug. 27, 1935. The luminescent material is formed in the nascent state during the formation of the glass itself. Thus carbonates or other fluxes which enter into the constitution of the glass are used for the formation of the material which then remains in the cryst. state in the glass. The luminescent glass may be combined with a special  $\text{Pb}$  glass which arrests cathodic radiations or x-rays.

**Insulation comprising spaced glass sheets** Lloyd V. Black (to Pittsburgh Plate Glass Co.) U. S. 2,025,710, Dec. 31. Between the edges of glass sheets there is placed a spacer of material such as cork or cardboard the pores of which are impregnated with a permanently plastic cement such as a pitch, shellac or pyroxylin compn. which serves to secure the spacer to the glass.

**Laminated glass** Archibald Renfrew (to Imperial Chemical Industries Ltd.) U. S. 2,024,389, Dec. 17. Sheets of glass are united with an intervening layer of a polymerization product, such as one from methyl acrylate or vinyl acetate, formed into a sheet in a partially polymerized and syrupy condition, freed from unpolymerized material by evapn. at a temp. below the b. p. of the latter and then interposed between the sheets of glass.

**Apparatus for making curved sheets of laminated glass** Wm. O. Lyle (to Duplate Corp.) U. S. 2,025,115, Dec. 21. Mech. and operative details.

**Apparatus for working clay such as that for making large tile, etc.** D. Earl Child and James L. Child (to Hancock Brick & Tile Co.) U. S. 2,024,025, Dec. 10. Various details are described of an extruder, a pug mill, a subatm. pressure transfer between them, and associated features of app.

**Bricks, tiles, etc.** Max Perikiewicz Ger. 621,411, Nov. 7, 1935 (Cl. 806, 12 11). Addn. to 545,237 (C. A. 26, 2685). Molded clay, before it is dried and fired, is treated with a suspension contg. a natural or synthetic resin and a clay or a mixt. of clays, with or without a protective colloid, a flux or a glaze. In this way the color of the final products is not only levelled, as described in Ger. 545,237, but can also be modified as desired by suitable selection of the clay, etc., added to the resin suspension.

**Ceramic composition suitable for floor tiles** Andrew Malinovsky (to Malinite Corp.) U. S. 2,025,702, Dec. 21.  $\text{Al}_2\text{O}_3$  0.1-3,  $\text{MgO}$  0.1-8,  $\text{Na}_2\text{O}$  1, and  $\text{SiO}_2$  4-14 parts, on a dehydrated molecular basis, form a compn. of good strength and hardness. Oxides of Co, Cr, Cu, Fe, Mn or Pb may be used for coloring.

**Ceramic flux for whiteware, wall tile, etc.** Joseph H.

Wess (to Feldspathic Research Corp.) U. S. 2,024,407, Dec. 17. Partially kaolinized pegmatite is combined with feldspar, lime and flint in such proportions as to produce a flux having the characteristics of English Cornwall stone and contg.  $\text{SiO}_2$  70-74,  $\text{Al}_2\text{O}_3$  15-17, potash 2.5-4.5, soda 2.5-4.5, lime 1-6 and  $\text{Fe}_2\text{O}_3$  less than 0.1%.

**Ceramic materials** Norton Co. Fr. 786,241, Aug. 31, 1935. Granules of refractory material, e. g.,  $\text{Al}_2\text{O}_3$ ,  $\text{SiC}$ ,  $\text{MgO}$  or quartz, and a ceramic binder, e. g., a mixt. of clays, are mixed with water to form a fluid mass which is then beaten up in the presence of air to form bubbles of air therein. The bubbles are stabilized by an aq. ext. of sawdust, sulfonated castor oil, saponin, licorice or bark of saponaria tree. The mass is then dried and baked.

**Dielectric ceramic material** Porzellanfabrik Kahla. Fr. 787,337, Sept. 21, 1935. Substances such as  $\text{TiO}_2$  in different forms (amorphous  $\text{TiO}_2$ , titanic acid, brookite, anatase or alk. earth titanate) are added to ceramic compns. in amt. sufficient to give a dielec. constant independent of the temp.

**Ceramic ware** Wesley B. Flower U. S. 2,026,624, Jan. 7. A supply mixt. of ceramic material contg. excess liquid is agitated and is fed into the cavity of a substantially closed, rotating, liquid permeable mold, which may be formed of plaster of Paris or porous fired clay, and liquid is withdrawn into the liquid-permeable material of the mold. App. is described suitable for the manuf. of saucers, etc.

**Blue-coated ceramic articles** Frederick J. Iarncomb (to Corning Glass Works) U. S. 2,026,098, Dec. 31. A ceramic article to be blue-coated is exposed to the fumes of a halide of a metal of the W sub-group of the sixth group of the periodic system having an at. wt. between 95 and 185, such as W chloride, to produce on the article a coating of the trioxide of the metal, and is then subjected to reducing conditions (suitably with sulfimating gas) to reduce the trioxide to pentoxide. App. is described.

**Silica articles** Allgemeine Elektricitäts-Ges. Ger. 621,187, Nov. 2, 1935 (Cl. 32a 37) See Brit. 374,761 (C. A. 27, 3299).

**Molded articles containing calcium hydrosilicate.** Karl I. A. Fikund. Ger. 621,340, Nov. 5, 1935 (Cl. 806, 10 01). In the manuf. of tiles, etc., by molding and hardening mixts. contg. lime and sand, the lime is first mixed with sufficient water to slake it and then treated with steam at a higher pressure than the steam employed in the hardening process. The slaking of a part of the lime during the hardening process is thus prevented, and products of improved mech. properties are obtained. (C. A. 30, 6357).

**Molded articles containing calcium hydrosilicate.** Karl I. A. Fikund. Ger. 621,341, Nov. 5, 1935 (Cl. 806, 10 01). In the manuf. of pipes, plates, etc., by molding and hardening mixts. contg.  $\text{CaO}$  and a silicious material, an excess of water is used in prepg. the mixt., which is then molded by a centrifugal or vibrational process under such conditions that a part of the water is sepi. Products of more uniform properties are thus obtained. Various details are given.

**Flame furnaces for ceramic products** Soc. des isolants et réfractaires (Fidélité C. Le Coultre, inventor). Fr. 787,819, Sept. 26, 1935.

**Refractory bricks** C. Otto & Co. C. m. b. H. Ger. 621,283, Nov. 4, 1935 (Cl. 806, 12 04) See Brit. 427,781 (C. A. 29, 6784).

**Magnesia refractories** Frederic A. Harvey and Raymond F. Burch (to Harbison-Walker Refractories Co.) U. S. 2,026,568, Dec. 31. A minor proportion of a forsterite material is incorporated with magnesia refractory grains so that the grains are bonded by cryst. forsterite at temps. as high as 1600°.

**Abrasive** Gilbert E. Seil (to E. J. Lavino & Co.) U. S. 2,026,275, Dec. 31. An abrasive comprises heat-treated and cooled chrome ore material contg. chrome and its accompanying gang, in which the chrome particles are recryst. and the gang material is so distributed over the surface of the recryst. particles of chrome as to avoid any local concn. of gang.

Abrasive wheel Frank H. Manchester (to Wingfoot Corp.) U S 2,024,591, Dec 17. An abrasive wheel is formed of rubber with an outside abrasive material bonded to the rubber by a protein-latex compn such as one formed of casein and latex.

Flexible abrasive articles such as sandpapers, etc. Ralph C. Shuey (to Bakelite Corp.) U S 2,025,249, Dec 24. Abrasive grains are used with a binder comprising multiple layers one of which is of the drying-oil varnish type adherent to the base and another of the heat-hardening phenol-aldehyde type insolubilized by heating and forming a superficial protective coating.

Abrading material suitable for grinding disks or wheels. Theodor Pohl and Josef Schneider U S reissue 19,502, Dec 31. A reissue of 1,996,849 (C A 29, 12244).

Enamel Firmutana Metallkeramik G m b H Ger 619,000, Sept 23, 1935 (Cl 48c 1). Enamelled objects of large area are obtained by enameling sections in a muffle furnace, joining the sections by welding, etc., cleaning the joinings or seams with sand, covering them with enamel powder and firing with a portable heater.

Enameling Walter Becker. Ger 618,613, Sept. 12, 1935 (Cl. 48c. 3). Ornaments, etc., are coated with a colorless or colored enamel flux and heated. The heating is continued after the flux begins to melt and flow, till it begins to crack and form an ornamental pattern. Ag may be treated in this manner.

Enameling Alexander Kneidl Ger 618,612, Sept. 12, 1935 (Cl 48c 1). A process for enameling Fe or steel without using a ground enamel consists in coating the metal with a layer comprising a flux and a spinel forming metal monoxide or sesquioxide, or a metal compd, forming these when heated, heating the layer to glowing in an atm poor in O or with exclusion of air, cooling, applying the enamel and reheating. The flux may be  $B_2O_3$  or fluor spar and the oxide may be  $FeO$  or  $Fe_2O_3$ .

Enameling and glazing Franz M. Wirtz and Reimbold & Strick G m b H Brit 434,115, Aug 11, 1935. Metal is coated with enamel, glaze, etc., and then passed through, or inserted and removed from, a chamber in which the burning-in temp is maintained by any suitable device. The process may be used for enameling, etc., thin Cu or Al foil. App is described.

## 20-CEMENT AND OTHER BUILDING MATERIALS

J C WITT

The classification of mixed cements Rudolf Barta Chem Listy 29, 153 5(1935)—After making a survey of the chem and phys characteristics of cements prep'd throughout the world, B. proposes and presents a table of classifications in which the groups are based upon (1) the clinker base as portland, Roman, aluminaceous, (2) the no. of accessory ingredients, (3) the quality of the mixt., (4) the percentage of the constituents, and (5) the method of prep'n. A sample code gives any position in the table, and since the chem analysis of a cement places the cement in a fixed position in the table, the cement can be designated by the code no. which will convey a picture of the characteristics and position of the cement to anyone acquainted with the table. Frank Marech

The heats of hydration of cements O. Kallauer and B. Burgl Chem Listy 24, 238-43(1935)—The heats of hydration and of soln. were measured in a calorimeter for 12 cements manuf'd in Czechoslovakia. The heat of hydration of an aluminous cement was 83, 89, 94 cal per g at the end of 3, 7 and 24 days, resp., at the same time a special cement for hydraulic application yielded 52, 55 and 57 cal per g, resp., the remaining 10 cements yielded heats of hydration between these limits. During the 48 hrs immediately after the cements are mixed with water, the temp of the mixt. rises to a max and returns to room temp. Each temp-time curve is characteristic for a specific brand of cement. The aluminous cement showed a sudden rise to 105° and a sudden fall in temp. from 5 to 15 hrs after being mixed. The smallest rise of 20° occurred in an ordinary portland cement and lasted about 40 hrs. The course of the temp. drop (after the max has been passed) indicates that in special cements and for cements prep'd in tall ovens the evolution of heat during the hardening of the cement is slow, mild and of long duration. Since the results indicate a direct relation between the heats of hydration and the max. temp. it may be possible under adiabatic conditions to est. the heats of hydration from the temp. max on the temp-time curve. F. Marech

Measurement of the heat developed during the hardening of cement. Stig Gieritz-Hedström Ida 1935, 75-6, cf C A 28, 6267—Addnl heat-development detns for pure and mixed cements are given. E. M. Symmes

Behavior of hydrated and set pozzuolana cements at high temperatures Corrado Vittori and Angelo Cereseto Ann chim applicata 25, 392-7(1935)—Test blocks of portland cement and pozzuolana cements were allowed to set 7 months in water, then dried at 2-300°, and finally heated for 1 hr at temps of 600°, 700°, 800°, 900° and 1000°, the samples being at all times in a CO<sub>2</sub> free atm. Samples were then exposed to air. The portland cements

all show cracks, which increase with the increased temp. of heating, whereas the pozzuolana cements are free from cracks. These results confirm those of Nagai (C A 26, 2841). This resistance to cracking of pozzuolanas is due to the presence of  $2CaO \cdot Al_2O_3 \cdot 7H_2O$  which loses its water of crystn. without undergoing any change in crystal structure. A. W. Conner

The annular deposit in the cement kiln III T. Yokoi J Soc Chem Ind, Japan 38, Suppl binding 192-6(1935), cf C A 29, 8273—The cause of the annular ring formation was exam'd chemically and physically, and a solution of the problem was attempted by the application of the structural arch theory. Expts showed that a raw meal high in  $SiO_2$  and low in  $CaO$  is suited for the prevention of the ring formation, and in order to be able to predict a ring-safe zone, the "residual lime-silica modulus" is suggested. When this modulus

$$\left( \frac{\text{Total } CaO \text{ (mols.)} - CaO \text{ in } CaO \cdot Al_2O_3}{SiO_2} \right)$$

was less than 3.19, no trouble was experienced with ring formation. The methods for the removal of the ring which are being used at present are critically discussed and some improvements are suggested. K. K.

Economic balance of waste-heat boilers attached to cement rotary kilns Shun-ichi Uchida J. Soc Chem Ind, Japan 38, Suppl binding 581-3(1935)—The overall heat-transfer coeff  $H$  (kg cal/sq m hr degree) based upon the outside surface of the boiler tubes (water tube boiler) and the gas velocity  $u$  (m/sec.) were found to be related by  $H = 12.4u^{0.44}$ , where the value 0.43 covers the resistance to heat flow due to cement dust and boiler scale, and the value 12.4 partly covers the heat transfer by radiation due to powder coal flame. Equations for the pressure drop through tube banks, and the most economical mean velocity and temp. difference at the hot gas exit, are also given. An illustrative example is appended. Karl Kammermeyer

The preparation of concrete and the problems related thereto V. Bahrner Tek Tid Uppl C, Aems 65, 73-9, 86-8(1935)—The factors to be considered in the prep'n of various types of concrete are outlined. The effects of the ratio of cement to water (water-cement no.), the mixing period, uniformity, hardening temp., after-treatment and age on the mech. properties of concrete are discussed. The general formulation of concrete mixts and the resulting consistencies are shown graphically. Diagrams are also given for appropriate sand-stone mixts. D. Thuesen

The compression test of concrete with small test pieces II Comparison of the compression of small and common

**mold concrete using different aggregates.** Yoshiaki Sanada *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 555-7 (1935); cf. *C. A.* 29, 313<sup>1</sup>—Exptl results are presented. Conclusions: The strength ratio of 15 × 30 cm. and 5 × 10 cm. concrete samples shows no change with age but is changed by the water-cement ratio. The relation between the compression of these samples is given by  $C_p = K_p' \times C_c$ , where  $C_p$  is the compression of the 15 × 30 cm. sample,  $C_c$  that of the 5 × 10 cm. sample and  $K_p'$  is a const. detd by the water-cement ratio and the aggregates. III Comparison of the compression of concrete and plastic mortar *Ibid* 557-8—The exptl results are presented and show that the effect of the water-cement ratio on the compressive strength of both concrete and plastic mortar increases with the amt. of cement used in the mixes. The max. strength of concrete exists at a certain water-cement ratio, but that of plastic mortar moves toward a slightly higher ratio as the age increases.

Karl Kammermeyer  
**Physicochemical aspects of asphalt pavements.** Energy relations at interface between asphalt and mineral aggregate and their measurements. Charles Mack *Ind. Eng. Chem.* 27, 1500-5 (1935)—Surfaces and their energies play an important part in a system asphalt-mineral aggregate. It is shown that hydrophilic solids have energy relations at their interfaces with pure liquids of the same order as at the interface between water and the same liquids. It is also shown that, with few exceptions, the vol. of a powder settled in a liquid is proportional to the interfacial tension of the liquid against water. By applying these facts to a study of 6 asphalts and 3 aggregates, it is found that the interfacial tensions between asphalts and solids vary with the type of asphalt and with the type of aggregate. This knowledge can be used to predict the behavior of asphalt pavements under the action of water.

Marion E. Headington  
**The drying of solids.** XV The drying of wood. S. Kamei, M. Takimoto and Y. Urakami *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 534-44 (1935), cf. *C. A.* 29, 7704<sup>1</sup>—Drying expts. were carried out on pine, and cypress wood with the vaporizing surface tangential to the tree rings. Considerable exptl. data are presented for drying velocity, total H<sub>2</sub>O evapd., free H<sub>2</sub>O content, air velocity and contraction. Cypress wood behaves like soap, e. g., the const. drying period is absent. The less dense pine wood shows all 3 drying stages like clay and paper pulp. Expts. were also carried out on pine wood with vaporization taking place from the radial and cross-section. The drying velocities for all 3 sections in the const. period were almost equal. In the retarded period the velocity was greatest perpendicular to the longitudinal section, medium in the radial and longitudinal direction and lowest in the direction tangential to the rings. The contraction decreased in the order tangential to rings, radial and longitudinal direction. Karl Kammermeyer

**Cement industry in Poland in 1934 (Kownacki).** 19. Sizing and sepg. fines from crude materials such as cement (U. S. Pat. 2,026,318) 1. Colored coating compns. [for cement] (Fr. pat. 787,787) 19. Heat-exchange app. for cooling hot granular material such as cement clinker (U. S. Pat. 2,024,934) 1.

**Cement.** I. G. Farben und A.-G. (Karl Dietz and Karl Frank, inventors). Ger. 618,749, Sept. 14, 1935 (Cl. 22, 1). Acidproof cement or stone is made by mixing water glass to a paste with a substance reacting strongly with the alkali or SiO<sub>2</sub>, such as NaF, BF<sub>3</sub>, BaO, terephthalyl chloride or benzoic acid anhydride. These substances are powdered and coated with paraffin or wax to delay hardening and ensure even mixing.

**High silica hydraulic cement.** Edward W. Rice (to Santa Cruz Portland Cement Co.). U. S. 2,026,064, Dec. 31. A mixt. of ground raw cement materials contg. SiO<sub>2</sub> and lime in about a 5 to 1 ratio is calcined at a temp. of about 540° or somewhat higher but below the temp. of incipient fusion, to activate the silica while calcining the

lime, the calcined material is mixed with several times its wt. of portland cement clinker, and the mixt. is ground to portland cement fineness. Cf. *C. A.* 29, 313<sup>1</sup>.

**Articles such as those of cement mixtures having a porous cellular structure.** Gustav Adolph and Theodor Pohl (to Leichtbaustoff-Ges. m. B.) U. S. 2,024,791, Dec. 17. Materials capable of effecting the liberation of O<sub>2</sub>, such as H<sub>2</sub>O<sub>2</sub> and NaOCl, are added in stoichiometrical proportions to masses such as cement, sand, water and soap mixts. Cf. *C. A.* 29, 491<sup>2</sup>

**Sintering material such as raw cement mixtures.** Thorne E. Lloyd (to Dwight & Lloyd Sintering Co.) U. S. 2,024,176, Dec. 17. Finely divided material such as a raw cement mix is prepd. for sintering by mixing with a small proportion of oily material such as a mineral oil and with water (insufficient to render the mixt. fluid) and rolling the mixt. to form it into pellets.

**Tunnel kiln suitable for burning cement.** Nicolas Kyriacou U. S. 2,024,048, Dec. 10. Structural and mech. details of a furnace having a conveyor of the reciprocatory type a portion of which is surfaced with refractory material for receiving and reflecting heat. Cf. *C. A.* 29, 3503<sup>3</sup>

**Rotary-drum apparatus for treating solids with gases as in cement kiln operation.** etc. Mikael Vogel-Jorgensen (to F. L. Smith & Co.) U. S. 2,024,453, Dec. 17.

**Various structural, mech. and operative details.**

**Apparatus for lining pipes with cement mortar.** Gerald Haskins and Samuel T. Farnsworth (to Haskins Patent Pipe Linings Ltd.) U. S. 2,026,470, Dec. 31. Various details of an app. of the projectile type.

**Mortars or binders containing rubber.** I. G. Farben und A.-G. Fr. 787,236, Sept. 19, 1935. Mortars or binders suitable for mastics, masonry, rough plaster, undercoating for paints and vibration dampers for cars are made by treating aq. emulsions of rubber, vulcanized or not, with powd. cements or binders so as to obtain mortars which contain basic substances sol. in water or capable of being emulsified and, besides, an excess of substances slowly sol. in water and capable of reacting with the alkalies. These substances react with one another so that the basic character of the mortar is neutralized slowly and the excess of substances which react with the alkalies produce afterward the coagulation or hardening of the mortar by sepn. of the acid or in any other manner, the water of the mortar being bound in the form of gel or in any other form in the final solid product. Thus, a cement composed of borax 4, p-toluenesulfonyl chloride 12, quartz powder 50 parts is mixed with an emulsion of rubber and powd. alkali silicate 20 parts, or borax 8, Na<sub>2</sub>SiF<sub>6</sub> 12 and quartz powder 55 parts is mixed with an emulsion of rubber and Na silicate 25 parts.

**Glaze.** N. V. Nederl. Fabrick van Betonemallic "Tortoliet." Ger. 618,781, Sept. 14, 1935 (Cl. 80b, 23 04). A cold glaze for concrete, walls, etc., consists of cement, soap and a cement-setting accelerator such as ZnCl<sub>2</sub> or CaCl<sub>2</sub>.

**Concrete.** Akt. Ges. Hunziker & Cie. Zürich Baustoff-fabriken Brugg & Olten. Fr. 787,391, Sept. 21, 1935. Concrete of particularly high resistance and chemically insensitive is obtained by adding a material contg. SiO<sub>2</sub>, e. g., quartz, to the constituents and afterward hardening under a pressure of steam. Up to 6% of sol. glass may be added to the water used.

**Light weight porous concrete.** Erik B. Bjorkman. U. S. 2,026,207, Dec. 31. For making preformed concrete of higher compressive strength than 1400 lb. per sq. in. and a lower d. than about 110 lb. per cu. ft., a stream of molten blast-furnace slag is mechanically disintegrated into drops which are solidified and cooled without quenching in water to produce a granulate of hard porous particles capable of developing hydraulic activity on the surface, water is added to the particles, the moist material is consolidated under a pressure in excess of 1500 lb. per sq. in. to form units such as bricks or tiles of the desired shape, the pressure on the units is then immediately released, and the units are cured.

**Curing concrete.** Dozier Finley and Wm. R. Grogg



mineral-matter-free basis A C Fieldner, W. A. Selvig and W. H. Frederic. *Bur. Mines, Rept. of Investigations* No. 3296, 22 pp (1935).—The fixed C (dry and free from mineral matter) of 316 typical Am. coals is plotted against their B. t. u. (moist and free from mineral matter). When used in conjunction with the proposed A S T M specifications for classification of coal by rank, shown tabularly, the chart enables the rapid detn. of rank of typical American coals. Alden H Emery

Alteration of coal seams in the vicinity of igneous intrusions, and associated problems Henry Briggs *Trans Inst Mining Engrs* 90, 114 18(1935). cf C A 29, 7044—Discussion Alden H Emery

Study of Roumanian coals by thermal fractionation of the gaseous products of pyrolysis Jean Rudic *Am Combustibles Liquides* 10, 889-921(1935). cf C A 29, 3493—The method of Lebeau (C A 28, 3215) was applied to 10 Roumanian coals. The yields of volatile products at various temps., microscopic exam. and the action of solvents lead to the conclusion that 6 of the samples are lignites, 3 bituminous coals and 1 is peranthracite. G Cabaigart

Coal from the Saar region W Gollmer *Gas u. Wasserfach* 78, 917-22(1935)—There appears to be a general belief in Germany that the Saar coals give a poorer coke than Ruhr coals. Coke produced from this coal alone, without grinding, has porous structure, due to the high volatile matter content of this coal, leading to a higher reactivity but a somewhat lower coke strength. The increase in gas and by-product yield together with improvement in coke qualities justifies the grinding and mixing of Saar coal before carbonization. Mixt with low-temp. coke or other coals gives an excellent coke. R W Ryan

The grading and classification of coal. H Eustace Mitton and D T Davies *Trans Inst Mining Engrs* 90, 3-8, 45-61(1935). cf C A 29, 7044—The breakage of coal in mining and handling and crushing for marketing are discussed. Alden H Emery

Coal's new industry—the production of oil F S Summitt. *Gas J* 212, 711 12(1935).—Hydrogenation is discussed. P J. Wilson, Jr.

The new preparation plant at the Rising Sun Colliery of the Wallend & Hebburn Coal Co. Ltd. Kenelm C Appleyard. *Trans Inst Mining Engrs* 90, 37-61(1935).—The plant, consisting of the following, is described: dry cleaning, wet washing, de-dusting, flotation of dust, filtration, thermal drying, water clarification, blending and mixing and dry and wet screening. Alden H Emery

Modernization of coal-preparation plants continues, without striking innovations Henry F Hebley. *Mining Met.* 17, 36-7(1936). Alden H Emery

Measurement of the expansion of coal during carbonization. V. J. Altieri. *Am Gas J* 143, No. 6, 43(1935). cf C. A. 29, 7814—A unidirectional coal-expansion tester is described. Joseph H Wells

The development of coal hydrogenation by Imperial Chemical Industries, Ltd. Kenneth Gordon. *Gas J* 212, 722-6; *Colliery Guardian* 151, 985-8; *Petroleum Times* 34, 547-58(1935).—Historical. P. J. Wilson, Jr.

The complete solution of the fundamental matter of a coal in benzene A. Gillet and A. Priot. *Bull. soc. chim. Belg.* 44, 604-12(1935).—At high pressures and temps. complete soln. is attained. R. E. DeRight

Fusain content of coal dust from an Illinois dedusting plant. Gilbert Thiesen. *Am. Inst. Mining Met. Engrs. Tech. Pub. No. 664*, 12 pp.(1936).—Because of its friable nature, fusain is concd in the fine coal dust. The very fine dust, -200 mesh, produced during the prepn. of coal, is composed mainly of fusain. The coking properties of the fine sizes of Ill. coals may be improved by dedusting. The P content of the coal is reduced at the same time. Alden H Emery

The pneumatic dedusting of coal S. R. Berrisford and R. H. Allen. *Trans Inst. Mining Engrs.* 90, 139-9, 140-60(1935); cf C A 29, 8285—Discussion Alden H Emery

The response of coal-cleaning practice to the demands of modern mining Geo Raw and F. F. Ridley. *Trans Inst. Mining Engrs* 90, 9-37, 45-61(1935)—The main factors controlling the type of treatment are (1) range of sizes and ash content of each, (2) free moisture, and (3) the proportions, ash contents and sp. gr. of the constituent portions of coal and impurities. Different methods of wet washing, dry cleaning and float-and-sink sepn. are described. Alden H Emery

The auto-ignition process in coal W Bielenberg. *Angew. Chem.* 48, 779-81(1935)—A parallelism between certain properties of a coal and its auto-ignition behavior does not exist, although several factors, such as brittleness, pyrite content, catalytic action of mineral constituents, etc., may exert contributing influences. The auto-ignition depends upon the easy oxidation of the coal at ordinary temps. and the occurrence of heat storage depending upon the reaction velocity. The dissipation of the heat of reaction may be very slow as the thermal cond. of coal is very low, i. e., about 0.3 for lignite. The app. of Dennstedt (see C A 3, 1453) was used for the detn. of the autooxidation-time curves of coals. The intervals to the ignition point increase greatly with decreasing initial test temp., and the shape of the curve becomes increasingly flatter at the front end. If the initial temp. is sufficiently low, the ignition temp. may not be reached at all, as all of the oxidizable substance may have been used up before the temp. of the coal reaches the ignition point. Auto-ignition tests artificially accelerated by higher temps. are not reliable. Only such spots in coal piles will show auto-ignition, where the heat insulation is excellent and O<sub>2</sub> has access. Autooxidation curves and six references are given. Karl Kammermeyer

Heat conductance of a layer (of coal) P T Kolesnikov. *Podzemnaya Gazifikatsiya Uglei* 1935, No. 2, 9-11—Actual measurements of heat conductance were carried out in various parts of an underground coal block undergoing gasification. The problem was complicated by the presence of cracks in the block through which gases circulated. The heat cond. was found to be 0.12-0.15 cal/s per sq m per hr. S L Madorsky

Steaming in horizontal retorts G L Braidwood. *Gas J* 212, 701-10(1935), *Gas World* 103, 567-75—The results at Beckton are described. In a 12-hr. carbonizing period with 3-hr. steaming, the steam consumed amounted to 11.2% by weight of the coal carbonized, and the decomposition efficiency was 27.6%. An increase of 7.2 therms per ton of coal of 480 B. t. u. was secured. The extra fuel consumption was between 220 and 620 lb. per ton of coal carbonized. With a 10-hr. carbonizing period and 2-hr. steaming only 16.3% decompos. was secured. The type of charging machine has probably the greatest influence on the conditions favorable to good results. The charges should be uniform and tapered at the gas end. The H<sub>2</sub>S content of the steamed gas was increased. The work indicated that the cost per therm of unpurified gas, after deducting the credits for coke and by-products, was increased by 0.3 d. The experiences at other plants, described in the discussion, did not agree with the Beckton results because of differences in the conditions. P J. Wilson, Jr.

Coal losses due to underground fires at Kuzbass. K. N. Shechepot'ev. *Podzemnaya Gazifikatsiya Uglei* 1935, No. 2, 11-14—Because of spontaneous combustion in the mines of easily combustible coal, losses amount to 50% of the amt. produced. To avoid these losses underground gasification of the coal is recommended. S L Madorsky

Electro hydrogenation of coal under conditions of underground gasification. V. V. Filatov. *Podzemnaya Gazifikatsiya Uglei* 1935, No. 2, 22-8—A discussion of Bergius method, involving production of H and hydrogenation of coal, under conditions of underground operation. S L Madorsky

Heat and gas calculations underlying the project of underground gasification (of coal) V. V. Pomerantsev, N. I. Sazonov and S. N. Sukrin. *Podzemnaya Gazifikatsiya Uglei* 1935, No. 1, 9-15—A discussion of gas

movements, heat losses and gas compn involved in under-  
ground gasification of coal S L Madorsky

Further experiments on underground gasification of  
Moscow coals P T Kolesnikov *Podzemnaya Gazifika-  
tsiya Uglei* 1935, No 1, 15-20—Moscow coals are  
low-grade, they contain 30-34 moisture, 25-30 ash and  
36-45% combustibles Expts were carried out on under-  
ground gasification of the Kruptovsk deposit, lying about  
20 m deep, by blowing air for combustion through borings  
into a body of coal embracing about 250 tons, and collecting  
the coal gas from another series of borings S L M.

Results of the third experiment (on underground  
gasification) of Moscow District coals P T Kolesnikov  
*Podzemnaya Gazifikaitsiya Uglei* 1935, No 4, 29-34—  
Gasification was carried out on an unbroken block of coal  
The gas obtained was nonuniform in compn and had an  
av heat value of 1000 Cals per cu m S L M.

Experiment on underground gasification of coal in the  
Lenn-Kuznetzk District (Kuzbas) Petrovichev. *Pod-  
zemnaya Gazifikaitsiya Uglei* 1935, No 1, 31-5—The  
compn of the coal is C 79.12, H 5.94, O 12.28, N 2.38,  
S 0.28 and volatile matter 43.62% The wt of the block  
subjected to treatment was approx 1500 tons After the  
fire was started underground, air was blown into the coal  
through drifts The chem reactions taking place in the  
gasification are discussed in the light of the compn of the  
resulting gas S L Madorsky

Method of underground gasification of coal by means of  
pit holes S I Buyalov *Podzemnaya Gazifikaitsiya Uglei*  
1935, No 2, 3-9 S L Madorsky

Fundamentals involved in the projecting of underground  
gasification of coal F M Ivanov *Podzemnaya Gazifikaitsiya Uglei*  
1935, No 2, 18-20—A discussion

Economic basis for underground gasification of coal  
by the Zhuravlev method T V Ovchinnikov *Pod-  
zemnaya Gazifikaitsiya Uglei* 1935, No 2, 20-2—The  
Zhuravlev method consists in duplicating in underground  
gasification the ordinary gas-generator method The air  
or steam is blown from underneath the coal block, through  
holes in the block, and the resulting gas is forced to the  
surface through shafts In one expt in the Chelyabinsk  
coal deposit the gas produced by this method had the  
following compn: CO<sub>2</sub> 9.61, CO 22.05, CH<sub>4</sub> 2.49, C<sub>2</sub>H<sub>6</sub>  
0.35, H<sub>2</sub> 18.15, H<sub>2</sub>S 0.17, N<sub>2</sub> 47.00 and NH<sub>3</sub> 0.18%  
heat value of the gas is 1420 Cals per cu m The yield of  
dry gas is 2 cu m per kg of coal S L Madorsky

Economics of underground gasification of coals T V  
Ovchinnikov *Podzemnaya Gazifikaitsiya Uglei* 1935, No  
4, 34-9 S L Madorsky

Second series of experiments on underground gasifica-  
tion of coal at Kuzbas B P Grindler *Podzemnaya Gazifikaitsiya Uglei*  
1935, No 4, 3-6—The experience  
gained in a previous test on underground gasification of  
coal (C 29, 67.28%), which lasted for 6 months, served  
as a basis for another test The difficulties encountered  
in the first test, particularly in regard to uniformity of  
the gas produced, were eliminated here Compn of the gas  
averaged CO<sub>2</sub> 11.10, CO 12.80, methane and unsatd  
hydrocarbons 1.80, H<sub>2</sub> 17.25, H<sub>2</sub>S 0.30, N<sub>2</sub> 55.55 and  
H<sub>2</sub>O 1.20% It had a heat value of 1035 Cals per cu m  
S L Madorsky

Calculation of heat losses in underground gasification  
of coal P V Melent'ev *Podzemnaya Gazifikaitsiya Uglei*  
1935, No 4, 7-12 S L Madorsky

Underground gasification of coal by means of double  
pit holes S I Buyalov *Podzemnaya Gazifikaitsiya Uglei*  
1935, No 4, 14-21—A discussion S L M.

Results of experiments (on underground gasification of  
coal) at Krutov shaft P A Manukyan *Podzemnaya Gazifikaitsiya Uglei*  
1935, No 4, 22-8—A discussion of  
causes underlying the failure of these expts S L M.

First generator gas under conditions of underground  
gasification (of coal) P T Kolesnikov. *Podzemnaya Gazifikaitsiya Uglei*  
1935, No 4, 39-41—Gasification was  
carried out on low-grade coal contg 34.57 moisture and  
28.26% ash The resulting gas contained 6-13% CO,

5-9% H<sub>2</sub> and approx 0.2% CH<sub>4</sub> It had a heat value of  
about 1000 Cals S L Madorsky

The development of future possibilities of gas as an  
industrial fuel Joseph B. White. *Gas J* 212, 642-6,  
712-13(1935), *Gas World* 103, No 2681, *Ind. Gas Suppl*  
113-19—Applications of gas in the ferrous and nonferrous  
metal industries P J Wilson, Jr.

The place of gas in German energy supply. L Winkler.  
*Gas u Wasserfach* 78, 815-8, 876-80(1935)—A discussion  
of competition of gas with electricity and with solid fuels  
A gas of higher heating value than the present 430-  
450 B. t. u. gas is advocated, as offering a lower unit cost  
Low-temp distn is advocated so as to produce larger  
vols of liquid fuel, and a gas of higher heating value, which  
may be scrubbed to remove heavy hydrocarbons. Motor-  
fuel synthesis from manufd gas by the Fischer process is  
also advocated, and attention called to yield of gas of  
higher heating value from this process in addn to motor  
fuel R. W. Ryan

Advances in dry-gas purification A Rettemmiser  
*Gas u Wasserfach* 78, 848-9(1935)—For regeneration of  
fouled gas-purifying material, a tower purifier contg the  
fouled material is isolated from the system and gas contg  
a regulated amt of O recirculated through the tower and  
a water-cooled condenser to absorb the heat of reaction  
and prevent the regeneration temp exceeding 40°. The  
O content of the recirculated gas is measured and controlled  
by an O recorder At first the O content is kept low, but  
is gradually increased to approx 5%. This process approx-  
doubles the purifier capacity This principle was originally  
suggested in 1907 by Doherty (U. S. pat. 843,624, C. A.  
1, 918) but did not find general application because of the  
absence of a suitable O recorder-controller to ensure  
safe and automatic control R. W. Ryan

Gas-dehydration experience in England G. W.  
Anderson *Gas u Wasserfach* 78, 828-33(1935).—The  
experience of the Gas Light and Coke Co. of London with  
gas dehydration by means of CaCl<sub>2</sub> solns. is reviewed  
At first, rotary brush washers were used for contacting  
gas with the soln, but static multilamin washers are now  
used for large plants and tower scrubbers for smaller plants,  
resulting in lower costs Total operating charges, includ-  
ing capital charges, vary from about 0.12¢ per M. cu ft  
for a plant treating 100,000,000 cu ft of gas per day to  
0.3¢ for a plant treating only 1,000,000 cu ft of gas  
per day In most cases the dehydration plant is at holder  
inlet, Beckton being the most important exception The  
gas-pressure drop through the dehydration plant was 1 to  
3 in. (water) Oil films are used in water-sealed holders  
so that the dew point of gas stored in these holders in-  
creased only about 1° to 1.5° in winter and 2° to 3° in sum-  
mer, although very much higher increases were noted in  
those holders provided with wooden framing for support-  
ing the holder crown A special petroleum distillate was  
used for filming, and this is said to be better than gas oil  
Dehydration causes a loss of 1/4 to 1% of the gas vol,  
but the Gas Referees allow the actual dew point of the  
gas as distributed to be taken into account in calcg the  
heating value of the gas Only 2 trucks are now required  
to collect condensate from the 33,000 drops on about 5300  
miles of distribution system, as compared with 10 used  
before dehydration Only a very slight increase in un-  
accounted for gas was noted The no of meter changes  
has greatly decreased, as have stoppages of services. Re-  
cently gum has appeared in both dehydrated and unde-  
hydrated gas-distribution systems, and a marked increase  
in no of stoppages has been noted, but no direct connec-  
tion between gas dehydration and gum formation could be  
established R. W. Ryan

Electric current from gas Gustav Josse *Gas u Wasserfach*  
78, 885-91, 903-6(1935)—A discussion of the  
use of gas for generating elec power in decentralized  
plants A suggestion is made that gas be generated  
outside of a city and supplied to a series of plants within  
the city When steam plants are considered, the use of  
gas shows the highest increase in efficiency with the older  
type of boilers and a much lower increase with modern  
boilers Gas offers special advantages for small boilers,

because of its greater flexibility and lower labor costs. For large boiler plants, the gas must be very low in price or used for special purposes, such as peak loads or reserve equipment. Various types of gas burners are discussed and illustrated. The use of gas engines of several types is also discussed. Such engines find applications in areas where cheap coke-oven gas is available. R. W. Ryan

Testing the tightness of high-pressure gas lines. P. Eduard Quintes. *Gas u. Wasserfach* 78, 900-10 (1935).—A compressed gas cylinder is buried at the same depth as the gas line and both the cylinder and the line are brought to some such pressure as 20 atm. A differential manometer between the cylinder and line then permits detection of any large leaks in the line. Small leaks can be evaluated by releasing sufficient gas from the cylinder so as to equalize the manometer reading and measuring this gas volume in a gas buret, this information serves for the calculation of the exact amount of leak. R. W. Ryan

Economic and hygienic aspects of heating with gas. E. Vogel. *Gas u. Wasserfach* 78, 922-9 (1935).—Special advantages in the use of gas for heat are the ease with which it may be regulated and the proper temperature maintained, the ease of using space heaters in each room, the possibility of supplying gas for other household purposes at a lower rate and cleanliness in use, etc. The danger of poisoning by gas is no greater than with solid fuels as suitable safety devices can be used with gas but not with solid fuels. R. W. Ryan

Gas-cleaning and sludge-recovery system. Fred Wille and Albert Mohr, Jr. *Blast Furnace & Steel Plant* 23, 529-32 (1935).—Hot  $H_2O$  is more efficient than cold  $H_2O$  for washing gas. This increased efficiency is related to the smaller size of the atomized  $H_2O$  particle resulting from the combined effect of decreased viscosity and decreased surface tension with increased temperature. A gas-cleaning and sludge-recovery system using hot  $H_2O$  and settling cones designed to clean 80,000 cu. ft. of gas per min. is described. The  $H_2O$  consumption is less than 22 gal. per 1000 cu. ft. of gas. The waste  $H_2O$  has less than 10 grains per gal. of suspended solids. The cleaned gas has less than 0.015 grain of dust and less than 1.0 grain of moisture per cu. ft. F. G. Norris

Gas-works lubrication. A. J. Walker. *Gas J.* 212, 909-11 (1935).

Developments in gas-making process for peak loads. Leon J. Wullen. *Am. Gas J.* 143, No. 5, 23-4, 74 (1935).—Substitute gases are used to augment the supply of coal or natural gas. Joseph H. Wells

Single-stage coke-oven gas exhausters, gas boosters and air blowers. W. Vincent Treeby. *Gas World* 103, 544-8 (1935).—A description, including the operating instructions, of a typical gas-exhauster installation.

P. J. Wilson, Jr.  
Thermostatic water quantity regulation with gas water heaters. Walter Brümmerhoff. *Gas u. Wasserfach* 78, 932 (1935).—A description of a thermostatically controlled combination device for shutting off gas in case of water-supply failure and for regulating water flow. This regulator permits conservation of heat in water supplied to a heater, an exact control of the temperature of the water from the heater, elimination of heat waste due to slow initial rate of water heating and automatic starting and stopping of the heating device according to the temperature of the preheated water supplied to the heater. R. W. Ryan

Gum deposits from illuminating gas. B. Th. Tjabbes. *Chem. Weekblad* 32, 521-6 (1935).—A review of the formation of liquid and vapor-phase gums. NO was detected in city gas by the Guyer and Weber method (C. A. 28, 880\*); NO was oxidized by  $H_2SO_4-KMnO_4$ . Good results were obtained on NO removal by dry boxes at the Groningen gas works. The removal was more satisfactory at low temperature (winter) than at high temperature. Fresh bog ore frequently gives off NO to gas (or to  $CO_2$ ), not to air or to  $N_2$ . Samples of ore from different origins showed this effect to a different extent but addition of 10% active ore to 90% of an inactive species caused a NO yield many times higher than that expected from the active ore alone. The NO yield from 4 parts bog ore + 1 part Lux mass

was for a few days 6 to 8 parts per million cu. ft. of gas. The above NO evolution can possibly be explained from bacterial action; actually the NO evolution decreased materially by addition of 0.2% PhOH to the gas and 1% PhOH stopped it altogether. Use of  $CH_3O$  in the gas was somewhat less effective. B. J. C. van der Hoeven

Vapor-phase gum. W. F. Thorne. *Gas J.* 212, 905-6 (1935).—The N in a sample of vapor-phase gum was present as  $NH_4Cl$  and as the  $NH_4$  salt of an organic acid. The reaction of the gum with resorcinol and  $H_2SO_4$  indicates the presence of the  $\alpha-C_6H_4(C-)_2$  grouping. Conclusion: The gum is similar in formation and constitution to the ordinary coumarone-indene resin. P. J. Wilson, Jr.

Conversion of hydrogen sulfide from coal gas into concentrated sulfuric acid. Hoelzer. *Gas u. Wasserfach* 78, 894-5 (1935).—An exptl. plant, scrubbing about 260,000 cu. ft. of gas daily, has been used for the direct removal of  $H_2S$  from gas, by means of  $NH_4$  liquor. The  $NH_4$  sulfide and carbonate formed in this way are decomposed in an eliminator at  $96^\circ$  into  $CO_2$  and  $H_2S$  as well as an  $NH_4$  soln., the latter being returned to the scrubber. The acid gases from the eliminator, containing about 14%  $H_2S$ , 32%  $CO_2$ , 5%  $NH_3$  and 49%  $H_2O$ , are cooled to remove ammonia, washed with  $H_2SO_4$ , and the resulting mixture of about 30%  $H_2S$  and 70%  $CO_2$  is sent to the contact  $H_2SO_4$  plant. It is possible to reduce the  $H_2S$  content of the gas to about 85 grains per 100 cu. ft., complete removal of  $H_2S$  would be unprofitable, and the 70% removal appreciably lightens the load on the dry purifiers. R. W. Ryan

Simple methods of determining olefins and paraffins in coking gas and its ethylene fraction. P. K. Sakmin, *Khimistoi* 7, 487-93 (1935); cf. C. A. 29, 3214, 3262\* 6732\*.—Full details are given for the analysis of mixtures of  $CH_4$  and  $C_2H_4$ ,  $C_2H_6$  and  $C_3H_8$ ,  $CH_4$ ,  $C_2H_4$  and  $C_2H_6$ ,  $C_2H_4$  and  $C_3H_8$ ,  $C_2H_6$  and  $C_3H_8$ , and  $C_2H_4$ ,  $C_2H_6$  and  $C_3H_8$ . The procedures are based on the methods previously described. Chas. Blanc

The synthesis of liquid hydrocarbons from natural gas. III-IV. Theoretical discussion of the pyrolysis of methane and of the formation of acetylene from methane. Kyuhei Kobayashi, Ken-ichi Yamamoto, Heishichi Ishikawa and Sukekazu Hinonishi. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 550-5 (1935); cf. C. A. 29, 1959\*.—The theoretical discussion is based upon a coordination of the published work. Exptl. work was carried out in the range of 800-1300° and with a  $CH_4$  content of the raw gas of 68.9, 46.3 and 39.8%. The decomposition of  $CH_4$  is accelerated at higher temperatures with an increase of the  $H_2$  and  $C_2H_2$  content in the exhaust gas. A considerable quantity of  $C_2H_4$  is formed between 1100° and 1200°, but it diminishes rapidly at higher temperature. The effect of different heating times between 0.05 and 0.15 sec. is very small. With 39.8%  $CH_4$ , at 1300° and 0.15 sec., the max. amt. of  $C_2H_4$  produced is 17.4% based on the total  $CH_4$  and 26.4% based on the reacted  $CH_4$ , while 7.2% of  $CH_4$  changed to  $C_2H_4$ , giving a total conversion of 37.3% based on the reacted  $CH_4$ . Conclusion:  $C_2H_4$  formation from  $CH_4$  will be more efficient above 1300°. Exptl. data are presented. K. K.

Determination of naphthalene. H. A. J. Pieters and K. Penner. *Chem. Weekblad* 32, 566-8 (1935).—Naphthalene picrate at 20° is not noticeably sol. in picric acid soln. regardless of the concentration of the latter as long as it is above 0.148%, the strength corresponding to the amount of free picric acid in equilibrium with solid naphthalene picrate. The concns. tested ran up to 0.4%; the filtered soln. was titrated with 0.02 N alkali and cresol red-bromothymol blue mixed indicator. The vapor pressure of  $C_{10}H_8$  over picrate soln. depends on the temperature and concentration of the picric acid:  $P_0 \times N_0 = k = f(t)$ . For accurate  $C_{10}H_8$  work the soln. should be cooled to 4°. Benzene picrate ppts. only at  $C_{10}H_8$  concns. of more than 140 g. per cu. m. air. This benzene concn. is well beyond that present in coke-oven gas. B. J. C. van der Hoeven

Ammonia recovery at Nantes. C. Cooper. *Gas J.* 212, 844-8 (1935).—The vertical chamber oven plant is operated with continuous steaming, the admission of



steam being 4% by wt. of the coal. The gas is cooled in the first stage of the primary condenser to 70°, and the condensate is either used in the NH<sub>3</sub> scrubbers or discarded. The condensate from the remaining condenser compartments is continuously recirculated by spraying into the hot gas from the retorts. The NH<sub>3</sub> scrubbers consist of a Livesey washer and a 12-compartment Multifilm washer, in series, both operated at a temp of 15°. They deliver a liquor contg 80 g per l NH<sub>3</sub>, which represents 70 to 75% of the NH<sub>3</sub> in the gas. From 4 to 5 grains of NH<sub>3</sub> per 100 cu ft of gas goes on to the purifiers.

P. J. Wilson, Jr.

Catalytic hydrogenation of phenolic oil of low-temperature tar. III. Effect of catalysts. Shingo Andô. *J Soc Chem Ind, Japan* 38, Suppl. binding 567-9(1935), cf. *C. A.* 29, 912. —One hundred g. of sample oil and 5 g. of catalyst were heated with 4.46% by wt. of H<sub>2</sub> for 1 hr. in a 0.6-l shaking autoclave at 470° and with an initial H<sub>2</sub> pressure of 100 atm at 0°. In all expts the liquid-product fractions boiling below 170° were colorless and volatile with gasoline odor. The distillates between 170° and 300° were colorless or yellow. Twenty-one catalysts were tested. The yield of gasoline fraction was highest with a Mo catalyst (MoO<sub>3</sub>) followed by SnCl<sub>4</sub>, ZnCl<sub>2</sub>, NiO and CoCl<sub>2</sub>, while some metallic oxides were almost inactive. Phenolic oils contained in the 170-220° fractions were those formed by dealkylation of higher phenols, but no catalyst was observed which accelerated this reaction in a specific manner. The unconverted phenolic oil, sep'd from the products, was found to be desulfurized to a remarkable extent, especially with Mo compds, NiO, Fe<sub>2</sub>O<sub>3</sub> and CoS, and this oil is considered suitable for the manu' of synthetic resins, etc. Exptl. results are presented.

Karl Kammermeyer

Hydrogenation of high-temperature tar and distillates. C. M. Cawley. *Gas J.* 212, 571-2(1935); *Gas World* 103, 531-2. —The amenability of tar to hydrogenation decreases with increasing temp. of carbonization. Thus the economics of the hydrogenation of coke oven and horizontal retort tars should be carefully considered before treatment is undertaken.

P. J. Wilson, Jr.

Does hydrogenation pay? F. G. Lupschler. *Gas J.* 212, 727-9(1935). —The min. production costs for the hydrogenation of tar to motor fuel are est'd at 8.07 d per gal. (Imperial), an av. yield of 85% is assumed. The plant cost exclusive of gas plant and auxiliaries decreases from £3000 to £2500 per ton of tar hydrogenated with increase in the capacity. Under present conditions only the protective methods of the European governments keep hydrogenation alive.

P. J. Wilson, Jr.

Coke oven manager's association. Inaugural address. N. L. Tyler. *Gas World* 103, No. 2679, Coking Sect. 116-18(1935). —T. discusses the disposal of the products from by-product coke ovens.

P. J. Wilson, Jr.

The future of coke-oven development with special reference to coke marketing. G. E. Foxwell and W. L. Boon. *Gas World* 103, No. 2679, Coking Sect. 113-26(1935). —The by-product coking industry is faced with the impossibility of expansion in the blast-furnace industry. The domestic coke market is the obvious outlet.

P. J. Wilson, Jr.

The marketing of coke. Domestic coke boilers for small installations. F. M. H. Taylor. *Gas J.* 212, 906-8(1935). —The equipment is described.

P. J. Wilson, Jr.

Determination of volatile matter in coke. A. P. Shakhno and M. D. Zhukovskaya. *Zavodskaya Lab.* 4, 874-7(1935). —A comparative study of proposed methods for the detn. of volatile matter in coke with the use of European and American elec. and gas furnaces produced unsatisfactory results because of the excessive oxidation of coke. In all cases better results were obtained by heating a sample in an atm. of pure N<sub>2</sub>. The best results were obtained by heating a sample in a Pt crucible with an alc. burner for 15 min. by conducting pure N<sub>2</sub> directly into the crucible. By this procedure the oxidation was reduced from 0.79-1.13% to 0.16-0.48%. Chas. Blanc

1 Changing to gas fuel improves Columbia tool steels (Foster) 9 Rotary-drum app. for filtering and dewatering slimes such as those from coal (U. S. pat. 2,024,338) 1 Exhaust steam condenser (U. S. pat. 2,025,043) 1 Plastic masses [from coal like fuel] (Ger. pat. 618,566) 13 Segg. materials such as coal and stone (U. S. pat. 2,026,343) 1

2 Fuel Charles Michel. Fr. 787,222, Sept. 19, 1935. A radioactive material in soln. or the gaseous state is incorporated in fuels to intensify the phenomenon of ionization of the explosive mixt. before and during burning. Stabilizing fuels. Soc. d'études & réalisation dite Ereal. Fr. 787,470, Sept. 23, 1935. Hydrocarbons contg. di- and poly-olefins are purified and (or) stabilized by adding an aromatic compd. contg. in its mol. one or more SO<sub>2</sub>H groups so as to transform the di- and poly-olefins into resins and segg. the resins. The compds. used include benzene-, *p*-toluene- and  $\alpha$ - and  $\beta$ -naphthalene-sulfonic acids and they may be used in alc. soln. The free or combined S is also removed by the treatment. Fr. 787,471. Aliphatic compds. contg. SO<sub>2</sub>H, e. g., methyl- and ethyl-sulfuric acids are used. Fr. 787,472. App. is described for heating the hydrocarbons with the reagent continuously in countercurrent and distg.

4 Motor fuel Piero Buti. Fr. 786,336, Aug. 31, 1935. The fuel is composed of a homogeneous mixt. of EtOH (50-70) and a hydrocarbon distillate, the b-p. range of which is about 70-100° (22-42 parts). The mixt. is stabilized by benzene up to 8 parts. The EtOH may be replaced partly or completely by MeOH with or without a certain proportion of iso-PrOH or iso-BuOH.

5 Motor fuel Aldo Giunta and Paul Letournier. Fr. 787,590, Sept. 25, 1935. The turbidity of fuels contg. gasoline and EtOH caused by the fact that the EtOH is never anhyd. or does not remain anhyd. is produced at a lower temp., e. g., below the temp. of use of the fuel by the addn. of a 3rd substance which is sol. in gasoline, EtOH and water. Examples are piperidine 0.68, cyclohexanol 0.7, complex phenols 0.94, cyclohexanol and PhNH<sub>2</sub> 0.95, pure PhOH 0.95, benzylamine 0.95, iso-PrOH 1.07, benzyl alc. 1.48, AcOEt 2.15, paraldehyde 2.3, ethyl ether 2.9, HCOOEt 2.9, acetone 5.8 and C<sub>2</sub>H<sub>5</sub> 10.6%. Any of these will lower the turbidity pt. of a fuel contg. gasoline 100 and alc. 6.5 vols. from 19 to 9°.

6 Refining hydrocarbons for motor fuels. N. V. de Bataafsche Petroleum Maatschappij. Fr. 787,565, Sept. 25, 1935. Motor fuels contg. a high proportion of unsatd. or aromatic components are prep'd by bringing crude hydrocarbons into contact in a continuous manner, in the vapor phase and in the absence of H<sub>2</sub>SO<sub>4</sub>, with a sulfonic acid of a simple hydrocarbon, substituted or not by halogen, in soln. or suspension in an inert org. medium liquid at the temp. of operation, the vapors of the hydrocarbons being constantly mixed with steam during the refining. The concn. of the sulfonic acid (benzenesulfonic acid) in the org. solvent (a lubricating oil) is at most 10 and preferably below 5%, and the refining temp. is high enough to avoid intense hydration of the sulfonic acid.

8 Fuel briquets Arthur A. Roberts. U. S. 2,025,776, Dec. 31. A mixt. of powd. fuel such as powd. coal and a binder such as cement, bitumen, etc., is formed into briquets which are baked to decompose smoke-producing substances in the superficial portion of the briquets and the evolved vapors are passed through fresh solid carbonaceous fuel and condensed in the latter prior to its use in briquet manu'f. U. S. 2,025,777 relates to a process in which a combustion-controlling agent such as H<sub>2</sub>BO<sub>3</sub> or NaCl is dissolved in water and an emulsion prep'd. from this soln. and bitumen, with starch as an emulsifier, is used for wetting a mixt. of powd. fuel such as coal, sawdust, peat or lignite and hydraulic cement for making briquets (details and specified proportions of materials used being given).

9 Forming briquets of finely divided fuels Eugene Sanial (to Distillation a basse temperature et auto-

Use of Zn alloys for gas pipes (Cazaud, Petot) 9.

agglomeration des combustibles). U. S. 2,026,439, Dec. 31. App. and operative details are described.

Low-temperature carbonization. Alfred J. A. Herang Ger. 621,252, Nov. 6, 1935 (Cl. 100, 29). Finely subdivided fuel is conveyed on a band through a closed chamber in which it is heated sufficiently to yield a passy product. This product is coarsely granulated before it leaves the chamber. The granular product may be briquetted without addition of a binding agent. App. is described.

Electrical apparatus for testing the completeness of combustion of engine fuels, etc. Clarke C. Minter U. S. 2,025,121, Dec. 24. Various structural, elec. and operative details.

Combustion-control system for induced draft furnaces such as those burning coal. Albert G. Blodgett (to Riley Stoker Corp.). U. S. 2,026,159, Dec. 31. Various structural, tech. and operative details of a control system for fuel supply, etc.

Maintaining uniform combustion in engines such as those of automobiles. Wm. Harper, Jr. (to H. B. Motor Corp.). U. S. 2,025,205, Dec. 24. Successive charges of a compressed fuel mixt. are subjected to a plurality of turned interrupted sparks which vary oppositely in intensity with variations in temp. within the combustion space.

Destructive hydrogenation. International Hydrogenation Patents Co. Ltd. Fr. 787,520, Sept. 24, 1935. Carbonaceous materials are treated with hydrogenating gases at a high temp. in the presence of halogen or S compounds contg. metalloids which are solid in the free state at 18° or combined NH<sub>3</sub>. The compds. are added during or after the preparatory heating of the materials and are protected against decompos. until the moment they are incorporated with the materials by keeping the entry pipe for these compds. cool. Compds. used include CCl<sub>4</sub>, CHCl<sub>3</sub>, CH<sub>3</sub>I, PhBr, PhI, CBr<sub>4</sub>, SiCl<sub>4</sub>, SeI<sub>2</sub>, SeI<sub>4</sub>, AsCl<sub>3</sub>, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, S-Cl<sub>2</sub> and NH<sub>4</sub>Cl, and these may be used in conjunction with other catalysts, e.g., compds. of metals of the 6th group.

Hydrogenating coals and lignites. Compagnie des mines de Vaconne, Noeux & Drocourt. Fr. 787,211, Sept. 19, 1935. A complex material which contains the catalyst in a chemically fixed form is prep'd from coal or lignite and an appropriate catalyst (an alkali or alk. earth metal, or alloy or salt thereof), material which may be stocked without alteration and which is hydrogenated under the influence of heat and pressure in an atm. of H<sub>2</sub>. Thus, coal or lignite is heated with NaOH, dissolved in water or not, to about 250-330° and kept there for 15 min. and afterward hydrogenated.

Apparatus for separation of materials such as coal, etc., by the action of air currents. Mack E. Haworth (to Jeffrey Mfg. Co.). U. S. 2,026,633, Jan. 7. Various details of app. and operation.

Coal distillation to obtain low-boiling hydrocarbons. Georges F. Mochet-Dupont (to "Physical Chemistry Research Cr."). U. S. 2,025,882, Dec. 31. In coal distn., methyl groups are substituted for phenol groups by heating the coal to a temp. above that at which acetone decomposes and below about 600° in the presence of an alk. salt of an aliphatic acid such as Ca acetate adapted to supply the methyl groups, in the presence of an oxide of a metal of the Fe group serving as a catalyst. Cl. C. A. 29, 582<sup>1</sup>.

Horizontal retorts for distilling coal. Fours Fieard Fr. 787,581, Sept. 25, 1935. Means for injecting steam is described.

Apparatus for distilling or calcining coal, schist, peat or wood. Steffe Ltd. Fr. 786,114, Aug. 27, 1935.

Treating furnace flue dust. Ervin G. Bailey and Ralph M. Hardgrove (to Fuller Lehigh Co.). U. S. 2,024,197, Dec. 17. Dust sep'd. from combustion products is returned to the furnace, and the incombustible portion is melted by the heat of the furnace and the molten product is withdrawn. App. is described. U. S. 2,024,188 relates to details of furnace construction.

Lignite briquet plant. Emil Dehl Ger. 618,775,

Sept. 17, 1935 (Cl. 103, 9 06). A device for obtaining the lignite dust is described.

Furnace with rotating hearth for carbonizing lignites, etc. Louis Granger. Fr. 786,408, Sept. 3, 1935.

Horizontal chamber furnace for coke and gas. Compagnie générale de construction de fours. Fr. 786,035, Aug. 24, 1935.

Steam generator and compensating superheater. Charles W. Gordon (to Superheater Co.) U. S. 2,026,130, Dec. 31. Various structural details.

Catalytic deoxidation of exhaust gases from Diesel engines. Wilhelm Wenzel (to I. G. Farbenindustrie A.-G.) U. S. 2,025,140, Dec. 24. The gases, while still hot, are brought into contact with a catalyst comprising Cu or organic.

Combustible gas substantially free from nitrogen oxides. Wm. W. Odell U. S. 2,025,071, Dec. 24. A carburized lean gas is obtained by heating a mass of combined refractory solid material such as checker work substantially to incandescence, then causing hydrocarbon material and some steam to react in contact with the heated material to form a rich gas contg. free, active H<sub>2</sub>, immediately commingling the rich gas and a lean gas contg. only about 0.1% of a N oxide and bringing the mixt. into contact with another portion of the heated refractory material to effect a substantial reduction of the N oxide. Exhaust engine gases are used and app. is described.

Inert noncondensing gas. Jav E. Teme U. S. 2,025,364, Dec. 24. A mass of charcoal is ignited and partially burned in a primary combustion chamber (of a described app.) until smoke and gases are removed, and the burning charcoal is transferred to a secondary combustion chamber and while still hot, air is forced through the glowing charcoal to form an inert gas free from O and smoke; the inert gas is drawn off, sep'd. from solid impurities, cooled and collected.

Method of fractionating coke-oven gas and like mixtures by liquefaction. Ges. fr. Linde's Eisenwerke A.-G. Ger. 621,453, Nov. 7, 1935 (Cl. 12, 1 01).

Cleaning gases. Gutehoffmühlwerke Oberhausen A.-G. Ger. 615,957, Sept. 19, 1935 (Cl. 183, 14 03). Soot deposited in solid form from the waste gases of furnaces heated by coal dust by subjecting the hot waste gases to a current of cool air.

Washing gases. N. V. Maschinenfabriek en apparaten fabriek "Meef". Fr. 786,431, Sept. 3, 1935. Light hydrocarbons and C<sub>2</sub>H<sub>4</sub> are ex'd. simultaneously by washing coke or lighting gas with strongly cooled (below 0°) washing oil. The hydrocarbons are sep'd. from the oil in the usual manner and the C<sub>2</sub>H<sub>4</sub> by strong cooling and crys't.

Purifying producer gas. Raphael Malhot. Fr. 787,632, Sept. 25, 1935. Long removable brushes made of metal wires, preferably in the form of a helix, are placed in the pipes leading from the producer so as to remove solid particles.

Freeing gases from sulfur compounds. Walther Saxner (to Gas Technik G. m. b. H.) U. S. 2,024,836, Dec. 17. A gas such as coke-oven gas is passed through a plurality of layers, arranged in series, of molded bodies of a S-absorbing mass such as may contain Fe(OH)<sub>3</sub> in which the absorbed S is set free by oxidation to regenerate the mass (the first layer traversed by the gas being regenerated more frequently than the second layer). Cl. C. A. 29, 4109<sup>1</sup>.

Coal-gas and water-gas generator. Carl Elver. Ger. 621,141, Nov. 2, 1935 (Cl. 24, 2 01).

Generator for making fuel gas from wood or other fuel containing water. Alois Jellik Austrian 143,660, Nov. 25, 1935 (Cl. 246). The generator is particularly designed for the supply of tar-free fuel gas to motors subjected to widely varying loads.

Rotary-tube feed device for gas generator. Wehlerwerk A.-G. Ger. 621,233, Nov. 3, 1935 (Cl. 246, 9).

Treatment of tars. Thermal Industrial & Chemical (T. I. C.) Research Co. Ltd. Fr. 787,511, Sept. 24, 1935. See Br. 432,998 (C. A. 30, 844<sup>1</sup>).

Coke. Octave C. M. Dozy-Hemault. Fr. 787,623,

Sept. 26, 1935 Coke is purified by treating it after leaving the furnace and while still hot with appropriate gaseous reagents, particularly  $\text{Cl}$ ,  $\text{HCl}$  and  $\text{HF}$ . Thus, a mixt. of  $\text{Cl}$  and  $\text{HCl}$  removes  $\text{S}$ ,  $\text{Fe}$  and  $\text{Al}$ , and  $\text{HF}$  removes  $\text{SiO}_2$  as  $\text{H}_2\text{SiF}_6$  at a lower temp

Coking coal Carl Stoll G m b H Ger 618,903,

Sept. 18, 1935 (Cl 10a 14). A device is described for compressing loose coal in a coke-oven chamber.

Coke oven Louis Wulputte. U. S. 2,025,967, Dec 31 Various structural details

Compound regenerative coke oven Wilhelm Muller Ger 618,969, Sept 19, 1935 (Cl 10a 504)

## 22—PETROLEUM, LUBRICANTS, ASPHALT AND WOOD PRODUCTS

W F FARACHER

Official instructions for the distillation test of petroleum products Anon *Ann fals* 28, 555-62 (1935) —Text of the Fr official method is given A. P.-C.

Viscosity of petroleum products Viscosity-temperature characteristics of Pennsylvania lubricating oils R E Hersh, E K Fisher and M R Fenske. *Ind. Eng. Chem* 27, 1441-6 (1935), cf *C A* 27, 534; 29, 1971 —The viscosities of a large no of Penna oils were detd at 100°F and 210°F. in modified Ostwald viscometers Equations are given for the relationship between the kinematic viscosities at 100°F and at 210°F for Penna and for naphthenic oils Viscosity-index tables, expressed in centistokes, are included It is recommended that this method be called kinematic viscosity index Marion E Headington

Viscosity-temperature relationship of Japanese petroleum oils II Takewo Kurosawa *J Soc Chem. Ind., Japan* 38, Suppl bunding 532-4 (1935), cf *C. A.* 29, 8307 —A Redwood viscosity table for Japanese petroleum oils is presented for a viscosity range 30-3000 and temps of 10°, 20°, 30°, 50°, 80°, 100° and 150°. The values were calcd from previously reported viscosity ratios Karl Kammermeyer

Petroleum solvents and naphthas E M Tobv. *Rubber Age (N. Y.)* 38, 85-8, 90 (1935), cf *C. A.* 29, 4851 —A discussion of the properties and uses of various petroleum solvents. C. C. Davis

The refining of mineral oils by selective solvents Marcel Gheorghiu *Mon pétrole roumain* 36, No 24, 1779-81 (1935) J R Strong

Oil sands as well as lime formations respond to proper acid treatment F B Plummer and R B Newcome, Jr. *Oil and Gas J* 34, No 23, 44-6 (1935); cf *C. A.* 30, 843 J R Strong

Oxidation of white oils Ralph W Dorate *Ind Eng Chem* 28, 26-30 (1936) —D carried out lab expts on the oxidation of white oils to det the chem mechanism involved One procedure consists in measuring the rate of absorption of oxygen at const pressure by a definite wt. of oil and the other in the withdrawal of samples for analysis during the reaction The rate of absorption of oxygen by moderately and highly refined oils is found to be a method of detg the resistance to oxidation E. W. Roth

Thermal polymerization process used by pure oil on olefin bearing gases. M B Cooke, H R. Swanson and C R. Wagner. *Oil and Gas J* 34, No 26, 57, 59, 60, 62 (1935). —The app used and the stocks suitable for polymerization are described The thermal polymerization process is described at length, with the actual operation procedure included J R Strong

Treating crude-oil emulsions in the Semmole area routine operation Harold K. Ihng *Oil and Gas J* 34, No 25, 49, 51, 53 (1935) J R Strong

Production-decline curve of oil well may be extrapolated by loss ratio Salvaun J. Pirson. *Oil and Gas J* 34, No 26, 34-5 (1935) —P. gives a math. justification of the loss rating method for extrapolating oil-well decline curves J. R. Strong

Refining of synthetic acids V. Varlamov, G. Oyat'eva and N. Davudova. *Moskolin Zhivoe Delo* 11, 494-5 (1935); cf *C. A.* 29, 7629 —Oxycids obtained by oxidation of petrolatum jells were refined by autoclaving 15% soln of acids with 10% excess of KOH in  $\text{H}_2\text{O}$ . (to prevent polymerization) at 180-200° for 1 hr. and at 290-3° for 43 min Similar dehydration with the

formation of unsatd acids was effected by autoclaving 1  $\text{C}_6\text{F}_6$  Ca soap in  $\text{C}_6\text{H}_6$  with stirring, at 250-300° for 1 hr. and 25 min Chas Blanc

New True-Vapor-Phase control unit uses tubular heater instead of original "stoves" Neil Williams *Oil and Gas J* 34, No 25, 38-9 (1935) —Description of a modern True-Vapor-Phase cracking unit. J. R. S

Viscosity and stability of drilling fluid C. W. Woolgar. *J. Inst Petroleum Tech* 21, 825-37 (1935) —The density of a drilling fluid at the bottom of the column proved in tests to be almost independent of the height of the column, and is not as important as the yield-point value This factor governs the stability of the mud, its ability to hold the cuttings and weighting material in suspension, its suitability for pumping and its sealing off and plastering effects It may be measured either by a shearometer or from the viscosity at 600 r. p. m. measured in a Stormer viscometer. E. E. Crandal

Hydrogenation of bituminous schists under ordinary pressure Jean Barlot *Compt rend* 201, 1137-8 (1935), cf *C. A.* 29, 9158 —On distg oil shale in a current of  $\text{H}_2$  or  $\text{CO} + \text{H}_2$  contg at least 60%  $\text{H}_2$ , at ordinary pressure and without previous drying or purification, at 500-600°, the yield of crude oil is increased by 5-10% This oil distils almost entirely (0.5-1.5% pitch) below 350° and contains 25-30% unsatd hydrocarbons With treated, Esthonian kukkerite, schists from Autun, Creveney and Chilly yielded, resp. 23, 13, 6.5 and 5% of crude oil, contg 0.34, 0.18, 1.45 and 0.40% of S Most of the S passed off as  $\text{H}_2\text{S}$  C. A. Silberrad

Coal hydrogenation gasoline plant at Billingham Anon *Engineering* 140, 440-2 (1935). E. H

Copper is economical for sweetening refinery and natural gasolines W. A. Schulze and A. E. Buell *Oil and Gas J* 34, No 22, 42, 44, 46 (1935) —Gasoline can be sweetened by the use of Cu salts The Cu salts oxidize the mercaptans to disulfides and are themselves reduced to the cuprous compounds (1)  $4\text{RSH} + 2\text{CuCl}_2 \rightarrow \text{RS-SR} + 2\text{RSCu} + 4\text{HCl}$ , (2)  $2\text{RSCu} + 2\text{CuCl}_2 \rightarrow \text{RS-SR} + 4\text{CuCl}$  (the  $\text{CuCl}$  can be reconverted to  $\text{CuCl}_2$  by  $\text{O}_2$  and  $\text{HCl}$ ), (3)  $4\text{CuCl} + 4\text{HCl} + \text{O}_2 \rightarrow 4\text{CuCl}_2 + 2\text{H}_2\text{O}$ , combining the above reaction gives (4)  $4\text{RSH} + \text{O}_2 \rightarrow 2\text{RS-SR} + 2\text{H}_2\text{O}$   $\text{CuCl}$  can be reconverted to  $\text{CuCl}_2$  simultaneously with the sweetening operation Some cracked gasolines discolor after Cu salts are used, but can be rendered color-stable if the sweetening operation is followed by a Na-S soln-wash. S and B. claim the following advantages over plumbite (1) saving in chem. costs, (2) no loss in gasoline, (3) no loss in octane no, (4) retention of natural gum inhibitors; and (5) quality of product. J. R. Strong

The "allowable boost ratio" G. D. Boerlage, L. A. Peletier and J. L. Tops. *Aircraft Eng.* 7, 306-8 (1935) —Allowable boost ratio is defined as boost pressure on sample at incipient knock, divided by boost pressure on standard fuel at incipient knock The authors prefer this test method for aviation fuels to the antiknock rating in octane nos M. W. Schwarz

Production of lubricating oils Separation of solvents and water in refining and dewaxing oils Eugene R. Smoley and Wheaton W. Kraft. *Ind. Eng Chem* 27, 1418-22 (1935) —A brief outline is given of solvent refining and dewaxing processes, used in the production of lubricating oils The importance of sepg solvent and water is indicated and the various methods of sepg them are discussed Marion E Headington

**Chemical investigation of mineral lubricating oils.** The action of chlorine and bromine on a medicinal mineral oil Emile André *Ann Combustibles Liquides* 10, 807-17 (1935) —A Prussian medicinal oil was chlorinated or brominated in  $\text{CHCl}_3$  soln., the products contg 8.5% Cl, and 11.0% Br, resp. The halogens were detd by igniting with a large excess of  $\text{CaO}$  G Calingaert

**Selection of lubricants for cold rolling of strip steel.** R. W. Moore *Iron Steel Eng* 12, 21-b (Dec., 1935) —A discussion of lubricants used in connection with the rolling of steel strip through finishing mills during the final stages of processing. The primary purposes of a roll oil are (1) to minimize friction between the strip and the work rolls of the mill, (2) to furnish a protective coating on the strip to prevent scratching when the surfaces of the metal are in contact, and (3) to resist atm. or other conditions to which the finished strip may be subjected prior to further processing. Vegetable oils, mineral oils and compounded oils are employed. Mineral oils are being used more and more. Those with viscosities of 100-200 sec at 100°F (37.7°C) are most widely used. Characteristics to be considered in the selection of the roll oil are (1) film strength, (2) thermal properties, (3) ease of application, (4) ease of cleaning, (5) C deposits, (6) clumping characteristics, (7) adhesiveness, (8) ability to spread and (9) economy. Each characteristic is discussed W. H. Boynton

**Use of tin compounds in lubricants.** P. C. Barbosa *Rev. chim ind (Rio de Janeiro)* 1935, 404-5 —Metallic Sn and Sn compounds, especially  $\text{SnO}_2$ , tin tetraethyl and tin ricinoleate in amts. up to 1% act as retarders of oxidation and sludge formation in lubricating oils. Ca ricinoleate is an aging accelerator O. W. Wilcox

**An attempt to raise the acetone yield from wood.** Hilding Bergström and K. N. Cedergaard *Ind* 1935, 76-8. —In dry distn. of wood, a layer of marble in pieces placed over the wood chips in the retort did not improve the acetone yield, the marble became coated with C.

**Determination of resinous matter in pyrogenous powder.** V. P. Zemlyanitsin *Zavodskaya Lab* 4, 877-8 (1935) —To det. resinous matter in pyrogenous powder in the production of  $\text{AcOH}$ , dissolve a 5-g. sample in 50 cc. of hot water, add 50 cc. of concd  $\text{HCl}$  and 1.5 g. of powdered activated C, and boil the mixt. for a few min. Filter through a weighed Gooch crucible, wash the ppt. with hot water free from  $\text{AcOH}$  and Ca salts, dry in a steam-heated drying oven to a const. wt., weigh, ignite and weigh again. The difference between the wt. of ppt. and the combined wt. of ash and C gives the wt. of resinous matter. The corrections for the ash content of activated C and its loss in wt. on boiling with  $\text{H}_2\text{O}$  and  $\text{HCl}$  must be introduced. The latter is obtained by drying the C at 100° and boiling with  $\text{H}_2\text{O}$  and  $\text{HCl}$  as described above, followed by drying and weighing. The detn. can also be made by using a tared filter paper, which, together with the ppt., is dried to a const. wt., ignited and weighed. Chas. Blanc

**Petroleum geology of Gondwana rocks of Southern Brazil (Oppenheim).** 8. Gas-woks lubrication (Walker) 21. App. for measuring the heat cond. of oils. (Lederer) 1. Use of wood tar from gas generators for the prepn. of plastic compds. (Losev, et al.) 13. Disposal of oil-field brines in the Ritz-Canton oil field, McPherson County, Kansas (Wilhelm, Schmidt) 14. Hydrogenation of tertiary alkyl phenols to form hydroaromatic alks [use as lubricants for textile fibers] (U. S. pat. 2,026,668) 10. Oxygenated org. compds. from hydrocarbons (U. S. pat. 2,024,954) 10. Exhaust steam condenser and oil separator (U. S. pat. 2,025,043) 1.  $\text{NH}_4$  sulfate [produced from sludge from refining hydrocarbons] (U. S. pat. 2,026,250) 18. App. for proportioning the flow of liquids as in refining petroleum oils (U. S. pat. 2,024,478-9-80) 1. Aminoanthraquinone compds. [for petroleum fractions] (Brit. pat. 434,906) 25. App. for distg. or calcining schist, peat or wood (Fr. pat. 780,114) 21. App. for lifting oil from wells (U. S. pat. 2,026,226) 1.

1 App. for sampling or visual examn. of oils (U. S. pat. 2,026,267) 1.

**Petrolatum refining.** Harry T. Bennett, Harris H. Hopkins and Jerry R. Marshall (to Mid-Continent Petroleum Corp.) U. S. 2,026,492, Dec. 31. U. Nitrate and anhyd.  $\text{AlCl}_3$  are added to the petrolatum to be treated, followed by agitation while heated and sepn. of resulting sludge.

2 **Breaking petroleum emulsions.** Claudius H. M. Roberts (to Tretolite Co.) U. S. 2,023,976, Dec. 10. Neutral phthalic olefin is used as a demulsifying agent.

**Breaking petroleum emulsions.** Melvin De Groot, Bernhard Keiser and Arthur F. Wirtel (to Tretolite Co.) U. S. 2,025,804, Dec. 31. A demulsifying agent is used comprising a poly keto fatty acid compd. such as may be derived by pressure oxidation of castor oil, suitably with linseed oil,  $\text{MeOH}$  and solvent naphtha. U. S. 2,025,805 relates to similar use of a keto fatty acid material, such as may be derived from castor oil, suitably with  $\text{C}_6\text{H}_6$  and  $\text{LiOH}$ .

**Breaking petroleum emulsions.** Charles N. Stehr (to Tretolite Co.) U. S. 2,026,195, Dec. 31. A demulsifying agent is used consisting of an anhyd. mixt., oil-sol. at least in high concns., comprising blown oil compds., such as those from castor oil 15-50, one or more acid salts of a sulfo fat acid such as an acid Na salt from sulfonated castor oil 2-15, an unsaponified S-free fat acid derived from sulfation of material such as castor oil followed by hydrolytic decompn. with splitting of sulfuric acid 20-60, and an aliphatic monohydric alc. such as iso-Pr alc. 2-25%.

**Breaking petroleum emulsions of the water-in-oil type.** Charles N. Stehr (to Tretolite Co.) U. S. 2,023,979, Dec. 10. A demulsifying agent is used which is produced by the super-oxidation of blown castor oil by the action of air at 200° and with continuation of the oxidation until the product is "semi-liquid" in consistency when freshly prepd., the oxidation being stopped just short of the point of kerosene-invol. U. S. 2,023,980 relates to the use of a demulsifying agent derived by the conventional oxidation (as by the action of air while heated) of a mixt. of castor oil and at least one semi-drying oil such as rapeseed oil in which the castor oil constitutes 40-80% of the mixt. U. S. 2,023,981 relates to the similar use of an oxidation product similarly derived from a mixt. of castor oil and sardine oil. U. S. 2,023,982 relates to like use of a demulsifying agent comprising blown castor oil or the like and dehydrated ricinoleic acid products such as may be formed by heating ricinoleic acid to slightly above 100°. Triethanolamine may be used as an acid neutralizing agent. Cf. C. A. 29, 7634 and following abstr.

**Breaking petroleum emulsions of the water-in-oil type.** Melvin De Groot and Bernhard Keiser (to Tretolite Co.) U. S. 2,023,993, Dec. 10. Demulsification is effected by use of dehydration products of ricinoleic acid of specified character such as may be produced by heating ricinoleic acid to above 100° and which are neutralized with a substituted basic amine such as triethanolamine and did. with a pine wood intermediate oil. U. S. 2,023,994 (Melvin De Groot and Arthur F. Wirtel (to Tretolite Co.)) A mixed demulsifying agent is used comprising dehydration products of ricinoleic acid such as specified in U. S. 2,023,993 in admixt. with blown fatty material such as blown castor oil and alkylated naphthalenesulfonic acid compds. in specified proportions. U. S. 2,023,995 (Melvin De Groot and Bernhard Keiser (to Tretolite Co.)) relates to the use of a demulsifying agent comprising an ester derived from a noncyclic nonfatty dibasic carboxy acid of the type  $(\text{CH}_2)_n(\text{COOH})_2$ , in which  $n$  is at least 5 and not more than 8, or castor oil or the like and an hydroxylated fatty acid compd. such as sebacic acid. U. S. 2,023,996 relates to similar use of a demulsifying agent comprising an hydroxyhendecenoic acid material of the formula type  $\text{XTCOOZ}$ , in which  $\text{X}$  is at least one O atom or at least one hydroxyl radical, or an acid residue,  $\text{T}$  is a hendecenoic acid residue and  $\text{Z}$  is an acidic H equivalent.

lent U S 2,023,997 relates to like use of an hydroxy-octadeca-9,11-dienonic acid material of the formula type XTCOOZ, in which X is at least one O atom or at least one hydroxyl radical or acid residue, T is an octadeca-9,11-dienonic acid residue and Z is an acidic H equivalent. U S 2,026,217, Dec. 31. Melvin De Groote and Bernhard Keiser (to Tretolite Co.) A demulsifying agent is used contg a hydroxylated, nonsulfo fatty acid compd of the type (R'TOH)COOZ, in which R' is a fatty acid hydrocarbon residue derived from a fatty acid or its glyceride, T is an aromatic residue attached directly to R', OH is a hydroxyl attached directly to T, COO is the carboxyl residue and Z is a "hydrogen-ion equivalent," such as the triethanolamine salt of phenol stearic acid. U S 2,026,218 relates to the use of a demulsifying agent comprising a sulfo compd derived by sulfonation of an unsatd oxy fatty material, such as a salt or ester of sulfobutenecenoic acid. U S 2,026,219 relates to the use of a demulsifying agent comprising a sulfite addn product derived by reaction between a water-sol sulfite such as NaHSO<sub>3</sub> and an unsatd reactive fatty material such as linseed oil.

Oxidized product of hydroxylated fat acids suitable for demulsifying water-in-oil petroleum emulsions. Melvin De Groote and Bernhard Keiser (to Tretolite Co.) U S 2,025,803, Dec. 31. Water insol hydroxylated fat acids obtained by the hydrolytic decompn of linseed oil type fat acid sulfates are blown with air or O until substantial oxidation takes place (suitably for several hrs at a temp of about 150°).

Producing steam for use in petroleum oil refining. Edward H. Clendenn (to Standard Oil Development Co.) U S 2,025,724, Dec. 31. Various features of app and operation are described.

Use of sulfur dioxide for extraction of petroleum oils. John T. Rutherford (to Standard Oil Co. of Calif.) U S 2,024,476, Dec. 17. Concd exts of unsatd. and aromatic constituents are obtained by extg a petroleum oil, such as a topped crude oil pressure naphtha, with SO<sub>2</sub>, the extd. oil is sepd. from the SO<sub>2</sub> and the ext. being admixed with a petroleum oil which has been denuded of the greater part of its unsatd. and aromatic compds., and the mixed oils are subjected to a second extn. with SO<sub>2</sub>.

Ammonium sulfate and denaturant from acid sludges produced in petroleum refining. John T. Rutherford (to Standard Oil Co. of Calif.) U S 2,025,401, Dec. 24. A sludge from petroleum oil refining is hydrolyzed with about 4 times its quantity of a mother liquor contg (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub> to sep. the material into an acid oil suitable for use as a denaturant after neutralization and distn. and the aq. phase resulting from the hydrolysis, after sepg. the acid oils, is treated with NH<sub>3</sub> for the production and pptn. of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and the mother liquor remaining after sepn. of the (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> is returned to the first step of the process. An arrangement of app is described.

Pour-point-reducing material from petroleum residue. Harry Levin (to Texas Co.) U S 2,024,106, Dec. 10. A cracking still residue is cracked and extd. with a solvent comprising a mineral-oil fraction of higher-boiling range than petroleum naphtha, such as a lubricating oil, to dissolve the desired constituents, the soln. thus formed is sepd. and ddd. with petroleum naphtha to ppt. the desired constituents in solid form, and the ppt. is removed. The product imparts pour-point-depressing and color-imparting properties to mineral lubricating oils.

Cracking hydrocarbon oils. Carbon F. Dubbs (to Universal Oil Products Co.) U S 2,025,500, Dec. 24. Oil is heated to a cracking temp. under pressure in a heating zone and thence discharged into a vapor-sepg. zone maintained under superatm. pressure, whence vapors and unvaporized oil are separately removed; the vapors are dephlegmated in contact with charging oil and reflux condensate, and the removed unvaporized oil from the sepg. zone is combined with a sufficient quantity of the mixt. of charging oil and reflux condensate substantially to check the cracking reaction in the unvaporized oil to

prevent C formation, and the admixed oils are introduced into a flashing zone maintained under a lower pressure than the sepg. zone and vapors from which are dephlegmated to condense heavier fractions which are fed to the heating zone. App. is described. Application for this patent was filed Jan. 17, 1927. U S 2,025,501 (issued on an application filed Aug. 15, 1929) relates to a somewhat similar process in which incondensable gases sepd. from dephlegmated vapors are heated by indirect heat exchange with vapors prior to their dephlegmation and are passed into heat-exchange relation with a reflux condensate to reboil it, the reflux condensate is combined with such condensate as may be formed by the heat exchange between the gases and vapors. App. and various other operative details are described. Cf. C. A. 30, 848<sup>2</sup>.

Cracking hydrocarbon oils. Edward W. Isom (to Sinclair Refining Co.) U S 2,026,231, Dec. 31. Fresh oil such as fuel oil or gas oil is passed in a single pass through a heating zone such as a heated pipe coil or the like and thence into an enlarged vapor-sepg. zone, after being heated to a cracking temp. under pressure. The vapor-sepg. zone is maintained at a temp. and pressure sufficient to permit partial vaporization only and vapors are taken off and subjected to a 1st and 2nd refluxing, fresh oil is introduced into direct contact with the vapors in the 2nd refluxing and resulting reflux and admixed fresh oil are passed in a single pass through a sepg. heating zone and thence into the vapor-sepg. zone. Fresh oil being supplied to the 1st-mentioned heating zone is passed in indirect heat-exchange relation with the vapors in the 1st refluxing operation, and the oil in the 2nd heating zone is subjected to a vapor-phase cracking under a pressure lower than that maintained in the 1st-mentioned heating zone. App. is described.

Cracking hydrocarbon oils. Douglas P. Bailey (to Texas Co.) U S 2,026,505, Dec. 31. A condensate oil is heated to a high cracking temp. while passing through a heating coil and is thence passed into an enlarged cracking zone and vapor separator into which oil of a lower temp. is also introduced so as to subject the cooler oil to cracking; sepd. liquid is withdrawn from the enlarged zone at a rate sufficient to prevent accumulation, and sepd. vapors are passed to a secondary cracking zone for further cracking and from which liquid is also withdrawn to avoid accumulation and conducted to the enlarged cracking zone, vapors being passed to a fractionation zone for sepn. of a final light distillate and a reflux condensate which is passed to the heating coil. App. is described.

Cracking and coking hydrocarbon oils. Luis de Florez (to Texas Co.) U S 2,024,996, Dec. 17. A process for converting oil into a light distillate, heavy fuel oil and coke comprises heating the oil to a cracking temp. while passing it through a heating coil, introducing a portion of the stream of cracked products from the coil into a coking chamber, directly admixing a residual oil from a source extraneous to the system with the portion of the stream being introduced into the coking chamber, passing vapors from the coking chamber at a cracking temp. into a dephlegmating zone, introducing a stream of fresh charging stock into the dephlegmating zone for subjection to a cracking temp. by heat exchange with the vapors, introducing a further portion of the hot cracked products from the heating coil directly into the dephlegmating zone, withdrawing liquid from the dephlegmating zone as a heavy fuel-oil product, fractionating vapors from the dephlegmating zone to produce a condensate heavier than gasoline and passing condensate back to the heating coil, and condensing final light vapors from the fractionating zone. App. and various operative details are described.

Separating olefins from gaseous mixtures such as those from oil cracking. Harry T. Bennett (to Mid Continent Petroleum Corp.) U S 2,026,265, Dec. 31. Dichloroethyl ether is used as a selective solvent for the olefins.

Apparatus for cracking hydrocarbon oils in the vapor phase. I. Morris Levine (to Texas Pacific Coal & Oil Co.) U S 2,024,828, Dec. 17. Various details are described of an app. in which a chamber for the oil vapors

to be cracked is heated by gases passing around the chamber and through tubes extending through it.

Furnace and heating-tube system suitable for heating oils for cracking or distillation John C Black U S 2,026,120, Dec 31. Structural details

Return-bend fitting suitable for oil-cracking and refining apparatus John F Getzen (to Fred C Fantz) U S 2,024,572, Dec 17. Structural details

Return-bend fitting for high-pressure oil-cracking apparatus Herbert C Stockham (to Fred C Fantz) U S 2,021,399, Dec 17. Structural details

Return-bend fitting for oil-refining apparatus Fred C Fantz U S 2,024,347, Dec 17. Various structural details

Return-bend fitting for oil-still apparatus, etc. Samuel H Edwards (to Standard Oil Co of Calif) U S 2,026,674, Jan 7. Structural details

Refining hydrocarbon oils N V de Bataafsche Petroleum Maatschappij Fr 780,175, Aug 28, 1935 Nonparaffinic hydrocarbons are removed from oils by an extn with a mixt of 2 furalsdehyde (60) and  $C_2H_5$  (40%)

Refining hydrocarbon distillates Richard F Davis (to Universal Oil Products Co) U S 2,021,681, Dec 17. A distillate such as a cracked gasoline contg unsatd hydrocarbons and S compds is treated with  $SO_2$  in the presence of  $AlCl_3$  (suitably in the vapor phase) App is described

Hydrocarbon oil conversion Jean D Seguy (to Universal Oil Products Co) U S 2,025,532, Dec 24. Oil is subjected to conversion conditions of heat and superatm pressure in a heating coil and reaction chamber and then discharged into a reduced pressure chamber where vapors sep from non-vaporizable residue, the vapors are subjected to fractionation followed by condensation and collection of resulting distillate and uncondensed products, and the latter, following their sep from the distillate, are subjected to liquefaction to effect the removal of their higher-boiling constituents, and the recovered liquid is subjected to further conversion at a higher temp than that employed in the first-mentioned heating coil and the heated liquid is introduced into the reduced pressure chamber. App is described Cf C A 29,3507

Fractionating hydrocarbon oils. N. V. de Bataafsche Petroleum Maatschappij. Fr 780,038, Aug. 24, 1935. An oil is introduced wholly or partly in the liquid or vapor state into one or more fractionating columns, preferably almost midway between the top and bottom, and one or more fractions are withdrawn as vapor between the bottom and the point of admission and submitted in a sep zone to a partial condensation, while the heavier constituents formed during the condensation are returned to the 1st column, preferably at the same place from which the vapor is withdrawn from the column or near this place

Heating coils such as those used for hydrocarbon oil treatment Percival C. Keith, Jr (to Gasoline Products Co.) U. S. 2,025,667, Dec. 24. For increasing the heat-reflective capacity of selected parts of a tubular heating coil exposed to radiant heat, the selected parts are painted with a paint contg. a white pigment such as lithopone or  $CaO$  capable of being subjected to normal furnace conditions without material discoloration and a vaporizable vehicle such as water contg. Na silicate and the coil is heated to vaporize the paint vehicle and fix the pigment on the coil surface as a relatively thin adherent layer. App is described

Refining mineral oils. Bernhard R. Carney and Ralph H. Crosby (to Shell Development Co.) U. S. 2,026,213, Dec. 31. See Can. 350,629 (C. A. 29, 62609)

Distilling acid-treated mineral oils Brian Mead (to Standard Oil Development Co.) U. S. 2,025,766, Dec. 31. A sufficient quantity of an oil-sol. soap such as Na naphthenate is added to the oil practically completely to take up any acid-reacting substance evolved from any residue of the treating acid during the distn. of the oil and the distn. is effected in the presence of this added soap. An arrangement of app. is described

Separating naphthenic and paraffinic constituents of mineral oils James M. Whiteley, Jr (to Standard Oil De-

velopment Co.). U. S. 2,025,965, Dec. 31. The oil is extd with phenol in the presence of an added liquid (such as liquefied  $C_2H_6$  and  $C_3H_8$ ) having greater solvent power for paraffinic constituents than for naphthenic constituents

Increasing the oil production of oil wells Wm V. Vietti and Allen D. Garrison (to Texas Co.). U. S. 2,024,119, Dec 10 For increasing the proportion of oil and decreasing the proportion of water produced from an oil well, there is deposited in a water-wet sand adjacent the well a chemical ppt. such as a Ca salt of sulfonic sludge acid which acts to render the sand less permeable to water without substantially retarding the flow of oil to the well.

Treating oil or gas wells to increase production Leonard C Chamberlain (to Dow Chemical Co) U. S. 2,024,718, Dec 17 There is introduced into the well a water-sol agent such as EtOH capable of substantially lowering the surface tension of water and an acid such as HCl capable of forming water-sol salts with the earth formation

Treating mineral absorbent material such as that used in oil refining Geo R Lewers (to Nichols Engineering and Research Corp) U S 2,024,589, Dec 17. Various structural and operative details of a multiple hearth furnace are described

Purifying oils, waxes and synthetic resins Standard Oil Development Co. Brit 431,072, Sept. 6, 1935.

Heavy hydrocarbon oil, e. g., lubricating oils, resins, e. g., those obtained by polymerizing unsatd hydrocarbons or by condensing unsatd hydrocarbons with aromatic hydrocarbons, etc., are treated with light hydrocarbon solvents at within 80° F of the crit temp of the solvent, whereby the oil, resin, etc., is sep into 2 layers which are removed and oil, resin, etc., recovered therefrom. App. is described. In 434,714, Sept. 6, 1935, divided on 434,672, the oil, resin, etc., is treated with a light hydrocarbon solvent and sep. into layers contg. heavy and light fractions and the heavy fraction is further treated with the solvent at a lower temp. App. is described. In modifications of the process, the extn is effected in (1) sep stages of decreasing temp, in which the heavier fraction proceeds from 1 stage to the next and the lighter fraction is drawn off at each stage, or (2) sep. stages of increasing temp, the lighter fraction proceeding from 1 stage to the next in 1 direction and the heavier fraction in the opposite direction

Flange oil. Harry T. Bennett (to Mid-Continent Petroleum Corp.). U. S. 2,026,491, Dec. 31. A flange oil having a Furol viscosity between 200 and 375 comprises 50-25% of an adhesive residuum from a fractional distn of petroleum and having a Furol viscosity between 10,000 and 35,000 at a temp. of 50° and 50-75% of an adhesive residuum resulting from a cracking operation and having a Furol viscosity between 10 and 125 at 50°

Antiknock fuels. International Hydrogenation Patents Co. Ltd. Fr. 780,145, Aug. 27, 1935. Middle oils from any source are treated by H under pressure and in the presence of catalysts having an intense hydrogenating action and submitting, without the addn. of H, the fractions obtained (after removal of fractions b below 100° or below 150°) to a heating under conditions of temp, pressure and duration that a product is obtained poorer in H but without appreciable change of the mol. wt. of the whole of the final products. In an example a temp. of 420° and pressure of 200 atm. is used in the 1st step, and 500-520° and 80-100° in the 2nd

Antiknock fuel. The Pure Oil Co. Fr. 780,207, Aug. 29, 1935. Gases contg. olefins in a relatively dil. state are brought into contact with a solvent which easily dissolves the olefinic hydrocarbons but not the H and  $CH_4$ . The dissolved gases are sep. from the solvent, compressed and heated to polymerization temp., allowing a large part of the olefins to be converted to liquid fuel which is sep. from gaseous constituents.

Antiknock fuel. N. V. Nieuwe Oetropi Maatschappij. Fr. 787,664, Sept. 25, 1935. Crude oil is fractionated to give a light fraction from direct distn., a fraction of naphtha and a heavier fraction than the naphtha fraction.

The 1st fraction is passed to a sep. condenser. The 2nd fraction is vaporized, superheated and mixed with a gas serving as heat carrier in a reaction zone. The conversion products are brought into contact with a washing fluid to ext. the tar and C and part of their heat. The 3rd fraction is brought into contact with the reaction products in the washing operation. App. is illustrated.

Cracked motor fuel. Wm M. Stratford (to Texas Co.). U. S. 2,024,117, Dec. 10. Cracked petroleum naphtha is extd. with a mixt. of furfural and water proportioned to remove about 10% by volume of the original naphtha as an ext. contg. gum-forming constituents and undesirable S compds. without substantial impairment of the antiknock properties of the naphtha.

Stabilizing cracked motor fuel. Frederick B. Downing and Herbert W. Walker (to E. I. du Pont de Nemours & Co.). U. S. 2,025,192, Dec. 24. About 0.001-1.0% of a dihydronaphthalene is added to inhibit gum formation. Cf. C. A. 30, 279.

Refining cracked gasoline. John C. Black (to Gasoline Products Co.). U. S. 2,025,409, Dec. 24. The material is subjected, in vapor phase, to partial condensation to produce a 1st condensate requiring chem. treatment and a 2nd vapor which requires no chem. treatment and which represents at least 20% of the mixt. being treated, the 2nd vapor is sep'd. into liquid products substantially free of normally gaseous hydrocarbons and into a gas, the first condensate is chemically treated while in substantially the same conditions as when sep'd. and the treated condensate is blended with the liquid products sep'd. from the 2nd vapor. An arrangement of app. is described.

Refining a sulfur-containing cracked gasoline distillate. Jack F. H. Taylor and Harold I. Lounsbury (to Shell Development Co.). U. S. 2,025,255, Dec. 24. The distillate is divided into a series of fractions of successively higher b. p., one of the limits of one of the b. p. ranges being so selected as substantially to correspond with a b. p. range limit of one of the sulfurous fractions in which S-bearing substances are conc'd. as the result of cracking and so as to include the sulfurous fraction in the oil fraction of corresponding b. p. range. An arrangement of app. is described.

Filter suitable for gasoline. Jean Zwicky (to Zwicky Ltd.). U. S. 2,024,753, Dec. 17. Structural, mech. and operative details.

Storage tank suitable for holding gasoline, etc. James O. Jackson. U. S. 2,026,279, Dec. 31. Structural features.

Oil or gasoline storage tank with a floating roof. Harry L. Babcock (to Graver Tank & Mfg. Co.). U. S. 2,024,327, Dec. 17. Various structural details.

Dewaxing hydrocarbon oils. Bernhard Y. McCarty and Wm. E. Skelton (to Texas Co.). U. S. 2,024,107, Dec. 10. A wax-bearing oil such as a lubricating-oil fraction is mixed with a selective solvent composed of o-dichlorobenzene and an aliphatic ketone, such as acetone or methyl ethyl ketone, having wax antisolvent properties such as those of acetone in such proportions that at temps. of about -22° the mixt. has substantially complete solvent action on the oil but substantially no solvent action on the wax; the mixt. is chilled to ppt. the wax, the wax is sep'd. and the solvent is removed from the dewaxed oil. Cf. C. A. 29, 3149.

Removing wax from oil. Robert E. Wilson (to Standard Oil Co. of Ind.). U. S. 2,026,336, Dec. 31. Waxy oil diss. with a normally gaseous diluent such as liquefied propane is cooled in a closed chll. chamber by condensing vapors above the mixt. in the chamber so that the pressure in the chll. is lowered and the vaporization of the diluent is increased, and the cold-condensed diluent is returned to the body of diss. oil wax mixt. Wax slurry is passed to a filter. App. and various operative details are described.

Separating waxes from oils. Standard Oil Development Co. Fr. 787,163, Sept. 18, 1935. The oil is diss. by a solvent of sp. gr. less than that of the wax to be eliminated, a "sepn. additive compd." is added of the type produced by the passage of an a. c. of high frequency through an org. compd. having a relatively long straight

chain. The diss. mixt. is cooled to effect solidification of the wax and the solid wax is sep'd.

Apparatus and method for dewaxing oils by use of propane, butane, etc. Donald E. Payne (to Standard Oil Co. of Ind.). U. S. 2,026,651, Jan. 7. Various structural and operative details are described.

Petroleum lubricating oils. John D. Fields. U. S. 2,024,968, Dec. 17. For conditioning low-gravity petroleum oils for removal of S from them, the oil is heated in a closed container with cellulosic vegetable material such as wood, cane refuse or hemp to a temp. sufficient to effect decompn. of the vegetable material, under a pressure (suitably about 100-140 lb. per sq. in.) not exceeding 140 lb. per sq. in. for a sufficient time to cause breaking of the bond linking the S to H and C in the oil so that the S can be readily removed from the resulting distillate and residual oil, at least in substantial part, by washing with water.

Lubricating oils. Hermann Zorn, Gerhardt Hofmann and Walter Rousinsky (to I. G. Farbenind. A.-G.). U. S. 2,025,490, Dec. 24. A vegetable fatty material such as soybean oil or olive oil is either heated to 300-500° in the presence of a catalyst having a splitting action such as oxides of Cu and Al or is treated with H at 200-300° under a pressure of 50-300 atm. in the presence of a catalyst having a reducing action such as Cu chromite and the resulting product, after dehydration, is treated with a condensing agent such as AlCl<sub>3</sub> at a temp. of about 120° to produce a lubricating oil.

Lubricating oils. Standard Oil Development Co. Fr. 787,381, Sept. 21, 1935. Oils are purified by treating them with an undil. mixt. of liquid solvents composed of pure PhOH and crude phenol (or other member of the class comprising crude phenol and cresol) at a temp. below that at which the mixt. becomes completely miscible with the oil, sep'd. the layer of oil from the layer of mixt., and removing the dissolved solvent from the layer of oil.

Lubricating oils. The Standard Oil Co. of Ohio. Fr. 787,717, Sept. 27, 1935. Paraffin is removed from oils by treating them with SO<sub>2</sub>, alone or with the addn. of a volatile org. solvent while cooling the mixt. to about -15°.

Refining oils such as mineral lubricating oils. Lawrence M. Henderson (to Atlantic Refining Co.). U. S. 2,024,221, Dec. 17. A mixed aliphatic-aromatic amine such as phenylmethylamine is used as a selective solvent for sepn. of fractions resp. richer in paraffinic and naphthenic compds. than the material treated.

Filter for lubricating oils. Manufacture de machines auxiliaires pour l'électricité & l'industrie. Fr. 787,686, Sept. 26, 1935.

Apparatus for production of high-grade lubricating oils by heating oil with hydrogen-containing gas under pressure, etc. Wm. L. Gomory (to Standard Oil Development Co.). U. S. 2,025,354, Dec. 24. Numerous structural and operative details.

Apparatus (with a light-sensitive cell) for testing lubricating oils, etc. Mead Cornell and Charles C. Curtis (to Charles C. Broadwater). U. S. 2,024,720, Dec. 17. Various structural, light-regulating and operative details.

Lubricant. Standard Oil Development Co. Fr. 787,577, Sept. 25, 1935. A lubricant comprises a lubricating oil and a linear polymer of C<sub>2</sub>H<sub>4</sub>, not contg. O, e. g., 0.2-10% of hydrogenated polyvinylacetate or alkylated polychloroprene.

Lubricant. I. G. Farbenind. A.-G. Fr. 787,801, Sept. 30, 1935. A lubricant comprises a major portion of a lubricating oil and a minor portion (0.25%) of a residue which is solid at ordinary temp. and which is obtained by distn. under vacuum of a deasphalted hydrocarbon product contg. cyclic constituents. Thus, an addn. product is obtained by removing asphalt from the product of destructive hydrogenation of coal and distg. under vacuum, a red-brown resin being obtained as residue.

Lubricants from fatty oils. Primo Perucca. Ger. 621,443, Nov. 7, 1935 (Cl. 23c 1). See Fr. 747,016 (C. A. 27, 4919).

Penetrating lubricant suitable for use with leaf springs.

Roy D. King (to Socony-Vacuum Oil Co.). U. S. 2,025,517, Dec. 24. Pine fatty acids and defoliated graphite are used with a preponderating proportion of a nonviscous petroleum distillate.

Lubricating grease. Gus Kaufman (to Texas Co.) U. S. 2,024,990, Dec. 17. A normally solid lubricant suitable for use with ball or roller bearings comprises neat's-foot oil, an alkali metal soap of neat's-foot oil and 30-50% of mineral lubricating oil, in homogeneous and substantially dehydrated mixt.

Removing foreign matter from used journal-box waste oil. Wm. T. Bissell and Thomas W. Potter (to Journal Box Servicing Corp.). U. S. 2,023,988, Dec. 10. Na silicate about 600 lb and a 20% aq soln of Na aluminate about 5 gal are added to about 1000 gal of dirty oil (the combined proportion of the silicate and aluminate being 3.5-8% of the oil) and the mixt is agitated and heated to above 100° and a ppt is allowed to form and is sepd.

Apparatus for reclaiming oil such as that from internal-combustion engines by heating and filtration. Paul Williams (to Skinner Purifiers, Inc.). U. S. 2,023,986, Dec. 10. Various structural and operative details.

Apparatus for filtering and reclaiming contaminated oils. Frank M. Clark (to General Elec. Co.). U. S. 2,025,570, Dec. 24. Various structural and operative details.

Products resembling petrolatum. I. G. Farben und A.-G. Fr. 786,472, Sept. 3, 1935. Such products are obtained by mixing waxy polynuclear chlorinated hydrocarbons, oily and resinous polynuclear chlorinated hydrocarbons, small amts of oils such as paraffin or mineral oils and (or) small amts. of waxes such as ozocerite or artificial waxes. An example contains waxy chlorinated  $C_{18}H_{34}$  (50% Cl content) 49, chlorinated  $Ph_2$  (59% Cl content) 59, ozocerite 0.5 and paraffin oil 0.5 parts.

Apparatus for cooling and granulating asphalt. Standard Oil Development Co. Fr 787,734, Sept. 28, 1935. Asphalt and a liquid hydrocarbon b below 0° (propane)

are mixed and passed into a vessel of large dimensions so that the asphalt is solidified to small particles and the hydrocarbon is gasified.

Rapidly setting asphalt. Charles M. Daskin (to Standard Oil Development Co.). U. S. 2,025,709, Dec. 31. A rapidly setting liquid asphalt of approx. 200 viscosity Furol at a temp of 60° comprises a soln. of 70-83% of asphalt of 80/100 penetration (N. Y. penetrometer) at a temp of 25° derived from the cracking of petroleum oil under at least 350 lb. per sq. in. pressure with 30-17% of distillate obtained from cracking petroleum oil.

Asphalt from oil residues. Herman T. Swerissen (to Shell Development Co.). U. S. 2,026,073, Dec. 31. A mineral oil residue contg naphthenic soaps is treated with just enough inorg acid such as an acid sludge to reduce its naphthenic soap content as desired by decompn. of the soap, and the substantially neutral residue, contg products of decompn of the soaps, is subjected to an oxidizing treatment.

Asphaltic product from cracked oil residuum. Felton S. Dengler, Earl W. Gardner and Dudley H. Felder (to Texas Co.) U. S. 2,024,046, Dec. 10. Cracked residuum relatively high in unstable  $CCl_4$ -insol compds is heated and blown with air to remove low-boiling constituents to increase its hardness and raise its m. p. and mixed with an air-blown substantially uncracked asphaltic residue adapted to stabilize the unstable  $CCl_4$ -insol compds. in the cracked residuum and to inhibit their formation and segregation.

Treating asphalt to prolong its "adhesive life." Geo. R. Hoover (to American Rolling Mill Co.) U. S. 2,026,039, Dec. 31. Asphalt, while liquid and at an elevated temp, is blown with air to which  $NH_3$  is added.

Mixing and masticating apparatus suitable for use with asphalt mixtures. Burns A. Stewart (to Patent and Licensing Corp.) U. S. 2,025,077, Dec. 24. Mech. features.

## 23—CELLULOSE AND PAPER

CARLETON E. CURRAN

Molecular weight of cellulose. J. Marchlewska-Roczniki Chem 15, 331-7 (1935).—The mol. wts. of different samples of cellulose treated with acetone were detd. by the van Campen dynamic osmotic pressure method (van Campen, C. A. 25, 5331) (R. Obogi and L. Brodka, C. A. 29, 1696). The values range from 27,000 to 37,000. J. I. Matejczyk.

Solka, the purified cellulose—its development, characteristics and application to industry. C. S. Kaufman. Proc. Am. Assoc. Textile Chem. Colorists 1935, 338-60, Am. Dyestuff Repr. 24, 734-6 (1935).—Solka is a fiber ranging from 93 to 98% pure  $\alpha$ -cellulose. Applications in various lines are mentioned. W. H. Boynton.

The synthesis of butylcellulose. S. Ushakov and V. Konkova. Kunststoffe 25, 169-73 (1935), cf. C. A. 29, 3151.—In disagreement with Nowakowski (cf. C. A. 26, 6122) U. and K. have found it possible to butylate alkali cellulose with BuCl. Most of U. and K.'s expts proceeded as follows. Linters were mercerized by treating them for 24 hrs with NaOH solns. of 50, 40 or 20% concn, and then allowed to ripen for 24 hrs. Excess caustic was removed by pressing. The alkali cellulose so obtained was ground in a mortar and butylated by reacting with 5, 7, 10 and 20 mols. BuCl and 5, 7, 10 and 10 mols NaOH, resp. per C<sub>2</sub> cellulose unit. Butylation was carried out at temps. ranging from 110° to 125°. A Ag-lined autoclave was used and the duration of heating varied from 8 to 30 hrs. In a few expts the alkali cellulose was subjected to repeated (stepwise) butylation. In any case, the product was purified by successively washing with hot water, grinding up with water and pouring on a filter, steam distg off as much BuOH as possible and vacuum drying at 60-70°. Soln. in  $CH_2$  and, to a lesser extent, soln. in an 80%  $CH_2$ -20% alc. mixt., as well as yield of cellulose ether increased very rapidly as the

duration of heating was lengthened up to 12 hrs. and more slowly thereafter. The temp. of heating in these expts was 123-5°, 10 mols. of BuOH and NaOH being used per C<sub>2</sub> unit. Similar results were obtained on increasing the amt. of BuCl up to 10 mols. while holding the butylation temp. at 123-5° for 12 hrs. It may be said that in general a butylation temp. of 123-5° gives somewhat better results than 110°. A mercerizing caustic contg. 50% to 40% NaOH was preferred as otherwise pressing the alkali cellulose caused excessively large caustic losses. Stepwise butylation at 123-5° yielded the most highly butylated product with a benzene soly of 93.9%. Films prepd from 8% benzene solns. possessed excellent resistivity to water, strong  $H_2SO_4$  and 20% caustic. The relatively poor mech. strength of such films is apparently ascribable to the Bu radical itself (cf. Frank and Caro, C. A. 24, 4270). J. W. Perry.

A method for treating celluloid wastes. S. Rozenblyum. Plasticheskie Massy 1935, No 3, 29.—Waste celluloid is softened with addn of soap and talc and pressed into blocks which can be sliced and used again.

Synthetic textile fibers. H. Roche. Silk J. 12, No 139, 40 (1935).—A review is given of processes for fibers other than viscose and cellulose acetate fibers. H. E. S.

Standard types of defiberizers. A. A. Khlebnikov, Ya. G. Rozental and P. E. Pimenov. Leningrad. Oblast. nol. Secet Nauch. Inzhenerno-Tekhn. Obshchestva Trelyuzno-Bumazhnol Prom (Problems of Pulp-Paper Production) No 4, 42-55 (1935).—Screw defiberizer is considered the most satisfactory of the continuous-action type of defiberizers. A four-press hydraulic defiberizer is considered more economical than a screw defiberizer. Chas. Blanc.

Method of analysis and composition of pumiceous defiber stones. M. I. Koffman. Bumazhnaya Prom



14, No. 9, 28-35 (1935).—The compn of several grades of Norwegian delbering stones was detd by a method of mech decompn and microscopic analysis. The working top layer is composed of ground corundum-pumice 60-1, Portland cement 16-29, Na<sub>2</sub>SO<sub>4</sub> 10.6-14.5 and org matter 0.2-0.4%. The loss in wt on ignition is about 8%. The core contains feldspathic sand 55, sawdust 4-5, Portland cement 18-25 and Na<sub>2</sub>SO<sub>4</sub> 8-14%. The loss in wt is about 7%. Chas. Blanc.

Results attained in sulfite digesters by combined acid and gas circulation. O. Kreusler. *Svensk Papperstidn* 38, 630-61 (1935).—A historical resumé. W. S.

Formulas for calculation of wood-pulp production. P. I. Borovoy. *Leningrad Oblasnii Sovet Nauch. Iskuzhenno-Tekhn. Otshektr. Tsellyulozno-Bumazhnoi Prom. (Problems of Pulp-Paper Production)* No. 4, 55-63 (1935).—A math treatment of various phases of wood-pulp production. Chas. Blanc.

Viscose pulp and viscose pulp woods. VII. Chemical compositions of Japanese and Manchurian birches. Masuzo Shikata and Hideo Omata. *J. Agr. Chem. Soc. Japan* 11, 879-8 (1935).—The wood of *Betula ermanii* Champ var. *communis* Kodz. produced in Karafuto contained H<sub>2</sub>O 8.12, alc-ether ext. 5.43, cold-H<sub>2</sub>O ext. 0.65, hot-H<sub>2</sub>O ext. 2.14, 1% NaOH ext. 29.77, mannann 0, galactan 0.74, pentosan 25.14, hemicellulose 25.68, total cellulose 53.25 (α-cellulose 70.52, β-cellulose 11.45 and γ-cellulose 17.50), crude protein 0.72, ash 0.24 and Lignin 21.50%. The wood of *Betula japonica* Sieb. produced in Karafuto and Manchukuo contained, resp., H<sub>2</sub>O 8.42, 9.16, alc-ether ext. 5.18, 6.02, cold-H<sub>2</sub>O ext. 0.89, 1.16, hot-H<sub>2</sub>O ext. 5.38, 10.31, 1% NaOH ext. 24.29, 28.14, mannann 0, galactan 0.51, 0.43, pentosan 21.01, 24.13, hemicellulose 21.52, 24.56, total cellulose 53.99, 54.38 (α-cellulose 70.13 and 67.53, percentage of total cellulose), crude protein 0.63, 0.49, ash 0.12, 0.53, and lignin 21.17, 21.28%. VIII. Chemical composition and cooking test of Manchurian fir, *Abies nephrolepis* Maxim. Masuzo Shikata and Kenkichi Tadokoro. *Ind.* 925-39.—*Abies nephrolepis* Maxim. contained H<sub>2</sub>O 10.99, alc-benzene ext. 4.10, 1% NaOH-sol matter 12.78, hot-H<sub>2</sub>O-sol matter 3.17, cold H<sub>2</sub>O-sol matter 1.12, total cellulose 49.57, α-cellulose 27.08, lignin 26.77, pentosan 12.92, mannann 5.54, galactan 0.79, hemicellulose 19.25, methoxyl 5.20, N 0.86 and ash 0.29%. It resembled in chem compn karafuto fir, *Abies sachalinensis* Fr. Schum. The Manchurian fir was cooked with 1% Ca and Mg bisulfite soln at 145° for 8 hrs under 75 lb/sq in., a comparatively good viscose pulp was obtained. Y. Kihara.

Effect of sodium hydroxide, sulfite, sulfite and carbonate on straw pulping. K. A. Dolgor. *Bumazhnyy Prom.* 18, No. 10, 21-35 (1935); cf. *Tsellyul. Nauch.-Issledovatel. Inst. Bumazhnoi Prom. Materialy* 1931, No. 1.—Rye straw (13.5% moisture content) was pulped 3 hrs at 140°, 155° and 170° with equiv. amts. of NaOH, Na<sub>2</sub>S, Na<sub>2</sub>SO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>S + NaOH, NaOH + Na<sub>2</sub>SO<sub>3</sub>, Na<sub>2</sub>SO<sub>3</sub> + Na<sub>2</sub>S, Na<sub>2</sub>SO<sub>3</sub> + Na<sub>2</sub>S + NaOH at a diln. of 8 parts of liquor to 1 part of straw. Conclusions: At 155° Na<sub>2</sub>SO<sub>3</sub> gave 54.07%, Na<sub>2</sub>S 48.48% and NaOH 42.89% of unbleached pulp. At 140° the yields are considerably larger, because of the increased contents of lignin (by 1.78%), cellulose (1.54%) and pentosans in the monosulfite pulp, and cellulose (1.58%), pentosans and lignin in the NaOH pulp. The best liberation of fiber and highest degree of wetness are effected with Na<sub>2</sub>S, followed by NaOH and then by Na<sub>2</sub>SO<sub>3</sub>. The degree of delignification decreases in the following order: Na<sub>2</sub>S + NaOH, NaOH, Na<sub>2</sub>SO<sub>3</sub> + Na<sub>2</sub>S + NaOH, Na<sub>2</sub>SO<sub>3</sub> + Na<sub>2</sub>S, Na<sub>2</sub>SO<sub>3</sub> + NaOH and Na<sub>2</sub>SO<sub>3</sub>. The decoloration of the stocks (dark brown) is greatest with NaOH and Na<sub>2</sub>S, and lowest (gray) with Na<sub>2</sub>SO<sub>3</sub> + NaOH and Na<sub>2</sub>SO<sub>3</sub> + Na<sub>2</sub>S + NaOH. The latter products can be used without bleaching in the manuf. of some grades of white paper. NaOH and Na<sub>2</sub>S act on straw lignin with the formation of salt-like products, which on acidulation sep. org. substances in the amt. nearly equiv. to that of Lignin dissolved in the pulping process. With Na<sub>2</sub>SO<sub>3</sub> Na lignosulfonate is formed. The action of

NaOH, Na<sub>2</sub>S, Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>SO<sub>3</sub> on S.O<sub>2</sub> in straw decreases in the order given. A large part of pentosans (about 50% in soln.) is decomposed, probably into the corresponding acids. With NaOH this decomposition leads to formation of resinification products incapable of giving furfural with HCl. At 140° Na<sub>2</sub>CO<sub>3</sub> causes considerable delignification and demerization of straw with solvation of 10% of available pentosans. The effect of Na<sub>2</sub>CO<sub>3</sub> in pulping with NaOH and NaOH + Na<sub>2</sub>S is probably insignificant, but is very strong in pulping with Na<sub>2</sub>SO<sub>3</sub> and Na<sub>2</sub>SO<sub>3</sub> + NaOH. The difficulties in bleaching NaOH and sulfate stocks increase with the higher temp. of pulping (from 140° to 155°). This may be explained by the formation of humic substances and products of pentosan resinification becoming partly fixed on the pulp fibers. Pulping with Na<sub>2</sub>SO<sub>3</sub> at higher temps. results in better bleachable stock. More easily bleachable stock of greater whiteness is obtained at 140° and 155° with Na<sub>2</sub>SO<sub>3</sub> + NaOH, followed by Na<sub>2</sub>SO<sub>3</sub> + Na<sub>2</sub>S + NaOH. The action of mixed alkalis is more favorable than that of any single reagent (NaOH + Na<sub>2</sub>S acts better than NaOH, and Na<sub>2</sub>SO<sub>3</sub> + NaOH acts better than Na<sub>2</sub>SO<sub>3</sub>), because of the more effective and milder seps. of incrustations. In the process of pulping with reagent's contg. S, the S compds. are oxidized (perhaps with the aid of certain catalysts) probably at the cost of decomposition of certain org. substances. Chas. Blanc.

The use of liquid sulfur dioxide in making cooking liquor. Feltz Bergström. *Finnisk Papper och Timber J.* 1935, 922-4.—The equipment for the transportation and use of liquid SO<sub>2</sub>, such as steel tank cars, storage tanks and valves, is described. By using liquid SO<sub>2</sub> the compn. of the cooking liquor can be regulated according to the requirements. E. E. Jukkola.

Standardization of beaters. A. M. Berlin, K. P. Belavin and P. I. Borovoy. *Leningrad. Oblasnii Sovet Nauch. Iskuzhenno-Tekhn. Otshektr. Tsellyulozno-Bumazhnoi Prom. (Problems of Pulp-Paper Production)* No. 4, 3-25 (1935).—Standardization of beaters for reworking of paper and newsprint wastes. *Ibid.* 26-35.—A comparative discussion of the construction and performance of various types of beaters with illustrations and math treatment. Chas. Blanc.

Production of fine paper from eiled and tarred rag waste. G. S. Tumpkin. *Bumazhnyy Prom.* 18, No. 10, 50-5 (1935), cf. Sokolov, *Tsellyul. Nauch.-Issledovatel. Inst. Bumazhnoi Prom. Materialy* 1931, No. 1, 36.—Canvas and 6-inch waste was cooked with 8-10% CaO and 2-3% NaOH at 3-4 atm. for 6-10 hrs., depending on the degree of contamination with oil and tar, and then bleached with 12% Ca(ClO)<sub>2</sub>. The stock after reworking was used in mixt. with 25% of bleached and 25% of unbleached sulfite pulp in the production of a good grade of cigarette paper. Chas. Blanc.

Production of vulcanized fiberboard. D. F. Lebedev. *Leningrad. Oblasnii Sovet Nauch. Iskuzhenno-Tekhn. Otshektr. Tsellyulozno-Bumazhnoi Prom. (Problems of Pulp-Paper Production)* No. 4, 64-102 (1935).—A comprehensive discussion of all phases of production of vulcanized fiberboard and recovery of waste products. Chas. Blanc.

Soln. of nitrocellulose (Tocman). 2. Hydrolysis of cellulose and its oligosaccharides (Frendenberg, Bloquist) 10. App. for drying continuous webs of paper (U.S. pat. 2,024,827) 25. Powd. substances [nitrocellulose and benzylcellulose] (Fr. pat. 757,233) 1.

Cellulose from lignocellulose materials. Walter H. Greenbridge and Eric V. Mellers (to Celanese Corp. of America). U. S. 2,024,689, Dec. 17. Material such as wood, straw or grass is etrid. at an elevated temp. with a solvent mixt. contg. a substantial proportion of water and at least 2 org. liquids such as acetone and EtOH which are chemically inert toward Lignin and which have substantially different dielectric constants due to the presence in the mixt. of groups of different type. Cl. C. A. 30, 82<sup>21</sup>.

Alkali cellulose; viscose. I. G. Farberindustrie A.-G.

Brit. 434,540, Sept. 4, 1935. See Fr. 784,935 (C. A. 30, 6119).

**Regenerated cellulose products** Henry Drevfus U. S. 2,025,940, Dec. 31. Regenerated cellulose materials, such as filaments, etc., are treated with sulfonic acids, such as sulfacetic acid or the like of at least 60% concn in the absence of inorganic acids, to give improved strength or elasticity or modified luster.

**Regenerated cellulose sheets, films, etc.** Daniel W. Loebe (to Johnson-Loebe Corp.) U. S. 2,025,930, Dec. 17. Urea phosphate is used as a treproofing addn and sheets or films of regenerated cotton cellulose contg. sufficient of the natural incrustations to waterproof the material are provided with a transparent, flexible, non-tacky coating such as a pyroxylin compn.

**Inhibiting distortion of regenerated cellulose films** Clarence E. Coleman (to DuPont Cellophane Co.) U. S. 2,025,376, Dec. 24. A roll of the film is aged in an atm. having a relative humidity of 30 to 40 for a time (which may be about 40 hrs. or longer) sufficient to relieve at least a large part of the internal strains present in the film.

**Cellulose derivatives** Walter F. Hoffman (to Cellosis, Inc.) U. S. 2,025,283, Dec. 24. Cellulose fibers such as aspen wood pulp together with a volatile liquid such as water and a foaming agent such as soapin are formed into a foam which is dried to form a fibrous distended porous fibrous mass and the latter is treated with a reactive fluid such as a nitrating or acetylating agent.

**Cellulose derivative compositions** Geo. D. Graves (to E. I. du Pont de Nemours & Co.) U. S. 2,025,048, Dec. 24. A cellulose deriv. such as cellulose nitrate or acetate or ethylcellulose is plasticized with a carboxylic acid ester of a lauryl ether of a polyhydric alc. such as lauryl myristyl acetate or stearate. Numerous examples are given.

**Films from cellulose derivatives** I. G. Farbenmd A. G. (Max Hagedorn, Wilhelm Schmidt and Paul Möller, inventors) Ger. 621,189, Nov. 2, 1935 (Cl. 3, 12 01). Thin films prep'd from cellulose derivs., e. g., films of a thickness below 200  $\mu$ , are improved in their mech. properties by treat'g with an inert nonaq. solvent until the softening agent originally present has been entirely or nearly entirely removed. Exptl. results are given.

**Use of cyclic ethers in plasticizing cellulose derivatives** Henry Drevfus. U. S. 2,025,044, Dec. 24. A cellulose deriv. such as cellulose acetate is used with a cyclic ether obtainable by condensing an alc. contg. at least 4 hydroxy groups such as erythritol alone or with an alc. contg. at least two hydroxy groups such as ethylene glycol. Various examples are given.

**Cellulose ester compositions.** The Distillers Co. Ltd., Harold A. Anden, Hains P. Strandger and Philip Eagleshead. Brit. 434,970, Sept. 12, 1935. Comps. contg. cellulose triacetate (I) as the chief constituent contain also a substantial amt. of a lactide or an anhydride of an org. acid having a b. p. of at least 200° at normal pressure, halogen derivs. of such lactides or anhydrides not being included. In examples, the comps. consist of powd. I 100 mixed with lactide from fermentation butyric and isobutyric chloride (II) 23, triacetate 12 and ethylacetamide 5 parts; or with maleic anhydride 18, a mixt. of equal parts of II and ethylene chlorohydrin 23 and *p*-toluenesulfonamide 6 parts; or with succinic anhydride 20, II 10 and cell. on red dye 0.3 parts.

**Cellulose organic and esters** Richard Müller, Martin Schenk, Wilhelm Weitz and Fritz Müller (to C. F. Roehm & Soehne G. m. b. H.). U. S. 2,024,881, Dec. 17. A cellulose material such as pretreated cotton linters is treated with an acylating agent such as  $\text{Ac}_2\text{O}$  in the presence of a lower aliphatic sulfonic acid, such as chloro- (or *n*-) *m*-thanesulfonic acid which contains more than one org. and radical and which serves as a catalyst for promoting esterification of the cellulose. Several examples with details are given.

**Stabilizing organic esters of cellulose.** Camille Drevfus and Clifford I. Haver (to Celanese Corp. of America). U. S. 2,023,900, Dec. 31. An ester such as a cellulose

acetate, formate, propionate or butyrate which has been prep'd. in the presence of a S-bearing acid catalyst, such as  $\text{H}_2\text{SO}_4$ , is sepd. from the primary soln. in which it is formed, dissolved in a solvent such as glacial  $\text{HOAc}$  and a sol. Ba salt such as Ba acetate is added.

**Refining organic acid esters of cellulose.** Dennis E. Northrop and Robert C. Burton (to Eastman Kodak Co.). U. S. 2,024,246, Dec. 17. For refining an ester such as a cellulose acetate, it is dissolved in an aq. acetone, the soln. is filtered and the ester is substantially completely ppt'd by gradually adding the filtered soln. to an agitated aq. acetone in which the ester is substantially insol. and which is maintained at approx. const. concn. during the pptn. App. is described.

**Extraction of cellulose esters** Cyril J. Stand and Edward C. Yackel (to Eastman Kodak Co.). U. S. 2,024,060, Dec. 17. For fractionally extg. a cellulose ester contg. an acyl group such as cellulose acetate of low viscosity from mixed cellulose acetates, a mixt. of acetone and water or  $\text{HOAc}$  and water is used.

**Cellulose diglycolic acid ester, etc.** Carl J. Malm and Charles R. Fordree (to Eastman Kodak Co.). U. S. 2,024,238, Dec. 17. By reaction upon a cellulose material such as regenerated cellulose, or on a cellulose acetate, with diglycolic anhydride, in the presence of a tertiary org. base such as pyridine, an ester is obtained such as cellulose diglycolate or cellulose acetate-diglycolate, suitable for various uses.

**Cellulose of acetate compositions** Geo. Schneider (to Celanese Corp. of America). U. S. 2,023,957, Dec. 31. A compn. resistant to discoloration when heated comprises cellulose acetate with the addn. of about 0.01-0.3% of a naphthene acid salt such as the Ca salt.

**Plated sheets of cellulose acetate or the like** Joseph H. Brown (to Celluloid Corp.) U. S. 2,023,568, Dec. 24. Substantially permanently plated sheets contg. an org. cellulose deriv. such as cellulose acetate and which also may contain a plasticizer such as dibutyl phthalate are formed so as to be extensible without loss of their plated form.

**Cellulose triacetate compositions** The Distillers Co. Ltd., Harold A. Anden and Hains P. Strandger. Brit. 433,311, Sept. 19, 1935. Cellulose triacetate is mixed with cyclohexanone, Me cyclohexanone, cyclohexanvl acetate, Me cyclohexanvl acetate, benzyl acetate or Et Ac glycolate in such quantity that the mass, when afterward heated to 150-190°, becomes viscous and capable of being molded or rolled.

**Mixed esters of cellulose** Thomas F. Murray, Jr., and Cyril J. Stand (to Eastman Kodak Co.). U. S. 2,024,658, Dec. 17. In making a mixed ester contg. acetyl and higher aliphatic acid radicals such as those of propionic, butyric or crotonic acid, in cases where the mixed ester does not form by the initial esterification both contain grain or lumps, a small proportion of  $\text{Ac}_2\text{O}$  is added to the dope to eliminate the grain or lumps.

**Cellulose acetate isobutyrate.** Carl J. Malm and Charles L. Fletcher (to Eastman Kodak Co.). U. S. 2,024,651, Dec. 17. In prep'g. cellulose acetate isobutyrate in fibrous form, cellulose such as cotton linters is treated with an esterification bath which is rendered non-solvent of the cellulose acetate isobutyrate formed by the presence of about 60-80% isobutyryl based on the total acyl content, the remainder of the acyl content being acetyl.

**Partially de-esterifying acetone-soluble cellulose acetate-propionate and acetate-butyrate.** Carl J. Malm and Charles L. Fletcher (to Eastman Kodak Co.). U. S. 2,023,583, Jan. 7. A material the acyl content of which comprises at least 15% propionyl or butyryl is treated with a hydrolyzing agent such as aq.  $\text{HOAc}$  soln. contg.  $\text{H}_2\text{SO}_4$ , and the hydrolysis is terminated before the total acyl content of the acetate has been reduced to less than 5% to obtain a product which is suitable for various uses.

**Treatment of cellulose esters and ethers.** Gero Lindan and Georg Frank. Ger. 621,047, Nov. 1, 1935 (Cl. 3, 12 01). Cellulose ethers and esters are caused to swell or are brought into soln. or dispersion by treat-

ment at atm temp with aq solns of salts of aromatic hydroxycarboxylic acids, e g, salicylic acid. Org solvents may be added to the aq salt solns.

**Treating cellulose material such as that for making viscose.** Emil Scheller (to E I duPont de Nemours & Co.) U S 2,026,068, Dec 31. The material is treated with active Cl to the extent of "about a half to three-fourths bleach," then washed with an alkali boil in the absence of O at a relatively high temp, and bleaching is completed with alkali peroxide solns, and the material is then washed, soured with a dil acid soln and finally washed with water which is slightly alk. in the last stage of the washing.

**Pressure indicating device suitable for use with viscose flowing through pipes.** Ralph E Valentine (to Dupont Cellophane Co.) U S 2,024,752, Dec 17. Various structural and operative details.

**Recovery of waste viscose.** Comptoir des textiles artificiels (Soc anon.) Fr 780,270, Aug 30, 1935. Waste viscose strongly swollen with water is submitted to a treatment with AcOH to replace the water by AcOH and then converted to triacetate by Ac<sub>2</sub>O. The replacement of water by AcOH may take place in a battery of diffusers.

**Artificial sponges from viscose.** Walter Johannes (to Winthrop Chemical Co.) U S 2,026,177, Dec 31. Viscose contg a pore-forming material is pressed into a tubular mold, coagulated in the mold by heat externally applied, the coagulated mass is removed from the mold, the pore-forming material is then removed and the coagulated mass is cut into pieces perpendicularly to its longitudinal axis. App is described. Cf. C. A. 29, 6758<sup>1</sup>.

**Viscose rayon.** James W Humphrey (to Viscose Co.). U S 2,025,868, Dec 31. For increasing the tensile properties of viscose rayon, there is added to the viscose soln, prior to spinning, a substituted hydroxybenzene in which a H atom of the benzene ring has been substituted by an alkyl group, such as *m*- or *p*-cresol in an amt of 1-2% the wt of the viscose soln (most of the added material being lost upon spinning).

**Increasing the tensile strength of viscose rayon.** James W Humphrey and John W Pedlow (to Viscose Co.) U S 2,024,041, Dec 10. There is added to the viscose soln used an aromatic deriv. of an aliphatic alc in which the aromatic group replaces a H atom of the alc, other than that of the OH group, such as anisyl alc, phenylethyl alc or benzyl alc, most of which does not remain in the extruded filament but is lost during the spinning operation, but which serves to facilitate the production of a product of increased tensile strength.

**Rayon.** Alsa Soc anon. Fr 787,585, Sept 25, 1935. In making rayon having tubular filaments from viscose, the thread has a short path (25 cm.) through the coagulating bath and a long path through the air before being wound. Fr 787,586. The lower the unitary titer of threads made of tubular filaments the higher is the concn of H<sub>2</sub>SO<sub>4</sub> and Na<sub>2</sub>SO<sub>4</sub> in the coagulating bath used. Cf. C. A. 29, 3513<sup>2</sup>.

**Rayon, etc.** I G Farbenindustrie A-G. Brit 434,547, Sept 4, 1935. See Fr. 752,152 (C. A. 29, 1636<sup>4</sup>).

**Apparatus for rayon manufacture.** Earle J R Beatty U S 2,024,962, Dec. 17. An app is described having a spinneret in a coagulating bath and a device with entrance and discharge openings of different size with which the filaments come into contact and through which they pass, the discharge opening being smaller and of a size less than the combined diam. of the rayon filaments after their extrusion from the spinneret.

**Apparatus for making rayon.** I G Farbenindustrie A-G. Brit. 434,881, Sept 9, 1935. The thread that leaves the spinning bath under the draft of a galeite is allowed to fall freely onto a plate having a rotary and an addnl. reciprocating movement, or movement with a reciprocating component, so as to form annular crosswise layers, and the ratio of the period of the rotary movement to that of the addnl. movement is adjusted so that at most only 1 addnl. movement occurs at every half revolution, while the period of natural oscillation of the freely hanging

thread is adjusted to equality or substantial equality with the period of the addnl. movement or a harmonic thereof.

**Apparatus for spinning rayon.** Edward Wurtz. Ger 618,960, Sept. 19, 1935 (Cl. 29a 6 01).

**Filaments, films, etc., of cellulose, organic derivatives such as cellulose acetate.** Wm I. Taylor (to Celanese Corp. of America) U S 2,025,902, Dec. 31. For increasing the tenacity of the material, it is subjected to swelling by treatment with glycerol or diethylene glycol at temps. of 120-170° or 75-90°, resp., and to stretching of 65-80% of the length of the material in the presence of the treating substance.

**Artificial threads, films, etc., from cellulose acetate.** Wm A Dickie and Percy F C Sowter (to Celanese Corp. of America) U S 2,025,730, Dec 31. A soln of cellulose acetate is extruded through a suitable orifice into a coagulating bath contg a relatively high proportion of a solvent or plastifying material and the product is stretched while in a plastic state with only a portion, at most, of the stretching force acting on the material immediately on extrusion. App is described.

**Artificial filaments, yarns, films.** British Celanese Ltd and Percy F C. Sowter. Brit. 434,953, Sept. 9, 1935. A soln of an org. deriv. of cellulose in a volatile solvent is extruded into a liquid coagulating medium that contains a softening agent for the cellulose deriv. that has a b p above 100° and is maintained near or above the b p. of the volatile solvent. Suitable softening agents are those of Brit 340,325 (25, 4708). In an example, an MeCO soln of cellulose acetate is spun into an aq 30% soln of glycol monoacetate at 55°.

**Cellulose sulfonic acid.** Geo W. Rigby (to E. I. du Pont de Nemours & Co.) U S 2,025,073, Dec 24. Cellulose such as cotton linters is treated with pyrosulfuric acid in the presence of a tertiary amine such as pyridine.

**Artificial staple fibers.** Fred Ferrand. Brit. 435,384, Sept. 19, 1935. In a method of fibers where a coagulable soln fed to an extrusion nozzle is subdivided at regular intervals in such manner that the soln. is extruded from the nozzle as sepd. or staple fibers, the subdivision is effected in or at the perforated cap of the nozzle. App is described.

**Utilizing values of waste sulfite liquor.** Peter A Paulson U S 2,025,891, Dec. 31. Waste sulfite liquor is evapd and the steam thus generated is brought into indirect heat exchange with the cooking liquor of a digester, heat for the evapn. being generated by burning the concd residue of the liquor. App is described.

**Paper pulp.** André Bergès. Fr 787,386, Sept. 21, 1935. The pulp is purified by causing it to enter a fixed circular vessel at a high speed and tangentially, cone-shaped separators being mounted in the vessel.

**Wire faced roll for paper making apparatus.** Einar I Flateboe U S 2,025,445, Dec 24. Structural details.

**Drier for paper manufacture.** Franz Hassmann. Ger 618,664, Sept. 13, 1935 (Cl. 55d. 14 30).

**Felts or aprons for drying paper.** James K Diamond (to Clipper Belt Lacer Co.) U S 2,025,275, Dec 24. Structural details.

**Apparatus for drying continuous running webs of paper.** Wm. Whiting (to Whiting Paper Co.). U S 2,024,079, Dec 10. Various structural, mech and operative details.

**Bacterial product suitable for use in making sheets or as a filler in paper.** Joseph R Sanborn (to International Paper Co.) U S 2,026,253, Dec 31. Growths formed from cultures of slime forming microorganisms such as *Oidium lactis* and *Monilia candida* are digested with a ZnCl<sub>2</sub> soln to form a viscous mass, and the mass is re-generated with water into a desired form such as a substantially transparent sheet.

**Coating paper to render it greaseproof.** Joseph H Swan, 3d (to Gardner-Richardson Co.) U S 2,025,788, Dec 31. A coating is prepd by adding an aq CH<sub>3</sub>O soln to an aq alk. casein soln, to form a mixed soln contg about 10% as much CH<sub>3</sub>O as casein on a dry wt basis, and then adding the CH<sub>3</sub>O casein soln to rubber latex emulsion with agitation to avoid coagulation of the latex.

Composition suitable for coating paper. Webster E. B. 1 Baker (to Stein, Hall & Co.). U. S. 2,024,123, Dec. 10. For making a paper coating of substantially insol surfacing material, a starch-contg material is treated (as with HOAc) to produce weak esterification and dextrinization of the starch constituent, a volatile alk substance such as  $\text{NH}_3$  is added, and the material is mixed with a dry sol Na silicate, dissolved in water, mixed with china clay, the coating is applied to paper and is heated to volatilize the alk substance and cause generation of hydrated silicic acid by hydrolysis upon concn of the soln.

Draining press for continuous or individual sheets of fibrous material such as paper. Antoine Valentin U. S. 2,024,616, Dec. 17. Structural, mech and operative details.

Apparatus for treating paper webs with sizing liquids, etc. Harold R. Rafton (to Raffold Process Corp.) U. S. 2,024,248, Dec. 17. Various structural, mech and operative details.

Sized paper. Akt-Ges für chem Ind (Rannnersdorf), Felix Noss and Wilhelm Goldlust Austrian 143,323, Nov. 11, 1935 (Cl. 55c). Animal glue hardened by treatment with  $\text{CH}_2\text{O}$  is dispersed in paper stuff and then pnd on the fibers, e. g., by addn of alum or  $\text{Al}_2(\text{SO}_4)_3$ . The treatment of the glue with  $\text{CH}_2\text{O}$  should be effected in a medium of H ion concn below pH 5, and may be carried out in the presence of the paper stuff

or as a sep. operation. Various methods of procedure are indicated.

Cigaret paper. Schoeller & Hoesch G. m. b. H. Fr. 786,460, Sept. 3, 1935. A consistent white ash is obtained by impregnating the paper with an aq. soln. of the products of hydrolysis (only slightly or not sweet) of polymerized carbohydrides, the hydrolysis being carried far enough to produce not only mono- but also di-, tri- and tetra saccharides sol in water, and also of a small amt of salt. Fr. 786,461. The combustion of cigarettes is improved by impregnating the paper with a soln of Na stearate, to which beeswax or other wax may be added in the form of an emulsion. Fr. 786,462. The aroma of cigarette is improved by adding beeswax in aq. emulsion or a soln of honey or both to the paper.

Multi layer paper or cardboard or the like. Ernst Fues (to Anthony W. Deller). U. S. 2,024,635, Dec. 17. A wet paper web has applied to it a hydrated cellulosic slippery mass having a fibrous structure such as one of wood fibers and filler and the treated web is finished to form an integral product.

Multi ply reinforced paper suitable for box corner stay strips, etc. Edward C. Smith (to American Reinforced Paper Co.) U. S. 2,026,194, Dec. 31. Various details of paper sheets united by adhesive and enclosing reinforcing unspun filaments.

## 24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE AND C. G. STORM

Coal dust and rock dust as constituents of air (Stipaniz) 13.

Smokeless powder. Willard de C. Crater (to Hercules Powder Co.) U. S. 2,024,128, Dec. 10. A double-base smokeless powder contains nitrated flaked starch retaining substantially its original flaked form, together with nitroglucerin, etc.

Propellant nitrocellulose explosive. Geo. C. Hale and Donald R. Cameron. U. S. 2,026,531, Jan. 7. Nitrocellulose is used with TNT 10-15 and triacetin 2-10%.

Purifying normally solid organic explosive compounds. Wm. A. Smith (to E. I. du Pont de Nemours & Co.) U. S. 2,024,396, Dec. 17. Purification of compds such as tetryl, trinitrobenzene, trinitrotoluene, tetranitroamline and pentaerythritol tetranitrate is effected by crystrg from a neutral org. solvent such as ethylene glycol having a b. p. above 100°, which is miscible with water and nonreactive

with the compd and the vapors of which are relatively noninflammable.

Wrapping plastic explosive material such as blasting explosives. Albertus H. Haupt (to Imperial Chemical Industries Ltd.) U. S. 2,026,532, Jan. 7. App and various operative details are described.

Priming mixture for explosives. Alfred Weale (to Imperial Chemical Industries Ltd.) U. S. 2,024,406, Dec. 17. See Can. 348,291 (C. A. 29, 3518).

Blasting-cap charges. Edmund T. Lednum (to E. I. du Pont de Nemours & Co.) U. S. 2,024,588, Dec. 17. See Can. 348,291 (C. A. 29, 3518).

Blasting cartridge. Charles B. Officer (to Safety Mining Co.) U. S. 2,024,247, Dec. 17. Structural details.

Match-igniting compositions. Sven Harald Ledum. Ger. 621,050, Nov. 1, 1935 (Cl. 78b 2). This corresponds to Brit. 355,901 (C. A. 27, 1177), but describes sp. compns.

## 25—DYES AND TEXTILE CHEMISTRY

L. A. OLNEY

Precision in the analysis of dyes. M. G. Martin. Rec. gen. met. color. 39, 423-8 (1935).—The use of math formulas as an aid is given.

Method for the identification of vat dyes. G. Buzziferraris. Boll. assoc. ital. chim. tessile 11, No. 1, 1-6 (1935); *Chimie & industrie* 34, 1182.—The method, based on the analogy of color and sound, consists in observing changes in tone. Of the various reagents which can modify chiefly the tone of vat dyes, 2 are particularly simple to use, viz., hyposulfite in alk. soln and  $\text{Co}^{2+}$   $\text{Be}^{2+}$   $\text{H}_2\text{SO}_4$ . On applying a drop of the hyposulfite soln to the sample, the tone will be either (1) increased, or (2) decreased or unchanged; the same observation is made with  $\text{H}_2\text{SO}_4$ . Dyes are thus divided into 4 groups, and the identification of the particular dye is sought by comparison with known dyes of the same group which give the same variation in tone. The comparison is facilitated by use of 3 auxiliary reagents: 41°  $\text{Be}^{2+}$ ,  $\text{HNO}_3$ ,  $\text{SnCl}_4$ , and Na hyposulfite in acid soln. A. P.-C.

Degradation of colors under the influence of light as a

function of concentration. J. Pinte. *Chambre Commerce Roubaix Lab. recherches ind.* 1935, No. 25, 10-13; *Chimie & industrie* 34, 923, cl. C. A. 28, 5989.—In the case of dyestuffs applied to fibers there is a concn. at which they exhibit max. sensitiveness to the action of light, and which is known as the "mean crit. tone." Its value is approx. 55% for yellows, 45% for oranges, 40% for reds and 35% for violets, blues and greens. The fastness of a color improves the further it is from the mean crit. tone, whether it tends toward 0 (abs. black) or toward 100 (white). If the dyestuff is very pure, its mol. structure offers a point at which absorption is max.; exposure to sunlight, therefore, as an abs. general rule, has for effect suppression of the dominant. If the dyestuff is impure, destruction of the color occurs more slowly. Beyond the mean crit. value, the fact that the color curve tends to a deeper and deeper gray accounts for the increased fastness to light. At decreasing concns. there is a smaller no. of dyestuffs mol., and hence sunlight has less effect. By means of a systematic study of

dye stuffs at different concns by means of a "fade meter," curves may be established which show, with a satisfactory degree of approximation, the degradation of the mean tones, when the crit pt and the max concn. (which generally is 3 times smaller) are known. It follows, on the other hand, that the light fastness of dyes must not be compared at different concns., all comparative tests should be made at the previously detd mean crit. tone.

#### A Papineau-Couture

Fading of dyestuffs I Photochemical decomposition of malachite green and crystal violet Kenji Iwamoto *Bull Chem Soc Japan* 10, 420-5 (1935).—Malachite green oxalate (2 g) exposed to direct sunlight for 2 months under Vita glass gives 0.51 g of *p*-dimethylaminobenzophenone, m 90.5-01°. Similar treatment of crystal violet oxalate (2 g) gives 1.18 g of Michler's ketone, m 173-4°. When sealed in Vita glass tubes these dyes give the corresponding leuco compounds W. J. P.

Wool dyeing—dyeing methods and formulas employed with various colors C H Young *Am. Dyestuff Repr* 24, 735-40 (1935) W. H. Boynton

Silk filaments' shape and size cause two tone dyeing. F A Mennerich *Am Dyestuff Repr* 24, 723-4; *Am Silk Rayon J* 54, 17, 19, *Textile Bull* 49, No 15, 12, 30 (1935).—See C A 29, 40461. W. H. Boynton

Suppression of alkaline boiling in the bleaching and dyeing of vegetable fabrics Raymond Vidal *Revue ind. color* 39, xlvii-xlviii (1935).—Use of NaOCl plus a fatty acid gives a stronger and more elastic fabric. It suffices to have a simple passage of the fabric through a cold bath of 0.5% detergent acid for 1 to 2 mm., to hydrophilize the fabric, and thus suppress the 8 to 12 hours' operation of alk. boiling Halsey E. Silliman

Staybrite steel in the dyeing industry F. Godber *Textile Mfr* 61, 512 (1935).—The qualities of Staybrite steel compared with other materials for plant construction and use are cited. In respect to this type of steel, whereas stainless steel has about 13% Cr and 0.3% C, stainless iron, 13% Cr and 0.1% C, Staybrite is a group of special steels, which besides having a high Cr content, have a high Ni content and also other metals. It differs from stainless steel in that it does not harden from quenching, it needs no heat treatment and avoids the complications of disintegration at both heavily manipulated joints and welded joints Halsey E. Silliman

Flax yarn qualities and types VII S. A. G. Caldwell *Textile Mfr* 61, 459 (1935), cf C A 30, 8637.—The mech. properties of flax yarns are shown by stress-strain diagrams. Halsey E. Silliman

Isolation of long silk filaments. Thomas Lonsdale *Silk J.* 12, No 139, 39 (1935).—A method is described of obtaining cocoon threads and single ultimate filaments of silk in long lengths Halsey E. Silliman

The isolation of cystine from wool hydrolyzates Gerrit Toennes and Mary A. Bennett *J. Biol. Chem.* 112, 39-50 (1935).—A suitable hydrolyzing agent for the prepn of cystine from wool is 50% H<sub>2</sub>SO<sub>4</sub>. The humin fraction can be removed from the hydrolyzate by salting out with Na<sub>2</sub>SO<sub>4</sub> and the cystine is rapidly pptd by cupric ion without oxidative or reductive changes. The best yield obtained was equiv to 44% of the total S content of the wool. Because of racemization the stage at which the amt. of L-cystine available for isolation is at its max. does not coincide with the completion of hydrolysis and a nebulizer test or a cyanide-nitroprusside test corresponding to the total S content is no proof of completeness of hydrolysis as far as liberation of cystine is concerned. No relation was found between the optical rotation of the hydrolyzate and the state of cystine under different conditions. Forty-two references A. P. Lothrop

Measurement of electrolyte content of textiles A. A. New *Textile Mfr* 61, 477-7, 508 (1935); cf *Ibid.* 58, 69 (1932).—The method has been developed more fully and successfully applied as a routine test for more than 2 years to all types of textiles used as insulation material. Details of the method, diagram of the app. used, and curves to det. the proper time of extn for the sp. cond. and pH data are given. Five g. of textile material is extd in 100

milliliters of H<sub>2</sub>O for 1 hr., and a portion of the ext. is placed in a cond. cell and the resistance detd. The math methods of calcg the results are also given. The sp. cond. of the aq. exts. of the following materials, in microhmhos at 25°, was found to be unwashed gray cotton yarn or sheeting 870-1000, dyed cotton 50-200, wool, dyed and undyed 100-200, natural silk 50-200, acetate rayon 30-100, jute 100-300 and washed gray cotton yarn or sheeting 50-100 Halsey E. Silliman

A new apparatus for the evaluation of waterproofed textiles Bundesmann *Melliand Textilber* 16, 128-31, 211-13, 331-2, 663-4, 739-40, 792-4 (1935).—The app. works on the following principle: the test pieces of the waterproofed fabrics are subjected to a continuous spray of rain from above, while being wiped at the same time by rubber wipers from below. The amt. of moisture taken up by the test piece is detd., as is the amt. going through the test piece. The testing equipment consists of 3 parts, a water tank to produce rain, 4 containers, over which the test pieces are stretched, mounted on a revolving cross arm and the driving mechanism. The cylindrical water tank is placed 150 cm. over the test pieces. The cross section of the tank is 1200 sq. cm., it has on its bottom plate 300 holes, each 0.4 mm. in diam. The size and the velocity of the drops can be regulated by the height of the water column in the tank. For the standard test a height of 7.5 cm is recommended; this gives a spray corresponding to a very severe shower. The test pieces, about 15 X 15 cm., previously conditioned at 65% relative humidity and 17° for at least 12 hrs. are weighed and then stretched over the 4 cup-like containers, 9.8 cm. in diam and 7.6 cm. high. The fabric is kept taut over the cup by stainless steel bands. The surface of the test piece subjected to the rain is 75 sq. cm. There is a faucet at the bottom of each cup. The cups are on a cross arm which revolves around a vertical shaft, 5 r. p. m. The circle described by the cup has an area of 1100 sq. cm. so that the test pieces are within the area of the spray. In each cup 4 rubber blades are pressed against the underside of the test pieces. These are so arranged that the test pieces are rubbed 10 times during each revolution. The driving mechanism consisting of a motor and speed reducer are in a waterproof housing. The entire assembly is mounted on rails, so that the fabrics can be placed over the cups outside of the rain, the motor started and then the assembly pushed under the rain. The test may be carried out for 5-30 min. At the end of the test the test pieces are removed from the cups, the surface water is shaken off 4 times, and then the fabrics are weighed. It is shown by a great no. of test results that this app. and method give reproducible figures for the detn. of the water resistance of impregnated wool, silk and leather. Photographs of the app. and various test pieces are given J. A. Szilard

Textile finishing from a chemical engineering standpoint J. E. Howarth *Textile Mfr* 61, 513 (1935).—Examples are given of the principles in the design and choice of equipment for various processes Halsey E. Silliman

Tinsel fabrics S. Bergneon *Textile Mfr* 61, 510 (1935).—The special qualities and limitations of lame fabrics which call for adaptations in scouring, dyeing, finishing and packing are described Halsey E. Silliman

An electrical measurement of moisture content of fabrics J. L. Spencer-Smith *Textile Mfr* 61, 494 (1935).—In methods of this type, based on the change of dielec. const. of the material, the material is placed in the field of a condenser and the increase of capacitance then is due to the difference of the material in a dry state and when the moisture is included Halsey E. Silliman

Characteristic defects of hosiery from mercerization J. Pinte and A. Delhalle *Revue ind. color* 39, 472-89 (1935).—A series of tests are described, illustrated by diagrammatic curves Halsey E. Silliman

The influence of metals in bleaching A. H. Pettinger *Textile Mfr* 61, 472 (1935).—In peroxide bleaching, it is essential to avoid Fe, Cu and Cr salts. Sn and Ti have no deleterious effects. Monel metal vessels can be used Halsey E. Silliman

Test to determine durability and resistance to water of certain umbrella fabrics. Viola Sykes and Katharine Cranor. *Am. Dyestuff Repr.* 24, 725-6 (1935).—Various umbrella fabrics were tested as to durability and resistance to water. All the fabrics tested contained Al, but the degree of surface waterproofing was not determined by the amount of ash contained in a fabric. None of the treated fabrics possessed the strength of the original fabric but cotton offered the greatest resistance to folding endurance and edge breaking. Closeness of weave, thickness of fabric, diam. of yarn and twists per in. influence water resistance. Twenty-four references.

Wool-scouring problems. A. T. King. *Textile Mfr.* 61, 483-4, 486 (1935).—A crit. exam. is given, confined to worsted yarns and only clear wools, of the requirements in scouring, in efficiency and in economy and from a phys. and chem. view of the fiber and subsequent processing of the material. The preservation of fiber strength is taken as the main factor of efficiency. H. E. S.

Reclaiming chlorinated dry-cleaning solvents by adsorption. Lawrence E. Stout and Arthur B. Tullman. *Ind. Eng. Chem.* 28, 225 (1936).—The authors in expts. carried out in the lab. and in a com. dry-cleaning plant with an azeotropic mixt. consisting of 70 mole percent  $\text{CCl}_4$  and 30 mole percent  $\text{C}_2\text{H}_5\text{Cl}$  as dry-cleaning solvents substituted adsorption by means of commercially obtainable adsorbing agents such as activated C, silica gel and activated magnesia for the customary distn. process to reclaim contaminated dry-cleaning fluids. The results indicate that activated C, silica gel and activated magnesia are excellent economical adsorbing agents, each having specific properties, in a combination of adsorption and filtration of contaminated chlorinated dry-cleaning solvents. E. W. Roth.

Rubberizing of balloon fabrics (Panem) 30. Prepn. of water for the textile industry (Ornstent) 14. Hydrogenation of tertiary alkyl-phenols to form hydroaromatic alcs. [use as lubricants for textile fibers and as dye intermediates] (U. S. pat. 2,026,668) 10. Rotary-drum app. for filtering and dewatering slimes such as those from wool (U. S. pat. 2,024,358) 1. Pressure filter and pump for filtering solvent liquid such as that used in "dry cleaning" (U. S. pat. 2,024,463) 1. Dyeing skins, etc. (Ger. pat. 618,902) 29. Azo-, amino- and acylamino-N-nitroamines (Brit. pat. 434,917) 10. Anthraquinone compds. and dyes (Fr. pat. 786,103) 10. 1,3-Dibromo-2-aminoanthraquinone-6- or -7-sulfonic acid (U. S. pat. 2,025,169) 10.

Dyes. I. G. Farbenind. A.-G. (Paul Wolff and Friedrich Heim, inventors). Ger. 621,476, Nov. 7, 1935 (Cl. 22c 7/02). One mol. of *p*-sulfobenzaldehyde or of a *p*-halobenzaldehyde is condensed with 2 mols. of an  $\alpha$ -substituted indole, and the leuco compd. so obtained is successively sulfonated and oxidized. The sulfonation step may be omitted if a sulfonic acid of an  $\alpha$ -substituted indole is used. The halogen atom or the  $\text{SO}_3\text{H}$  group linked to the benzaldehyde residue is then exchanged by fusing the product with a *p*-alkoxyaryl amine. Blue acid dyes for wool are obtained. Sp. processes are described.

Metalized dyes. I. G. Farbenind. A.-G. Fr. 787,428, Sept. 23, 1935. Dyes of formula  $\text{X(Y)NRN:NRZ}$ , where R is the radical of a diazotization component, R' is a coupling component of azo dyes which may contain azo groups, X and Y are H, alkyl, aralkyl, aryl or acyl, Z is OH or  $\text{NH}_2$  and -N(Y)X and Z are ortho to the azo bond, are converted to complex metal compds. in known manner. Thus 1-methyl-3-aminobenzene-4-azo-1-(4'-sulfophenyl)-3-methyl-5-pyrazolone is treated with Cr formate giving a product which dyes wool fast brownish yellow shades from an acid bath. Several examples are given.

Pigmentary dyes. I. G. Farbenind. A.-G. Fr. 787,453, Sept. 23, 1935. Dyes which are fast to oil, alc. and solvents are prep'd. by coupling diazotized amines of the formula  $\text{ROSCCH}_2\text{C}(\text{NH}_2)\text{CXCH}_2\text{CY}$ , where R is

1 alkyl, aralkyl or aryl and these may contain nonsolubilizing substituents, X and Y are H, alkyl, alkoxy, aralkyl, aryl, aralkoxy, aryl or halogen, and these may contain nonsolubilizing substituents, and Y can only be the same as X when X is H or alkyl, with an anilide of 2,3-hydroxynaphthoic acid having at least 2 alkyl groups as substituents in the  $\text{C}_6\text{H}_4$  ring. Thus, 3-amino-4-methyl-diphenyl sulfone  $\rightarrow$  2-hydroxynaphthalene-3-carbonyl-amino-2',5'-dimethylbenzene forms an orange-red ppt. which may be used as a paste or powder. Other examples are given.

Azo dyes. Miles A. Dahlen (to E. I. du Pont de Nemours & Co.) U. S. 2,025,094, Dec. 24. Dyes which have good fastness to washing, light and Cl are formed from components such as diazotized 4-nitro-2-aminonitroisole and 3,4,5-trimethoxyaniline of  $\beta$ -hydroxynaphthoic acid or the like.

Azo dyes. Arthur H. Knight (to Imperial Chemical Industries Ltd.) U. S. 2,025,211, Dec. 24. Azo dyes which are suitable for dyeing cellulose acetate are formed with 1- $\beta$ -hydroxyethylamino-5-naphthol as end component which may be coupled with various aromatic diazo compds. Several examples are given.

Azo dyes. Ernst Fischer (to General Aniline Works) U. S. 2,025,582, Dec. 24. Red dyes insol in water and which may be used for the prepn. of lakes fast to oils and light, and which are suitable for coloring natural or synthetic rubber are obtained by coupling diazotized 3-amino-4-methylbenzene-1-sulfonamide or the diazo deriv. of the corresponding methylamide, ethylamide, butylamide or cyclohexylamide or the like with 2',3'-hydroxynaphthyl derivs. such as the 1- or 2-aminonaphthalene derivs. an aminomethoxybenzene deriv. or the like (numerous examples being given).

Azo dyes. Max A. Kunz, Gerd Kochendoerfer, Kuno Maurach and Walter Lumbacher (to General Aniline Works) U. S. 2,025,991, Dec. 31. Dyes generally producing violet to blue to black shades and suitable for combination with Cr on wool are produced by coupling one mol. proportion of a diazo compd. with one mol. proportion of a monohydroxypyridinonaphthalene or by coupling one mol. proportion of a tetrazo compd. with two mol. proportions of a monohydroxypyridinonaphthalene, where either the diazo (or tetrazo) compds. or the monohydroxypyridinonaphthalenes used or both components contain at least one sulfonic acid group and the components are selected so that the resulting dyes are capable of forming complex metal compds. Several examples with details are given.

Azo dyes. I. G. Farbenind. A.-G. (Gerhard Schrader, inventor). Ger. 618,809, Sept. 10, 1935 (Cl. 22a. 1). Dyes insol. in water are obtained by coupling *o*-hydroxynaphthothiazole carboxylic acid arylides with diazotized arylamines in the substance or on the fiber, neither constituent contg. soly.-inducing groups. Thus, cotton is soaked in 1-(2'-methyl-7'-hydroxy-1',5',1'-naphthothiazole-6'-carbonylamino)-2-methylbenzene dissolved in alkali. The cotton is then treated with a soln. of diazotized 1,4-diethoxy-2-benzoylamino-5-aminobenzene. A fast blue effect results. Other examples are given.

Azo dyes. I. G. Farbenind. A.-G. Fr. 786,042, Aug. 24, 1935. Dyes are prep'd. by coupling diazotized 5-nitro-3-amino-1-x-benzene-3-sulfonic acids, (in which x is a nonacid substituent and the  $\text{SO}_3\text{H}$  group is esterified or contains an  $\text{NH}_2$  group, substituted or not) with 2-amino-naphthalenesulfonic acid or one of its substitution products, in acid medium. Thus, 5-nitro-2-amino-1-methoxybenzene-3-sulfonethylaniline  $\rightarrow$  2-amino-8-hydroxynaphthalene-6-sulfonic acid dyes wool in clear blue shades insensitive to artificial light. Several examples are given.

Azo dyes. I. G. Farbenind. A.-G. Fr. 786,086, Aug. 26, 1935. Dyes contg. metal are prep'd. by treating 8-hydroxyquinoline with agents furnishing metal and combining the metal compds. obtained with diazo compds. free from groups capable of forming complex metal compds., the constituents being chosen so that the dyes contain at least one acid group. Thus, the compd. obtained from 8-hydroxyquinoline and Cr formate is

combined with diazotized metanilic acid. The dye gives a fast brown-yellow on leather and orange shades on wool.

**Azo dyes.** I. G. Farbenind. A.-G. Fr. 786,193, Aug. 28, 1935. Dyes are prepared by coupling diazotized 3-nitro-4-chloro-2-aminophenol with 1-naphthol-3,8, or 4,8-disulfonic acid, and, if necessary, the dyes are treated with chroming agents. Bordeaux-red shades are obtained on wool before chroming and deep blue after chroming.

**Azo dyes.** Imperial Chemical Industries Ltd. Fr. 787,714, Sept. 27, 1935. Dyes sol. in water for acetate silk, wool, silk, silk charged with Sn, nitrocellulose lacquers, etc. colors and rapid drying remakes and oils are prepared by condensing a diazotized  $\beta$ -nitroaniline of the  $C_{10}H_7$  series with a  $N$ -sulfatoethyl (or propyl)- $N$ -3-alkoxyethyl deriv. of an amine of the  $C_{10}H_7$  series having a coupling position free, the resulting dyes contain no solubilizing groups other than the sulfato group. The dyes may also be prepared by coupling with the corresponding  $N$ -hydroxyethyl (or propyl) compd. (I) and afterward treating with concd.  $H_2SO_4$ . The compds. I are obtained by condensing a  $N$ -3-alkoxyethylamine with an ethylene or propylene chlorohydrin or with an alkylene oxide or by condensing a  $N$ -hydroxyethyl (or propyl)aniline with a 3-chloroethylalkyl ether or a toluene- $p$ -sulfonic-3-alkoxyethyl ester. Examples are given of the prepn. of  $N$ -3-hydroxyethyl- $N$ -3-ethoxyethyl- $m$ -toluidine, b. 183°,  $N$ -3-hydroxyethyl- $N$ -3-methoxyethylamine, b. 181-3°,  $N$ -3-hydroxyethyl- $N$ -3-methoxyethyl- $m$ -amino- $p$ -cresol methyl ether, b. 192-4°, also their sulfonic esters and of dyes prepared from these esters and diazotized  $p$ -nitroaniline,  $\alpha$ -chloro- $p$ -nitroaniline, 2,4-dinitroaniline and 6-chloro-2,4-dinitroaniline.

**Azo dyes.** See pour l'ind. chim. a. Bile. Fr. 786,106, Aug. 27, 1935. Various azo compds. are coupled with phenols and their derivs. capable of coupling in the 2 or 6-position and which are substituted in the 3- and 4 positions by an atom of halogen, alkyl, O-alkyl or NH-acetyl groups, with the exception of products in which the 3- and 4 positions are occupied by 2 alkyl groups or by an alkyl group and an atom of halogen. Thus, sulfanilic acid  $\rightarrow$  3,4-dichlorophenol dyes wool orange-yellow and 1-amino-naphthalene-4-sulfonic acid  $\rightarrow$  3,4-dichlorophenol a red-brown.

**Azo dyes.** See pour l'ind. chim. a. Bile. Fr. 787,692, Sept. 26, 1935. Dyes are prepared by coupling diazotized aromatic amines containing a nitro or acetylamin group in the para and an SO<sub>2</sub>R group (R is alkyl or aralkyl, substituted or not) in the ortho position with respect to the diazotizable group, with amines of the  $C_{10}H_7$  and  $C_{11}H_9$  series and their substitution products capable of coupling. The NO<sub>2</sub> group, if present, may be reduced, acetylated, alkylated or diazotized and coupled with appropriate components. Thus, 2-amino-5-nitrophenyl ethyl sulfone  $\rightarrow$  ethoxyethylamine dyes acetate silk in pure violet-red shades. Several examples are given.

**Azo dyes.** See pour l'ind. chim. a. Bile. Ger. 618,749, Sept. 14, 1935 (Cl. 22a 2). Dyes contain one or more metals and are free from COOH groups are obtained by treating azo dyes free from COOH groups and groups containing SO<sub>3</sub>, such as HSO<sub>3</sub>, HSO<sub>3</sub>, SO<sub>2</sub>NH<sub>2</sub>, etc., and in which the coupling C atom is ring-bound, with hydrides of metals in the presence of alkalis. Thus, the dye from diazotized 2-amino-1-naphthol and 2-naphthol is heated with Cr(OH)<sub>3</sub> in the presence of KOH to give a product imparting a blue-violet color to Japanese lacquer, suitable for coloring Al foil. Other examples and a table of dye components, metals and colors produced are given.

**Azo dyes.** See pour l'ind. chim. a. Bile. Ger. 621,304, Nov. 6, 1935 (Cl. 22a 1). Diazotized 4-nitro-2-chloroaniline is coupled with an  $\alpha$ -alkylsulfonic acid of a primary amine of the benzene series containing at the most one additional substituent, which must not be SO<sub>3</sub>H and must not be in  $m$ -position to the NH<sub>2</sub> group. The alkyl-sulfonic group is split off in known manner after the coupling. Orange or red dyes applicable to cellulose esters and ethers are obtained. Examples are given.

**Azo dyes, dyeing.** I. G. Farbenind. A.-G. (Max Balthes, Kurt Bricsewitz and Arthur Zitscher, inventors). Ger. 621,078, Nov. 1, 1935 (Cl. 22a 1). See U. S. 1,992,461 (C. A. 29, 27357).

**Chromatable azo dyes.** Georges Kopp and Pierre Petitcolas (to Compagnie nationale de matieres colorantes et manufactures de produits chimiques du Nord reunes etablissemens Kuhlmann). U. S. 2,024,864, Dec. 17, 1935. Dyes of various shades capable of being after-chromed are obtained by diazotizing an initial material such as 1-alkoxy-2-amino-6-nitrobenzene substituted in the 4-position by a nitro, sulfonamide or sulfonic acid group and coupling with a naphthol, phenol, naphthylamine, aminonaphthol, pyrazolone or  $\beta$ -diketone. Various examples are given.

**Metal compounds of azo dyes.** I. G. Farbenind. A.-G. (Hans Krmkalla and Walter Kuhne, inventors). Ger. 621,473, Nov. 7, 1935 (Cl. 22a 2). Salts of haloacetylated lower aliphatic acids with metals such as Cr or Cu are used as the source of metal. These salts react more quickly than those ordinarily used. Examples are given.

**Metaliferous azo dyes.** I. G. Farbenind. A.-G. (Guido von Rosenberg, inventor). Ger. 621,079, Nov. 1, 1935 (Cl. 22a 1). The azo dyes are obtainable by coupling 1 mol. of tetrazotized 3,3'-dimethoxy-4,4'-diaminodiphenyl with 2 mols. of a naphthol are heated with a heavy-metal salt of a carboxylic acid of high mol. wt., e. g., with a Cr, Cu, Fe, Ni, Co or Pb stearate, oleate, laurate, naphthenate or sebacate. The reaction may be effected in the presence or absence of an org. solvent, e. g., xylene or trichlorobenzene. Pigment dyes, generally violet or blue, are obtained. Sn processes are described.

**Azo dyes containing metal.** I. G. Farbenindustrie A.-G. Brit. 435,040, Sept. 13, 1935. Addn. to 434,777 (C. A. 28, 4919). The dyes are made by treating  $\alpha$ -hydroxyquinoline (I) or its derivs. with an agent supplying heavy metal and combining the product with a diazo compd. free from groups that form complex metal compds., the components being so chosen that the resulting dyes contain at least 1 acid group. In examples, (1) the Cr compd. obtained by boiling a HCl soln. of I with Cr formate, fluoride, acetate or sulfate is coupled with diazotized metanilic acid in alk. soln., the compd. from I and (AcO)<sub>2</sub>Fe may be similarly coupled, the products are suitable for dyeing leather, and (2) the Cr compd. obtained by boiling a neutral soln. of I-5-sulfonic acid with Cr formate is coupled with diazotized  $p$ -nitroaniline in AcOH soln., the product dyes wool.

**Copper-containing azo dye.** Imperial Chemical Industries Ltd. and Mordecai Mendoza. Brit. 435,315, Sept. 19, 1935, Fr. 789,445, Sept. 3, 1935. The dye is made by coupling 2 mol. proportions of diazotized 6-chloro-2-aminophenol-4-sulfonic acid with 1 mol. proportion of the urea of Jacod in aq. alk. med. soln., e. g., Na CO<sub>3</sub>, and treating the resulting dye with Cu or an agent yielding Cu, e. g., CuSO<sub>4</sub>. It dyes cotton blue-red shades fast to light.

**Monoazo dyes.** Henry Jordan and Miles A. Dahlen (to E. I. du Pont de Nemours & Co.). U. S. 2,024,358, Dec. 17, 1935. Dyes of various colors (various red shades in most of the examples given) which are suitable for development on the fiber and which can be discharged to a pure white, suitable for dyeing cellulose esters such as cellulose acetate, are formed by diazotizing  $p$ -aminodimethylbenzamide or other amine of the general formula R<sup>1</sup>NR<sup>2</sup>NR<sup>3</sup>NH<sub>2</sub> in which R<sup>1</sup> and R<sup>2</sup> represent H or alkyl groups when X represents CO and R<sup>3</sup> represents an alkyl group and R<sup>1</sup> represents H or an alkyl group when X represents SO<sub>2</sub>, and R<sup>3</sup> represents a phenylene radical, and by coupling with  $m$ -toluidine, cresidine or other amine of the benzene or naphthalene series having a free  $\beta$ -position to the amino group. Numerous examples are given.

**Trisazo dyes.** I. G. Farbenind. A.-G. (Hermann Klingenstein, Eugen Glettenberg and Oscar Goos, inventors). Ger. 621,186, Nov. 2, 1935 (Cl. 22a 10). See Fr. 765,752 (C. A. 28, 70349).

**Mordant azo dyes.** Durand & Huguennin A.-G. Brit. 434,918, Sept. 11, 1935. Addn. to 432,287 (C. A. 29, 33619). Dyes are made by coupling H acid in an acid

medium with a diazotized aminoazo compd. of type  $R_1N:NR_2NH_2$ , then in an alk. medium either with a diazo compd. of the same type or with a diazo compd. of an amine  $R_1NH_2$ , where  $R_1$  is a  $C_{6H_5}$  nucleus which may contain substituents, particularly  $SO_3H$ ,  $NO_2$ ,  $COOH$  or halogen,  $R_2$  is a sulfonated  $C_{6H_4}$  nucleus and  $R_3$  is a  $C_{6H_5}$  nucleus that contains  $OH$  and  $COOH$  in *o*-position and may contain further substituents, e. g.,  $Cl$ ,  $CH_3$ , halogen. They yield fast blue-green to green shades in chrome printing on cotton. In examples, 3-nitroaniline-4-sulfonic acid — salicylic acid (reduced) — (alk.)  $HCl$  — (alk.) *o*-, *m*- or *p*-sulfamic acid, 4-chloroaniline-3-sulfonic acid, aniline-2,4-disulfonic acid or 3-amino-4-sulfolobenzeno-azo-salicylic acid.

**Vat dyes** Wilhelm Eckert and Otto Braunsdorf (to General Aniline Works). U. S. 2,026,026, Dec. 31. Dyes, examples of which dye cotton from a hyposulfite vat gray to brown shades, are obtained by heating, to about 140–250°, an arylamino substituted 1,4,5,8-naphthylenedi(arylimidazole) with an acid-condensing agent such as  $AlCl_3$  either alone or mixed with a flux such as  $NaCl$ . Several examples with details are given.

**Vat dye paste for textile printing** Herbert A. Lubs and John E. Cole (to E. I. du Pont de Nemours & Co.) U. S. 2,024,973, Dec. 17. An unsubstituted alkali metal anthraquinone-sulfonate is used as an assistant. U. S. 2,024,974 relates to the similar use of an alkali metal anthraquinone-sulfonate and gives numerous examples and U. S. 2,024,975 relates to the addition of anthraquinone-2-sulfonic acid or an  $NH_4$  or alkali metal salt of this acid to various printing pastes, and also gives numerous examples.

**Separating vat dyes** I. G. Farbenindustrie A.-G. Brit. 434,815, Sept. 9, 1935. Addn. to 341,357 (C. A. 25, 5040) and 369,604 (C. A. 27, 2308). Sepn. of mixts of isomeric 1,4,5,8-naphthylenediarylimidazoles, obtainable according to Brit. 257,244 (C. A. 20, 1527), 265,232 (C. A. 22, 322) and 265,904 (C. A. 22, 503) into their constituents is effected by first heating with an alkali metal compd. of strong alk. action, except caustic alkali, e. g., sodamide,  $NaOLi$ , in presence of a  $H_2O$ -miscible solvent or diluent, e. g., alc., then sep. the compds. obtained by means of their different solubilities and recovering the vat dyes from these sep. compds.

**Anthraquinone vat dyes** Max Utringer and Max Bommer (to Soc. pour l'ind. chim. A. Bille). U. S. 2,026,150, Dec. 31. See Brit. 414,620 (C. A. 29, 612).

**Vat dyes of the anthraquinone series.** Paul Nawia-ky and Berthold Stein (to General Aniline Works). U. S. 2,025,546, Dec. 24. Dyes giving green shades of good fastness on cotton are obtained by heating compds. such as 1,2'-dianthraquinonyl sulfide or a chloro deriv. with  $ZnCl_2$  (suitable at about 300° and in the presence of a catalyst such as  $Cu$  chloride). The crude dyes may be purified by treatment with alk. solns. of dextrose or by recryst. or by dissolving in  $H_2SO_4$  and subsequent fractional pptn.

**Dyes formed by heating  $\alpha$ -cyanoarylamides with metal compounds.** Joseph F. Thorpe, Reginald P. Linstead and John Thomas (to Imperial Chemical Industries Ltd.) U. S. 2,025,791, Dec. 31. By heating an  $\alpha$ -cyanoarylamide such as  $\alpha$ -cyanobenzamide to 200–300° in the presence of an oxide, sulfide or carbonate of a multivalent metal such as those of Fe or Mg, or with the free metal, dyes are obtained which appear to be dehydration and condensation products of intermediately formed compds. such as 1-hydroxy-3-imidazoindole.

**Anthraquinone dyes.** Chemische Fabrik vorm. Sanda Ger. 621,369, Nov. 6, 1935 (Cl. 223, 3 02). Water-sol. blue, violet or brown wool dyes are prepd. (1) by introducing a  $SO_3H$  group or groups by standard processes into the known  $N,N'$ -dianthraquinonyldiiminodiazol compds. obtainable by oxidizing arylaminoanthraquinones or (2) by oxidizing a sulfonated arylaminoanthraquinone, unsubstituted in the aryl residue in *p*-position to the  $NH$  group. Method (1) may be effected with oleum at about 45°; method (2) may be effected with  $H_2SO_4$  and  $MnO_2$  at atm. temp. Examples are given.

**Anthraquinone dyes.** Chemische Fabrik vorm. Sanda. Fr. 787,400, Sept. 21, 1935. Bright dyes are

prepd. by condensing aromatic bases of which the 2 positions ortho to the  $NH$  group are substituted, and which may also contain other substituents, with anthraquinone derivs. having one or more groups replaceable by an arylamino group, and treating the products obtained by the usual methods for anthraquinone dyes. Thus, they may be sulfonated or treated with aldehydes in acid or neutral medium or converted to anthrapyridines, anthrapyrimidones or anthrapyrindones. In an example *C*-carbethoxy-2-methyl-4-bromoanthrapyrindone is heated with 1-amino-2,4,6-trimethylbenzene in the presence of  $AcOK$  and  $Cu$  and the product is sulfonated giving a product which dyes wool bright yellowish red shades. Several examples are given.

**Benzanthrene dyes** Soc. pour l'ind. chim. A. Bille. Fr. 787,448, Sept. 23, 1935. Dyes are prepd. by treating the *B*-1-bromo-2-methylbenzanthrones prepd. by the process of Fr. 787,175 (C. A. 30, 1070) with alk. condensing agents, if desired after causing the atom of Br to react with appropriate compds. of S, Se or 1-aminoanthraquinone.

**Chromiferous dyes** Soc. pour l'ind. chim. A. Bille. Brit. 434,835, Sept. 10, 1935. See U. S. 1,990,257 (C. A. 29, 1906).

**Tricarbocyanine dyes, intermediates.** Leslie G. S. Brooker (to Kodak Ltd.) Brit. 435,232, Sept. 18, 1935.

**Pyridinium quaternary salts** are prepd. by causing pyridine (1) to react with a 2-iodopyridine alkyl halide or with a 2-iodoquinoline alkyl halide. Dyes are prepd. by condensation of these salts with quaternary cycloammonium salts having a reactive alkyl group, the pyridinium ring appears to undergo fusion, 2-aminopyridine or 2-aminoquinoline alkyl halides being left as by-products. Alternatively, dyes are prepd. by condensing a quaternary cycloammonium salt having a reactive alkyl group with pyridine in the presence of 2-iodopyridine alkyl halide or 2-iodoquinoline alkyl halide. Among examples, 1-methylbenzothiazole ethyl *p*-toluenesulfonate, 2-iodoquinoline ethiodide and *f* are heated together to form the dye 2,2'-diethylthiobenzocarbocyanine iodide.

**Sulfur dyes, intermediates.** Soc. pour l'ind. chim. A. Bille. Brit. 435,342, Sept. 19, 1935. An  $N$ -aryl deriv. of a hydrocarbon that contains at least 3 condensed rings or of a carbazole contg. the nucleus of such a hydrocarbon, or a hydroxy deriv. of such an  $N$ -aryl deriv., is treated with S or an agent yielding S to produce sulfurized dyes. Among examples, (1) *p*-hydroxyanthracene is condensed with *p*-aminophenol to give 2-(4'-hydroxyphenyl)aminoanthracene which is heated with S in cyclohexanol, and (2) quinoxalinecarboxamide is condensed with the carbazole derived from 1-phenylaminoanthracene and, after reduction, the leuco compd. is sulfurized. Examples of dyeing cotton with the product of (1) in the usual  $Na_2S$  bath and with the product of (2) in a hyposulfite vat are given.

**Pigments of the indanthrene series.** Frank W. Johnson (to E. I. du Pont de Nemours & Co.). U. S. 2,026,657, Jan. 7. Highly dispersed dyes easily reducible to the leuco form and which are suitable for dyeing cloth by the "pigment pad-jig reduction" process are produced by treating a  $H_2SO_4$  soln. of a compd. of the indanthrene series such as *N*-dihydro-1,2,2',1'-anthraquinonazine with  $Cl_2$  and then with a sol. cycloaliphatic compd. such as cyclohexanol under such temp. conditions that the  $H_2SO_4$  is substantially inert. Several examples with details of procedure are given.

**Dye printing pastes** Hermann Berthold (to General Aniline Works). U. S. 2,024,502, Dec. 17. A mercapto-benzothiazole or a mercaptobenzimidazole is used as a stabilizer. Several examples are given.

**Printing pastes** I. G. Farbenind. A.-G. Fr. 788,012, Aug. 24, 1935. Printings of used, azo dyes are obtained on fiber by using an azo dye prep. contg. the usual thickening agents and a compd. capable of liberating acids during steaming and developing the dyes by steaming. Suitable compds. liberating acid include  $K$  fluoro-sulfonate,  $Na$  bromoacetate,  $K$  salt of the mono-*Me* ester of oxalic acid and  $Na$  acetoxyethanesulfonate.



Printing pastes, printing fabrics. E. I. du Pont de Nemours & Co. Brit. 434,736, Sept. 9, 1935. The pastes comprise as principal component a yellow or orange vat dye having only moderate printing qualities and as subsidiary color component a yellow or orange 1,8-bis-(acylamino) anthraquinone exhibiting a greater vat solubility than the principal dye. Alternatively, the principal and subsidiary components may each be a mix of suitable vat dyes as specified. Among examples, (1) a soln. of crude 1,8-dibenzamidoanthraquinone (1 part) in 95% H<sub>2</sub>SO<sub>4</sub> at 5° is poured into ice water, filtered and washed free from acid, the filter cake is suspended in H<sub>2</sub>O, warmed to 80-85° with addn of a dil. alk. hypochlorite soln., so maintained for 2-4 hrs., again filtered and washed free from alkali, then made into an aq. paste with addn of "Leucanol" or other wetting agent, milled and mixed with a similarly prep'd paste of 1,8-dibenzamidoanthraquinone (2 parts), and (2) cotton fabric is printed with an aq. paste contg. wheat starch, British gum, gum tragacanth, K<sub>2</sub>CO<sub>3</sub>, Na formaldehydesulfonate, glycerol and the dye paste obtained in (1) and the printed fabric is dried, steamed for 2-8 min. at 101-102°, oxidized in dil. Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> soln. or in cold running H<sub>2</sub>O, soaped, rinsed and dried.

Arylides of 4-hydroxybiphenyl-3-carboxylic acid. Oskar Haller and Hermann Morechel (to General Aniline Works). U. S. 2,025,587, Dec. 24. See Ger. 618,213 (C. A. 30, 4709).

Dioxazine compounds. Georg Kalscheur and Werner Zerweck (to General Aniline Works). U. S. 2,024,525, Dec. 17. By the reaction of 2 mol. proportions of a nitro- $\alpha$ -naphthol with the benzene or naphthalene series on 1 mol. proportion of a halogenated quinone of the benzene or naphthalene series, products are obtained such as dichloro-di- or tetra nitro-triphenoldioxazine which dye cotton from an acid bath brown-orange shades.

Aminoanthraquinone compounds. I. G. Farbenindustrie A.-G. Brit. 434,906, Sept. 11, 1935. 1,4-Diaminoanthraquinones in which the 1- and 4-groups may be substituted by alkyl, aralkyl, cycloalkyl or aryl or heterocyclic groups are obtained by condensing leuco-1,4-diamino-, 1,4,5-triamino- or 1,4,5,8-tetraaminoanthraquinones in the absence of reducing agents with at least 2 mol. proportions of a primary amine free from salt-forming groups, or with at least 1 mol. proportion of each of more than 1 different such amines. The condensation takes place at atm. pressure, if desired in the presence of a diluent and if in the presence of air gives the substituted amino derivatives and if not gives the leuco compounds, which may be oxidized with air, O, FeCl<sub>3</sub> or H<sub>2</sub>O<sub>2</sub> and in presence of Cu. The products are suitable for dyeing cellulose esters, artificial combs, lacquers, waxes, oils and petroleum fractions. According to examples, leuco-1,4-diaminoanthraquinone is condensed with BzNH<sub>2</sub>, cyclohexylamine, PhNH<sub>2</sub>, ethanoloamine and benzylamine.

Quaternary ammonium compounds. Gytung. I. G. Farbenindustrie A.-G. Brit. 435,388, Sept. 20, 1935. Polyethylenepolyamine mixtures contg. substantial proportions of compounds of the formulas H<sub>2</sub>N(C<sub>2</sub>H<sub>4</sub>NH)<sub>2</sub>, C<sub>2</sub>H<sub>5</sub>NH<sub>2</sub>, and (C<sub>2</sub>H<sub>5</sub>NH)<sub>3</sub>, in which  $n$  is greater than 2, are converted into quaternary NH<sub>4</sub> compounds by treatment with alkylating or aralkylating agents. In an example, a mix is freed by distn. from the fraction boiling up to 220° under 10 mm. pressure and the residue is treated with Me<sub>2</sub>SO in an aq. medium kept alk. with Na<sub>2</sub>CO<sub>3</sub>. Alkylation may be effected in 2 stages, tertiary amines being formed in the 1st stage by treating the starting material with an alc. and HCl, and quaternary NH<sub>4</sub> compounds being formed in the 2nd stage by further alkylation. The products are useful for after-treating cellulose materials dyed with substantive dyes, to improve the fastness of the dyes. Cf. C. A. 29, 4375.

Arylamides (dye intermediates). Herbert A. Lubs, Emmet F. Hinch and Miles A. Dahlen (to E. I. du Pont de Nemours & Co.). U. S. 2,025,116, Dec. 24. An alkylmercaptopyrrolidine such as a toluene soln. of  $\beta$ -methylmercaptopyrrolidine is caused to react with a hydroxyaryl carboxyl chloride, or a hydroxyaryl carboxylic acid such

as 2,3-hydroxynaphthoic acid in the presence of a dehydrating agent such as PCl<sub>5</sub>, to produce arylamides suitable for use in the manufacture of ice colors and insol. azo pigments. Various examples with details of procedure are given. U. S. 2,025,117 relates to azo dyes of various colors produced by coupling a diazotized arylamine with an alkylmercaptopyrrolidine of 2,3-hydroxynaphthoic acid. Numerous examples are given.

Intermediates for polymethine dyes. Imperial Chemical Industries Ltd. Ger. 621,494, Nov. 7, 1935 (Cl. 22-3). See Brit. 344,499 (C. A. 26, 315).

Dyed cellulose esters. Soc. pour l'ind. chim. à Bâle Ger. 620,246, Nov. 11, 1933 (Cl. 8m. 2.02). Cellulose is impregnated with a dye which contains an acid group and has only a low or no affinity for cellulose. The latter is then acylated, whereby a cellulose ester dyed fast to washing is obtained. Patterns may be produced by applying the dye and (or) the acylating agent to parts only of the material. The dye must be one which is not converted into a nondyeing compound under the acylation conditions. Examples are given in which cellulose is acetylated after it has been impregnated or printed with the following dyes: Kiron red G, Alzarna fast green and Kiron red 6B.

Dyeing. Daniel Bertho and Richard André Ger. 621,038, Oct. 31, 1935 (Cl. 8m. 12). In producing azo dyes on vegetable fibers or silk with the aid of 2,3-hydroxynaphthoic arylides, the dyed materials are rinsed in the cold, boiled in a circulated soap soln., and then subjected to a stamping or ponding treatment in a hot soap soln. which is gradually displaced by water. The materials may be packed in sacks for the stamping treatment or treated in a loose form, and the soap soln. used may contain fullers' earth or like mineral or a fat solvent, e.g., CCl<sub>4</sub> or CHCl<sub>3</sub>. The treatment removes unfixed dye from the materials, and yields dyings fast to wet rubbing.

Dyeing. Soc. pour l'ind. chim. à Bâle Ger. 621,359, Nov. 6, 1935 (Cl. 8m. 2.01). The known products obtained by treating alkylated cellulose, dyed or undyed, with heterocyclic compounds, contg. one or more groups :NCCX, where X is halogen, have been found to be inapplicable to indigo dyes provided that the application of the dye is effected at below 30° under otherwise usual conditions. Details are given of the utilization of this property in the production of white or colored effect threads in mixed fabrics.

Dyeing. I. G. Farbenind. A.-G. (Hans Kämmerer and Hermann Winkler, inventors). Ger. 621,451, Nov. 7, 1935 (Cl. 8m. 7). For dyeing animal fibers, use is made of the azo dyes obtainable by coupling 1-(3'-sulfonyl)-3-methyl-5-pyrazolone with the diazo compounds from 2-aminophenols substituted in the 4-position by halogen or NO<sub>2</sub>, and in the 6-position by halogen, NO<sub>2</sub> or CH<sub>3</sub>. Dyeing is effected by the single-bath process in the presence of a heavy-metal compound, e.g., K<sub>2</sub>CrO<sub>4</sub>.

Dyeing. Soc. pour l'ind. chim. à Bâle Ger. 621,452, Nov. 7, 1935 (Cl. 8m. 12). In producing azo dyes on the fiber, the material is first impregnated in the usual way with an arylide of an  $\alpha$ -hydroxy carboxylic acid which couples in  $\alpha$ -position to the OH group. It is then treated with a soln. of a diazotized amine of the formula ROCH<sub>2</sub>CONHR'NH<sub>2</sub>, where R is a phenyl residue which may be substituted, e.g., by Me or Cl, and R' is a  $\beta$ -phenylene radical substituted by two alkyl groups in  $\beta$ -position to one another. The acidity of the diazo soln. must be between pH 5 and 6.5. Blue or violet dyings are produced. Sp. processes are described.

Dyeing regenerated cellulose rayon. Imperial Chemical Industries Ltd. Ger. 618,329, Sept. 6, 1935 (Cl. 8m. 1.05). See Brit. 420,993 (C. A. 29, 8357).

Dyeing elastic cloth. Dogman, S. A. Fr. 787,224, Sept. 19, 1935. App. is described for stretching the cloth during dyeing.

Dyeing textiles. Deutsche Hydrierwerke A.-G. Brit. 434,911, Sept. 11, 1935. See Fr. 782,872 (C. A. 29, 7040).

Discharging dyings on wool with sulfonylates. Robert Haller (to Soc. pour l'ind. chim. à Bâle). U. S. 2,024,038, Dec. 10. See Fr. 780,588 (C. A. 29, 6077).

**Printing cotton.** Radmer & Co A-G (C. Bener, inventor). Ger 621,109, Nov. 1, 1935 (Cl. 8n. 5 01). Crude, bleached or mercerized cotton fabric is printed with a paste contg. an oxidizing agent and an ester salt of a leuco compd. of a vat dye, and the dye is then developed in the cold with  $H_2SO_4$  of parchmizing strength, e. g., 50°Bé. The coloring of the fabric is thus combined with the known processes for producing opalescent, linen-like or transparent finishes with the aid of  $H_2SO_4$ . Modified effects may be produced by using gum arabic as a thickening agent in the printing paste, whereby the printed parts are protected from the action of the acid, but the development of the dye is not prevented. As a further modification, the fabric may be mercerized after the  $H_2SO_4$  treatment. Sp. processes are described.

**Stripping dyed materials, sulfonium and phosphonium compounds.** I G Farbenindustrie A-G. Brit 434,810, Sept. 3, 1935. Dyes are stripped from textiles, leather, paper, etc., by the use of  $H_2O$ -sol. org. compds. derived from quaternary S or quaternary P and contg. at least 1 aliphatic residue of at least 6 C atoms, but no sulfonic groups, e. g., methylthiethylsulfonium hydride (I) and its Me sulfonic acid salt, diethyloctadecylsulfonium chloride (II), trimethyldodecylphosphonium bromide (III), dimethylethylphosphonium chloride (IV) and diethyldodecylphosphonium sulfate (V). I is made by causing methyl ethyl thioether to react with cetyl iodide and converting the product into the hydride, II by causing diethyl thioether to react with octadecyl chloride, dipropyldodecylsulfonium bromide by treating dipropyl thioether with dodecyl bromide, III by causing trimethylphosphine to react with dodecyl bromide, IV by treating dimethylethylphosphine with cetyl chloride, V by causing diethylethylphosphine to react with oleyl halide and converting the product into the hydride from which the corresponding sulfate is formed.

**Apparatus for bleaching and dyeing textiles.** Gustave Cotel. Fr. 787,841, Sept. 30, 1935.

**Apparatus (with a rotating applicator disk) for applying liquids such as dye solutions, etc., to yarns or filaments.** Wm. Whitehead (to Celanese Corp. of America). U. S. 2,025,079, Dec. 24. Structural and operative details.

**Device for testing materials for the fastness of their colors to light.** N. V. Philips' Gloeilampenfabrieken. Brit. 435,097, Sept. 13, 1935.

**Obtaining vegetable fibers.** Leo Nordmann. Ger. 621,115, Nov. 1, 1935 (Cl. 2n. 2). In the recovery of fibers suitable for spinning from bast-fiber material, e. g., flax straw, the air-dried or dried material is treated with  $O_2$  or ozonized air under such conditions that the material and (or) the gas is continuously or periodically agitated. Preferably, the temp. of the gas is reduced periodically to about 2°. The fibers and the incrusting materials are then sep'd by known mech. methods.

**Obtaining fiber from plant materials such as flax, jute, china grass, etc.** Joseph A. Manahan. U. S. 2,026,584, Jan. 7. For liberating plant fiber from connective tissue, the raw material is treated with water contg. an emulsifying agent such as a tallow soap and is subsequently treated with an aq. soln. of a mild alk. salt such as  $Na_2PO_4$  and is then rinsed and treated with a softening agent such as a dil. soap and  $CaCl_2$  soln. prior to spinning.

**Bleaching fibers.** B. Laporte Ltd and Isaac E. Weber. Brit. 434,809, Sept. 5, 1935. Vegetable textile fibers are bleached in a bath consisting of a peroxide to which a metaphosphate or  $HPO_4$  has been added.  $Na$  silicate,  $Na_2CO_3$ ,  $NaOH$ , etc., may be added to give the bath any desired alkali.

**Treating textile fibers.** Soc. Nobel Française. Fr. 787,907, Sept. 20, 1935. Textiles are given the elasticity of silk or wool by treating them with a soln. composed of the mixt. of a condensation product of urea and  $CH_3O$  with a condensation product of another compd. and  $CH_3O$ . The other compd. is preferably thiourea or dicyanodiamide.

**Oiling textile fibers.** I. G. Farbenind. A-G. (Joseph Nüsslein and Heinrich Ulrich, inventors). Ger. 621,396, Nov. 7, 1935 (Cl. 2n. 5). As lubricants for fibers, use is made of the condensation products of primary, secondary

or tertiary amines, contg. one or more OH groups, with carboxylic, sulfonic or sulfo-carboxylic acids of high mol. wt. Typical products are obtainable by condensing  $OH\cdot C_2H_4NH_2$ ,  $(HO\cdot C_2H_4)_2N$  or  $(HO\cdot C_2H_4)_3N$  with oleic acid. The products may be used as aq. emulsions.

**Preserving fibers.** Kammgarnspinneri Stöhr & Co A-G and Ehrhart Franz. Fr. 787,342, Sept. 20, 1935. Animal and vegetable fibers and rayon are preserved against the attack of parasites by means of inorg. compds. such as derivs. of P, Se, Sr, Ba and Zn, or org. substances such as derivs. of chloro phenols, thioureas, rhodanides, thiuronium salts and chloroanilides and organo-metallic compounds such as triphenylphosphine, triphenylstibine, their halides, oxides and salts of the corresponding acids. The products are applied when the receptivity of the material is high, e. g., during or after washing, bleaching, dyeing, sizing or oiling, according to the soly. of the treating agent in water, oil, etc.

**Soaking natural silk fibers to soften them.** Philip Kaplan. U. S. 2,025,980, Dec. 31. The fibers are soaked in a bath contg. high titer fats which have been sulfonated to the extent of 5% or more combined  $SO_2$ .

**Treating yarns.** Joseph Brandwood. Brit. 435,395, Sept. 16, 1935. Doubled textile yarns impregnated with rubber or analogous dispersions are made by impregnating the individual yarns under differential pressure, clearing them from excess dispersion by means of a gas under pressure and doubling while still moist. App. for impregnation is described.

**Producing fullness in yarns such as those of wool.** Geo. M. Pearsall. U. S. 2,025,175, Dec. 24. A roving is twisted to a higher degree than the normal roving twist, spun, subjected to the action of a boiling liquid, dried, twisted in the reverse direction to remove part of the twist imparted by the spinning, redrafted to displace the fibers longitudinally, and spun.

**Lubricating natural and artificial threads and filaments.** Aelheid G. Bouthuys (to American Enka Corp.). U. S. 2,025,434, Dec. 24. Various materials are treated with a stable, homogeneous "soln." consisting of a mineral oil, an emulsifying agent such as a mineral oil sulfonate in an amt. insufficient to inhibit the lubricating properties of the oil, addnl. emulsifying and lubricating agents including alkali metallic and org. base soaps such as K oleate and the triethanolamine soap of oleic acid and a substantially anhyd. hygroscopic solvent for the soaps, so that the solvent serves to absorb from the materials treated any moisture which would normally form an emulsion with the oils. U. S. 2,025,435 relates to similar compns. contg. oil, a mineral oil sulfonate, soaps and a solvent such as the monoethyl ether or monobutyl ether of diethylene glycol.

**After-treatment of rayon filaments.** Vereinigte Glanzstoff-Fabriken A-G (Hugo Elling, inventor). Ger. 621,395, Nov. 6, 1935 (Cl. 2n. 6 08). Freshly spun rayon filaments are given their first washing while they are wound on porous reels or on perforated reels having a porous coating. The filaments are then dried and re-wound, with twisting, onto similar reels, on which the after-treatment is completed. More uniform products are thus obtained.

**Rendering silk and rayon articles mat.** Fritz Schuster. Fr. 787,767, Sept. 28, 1935. The treating liquid is projected by aspersion onto the articles, particularly stockings, while these are mounted and stretched on a form.

**Finishing agents for textiles.** Farb- und Gerbstoffwerke Carl Flesch, Jr. Fr. 787,815, Sept. 30, 1935. Agents resistant to water contain in addn. to glue or other albuminoid materials, substances which cause sepn. of the glue, etc., in an insol. form and that only after a certain time or by the application of heat. Thus, to a soln. of paraffin, glue and  $Al(OAc)_3$ , is added  $SnCl_4$  and the mixt. is regulated to 16°Bé. The soln. is applied to cotton velour which is afterward heated to 120°. In another example  $CaCl_2$  is added to an emulsion of glue and spermaceti.

**Apparatus for fulling and other processing of textiles.** Kraft Göbel and Georg Friede. Ger. 618,640, Sept. 12, 1935 (Cl. 8a. 9 70). Addn. to 554,773 (C. A. 28, 12021).

Moore fabrics Albert Mellor and Ralph J. Mann (to Celanese Corp. of America). U. S. 2,024,177, Dec. 17. Various details of manufacture and use of a pressure surface for use in the production of moiré effect fabrics are described.

Transparent fabric from muslin, etc. Geo. Heberlein, Jr. (to Heberlein Patent Corp.). U. S. 2,026,129, Dec. 31. Cloth such as a cotton fabric is treated with a swelling agent such as  $H_2SO_4$  to render it transparent and is passed through successive jaws of a calender having at least 2 jaws, to render it noncurling. The material may also be mercerized.

Impregnating textile fabrics with metal compounds such as those for weighing and mordanting. Sidney G. Osborne (to Hooker Electrochemical Co.). U. S. 2,025,072, Dec. 24. A fabric such as silk or cotton is immersed in a solution of a metallic salt such as one of Pb, Sn or Sb in a nonaqueous solvent such as an organic solvent which is inert to the fabric, and the salt is hydrolyzed (suitably by water vapor and  $NH_3$ ) to convert it into a water-insoluble compound of the metal.

Treating cellulose esters. I. G. Farbenindustrie A.-G. Brit. 435,129, Sept. 16, 1935. In treating materials made of or containing cellulose esters with an alkali liquor, saponification of the esters is avoided by incorporating in the liquor a small proportion of aminoacetic acid, or a salt thereof. Among examples, a mixed fabric of cotton and acetate rayon is handled for 1 hr. in a boiling liquor containing Chicago blue 6B,  $Na_2CO_3$ ,  $Na_2SO_4$ , and Na aminoacetate to dye the cotton only.

Treating fabrics such as "tobacco cloth" to improve its resistance to sun and rain. Geo. H. Rhodes. U. S. 2,026,190, Dec. 31. A web of fabric is continuously

brought into contact with an acidulated solution of Pb acetate for a sufficient time to effect penetration and is then treated with a solution of  $Na_2Cr_2O_7$  to form Pb chromate on the fibers of the fabric. Apparatus is described.

Rotproof material. I. G. Farbenindustrie A.-G. (Fritz Siefert and Hans Kammerer, inventors). Ger. 618,783, Sept. 18, 1935 (Cl. 8 & 1). Cellulose material for sacks, fishing nets, sails, tents, etc., is made rotproof by treatment with mono- or poly-azo dyes containing at least 2  $NO_2$  groups in the molecule; the material is also fast to light and water. Thus, fishing nets are impregnated with 2,3-dihydroxynaphthyl-4-amino-2',4'-dinitrodiphenylamine and treated with diazotized 2-nitro-4-chloroaniline to render them rotproof. Other examples are given.

Apparatus for sizing warp threads. Otto Stoeger. U. S. 2,024,890, Dec. 17. An apparatus with a pump for supplying size from a container is provided with devices for indicating the viscosity of the size and its temperature, and for indicating the temperature of a hot-air chamber of the apparatus during the drying process.

Apparatus for drying continuous webs of cloth, paper or the like. Alphons O. Huxthal (to Proctor & Schwartz). U. S. 2,024,927, Dec. 17. Various structural, mechanical and operative details of a loop dryer.

Fat acid amides. Gustave Maubec. Fr. 787,641, Sept. 26, 1935. Amides such as stearamide are made by bubbling anhydrous  $NH_3$  through the fat acid previously melted and heated to 100–150°. The amides may be used for coating, softening or finishing textile fibers and leather.

Dry-cleaning fabrics. Warren T. Reddish (to Emery Industries, Inc.). U. S. 2,024,981, Dec. 17. An absorbent compound such as one comprising true mahogany Na sulfonate 50, oleic acid 25 and naphtha 25% is mixed with a dry-cleaning solvent such as naphtha.

## 26—PAINTS, VARNISHES AND NATURAL RESINS

A. H. SABIN AND CARLTON H. ROSE

Production and use of high grade Zn oxide (Breyer). 13. Constituents of natural phenolic resins (Haworth, et al.). 10. Plastic compounds, lacquers, etc. (Brit. pat. 435,058). 13. Colloidal compounds [used in making varnishes] (Fr. pat. 787,267). 13. Aminoanthraquinone compounds [for lacquers] (Brit. pat. 434,906). 25. Azo dyes [for lacquers, varnishes and oils] (Fr. pat. 787,714). 25.

Roll and bar mill for grinding paints, etc. Asbjorn Sonsthaugen. U. S. 2,024,073, Dec. 10. Various structural, mechanical and operative details.

Photometric apparatus for measuring the hiding power of paints, opal glasses and photographic emulsions. Raymond F. Hanstock, Louis A. Jordan and The Research Association of British Paint, Colour and Varnish Manufacturers. Brit. 434,136, Aug. 27, 1935.

Pigments. Geo. I. A. Stutz and Harlan A. Depew (to New Jersey Zinc Co.). U. S. 2,024,611, Dec. 17. In drying a wet pigment, the wet mass is heated under superatmospheric pressure until a sufficient amount of the associated liquid is vaporized to leave the pigment readily disintegrable, and the vapor is permitted gradually to escape while the material is under pressure.

Pigments from vat dyes. Maurice H. Fleischer and James Oglvie (to National Aniline & Chemical Co.). U. S. 2,026,623, Jan. 7. For producing a pigment, a vat dye such as flavanthrone or indanthrone is dissolved in  $H_2SO_4$  and the material is dropped in water of substantially the boiling temperature.

Driers for use in paints and varnishes. Wilhelm Krumpholtz (to Beck, Koller & Co.). U. S. 2,025,870, Dec. 31. For producing a finely divided light-colored drier, a metal hydroxide such as that of Co, Pb, Mn, Ca or Zn is precipitated at a temperature not above room temperature under nonoxidizing con-

ditions, the precipitate is dispersed in an oil such as linseed oil and the dispersed precipitate, without drying, is treated with a resinic, naphthenic or soap-forming acid or with a glyceride of such an acid at a temperature below 200° (suitably with rosin in "mineral spirits").

Varnishes. Dubois & Kaufmann G. m. b. H. Ger. 621,440, Nov. 7, 1935 (Cl. 22 & 2). Brown fattier prepolymer in known manner from nondrying oils, e. g., tram oil, is dissolved in a drying oil, e. g., linseed oil. Driers and pigments, e. g.,  $PbO_2$ , may be added. Rapidly drying compounds are obtained.

Varnishes and plastic compositions. I. G. Farbenindustrie A.-G. Fr. 787,690, Sept. 26, 1935. Varnishes and plastics are made by mixing linseed oil, wood oil, chlorinated rubber, natural or artificial resins or cellulose derivatives with viscous or solid chlorinated aromatic compounds containing at least 1 cyclic methylene group, e. g., chlorinated compounds of tetra- and decahydronaphthalene, acenaphthene, fluorene, benzanthrene, indene, etc. The mixture is preferably effected in the presence of solvents. Pigments, fillers, etc., may be added.

Bronzing lacquer. Denis J. Burke (to Commercial Solvents Corp.). U. S. 2,026,493, Dec. 31. A bronzing lacquer contains, as a gel preervative, an ester or salt of malic acid such as ethyl or Na malate.

Coating compositions. N. V. tot Voortzetting der Zaken van Pieter Schoen & Zoon. Brit. 434,870, Sept. 10, 1935. A binding agent for paints or lacquers is manufactured by mixing 1 or more raw or polymerized drying oils with an oil-modified polyhydric acid-phthalic anhydride resin and chlorinated rubber. Paints are prepared by mixing the binding agent, or its 3 constituents in any order, with the pigment. A seccative and a thinning-out agent may be added to the finished paints or lacquers.

## 27—FATS, FATTY OILS, WAXES AND SOAPS

E. SCHERUBEL

The investigation of saturated acids of completely hydrogenated oils by the method of fractional distillation I. Hardened cottonseed oil. Sei-ichi Ueno and Masayoshi Iwai. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 602-3 (1935).—Hardened cottonseed oil (I no. 107.3, sapon no. 189.5), was saponified, purified with Et<sub>2</sub>O and the mixed fatty acids were saponified. The mixed acids (neutralization no. 191.5, I no. 0.86, m. 63.5-4.3°, titer 60.6°) were esterified with MeOH contg. 2.5% HCl and the Me esters were fractionally distd. at 2 mm pressure into 14 fractions. The results of the distn. were checked by the fractional crystn. of the free fatty acids in alc. The quant. compn. was calcd. by use of the mean mol. wt. of the Me esters computed from the neutralization nos. and the theoretical mol. wt. of the pure esters. The compn. was found to be minute amt. of myristic acid, and about 17% palmitic, 76% stearic and 7% arachidic acids. Exptl. results are given.

Catalysis of hydrogenation III. Hydrogen number, a constant for fatty oils. A. S. Ginzberg and N. Yrasshevskii. *J. Gen. Chem. (U. S. S. R.)* 5, 1168-70 (1935). *C. C. A.* 30, 368<sup>1</sup>.—The H nos. of 32 kinds of fatty oils, fats and waxes were detd. by the method of hydrogenation previously described. IV. Hydrogen number, a new constant for essential oils. A. S. Ginzberg and E. Evdokimova. *Ibid.* 1252-4.—The H nos. of 34 essential oils were detd.

Use of the Wood light in analysis. Examination of Argentine olive oils under the Wood light and comparison with peanut, cottonseed, rape and sunflower oils of domestic origin. B. Berisso. *Rev. col. farm. nac. (Rosario)* 2, 59; *Anal. farm. bioquim.*, Suppl. 6, 89-90 (1935).—The adulteration of olive oil with 3% of any of the above oils can be detected by examn. of the fluorescence produced by irradiation with light of 365 mμ wave length.

Apple- and pear-seed oil. J. Pritzker and Robert Jungkutz. *Z. Untersuch. Lebensmittel* 70, 255-8 (1935).—Data are presented on the constants of these 2 oils. The compn. of the seeds are also tabulated, as well as some tests made on the total fat acids.

Adulteration of arachis oil with coconut oil. Henryk Ruebenbauer. *Przemysł Chem.* 19, 9-10 (1935).—Arachis oil contg. 10% of coconut oil can be distinguished from the pure product by its higher d, sapon, Reichert-Messl and Polenske values, and by its lower n and i no.

The fruits of *Ochna pulchra* as a source of vegetable oil. E. P. Phillips. *Farming S. Africa* 10, 337 (1935).—The fruits and seeds contain a greenish brown oil having an odor resembling that of valeric acid. The oil is suitable for soap making. The residual meal contains no alkaloids.

The occurrence of some new alcohols and hydrocarbons in sperm oil. (Preliminary report) Sei-ichi Ueno. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 608-11 (1935).—The oil used was a blubber (skin) oil of the sperm whale (*Physeter macrocephalus* L.). It was saponified, the unsaponifiable matter extrd. with Et<sub>2</sub>O, and fractionally distd. at 15 mm pressure. The distillate boiling below 150° had an Ac no. 168.0 and I no. (Wijts) 54.3. Refractionation, mol. wt. detns. and chilling tests on fractions, acetylation of fractions and fractionation of Ac derivs., as well as soly. detns. on the Ac fractions in MeOH were carried out and alcs. contg. 8, 10, 12 and 14 C atoms were found. Some hydrocarbons were sep'd. from the Na alcoholates by distn. at reduced pressure. They formed a colorless volatile liquid and had a fragrant odor.

Eel oil. Toyoki Ono. *J. Agr. Chem. Soc. Japan* 11, 773-80 (1935).—The properties of the eel oil in the spring were: sp. gr. 0.9330, acid value 2.11, sapon. value 197.01 and I value 146.22; in the summer they were, resp.,

0.9334, 6.20, 193.09 and 158.89. The higher I value of the oil of the eel in the summer may be due to the fact that the eel consumes a large amt. of food in the summer. The Carr-Price reaction of vitamin A could not be observed in the oils of the various part of the eel. A small amt. of vitamin A was found only in the liver oil. The said fat acids consisted of palmitic acid and a small amt. of myristic acid and stearic acid. The Me esters of unsatd. acids were distd. fractionally and sep'd. as bromide. Tetradecylene, eleic, somarinic, stearidonic and iwahine acid were proved.

Copra of Mozambique. Joao de Carvalho de Vasconcellos. *Anais inst. super. agron., Univ. tech. Lisboa* 6, No. 2, 63-73 (1934).—Analyses of 7 samples of well-dried (H<sub>2</sub>O less than 6%) Mozambique copra show oil contents of 66.06-68.85%, ash 1.74-3.18, crude protein 5.69-7.34, cellulose 8.02-10.03 and N-free ext. 8.73-12.40%. There is no correlation between the percentage of oil and percentage of N-free ext.

O. W. Wilcox. Orientation of molecules of beeswax. Paul Woog and N. Yannaqu. *Compt. rend.* 201, 1400-2 (1935).—Examined by x-rays the mols. of beeswax, when first built into the cell wall show very little orientation, this increases with time at a rate increasing rapidly with temp. Thus wax showing practically no orientation showed it distinctly after 23 days at 30°, and even more so after 3 days at 40°. Beeswax from hot countries is much more oriented than that from cold.

C. A. Silberrad. The use of palm oil in making hard soap. C. Bergell. *Seifenfabr.-Ztg.* 62, 688-9, 720 (1935).

Volumetric determination of unsaponified fat in soap. N. Spasskii. *Maslobitno Zivovoe Delo* 11, 352-3 (1935).—The Spitz-Hönig method of detn. of unsapd. fat in soap is time-consuming and gives high values. The following procedure is rapid and accurate to 0.01% for 0.2% contents of unsapd. fat in soap: Dissolve 10-20 g. of soap in 100-50 cc. of 60% alc. contg. 0.5% Na<sub>2</sub>CO<sub>3</sub>. Ext. the unsapd. and unsaponifiable matter with petr. ether as usual. Wash the ext. with 60% alc. contg. 0.5% Na<sub>2</sub>CO<sub>3</sub> and distil off the solvent. Dissolve the residue in 5 cc. of alc.-C<sub>2</sub>H<sub>5</sub> (1:1) and titrate the soln. with 1/10 N KOH in alc.-C<sub>2</sub>H<sub>5</sub> (1:1) to a faint pink. Introduce 5-10 cc. of the same KOH soln., reflux the soln. on a water bath for 15-20 min. and titrate back with alc. 1/10 N HCl.

Chas. Blanc. The determination of free alkali in soap. II. W. Poethke. *Fettchem. Umschau* 42, 180-2, 197-201 (1935); *C. C. A.* 26, 4493.—The soly. of Na or K soaps in PrOH-glycol mixts. suggested the use of such mixts. as solvents for acidimetric titrations of soaps. Preliminary work detd. the indicator error (amt. of standard alkali soln. required to produce a red color to phenolphthalein) of a series of PrOH-glycol, PrOH-H<sub>2</sub>O, glycol-H<sub>2</sub>O and PrOH-glycol-H<sub>2</sub>O mixts. The indicator error increased continuously with the glycol content of the glycol-H<sub>2</sub>O and glycol-PrOH mixts., while it passed through a max. at 95% PrOH in the PrOH-H<sub>2</sub>O mixts. Addns. of small amts. of water had a minor effect on the indicator error of PrOH-glycol mixts. Addn. of NaOAc or K palmitate to various of the solvent mixts. always markedly decreased the indicator error. By applying the proper corrections, it was possible accurately and easily to titrate known amts. of KOH in artificially prep'd. mixts. of KOH and K palmitate or K stearate. Total carbonate and caustic alkly. in soap can be detd. by dissolving the soap sample in alc., adding an excess standard acid, boiling to expel CO<sub>2</sub> and cooling before titrating with standard alkali. Other expts. established the error involved in attempting to titrate the NaOH in soaps contg. Na<sub>2</sub>CO<sub>3</sub> with a mixt. of 80 cc. PrOH and 15 cc. glycol as a solvent. With the help of the corrections thereby detd., it was possible accurately to est. the free caustic in soap contg. small amts. of Na<sub>2</sub>CO<sub>3</sub>. Perhaps analogous methods for detg. the KOH in K soaps contg. K<sub>2</sub>CO<sub>3</sub> could be worked out. Expts. for prep. Na

oleate and Na palmitate in cryst. form are described.

J W Perry  
Detergent action of soaps of elaidinized fatty acids and the cis trans isomerism of unsaturated fat acids. S H. Bertram and E C S Kipperman. *Chem Weekblad* 32, 624-7 (1935).—Drop nos were detd., by means of a stalagmometer, of soaps of K soaps of 9 unsatd. acids and Na oleate, with 15 different org solvents. The drop nos are proportional to the emulsive powers of the soaps. All K soaps of elaidinized acids have higher drop nos than the corresponding oleic acids, and the ratios of the drop nos of the elaidic acids to those of the corresponding oleic acids is roughly const. Drop nos of  $C_{18}$  oleic acids do not differ much from those of stearic acid, but the drop nos of the corresponding elaidic acids are considerably higher for the hydrocarbon solvents, therefore the oleic acids have to be considered as the *trans*- and the elaidic acids as the *cis*-isomers. The influence of a longer fat acid chain, of a hydroxyl group, and the position of the double bond in the soap mol are demonstrated and discussed. The "2,3-oleic acid" of the literature is shown to be 2,3-elaidic acid. Drop-no tests and comparative washing tests show that soaps from oils in the ordinary condition have smaller detergent powers than elaidinized soaps.

O W Wilcox

App for measuring the heat cond of oils (Lederer) 1. Butter, fats and fatty oils (Langton) 12 Exhaust steam condenser and oil separator (U. S. pat. 2,025,043) 1. App for proportioning the flow of liquids as in refining vegetable oils (U. S. pat. 2,024,478-9-80) 1. App for sampling or visual examn of oils (U. S. pat. 2,026,267) 1. Ammonoanthraquinone compds [for waxes and oils] (Brit pat 434,906) 25.

Fats, etc Swift and Co Ger 618,836, Sept 17, 1935 (Cl 534 1 01) Fats, oils or their mixts, especially lard, is rendered stable by adding 1-5% of crude cottonseed oil.

Denaturing fats or oils to render them inedible. John E Muth (to Standard Oil Co of Calif.) U. S. 2,025,954, Dec 31. A benzyl deriv. of a saccharide or of glycerol or a glycol is added to a fat or oil.

Neutralizing oils. The Sharples Specialty Co. Fr. 787-555, Sept. 25, 1935 App is described for continuously mixing fatty oils and an alkali at high speed in carefully proportioned quantities and for sepg. the soap and neutral oil at suitable temp.

Oxidation products of castor oil. Melvin De Groote and Bernhard Keiser (to Tretolite Co.). U. S. 2,025,806, Dec. 31. Castor oil is oxidized with air at a temp of

1 about 120-150° and under a gage pressure of 25-125 lb, with 10-20% of octadeca-9,11-dienoic acid glyceride as an autooxidizer-catalyst. U. S. 2,025,807 relates to a similar oxidation of castor oil with air of normal moisture content, with use of heneleoic acid glyceride as an autooxidizer-catalyst. U. S. 2,025,808 involves the similar use of  $\alpha$ -pinene as catalyst. U. S. 2,025,809 involves a like use of dipentene as catalyst.

Polymernizing glyceride oils. Wilhelm Krumhaar (to Beck, Koller & Co.). U. S. 2,024,103, Dec 10. For bodying, an oil such as linseed, soybean or coconut oil, is heated with a colloidal dispersed heavy metal such as Co, Mn or Fe until a viscous product is obtained.

Apparatus for treating fish to recover oil from them and to prepare them for canning. John M Devers (to Food Machinery Corp.). U. S. 2,026,022, Dec 31. Various structural, mech. and operative details.

3 Soap Herbert Krause Austrian 143,654, Nov. 25, 1935 (Cl. 23b). In the manuf. of soap from fat compns. contg. liquid fatty oils or fat acids having an I no of 70 or more, the tendency of the product to deteriorate during storage is restrained by adding to the soap small proportions of glycerol (or other alc) and a water-sol phosphate, preferably  $Na_2PO_4$ . These addns. are made at any stage in the manuf. of the soap following the removal of excess alkali and salt.

4 Soap preparations I. G Farbenind A-G. Fr. 787-785, Sept 28, 1935. Water-sol salts of aliphatic carboxylic acids having at least 10 C atoms and in which at least 1 atom of N, O or S is situated between the aliphatic radical and the COOH, are used alone or with other substances such as soaps, inorg salts, albuminous materials, vegetable gums, starch, fats, waxes, glycerol, tar or nicotine, instead of soaps and have a high stability to the action of lime. Examples of these compds are alkali salts of lauryl-propylaminoacetic acid, oleylsarcoside, sarcoside of palm oil fat acid, reaction product of dodecylamine and maleic anhydride, reaction product of  $\alpha$ -stearyl- $\beta$ -diethylene-diamine with  $CH_3ClCOOH$ , heptadecylaminoacetic acid, dodecylmercaptosuccinic acid, dodecylaminobenzoic acid, stearylbenzylaminopropionic acid, and dodecylphenylaminoacetic acid.

5 Continuous soap-crutching apparatus Robert V Burt (to Procter & Gamble Co.). U. S. 2,024,425, Dec. 17. Structural, mech. and operative details.

6 Washing agents. Chemische Fabrik Joli A Benckiser G. m. b. H. Fr. 786,332, Aug 31, 1935. Alkali salts of metaphosphoric acid or mixts of these salts with alkali pyrophosphates or other alkalies, such as  $Na_2CO_3$ , are added to soaps in amt. such that the H-ion concn of the product is not above  $pH = 8.5$ .

## 28—SUGAR, STARCH AND GUMS

J K DALE

Refining quality of raw sugar. Effect of manufacturing conditions. R. W. Harman. *Intern Sugar J.* 37, 471-3 (1935).—Although the fundamental cause of poor-quality sugar originates in the cane, the mfg. conditions are also often responsible. Poor clarification and poor sugar boiling often yield very low-quality raw sugars from good-quality cane. H. has devised a set of lab. methods to test the refining quality of these raw sugars. Since these tests duplicate refining conditions very closely their use has proved very effective in grading incoming raw sugars.

D. P. Langlois  
The present state of purifying sugar juices. K. Smolenski. *Gaz Cukrownicza* 77, 35-53, 69-87 (1935) —A review with numerous references. J. Wiertelak

Modern beet-juice clarification. O. Spengler. *Intern Sugar J.* 37, 476-7 (1935).—See C. A. 29, 64584.

D. P. Langlois  
Determination of mixtures of sucrose, invert sugar and glucose sirup by Kruisheer's method. Wacław Reicher. *Gaz Cukrownicza* 77, 5-18 (1935) —Kruisheer's method of analyzing sucrose, invert sugar and glucose sirup in mixts.

with each other (C. A. 23, 3520, 24, 3064) has been applied to confectionery. The results are quite satisfactory.

J. Wiertelak

8 Methods of determining invert sugar Jerzy Zaleski. *Gaz Cukrownicza* 77, 109-29 (1935).—The different methods of detg. invert sugar, as recommended by the VIIIth International Comm. Meeting for standardization of analytical methods in sugar research, held at Amsterdam in 1932, are described. J. Wiertelak

Different methods of defecation. II. K. Smolenski. *Gaz Cukrownicza* 75, 157-70, 185-207 (1934) (French summary); cf. C. A. 28, 22114.—An important factor in the reproducibility of defecation expts. performed on a factory scale is the alk. after first satn. which should be approx 0.08. Previous results were confirmed, showing that a higher dose of CaO results in quicker filtering and better quality of the juices, e. g., if 1.5% CaO (on the juice basis) is used, 10-15% more nonsugars are removed from the juice, than with 1% CaO. S. considers that nascent  $CaCO_3$  is the main cause of this improvement. Slow defecation, lasting 5 min., gives higher filtering rates and better

juices than quick defecation, lasting 0.5 min. Similarly quick defecation, at 80°, gives a better filtering rate than cold defecation at 40°, but only slightly better juices. Defecation performed according to Troye's method of mixing the diffusion juice before defecation with an equal vol. of first satn. juice gives considerably shorter filtering periods, but a lower quality of juice, than if a preliminary defecation is applied. J. Wiertelak

Use of the electrical sodium lamp in saccharimetry. E. Landt. *Intern. Sugar J.* 37, 474-6, *Centr. Zuckerind.* 43, 650-1 (1935) — The advantages of a simple polarimeter illuminated by means of a new elec. Na lamp of high intensity over the white light, quartz wedge compensation saccharimeter, which is almost universally used in the sugar industry today, are discussed. Such equipment has been used in England for many years. Two years ago such a polarimeter was supplied to a firm on the continent. Its performance has been highly satisfactory. D. P. Langlois

Ost and Wilkening's method of determining lignin applied to sugar beets. Anna Nowotwórna and Janina Włodowska. *Gas Cukrownicza* 76, 270-2 (1935) — Ost and Wilkening's method of detg. lignin in wood is modified for sugar beets. In the first step the total fiber of the beets is isolated according to Henneberg and Stohman and the lignin is then detd. in the usual way, except that previous to hydrolysis with 72%  $H_2SO_4$  the total fiber is extd. consecutively in a Soxhlet app. with  $Et_2O$ , 96%  $EtOH$ ,  $C_6H_6$ , and  $Et_2O$ . Four different samples of beets showed the following lignin content: (a) 0.368, 0.385%; (b) 0.467, 0.471%; (c) 0.341, 0.340%; (d) 0.416, 0.424%. J. Wiertelak

Microscopy of starches and their modifications. Otto A. Sjostrom. *Ind. Eng. Chem.* 28, 63-74 (1936) — A series of photomicrographs shows the appearance of the more important starches and their modifications, also the thin-boiling starches and dextrans, in their gelatinized and disintegrated forms. These pictures support the recent theory of "growth structure." W. H. Boynton

X-ray studies of nitrated starches. I. Maria Kolaczowska and Tadeusz Urbański. *Roczniki Chem.* 15, 339-42 (1935) — The authors obtained x-ray spectrograms of nitrated potato and sol. starch by the Debye-Scherrer method. Both show a marked resemblance and give the

same principal interference rings. Natural starch is more cryst. before the introduction of  $-ONO_2$  groups, while the structure of sol. starch changes regularly during nitration. J. F. Matejczyk

Treatment of molasses for the production of foods, fertilizers, etc. (Gaspar y Arnal) 12. Significance of K in the system of fertilizing sugar-beet rotations (Lut) 15. Sugar cane (Taggart, et al.) 15. Clarifying and removing coloring matter from aq. liquids [sugar solns.] (U. S. pat. 2,025,715) 1

Sugar. Allgauer Alpenmilch A.-G. Brit. 435,034, Sept. 9, 1935. See Fr. 769,800 (C. A. 29, 519).

Apparatus for concentrating sugar sap. Hermanus A. Jansse. Ger. 618,075, Sept. 17, 1935 (Cl. 89d) 1.

Crystallizers for sugar, etc. Walter J. Blanchard. Brit. 434,740, Sept. 9, 1935. A crystallizer comprises a soln. container within which rotates a drum having hollow radial arms arranged so that their paths overlap. Cooling water or steam may be admitted to the worm-driven drum.

Starch from corn. Robert E. Greenfield and Harold R. Baker (to A. E. Staley Mfg. Co.) U. S. 2,023,999, Dec. 10, 1935. Corn is steeped, cracked, dewatered by starch flotation, ground, washed to remove fibrous constituents and to form a starch-gluten water suspension, tabled to sep. starch from gluten, and the gluten is dewatered; the starch milk from the flotation is dewatered and the water is employed to steep addnl. corn, the dewatered starch is washed with fresh water, germs from the flotation are washed with the starch wash water, the germ wash water is returned to the flotation, the starch-gluten suspension is dewatered, the dewatered starch-gluten mixt. is washed with fresh water, the fibrous constituents are washed

with the starch-gluten wash water; a portion of the fibrous constituents wash water is returned to the flotation; water from the gluten is added to the dewatered materials to form a water suspension which can be tabled, tabled starch is taken up in water, the tabled starch is filtered, the filtered starch is washed with fresh water, a portion of the water from the filtered starch is employed for taking up addnl. tabled starch, and an addnl. portion of the water from the filtered starch is returned to the process. An arrangement of app. is described.

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## 29—LEATHER AND GLUE

ALLEN ROGERS

Wattle barks. IV. Tannin content of a variety of *Acacia mollissima* Willd. F. A. Coombs, W. McGlynn and M. B. Welch. *J. Proc. Roy. Soc. N. S. Wales* 68, 246-8 (1934); cf. C. A. 27, 439. — Analytical data are given for 17 samples of bark from a newly recognized and described variety that differs from the main species only in its slightly lower ratio of tannin to nontannin.

H. B. Merrill  
The vinegar eelworm in tan liquor. B. G. Peters. *J. Helminthology* 13, 159-62 (1935). — Eelworms (*Turbatrix aceti*) thrived in fermenting tan liquor of pH 5.0. There is no evidence of the active secretion of proteolytic enzymes by this worm and there is no reason to suppose that the worm causes damage to hides and skins during the tanning process. K. D. Jacob

Fat liquoring of animal skin. Edwin R. Theis. *J. Tech. Assoc. Fur Ind.* 6, 88-90 (1935). H. B. Merrill

Practical salting experiments in 1934. W. Grassmann and W. Hausam. *Ledertech. Rundschau* 27, 49-53, 57-62, 67-70 (1935); cf. C. A. 29, 2777. — Packs of calfskins in 3 cities were cured with NaCl contg. NaF or naphthalene, 2385 and 1376 skins, resp., including blanks bemused.

Small lots of skins (15-50) were cured with addn. of Zn salts and Preventol. Skins cured with NaCl contg. 1% NaF were slightly less stained than comparable skins without NaF, but the finished leather was not enough better to make the use of NaF advisable. Crude naphthalene was advantageous but the pure material was much

better; the addn. of naphthalene to curing salt at the beginning of warm weather is advisable. Preventol in brine followed by salting with 15% ordinary salt entirely prevented staining of the skins but the finished leather was only moderately better than that from the blanks. Preventol-treated salt was not effective. ZnO (1.25%) with salt was very effective and was better than Zn peroxide. Dry salted skins were much better than brined skins. I. D. Clarke

Effect of perspiration on leather. P. White and F. G. Caughley. *New Zealand J. Sci. Tech.* 17, 412-17 (1935).

—To show the effect of perspiration from the foot on the compn. of leather, socks made from vegetable-tanned lamb skin were worn for periods up to 30 days and analyzed. pH values of  $H_2O$  exts. of the leather increased from about 4.1 to 5.4, there was a marked increase in sol. N, a slight decrease in  $H_2O$ -sol. matter, and a marked darkening. The last 2 changes are attributed to oxidation promoted by the lowered acidity. H. B. Merrill

Fat acid amides for [coating, softening or finishing leather] (Fr. pat. 787,641) 25. Azo dyes contg. metal [for dyeing leather] (Brit. pat. 435,060) 25

Leather. Sebastiano Boccardo & Co. S. A. Austrian 143,631, Nov. 25, 1935 (Cl. 28b). White leather is obtained by lightly tanning hides with  $CH_3O$  soln. and com-

pleting the tanning with an acidified soln of water glass. The hides are washed between these treatments. If a soft product is desired, an alkali, e. g., soda, is added during the treatment with  $\text{CH}_3\text{O}$  in an amt. sufficient to establish a  $\text{pH}$  value of 7-9.8.

Chrome tanning. Arthur Weinschenk. Ger. 618,921, Sept 18, 1935 (Cl 28a 3). A complex Cr tanning compn is obtained by treating Cu or Zn borates with Cr salts and drying. Hydroxides, carbonates or basic carbonates of Cu or Zn may be dissolved in water, treated with  $\text{H}_2\text{BO}_3$  and Cr salts, and the product evapd to dryness. In an example,  $\text{CuSO}_4$ ,  $\text{H}_2\text{BO}_3$  and  $\text{Na}_2\text{CO}_3$  are dissolved in water and treated with  $\text{Cr}_2(\text{SO}_4)_3$  soln. and the product dried or evapd to the required strength.

Treating soft leather to render it more rigid. Arthur J. Beford. U. S. 2,026,453, Dec 31. The leather is impregnated with a hot soln of paraffin, colophony, Burgundy pitch and neat's-foot oil and compressed while heated.

Coating leather. Imperial Chemical Industries Ltd. Fr. 786,233, Aug 29, 1935. A finishing compn for leather is made by incorporating an aq emulsion of an aliphatic alc of long chain contg 10 C atoms or more, e. g., cetyl alc or one obtained by reducing lauric acid, with an alk soln of gum lac.

Dyeing skins, etc. I. G. Farbenind. A.-G. (Erich Lehmann, inventor). Ger. 618,902, Sept 18, 1935 (Cl 3m 10 02). Addn to 617,987 (C. A. 30, 878a). The

method of 617,987 for dyeing skins, hair and feathers is modified by replacing the salts of 1,5-dihydroxynaphthalene and ammodiphenylamine by salts generally of 1,8-dihydroxynaphthalene and aromatic or heterocyclic amines, their derivs. or substitution products. Thus, the salt of 1,5-dihydroxynaphthalene and 7-aminoquinoline is dissolved in water and given a small addn. of  $\text{H}_2\text{O}_2$ . On soaking in the soln white dog skin mordanted with Cr salt, a product with deep wine-red hair and bright red leather is produced. Other examples are given.

Gelatin from pigskins, etc. Chester H. Epstein and Nathan R. Gotthoffer (to Grayslake Gelatin Co.). U. S. 2,024,683, Dec. 17. The gelatin-yielding raw material is acidulated with citric or tartaric acid (suitably conjointly with an inorg. acid) and is then hydrolyzed.

Adhesive. Sergei Ushakov and Aron Endlin. Ger. 621,139, Nov. 2, 1935 (Cl 22a 2). As an adhesive for securing veneers under heat and pressure, use is made of finely powd vegetable-tanned leather waste, which has preferably been pretreated with warm water. Small proportions of softening, preserving or waterproofing agents may be added to the leather. Sp processes and compns. are described.

Dried glue. Roy C. Newton and Frank L. De Beukelaer (to Industrial Patents Corp.). U. S. 2,024,131, Dec. 10. Glue particles with a surface of jagged glue granules are obtained by dropping glue liquor upon granulated dried glue (various details of procedure being described).

### 30—RUBBER AND ALLIED SUBSTANCES

C. C. DAVIS

The role of  $\text{pH}$  in the latex industry. Maurice Dérivé. *Caoutchouc & gutta percha* 32, 17363-5, 17365-7 (1935), cf C. A. 28, 1889<sup>a</sup>.—Measurements of the  $\text{pH}$  values of fresh and stabilized latex, and the coagulation of both kinds of latex are discussed. C. C. Davis

Patent literature in the field of latex and rubber in 1933-1934. St. Reiner and O. Duditz. *Caoutchouc & gutta percha* 32, 17339-40, 17371-3 (1935); cf C. A. 30, 897<sup>a</sup>.—Forty-six French and English patents are described. C. C. Davis

Progress in x-ray research on rubber. Geo L. Clark. *Rubber Age* (N. Y.) 38, 79-80 (1935).—A review, with preliminary announcement of the discovery of a long spacing in gel rubber, which is absent in sol rubber. C. C. Davis

Contributors to rubber compounding progress. D. C. McRoberts. *India Rubber World* 93, No. 4, 45-7 (1935), cf C. A. 29, 8396<sup>a</sup>.—An illustrated description of the Philadelphia Rubber Works Co. at Akron, O., with special reference to the lab facilities. C. C. Davis

Purification of natural rubbers. III. A formula for the calculation of nonvolatile, water soluble substances in coagulated rubber and the application of the formula. Syozo Satake. *J. Soc. Rubber Ind. Japan* 8, 602-10 (1935), cf C. A. 29, 5300<sup>a</sup>.—Nonvolatile, water-sol substances (A) in coagulated rubber can be calcd by knowing the concn of nonvolatile, water-sol substance in the serum of coagulated latex and its degree of satn (com raw rubber = 100) of water (B) thus:  $A = (a/b) B$ , where  $a$  is the nonvolatile, water-sol solid in the serum of coagulated latex and  $b$  is the volatile substances. Expts on the syneresis taking place when coagulated latex was passed through rolls indicated that the degree of satn of water became approx 20%. Application of this formula to the removal of nonvolatile, water-sol substances of coagulated rubber prepd from dln of latex agreed well. Three samples of rubber (pale crepe, smoked sheet and LS rubber) of known content of water-sol substances were tested with this formula and agreed well. K. Kitsuwa

The acetone extraction of raw rubber. VII. The time of acetone extraction of raw rubber and the saponification values of the acetone extract and of the residue. Hidemaro Endô. *J. Soc. Chem. Ind., Japan* 38, Suppl

binding 514-17 (1935), cf C. A. 30, 323<sup>a</sup>.—Free fat acids and saponifiable matter were extd almost completely by acetone in 1-2 hrs. The sapon no. of the ext (A) decreased rapidly and reached a min after 12 hrs of discontinuous extn and after 16 hrs of continuous extn. This decrease is attributed to a gradual increase of extd non-acidic compds and unsaponifiable matter with increasing time of extn and the neutralization of acids by other compds. After the min point, A increased, probably as a result of a gradual increase of extd esters, formation of acids by oxidation and decomps of esters. The change in the sapon. no. of the acetone ext with increasing time does not result from a change in the type of compds gradually extd, but from a change of the ext in acetone soln during extn. This is in agreement with the facts found for the acid no. Exptl data are reported. Karl Kammermeyer

The oil resistance of rubber. II. Molecular polarization and dipole moment of purified natural rubber. Shû Kanbara. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 506-10 (1935); cf C. A. 29, 7121<sup>a</sup>.—Natural rubber was purified by Fummeier's method and dissolved in  $\text{C}_6\text{H}_6$  and  $\text{Et}_2\text{O}$ . The dielec. const.,  $n_D^{20}$  and  $d_4^{20}$  values and the concn of the solns were measured, and from these data the mol polarization and the dipole moment (I) were calcd on the assumption that rubber is dissolved in a mol state of ( $\text{C}_6\text{H}_6$ ), 1 or total and gel rubber, the I values were 2.45 and  $2.91 \times 10^{-18}$  e.s.u., resp., in  $\text{C}_6\text{H}_6$ . These values are of the same order as those for masticated crude rubber obtained by Ostwald. The impurities in rubber are probably interlocked mechanically with the rubber mol., and not combined chemically. Sol rubber in  $\text{Et}_2\text{O}$  and  $\text{C}_6\text{H}_6$  gave I values of 0.72 and  $2.79 \times 10^{-18}$ , resp. The values of total and gel rubber may be high because of autooxidation, and the true values for the rubber hydrocarbon may be considerably smaller than the reported ones. Circuit diagrams of the app used for detg the dielec. const. and the exptl data are included. K. K.

Fundamental rules for the technical application of chlorinated rubber. Guido Schultze. *Farben-Ztg.* 40, 1165-7 (1935).—A discussion of the correct formulation of chlorinated rubber protective coating compns, contg unsaponifiable plasticizers, pigments, etc., and methods which give the best practical results. J. W. Perry

Hydrazines as rubber softeners I Williams and C. C. Smith *Ind. Eng. Chem.* 27, 1317-21 (1935).—About 1% of PhNH<sub>2</sub> (I) plasticizes rubber, reduces the viscosity of its solns., and peptizes gel rubber in CCl<sub>4</sub>. Softening is faster at 75° than at 25°, but reaches the same final stage, which depends on the concn of I. Rubber softened to the same extent by either I or by milling gives the same tensile strength, which is much greater than when mineral oil or stearic acid is used as a softener. C black, clay, etc., retard the action of I. Viscosity reduction is less in gasoline than in CCl<sub>4</sub>, and is lessened by addn of EtOH. The softest product (like molasses) had a mol wt of 4500 (Staudinger's method) and a tensile strength of 115 kg per sq cm. Other asym hydrazines, salts of I, and its addn products with PhOH, CS<sub>2</sub>, heavy metal salts, etc., are also softeners. The action of I is probably not phys., since I cannot be recovered from the softened rubber. Since the softening appears to depend on the presence of O, it is suggested that O reacts with double bonds, thus either reducing the real mol wt by breaking C chains or the apparent mol wt by destroying centers of attraction between mols. I accelerates this process by combining with the oxidation products. Reducing agents (HI), however, are not softeners. The literature of viscosity reduction and rubber softening is reviewed.

Arnold M. Collins.

The properties of rubber coagulum and rubber films formed from it A van Rossem and Raden Soepardi Prawirodipoero *Gummi-Ztg* 48, 622-53 (1934).—A preliminary report. The remarkable properties of so-called Emka sheet, prep'd by the Emka process by compression of the coagulum, suggested an investigation of the coagulum itself. Sheets were prep'd in 2 ways: (1) by coagulation of latex cream with Na<sub>2</sub>SiF<sub>6</sub>, in which case coagulation was slow enough so that the next could be placed on a smooth glass surface and a coagulum of uniform thickness be obtained, and (2) by the method of Traube, in which the latex was poured on a layer of gelatin, and a coherent sheet thus obtained. Acid products diffused out of the gelatin, lowered the pH value and led to coagulation. Addn. of HCO<sub>2</sub>H to the gelatin accelerated the coagulation. The properties of the coagulated sheet obtained thus were studied: (1) *Water absorption on stretching under water*.—When a sheet was stretched under water, it absorbed up to 100% of its own vol of water, and the proportion absorbed was independent of the original water content and of the elongation. (2) *Syneresis*.—Compression of the coagulum released very little water, whereas kneading released a large proportion of water by syneresis. The washing process on the plantations may be regarded as a kneading process with irrigation by water, the only function of which is a pronounced cooling effect. (3) *Residual elongation*.—Measurements were made of the elongation remaining after sheets were stretched in a moist atm., and also when kept stretched until dry. In the latter case, the residual elongation was very high, and diminished only slightly on warming to 70°. Emka sheet also showed no shrinkage at 70°. (4) *Emka sheet showed, for raw rubber, an extraordinarily high tensile strength and modulus*. When wet sheeted coagulum was stretched to 300% and was dried under this tension, the tensile strength increased greatly in the direction of stretching, whereas it remained practically unchanged at right angles to this direction. Here there are certain analogies to calendered sheets having a calender effect, with the difference that calendered sheets shrink considerably at 70°, whereas stretched wet sheeted coagulum does not shrink. Accordingly there is a sort of reinforcement involved, the cause of which is still uncertain. Probably the phenomena are related in some way to an orientation of the long-fiber mols., which if true will be confirmed by x-ray studies in progress. Though other films from wet coagula, e. g., viscose and cellulose acetate films, show different properties in different directions, the phenomena are not the same as with rubber, for in the first case gel formation from a sol is involved, whereas in the latter case a gel is formed from a suspension of particles in an aq. serum.

C. C. Davis

Effect of oxygen absorbers in rubber. A. A. Somerville *Ind. Eng. Chem.* 28, 11-17 (1936).—A new chem. method for retarding the tendency of vulcanized rubber to crack when subjected to repeated dynamic strains, such as so-called flexing, is described. This new method is based on the theory that such cracking is an aftermath of oxidation by free O dissolved in the rubber and absorbed by the powders (cf. Neal and Northam, C. A. 26, 873), and involves the addn of org. substances which absorb O in the presence of a base. The more strongly a substance in alk. medium absorbs free O, the more it retards cracking from flexing, and substances of the activity of the photographic developer class have a notable effect. As simple examples, the results obtained with pyrogallol + ethanolaniline and pyrogallol + hydroquinone + ethanolaniline are described. The mechanism of this reaction is obscure and it is complicated by the fact that the O absorbers develop their max. effect in the presence of a catalytic antioxidant. The exp't results are of direct practical significance, since with O absorbers, higher proportions of C black can be used with no greater tendency to crack by flexing than with lower proportions of C black in the absence of such agents. The use of O absorbers also enables the use of lower proportions of S. C. C. Davis

A new gutta from South Africa. Anon. *Bull. Imp. Inst.* 33, 303-6 (1935).—The *Gutta* exts of bark and of twigs and leaves of *Gymnosporia acuminata* Syzys. (= *Celastrus acuminatus* Linn.) from Swaziland have the appearance and properties of gutta-percha or balata, and are quite distinct from rubber. The yield of 3.5% of gutta-like material from the twigs and leaves compares very favorably with that stated to be obtained in the East from the leaves of gutta-percha trees (*Palaquium* spp.). It is recorded that on the Govt. estates in Java a yield of 2.7% is obtained. On this basis, the yield of 10% obtained from the bark is very satisfactory. A. P. C.

A program of research with a view to improving carbon blacks for rubber. Cuvenc *Rev. gén. caoutchouc* 12, No. 116, 8-10 (1935). C. C. Davis

Further observations on variations of smoked sheet rubber. John Young and L. H. Rich. *Trans. Inst. Rubber Ind.* 11, 343-53 (1935); cf. C. A. 28, 462a. The light and dark grades (I and II, resp.) of smoked sheet already described (*loc. cit.*) were tested further for their aging properties and for the relative resistances to abrasion in road service of tires made from them. I and II gave, resp., in % ash, 0.136, 0.080; acetone ext., 2.54, 3.99; aq. ext., 1.59, 1.01. Photomicrographs (illustrated) showed that I gave the poorer dispersion of C black, both in a rubber-C black master batch and in a tire-tread mixt., whereas there was no difference between I and II after vulcanization. The dispersion in II was as good as that in I in less than 0.5 the time of milling. I, washed and dried I, masticated I and II were vulcanized according to a tire-tread formula, and the tensile strengths and moduli (at 300%) at 0°, 25° and 100° were det'd. The differences in phys. properties of the 4 samples at a given temp. were small, but the differences with change in temp. were great. This shows the importance of testing at a const. temp. Aging tests by the Geer oven and O-bomb methods showed small differences in the tensile strengths and in the resistances to abrasion (Williams app.), while in road wear tests the best wear was shown by the tire made from washed I, and in flexing tests (Rubber Service Laboratories Co. machine) I and II were almost the same, and better than masticated I and washed I. C. C. Davis

Reinforcement and stearic acid. Harry Barron *India Rubber J.* 90, 638-40 (1935).—An investigation was made of the effect of stearic acid (SA) on the mech. energy of vulcanizates composed of the base mixt.: rubber (10), S 3, diphenylguanidine 1.5, ZnO 5, C black 35, to which had been added 0, 0.5, 1, 1.5, 2, 2.5, 3 and 5 parts of SA, and which had been vulcanized at 141° to their optimum tensile properties. Another similar vulcanizate was prep'd from the same rubber previously ext'd. free of SA by acetone. The rubber contained approx. 1.5% SA. Graphs show the works of extension, works of retraction and hysteresis losses as functions of the elongations, and the



tensile strengths, elongations at rupture and energies as functions of the proportion of SA. For a given elongation, the work of extension (resilient energy) (WE) increased from 0 to 1.5% SA (normal content), remained practically constant to approx 4% (2.5% addn) and then diminished progressively. For a given elongation, the work of retraction (WR) increased from 0 slowly to a maximum 4% SA, above which it diminished very slowly. For a given elongation the hysteresis loss increased greatly to a max at 1.5% SA (normal content), then diminished slowly. The tensile strength increased from 0 to 1.5% SA (normal content), remained the same to 3.5% (2% addn) and then slowly diminished with increasing SA. The elongation at break increased progressively with increase in SA. Since SA has only a small effect on the total reinforcement but reduces considerably the reinforcement from internal friction and increases the WK value (true reinforcement as defined by B and Cotton, C A 26, 3908), it probably has a 2-fold part. As a softener, it acts as a lubricant and reduces the work expended in overcoming internal friction. It is also sol in rubber and reduces the interfacial tension, and according to the Gibbs adsorption theory there must be a concn of SA at the interface. There are unnumerable interfaces in a rubber-filler mixt, since not all air disappears from the surface of the filler particles in the rubber, and SA blooms to these internal interfaces. Here it decreases the interfacial tension between rubber and filler, and increases the wetting of the filler by the rubber, i.e., increases the true reinforcement. There are several known phenomena in support of this view, including particularly the acceleration of the rate of vulcanization by SA and the blooming of SA on a freshly cut surface of vulcanized rubber. C C Davis

Rubbenizing of balloon fabrics J Panem. *Rev gen caoutchouc* 12, No 116, 18(1935)—Present-day developments are discussed. C C Davis

The use of rubber in automobile construction Colin Macbeth *Avtushuk* 11, 197-204(1935)—Descriptive, with numerous diagrams. C C Davis

An impact cutting test for tire tread stocks J H Dillon *Ind Eng. Chem., Anal Ed.* 8, 68-71(1936)—The relations between the ease of cutting of vulcanized rubber of the tire-tread type and its tear resistance, resistance to cracking and hardness are discussed and an impact-cutting device, based on theoretical principles, is described and illustrated. C C Davis

The story of synthetic rubber J A Nieuwland *Proc. Indiana Acad Sci* 44, 17-21(1934)—A review covering for the earlier years all important investigations, but for the last few years only work on the polymerization of chloroprene. C C Davis

Chloroprene synthetic rubber (Soprprene) A L Klebanik, L G Teyurikh and I. M. Dolgopolskiy *Bull acad. Sci. U R S S* 1935, No 2, 189-226; *J. Research Assoc Brit Rubber Mfrs* 4, 505-6(1935)—In the continuous polymerization of  $C_3H_4$  in a weakly acid soln of  $CuCl \cdot NH_4Cl$  as catalyst, the product contains 75-80% of HC CCH  $CH_2$  (I) and 20-25% of the trimer (II) and higher polymers. The yield, calcd on the  $C_3H_4$ , is almost 100%, and a single passage of the gas converts about 40% of the  $C_3H_4$ . The optimum condition of the catalyst, the mechanism of the reactions and the main factors which affect this polymerization are discussed, and a method of sepn and absorption of the gas by solvents is described. A discontinuous method of synthesizing chloroprene (III), and methods for its continuous prepn from pure I and from mixts of I with  $C_2H_4$  are also described. The polymerization of III to the  $\alpha$ -polymer (IV) is then discussed, with particular reference to the conditions which hinder the formation of higher, balata-like polymers. A study was made of the ozonization and oxidation of polymers of III, the first method being applied to IV, to the  $\mu$ -polymer and to the  $\alpha$ -polymer. The chief product is succinic acid. The compn of II is discussed and the phys and chem. properties of the previously unknown acetylenic polymer,  $H_2C \cdot CHCH \cdot CHCH \cdot CH$ , the formation of which accompanies that of the divinyl, are described. Data are also given on the action of alk.  $NaOH$  on 2,4-

dichloro-2-butene (V), in which reaction 2-chloro-2-butene ethyl ether is formed. Similar methods are described for the synthesis of chlorobutanol and of dichlorobutenyl ether from chlorobutanol and V are described. In this reaction 2-chlorobutene-4-ol xanthate is also formed. A series of new syntheses from I is described, including a new synthesis of  $\alpha, \beta$ -dichlorobutadiene. The structure of the product and the method of polymerization are discussed, as well as the fixation of aliphatic org. acids by I in the presence of  $HgSO_4$  or a mixt. of  $HgO$  and  $BF_3$ , with formation of esters of 2-hydroxy-1,3-butadiene. The method of synthesis and the structures of the formates and acetates of 2-hydroxy-1,3-butadiene, and of the condensation product with maleic anhydride, and of their hydration products, are described. The combinations of EtOH and MeOH with I in the presence of alk. alc. are described. In these reactions 1-methoxy-3,4-butadiene and 2-ethoxy-1,3-butadiene are formed. The fixation of MeOH by I in the presence of  $HgO$  or  $BF_3$ , EtOH, and the hydrogenation of I to butadiene are described. Finally the formation of styrene by the polymerization of I is discussed. C C Davis

The use of substitute [factice] in rubber manufacture. I. H. Hurlston *Trans Inst Rubber Ind* 11, 295-301 (1935)—A review and discussion, with new expl. data. Tests of rubber mixts contg various org. accelerators, and with and without 2% white factice (I), showed that the latter has a pronounced tendency to retard scorching with mercaptobenzothiazole (II), Zn diethylthiocarbonate and Zn isopropylxanthate, less tendency with II diphenyl guanidine, and practically no effect with piperidine piperidylthiocarbonate, tetramethylthiuram disulfide (III) and tetraethylthiuram disulfide. At a temp corresponding to 5 lb per sq in steam pressure, increasing percentages of I increased progressively the rate of vulcanization, whereas at a temp corresponding to 25 lb per sq in steam pressure, the rate of vulcanization was retarded progressively with increase in I, and the tensile strength lowered progressively. The acceleration may be due to free alkali or S compds, while the retardation may be caused by decompn products of I (cf Baker, Bodger and Cotton, C A 26, 335). Tests show that most factices (white and dark) increase the tendency of rubber-III 7nO mixts. to scorch. Measured by the Griffiths extrusion plastometer (C A 20, 2094), I has very little effect on the plasticity of milled rubber, whereas a factice of the Vulcatac type (cf Auer, C A 23, 4370) has considerable effect. Tests show that brown factice (IV) can be used up to 5% in C black mixts used for belt and hose covers without affecting adversely tensile strengths, elongations and tear-resistances, whereas the resistance to abrasion diminishes with increase in IV. C C Davis

Balata resin I Crystalline components of Sarnum sheet balata resin Yoshio Tanaka, Tsutomu Kuwata and Toshio Suzuki *J. Soc Chem Ind, Japan* 38, Suppl. binding 504-5(1935)—From balata produced in Guiana were isolated cryst.  $\beta$ -amyrin acetate m 230-1°, an ester of isopulepol, m 195-6°, and balatol, m 100-1°, sapon no 121.0, benzoyl ester, m 121-15°, percentages of C 81.62, 82.13 (calcd from  $C_{27}H_{44}O_2$ : 81.98), percentages of H 11.32, 11.45 (11.19) and mol wt from sapon no 463.6 (calcd 468) Karl Kammermeyer

Synthesis of the derivs. from 2,4-dichloro-2-butene—use of by products from the synthesis of chloroprene (Klebanik, Chevchnalova) 10 Application of furaluril and its derivs to the manu of plastics [accelerators for rubber vulcanization] (Bruins) 13 Production of lamp-black from anthracene and naphthalene (Zmif) 18 Synthesis of  $\alpha, \beta$ -dichlorobutadiene and its polymerization (Klebanik, del) 10 Hydrogenation of tertiary alkyl phenols to form hydroaromatic alcs [use as softening agents in rubber compns] (U. S. pat. 2,026,668) 10 Pwd. substances [chlorinated rubber] (Fr. pat. 787,363) 1 Azo dyes [for coloring rubber] (U. S. pat. 2,025,582) 25 Mortars or binders contg rubber (Fr. pat. 787,236) 20 Aliphatic S compds [vulcanizing rubber] (Fr. pat. 787,310) 10 Plastic rubber deriv for molding (U. S.

pat. 2,024,987) 13. Activated C (U. S. pat. 2,026,355) 18.

**Rubber latex.** Metallgesellschaft A-G Fr. 786,458, Sept. 3, 1935. The deep coloration of latex is prevented or suppressed and its stability improved by the addn of stabilizers of alk. reaction, e. g.,  $\text{NH}_3$  or KOH in amt sufficient for stabilization, as well as small amts of compds of Zn, e. g., ZnO, alkali zincate, complex compds. of Zn and  $\text{NH}_3$  or salts of Zn such as carbonate, chloride or acetate, while avoiding the addn of any substance permitting vulcanization of the rubber.

**Preserving latex** I G Farbenind. A-G Fr. 787,466, Sept. 23, 1935. Amines contg at least 1 aliphatic radical of more than 8 C atoms or the corresponding quaternary  $\text{NH}_4^+$  bases or their salts or mixts, e. g., dodecylamine-HCl or diethylbenzyl-dodecylammonium chloride are incorporated with latex to prevent coagulation or corruption.

**Determining the rubber content of latex, etc.** John S. Ward and Samuel D. Gehman (to Wingfoot Corp.) U. S. 2,024,617, Dec. 17. An incandescent filament is observed through a green filter by looking through an extinction cell contg a rubber dispersion, and the extinction cell is moved in a light-proof housing across the field of vision from the extinction point on one side of the point of max. brightness to the extinction point on the other side, and the amt. of movement is indicated by a described device. Various features of app. are described.

**Rubber compounding** Albert A. Somerville U. S. 2,026,442, Dec. 31. In the manuf. of products such as tire casings, elec. insulation, etc., a rubber compn is vulcanized which contains a catalytic antioxidant (such as diphenylamine) effective to inhibit oxidation of the rubber by the O of the atm. to which it may be exposed, an org. O absorber (such as pyrogallol) effective to eliminate O initially present in the compn and an org. base (such as an ethanolamine) which is nonvolatile at the vulcanizing temp., is miscible with rubber and is effective to activate the O absorber. Numerous examples are given.

**Halogenating rubber** Nicolas Floresco and Max Pfunderer Fr. 787,217, Sept. 19, 1935. Rubber or natural gums are masticated and submitted at the same time to the action of ultraviolet rays, then treated with a solvent such as  $\text{CCl}_4$  and submitted to the action of halogen, such as  $\text{Cl}_2$ , the latter operation taking place in the presence of ultraviolet rays or very intense light, preferably short radiations.

**Chlorinated rubber** Eugen Molloy (to Chemische Fabrik Buckau) U. S. 2,023,017, Dec. 17. See Fr. 768,157 (C. A. 29, 6409).

**Chlorinated rubber** Bosnische Elektrizitäts-A-G (Noc. L. Müller and Oskar Siglhuber, inventors). Austrian 143,522, Nov. 11, 1935 (Cl. 39a). An aq. suspension of mechanically disintegrated rubber is chlorinated, preferably at below  $40^\circ$ , until a product contg 30-40% of  $\text{Cl}$  is obtained. This product is further disintegrated in water or an org. solvent, and then rechlorinated until its  $\text{Cl}$  content is about 70%. The final product is sol. in org. solvents.

**Chlorinated rubber** Rubber Producers Research Association Fr. 786,102, Aug. 27, 1935. See Brit. 424,561 (C. A. 29, 5693).

**Chlorinated rubber** International Latex Processes Ltd. and John McGavack. Fr. 787,333, Sept. 20, 1935. Latex is stabilized to prevent premature coagulation of the rubber at the moment of chlorination and  $\text{Cl}_2$  is then introduced at least until isolated particles of chlorinated rubber ppt. The latex may be stabilized by vulcanization or by oxygenating or by adding glucosides, Na alkyl-naphthalene sulfonates or the product obtained by sulfonating phenols and hydrocarbons in the presence of unsatd. aliphatic acids, etc. Cf. C. A. 30, 649.

**Chlorinated rubber.** Deutsche Gold- und Silber-Schneidanstalt vorm. Roesler Fr. 787,354, Sept. 21, 1935. Solns. of chlorinated rubber in solvents such as  $\text{CCl}_4$  and  $\text{CHCl}_3$  are treated with small amts of alkali salts of org. acids such as HCN, propionic, stearic and oleic

acids which are themselves sol. in the solvent used and their alkali salts are sol. to a certain degree. The chlorinated rubber when freed from solvent and dried is found to have a high degree of stability.

**Preserving rubber** Winfield Scott (to Rubber Service Laboratories Co.) U. S. 2,024,477, Dec. 17. Rubber is treated with a S. deriv. of a diaryl amine (such as that of *p*-ethoxyphenyl-*p*-naphthylamine) a phenyl radical of which contains a nuclear alkoxy substituent only.

**Preserving rubber** Louis H. Howland (to United States Rubber Co.) U. S. 2,026,380, Dec. 31. About 1% or less of a product of reaction of an alkali metal such as Na upon a ketone-aromatic amine condensation product such as that from acetone and diphenylamine is added to a rubber compn.

**Preserving rubber** Albert M. Clifford (to Wingfoot Corp.) U. S. 2,026,517, Jan. 7. The reaction product of an amino deriv. of a diphenylene oxide and an aliphatic aldehyde such as  $\text{CH}_3\text{CHO}$ , crotonaldehyde, acrolein or aldol is added to rubber compns. (suitably in a proportion of about 1-5%) Cf. C. A. 30, 649.

**Coloring rubber** Imperial Chemical Industries Ltd. Fr. 786,392, Sept. 2, 1935. A finely divided coloring substance such as Indigo LL, Caledon blue R, Durindone blue 4 BC or Monolith red R, and a supplying agent comprising a compd. having a long alkyl chain, *n* below  $100^\circ$  and preferably about  $50^\circ$ , e. g., stearic acid, cetyl alc. or the condensation product of sulfonated  $\text{C}_{12}\text{H}_{25}$  with  $\text{CH}_3\text{O}$ , are intimately mixed with rubber latex and a dispersing agent is added. The mixt. is dried and made into sheets or other forms.

**Coloring rubber, etc.** I G Farbenind. A-G Fr. 786,389, Sept. 2, 1935. Dyes obtained by coupling diazo-benzene compds. contg the group  $-\text{CON}(\text{R})\text{R}'$ , not in the position ortho to the azo group, with 2,3-dihydroxy-naphthoic amides which contain the group  $-\text{CONR}^2\text{R}^3$  in the 3 position and which may also contain a halogen or oxyalkyl ( $\text{R}, \text{R}', \text{R}^2, \text{R}^3$  represent H, alkyl, aryl, aralkyl or a hydro-aromatic radical, or they may form heterocyclic nuclear systems but  $\text{R}$  (or  $\text{R}^2$ ) must not be H when  $\text{R}^3$  (or  $\text{R}^2$ ) is H and  $\text{R}$  (or  $\text{R}^2$ ) an aryl group). Thus, 1-amino-2-methylbenzene-5-(carbonylaminobenzene)  $\rightarrow$  2,3-dihydroxy-naphthoic ethylamide colors rubber a bluish red. Several examples are given.

**Mixture of size and rubber** Georg Goll and Ernst Helft. Fr. 618,657, Sept. 13, 1935 (Cl. 39b. 5). Size or glue is mixed with high mol. fatty acids and the product mixed with rubber. In the examples, size is mixed with stearyl or dodecyl alc.

**Rubber coatings.** Johann Tengler. Ger. 618,592, Sept. 11, 1935 (Cl. 22k. 3). Unsatd. substances such as fish oil, tall oil, wood pitch, etc., are vulcanized and the product is dissolved in an org. solvent with rubber or old rubber. The soln. is revulcanized, mixed with a diluent, and used as a paint for coating articles with a rubber-like layer. The org. solvent used is preferably an oily, fatty or resinic acid.

**Rubber articles** Edmond Basset. Fr. 786,084, Aug. 26, 1935. A colloidal dehydrating agent capable of absorbing a large amt. of water, e. g., "bentonite," "wulkenite" or "ardmonte" is applied to a form which is repeatedly dipped into rubber latex and exposed to air between each dipping until the latex is partly dehydrated. The process is continued until sufficient thickness of rubber is obtained.

**Apparatus for cutting and weighing lengths of material from a continuous strip of material such as rubber for tire manufacture** Charles C. Cadden (to B. F. Goodrich Co.). U. S. 2,025,801, Dec. 31. App. and various operative details are described.

**Apparatus for making inner-tire tubes** Wm. W. Potter. U. S. 2,024,304, Dec. 17. Structural, mech. and operative details.

**Forming hollow articles such as tire tubes.** Frank A. Daly (to Wm. W. Potter). U. S. 2,024,149, Dec. 17. App. and various operative details are described.

**Elastic yarn comprising rubber and suitable for knitting.** Buntwell H. Foster (to United States Rubber Co.). U. S.

2,024,155, Dec 17 Structural details U. S. 2,024,156 also relates to details of manufacture of an elastic yarn.

Hydrorubber yarn Thomas H. Byrton (to North American Rayon Corp.). U. S. 2,025,025, Dec. 24. A spinning soln for the manufacture of soft-luster products contains viscose or cuprammonium cellulose and a small proportion of a hydronubber.

Printing plates comprising rubber compositions of different degrees of hardness in different layers Stanley A. Danser (to Vivian M. Stacy-Bush). U. S. 2,026,021, Dec 31 Various structural details.

Splicing rubber articles such as unvulcanized rubber tubes Jorgen I. Haase (to Wingfoot Corp.) U. S. 2,024,577-8, Dec 17 Various details of app. and operation are described.

Apparatus for retreading tires Howard L. Shaw, U. S. 2,024,888, Dec 17 Structural details.

Attaching rubber soles to shoe bottoms Alexander D. Macdonald (to Boston Blacking & Chemical Co.) U. S. 2,024,235, Dec 17 The attaching surface of the rubber sole is treated with an aq. dispersion cont. rubber and an org. rubber solvent such as toluene and  $\text{C}_2\text{H}_5\text{Cl}$ , and a rubber adhesive is applied to the overlaid portion of the shoe bottom, the comps. are permitted to dry, the deposited rubber material is activated by a rubber solvent such as  $\text{C}_6\text{H}_6$ , contg. rubber and the materials are united under pressure. U. S. 2,024,235 relates to a generally similar process of attaching leather soles to shoe bottoms after treating them with a soln. of a low-viscosity rubber in an org. solvent. U. S. 2,024,237 relates to cementing channel flaps on a shoe sole by a method generally similar to that of U. S. 2,024,235. Phenyl- $\beta$ -naphthylamine is used as an antioxidant.

Rubber-lined metal barrels Claude S. Beldin (to Pennsylvania Salt Mfg. Co.) U. S. 2,025,932, Dec 31. Various mfg. details.

Rubber-like polymerization products E. I. du Pont de Nemours & Co. Fr 787,151, Sept 18, 1935. 2-Buta-1,3-diene, substituted by halogen, is completely or partly polymerized by air or O<sub>2</sub>, heat, light, pressure or catalysts. The butadiene is made into an emulsion before polymerization and the rapidity or degree of polymerization is retarded or lessened by an anticatalyst or a solvent. The catalyst may be  $\text{H}_2\text{O}_2$  or peroxides of Pb, Na or benzoyl.

Rubber-vulcanization accelerators Clayton O. North (to Rubber Service Laboratories Co.). U. S. 2,024,470, Dec 17. An accelerator is formed by the reaction of  $\text{CS}_2$  on a preformed aldehyde deriv. of a Schiff's base such as that formed by the reaction of 1.5-3.0 mols. of an aliphatic aldehyde, such as butyraldehyde with 1 mol. of an aromatic primary amine such as aniline.

Rubber vulcanization accelerator Lorin B. Sebrell (to Wingfoot Corp.) U. S. 2,024,605, Dec 17 Vulcanization of rubber is effected in the presence of a mercaptothiazole such as mercaptobenzothiazole in combination with a compd. such as  $\text{NH}_3$ , an amine having a primary amine group such as ethyl or propyl amine or other aliphatic amine, or an alkyl aryl amine such as ethyl aniline, etc. U. S. 2,024,596 relates to mercaptan derivs. which are obtained by reaction of a mercaptothiazole with  $\text{NH}_3$  or an amine, suitable for use as accelerators.

Rubber vulcanization accelerator Waldo L. Semon (to B. F. Goodrich Co.). U. S. 2,025,276, Dec 31. A tetraaryl substituted thuram sulfide such as a diphenyl-di- $\beta$ -naphthyl thuram sulfide is used as a vulcanization accelerator.

Vulcanization accelerators The Rubber Service Laboratories Co. Brit. 434,506, Sept. 2, 1935. An isocyclic said org. base, e.g., decabicyclo-o- and - $\beta$ -naphthylamine, bornylamine, di- $\gamma$ -naphthylamine, carylamine, cyclohexylamine, dicyclohexylamine, cyclohexylmethylamine, methylbornylamine is caused to react with a mercaptoarythiazole or di- or poly-sulfide thereof, or with thuram di- or

poly-sulfide, or with arylthiocarboxylic acid disulfides, e.g., mercaptobenzothiazole, mercaptotolylthiazole, dibenzothiazyl di- and poly-sulfides, tetramethylthuram di- and poly-sulfides, tetraethylthiuramdisulfide, dithiobenzac acid disulfide, dithioisobutylic acid disulfide.

Vulcanization accelerators The Rubber Service Laboratories Co. Ger. 618,025, Sept. 21, 1935 (Cl. 398.8). An accelerator for rubber vulcanization is obtained by treating a ketoneamine with a mercaptoarythiazole. Diphenylguanidine may be added to the accelerator. In an example, the accelerator is produced by treating dacetone amine with mercaptobenzothiazole. Several other examples are given. Cf. preceding abstract.

Vulcanization accelerators E. I. du Pont de Nemours & Co. Brit. 434,506, Sept. 5, 1935. A 2 mercaptoarythiazole is caused to react with a halogenated ketone of formula  $\text{RCH}_2\text{XCOR}'$ , where X is a halogen atom and R and R' are alkyl groups. In examples 2 mercaptobenzothiazole is condensed with 2-chlorobutanone-3, 3-chloropentanone-2 and 2-chloropentanone-3 and details showing the accelerating properties of the compds. in a rubber mix are also given.

Dithiazyl disulfides Albert M. Clifford (to Wingfoot Corp.) U. S. 2,024,567, Dec. 17. For prep. a dithiazyl disulfide such as dibenzothiazyl disulfide, a thiazyl mercaptan such as 1-mercaptobenzothiazole is oxidized with  $\text{H}_2\text{O}_2$  in the presence of an inorg. acid such as  $\text{H}_2\text{SO}_4$ . Various examples are given.

Dithiazyl disulfides Albert J. Gracia (to Wingfoot Corp.) U. S. 2,024,575, Dec. 17. Dibenzothiazyl disulfide is prep'd by heating an aq. soln. of mercaptobenzothiazole and mixing with it an aq. soln. of  $\text{H}_2\text{O}_2$  and an amt. of  $\text{H}_2\text{SO}_4$  sufficient to neutralize the alk. soln., filtering, washing and drying the ppt. formed. Similarly, other related compds. may be obtained.

Hydraulic presses for vulcanizing, veneering, etc. Josef van Liempt. Brit. 435,012, Sept. 12, 1935.

Apparatus for vulcanizing belting Fernand Van Camp (to Soc. anon. des établissements J. Laroche-Lechat) U. S. 2,024,811, Dec. 17. Structural and mech. details.

Apparatus for vulcanizing tire casings John G. Ebenhaek. U. S. 2,024,279, Dec. 17. Various structural, mech. and operative details.

Apparatus of the "full circle" type for vulcanizing tires, etc. Leslie T. Vogt. U. S. 2,024,554, Dec. 17. Structural and mech. details.

Retreading vulcanizer for tires Charles E. Miller. U. S. 2,024,941, Dec. 17. Structural, mech. and operative details.

Manufacture and vulcanization of tire casings Laurits A. Laursen (one-fourth to Pearl F. Laursen) U. S. 2,025,993, Dec. 31. App. and various operative details are described.

Vulcanizing tire casings Laurits A. Laursen U. S. 2,025,992, Dec. 31. App. and various operative details are described.

Vulcanizing rubber Continental Gummi-Werke A.-G. Ger. 618,658, Sept. 13, 1935 (Cl. 398.7). Rubber objects are coated with rubber latex or a rubber-latex mixt. and vulcanized in the mold.

Rubber vulcanization. Th. Goldschmidt A.-G. (Hans Schneider, inventor). Ger. 618,756, Sept. 14, 1935 (Cl. 398.8). The vulcanization of natural or synthetic rubber, etc., is accelerated by an addn. of  $\text{PbO}$  and alk. earth sulfide to the vulcanization mixt. Examples are given.

Vulcanizing product Cela Holding S. A. Fr 786,267, Aug. 20, 1934. A vulcanizing agent for latex, particularly that used for impregnating textiles, contains the element (Zn and S) necessary for the vulcanization in the form of a colloidal dispersion. Thus,  $\text{Zn}(\text{OH})_2$  or a salt of Zn with a weak acid is dissolved in  $\text{NH}_3$  soln., mixed with a soln. of an org. substance such as albumin, casein, gelatin, agar-agar or blood serum and a  $\text{NH}_4$  polysulfide is added.

# CHEMICAL ABSTRACTS

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## I—APPARATUS, PLANT EQUIPMENT AND UNIT OPERATIONS

W. L. BADGER

Chemical plant construction of the present and future Hurlbut S. Jacoby *Chem. & Met. Eng.* 42, 674-7 (1935), cf. C. A. 29, 1477.<sup>1</sup> C. H.

Electro-ultrafiltration apparatus E. J. Czarnetzky *Science* 82, 625-6 (1935).—The app. was developed for sepg. Hg salts from Hg-protein solns and consists of a collodion-covered aluminum thimble contg. a Pt electrode, and a wide glass tube contg. another Pt electrode and with the opening of a short, wide side-arm covered with Cellophane. The glass tube is open, the thimble is closed with a rubber stopper connected to a vacuum line. Both are dipped into the soln and when the current is applied the gradient in potential discharges the protein from the collodion surface of the thimble and prevents clogging, which is the essential feature of the app. *Ch. C. A.* 21, 1074. J. H. Moore

Semimicro Cottrell boiling-point apparatus Mary L. Willard and Delena E. Crabtree *Ind. Eng. Chem., Anal. Ed.* 8, 79-80 (1936).—The app. is one-tenth the size of that referred to in C. A. 29, 381, and results accurate to 0.1° are obtained on as little as 5-cc. samples. J. H. Moore

An electrically heated melting point apparatus Edwin Doward and Michael J. Russo *Ind. Eng. Chem., Anal. Ed.* 8, 74-5 (1936).—The app. consists of a round battery jar contg. a resistance wire-wound test tube. The latter contains a smaller test tube in which the m-p capillary and thermometer are suspended. It is suitable for the detn. of m-p's up to about 310° with a reproducibility of within 0.5°. Allen S. Smith

An automatic recording balance D. S. Binnington and W. F. Geddes *Ind. Eng. Chem., Anal. Ed.* 8, 76-9 (1936).—The app. was designed for use in drying macerous products, and losses up to 17 g. on a 100-g. load are shown to  $\pm 0.01$  g. It is electrically operated and the chart will cover periods up to 6 days. Construction details are shown. J. H. Moore

A new type of calorimeter for the measurement of continuous heat effects W. Swietoslowski *Roczniki Chem.* 15, 342-9 (in French 349-50) (1935).—A flow calorimeter suitable for the detn. of such continuous heat processes as the hardening of cement is described. In principle the calorimeter consists of a labyrinth of concentric tubes through which water or other calorimetric liquid circulates from the outside to the middle, to avoid loss of heat. The system evolving heat is placed in the central portion and the temps. of the liquid at the entrance and exit are measured. The effect is calcd. from the temp. difference and the amt. of liquid flowing. M. W.

Development of an electric light calorimeter Th. W. Schmidt. *Z. Instrumentenk.* 55, 339-48, 357-67 (1935).—A math. discussion of the errors as compared with calorimeters using a gas lamp. The new calorimeter has 0.4% error but is not so satisfactory for plant use. Thirty-eight references. Ann. Nicholson Hurd

An inexpensive ball mill. Laurence L. Quill. *Ind. Eng. Chem., Anal. Ed.* 8, 27 (1936); cf. *Furnst. C. A.* 29, 1727. L. L. Quill

A precision pycnometer for liquids. S. T. Yuster and L. H. Reyerson *Ind. Eng. Chem., Anal. Ed.* 8, 61-2 (1936).—The pycnometer described is believed to eliminate difficulty of thermostating both liquid and container, evapn. losses at ground-glass joints, loss of liquid during filling and the difficulty of detg. the d. at some definite

temp. Sizes from 0.1 to 25 cc. were used. It was possible to check wts. to 0.1 mg. Allen S. Smith

A differential refractometer D. Rau and W. E. Roseveare *Ind. Eng. Chem., Anal. Ed.* 8, 72-3 (1936).—The app. described was designed to be a rapid and reliable instrument for detg. concns. of solns too dil. to permit the use of the Pulfrich or immersion refractometers. It is similar in principle to the Ketteler and Haber-Löwe instruments but is more practical because of a new method of observing and measuring the deflection and of detg. the zero reading. It has been used to det. the concns. of aq. solns with  $n_D$  from 0.0004 to 0.002 greater than pure water with an accuracy of 0.1%. Allen S. Smith

Drum dryers Charles R. Harte, Jr. *Ind. Eng. Chem.* 28, 7-10 (1936).—A review describing types, uses and economic factors. F. W. Tillitson

The use of a new valve for the flow of gases through a slit V. Kunzl and J. B. Slavik *Ann. Physik* 24, 409-20 (1935).—The new valve consists of a glass tube with an external diam. 1.95 cm., internal diam. 1.75 cm., which has spontaneously split longitudinally to a length of 2.3 cm. The slit varies (0.58-2.3)  $10^{-4}$  cm. under different pressures. The flow of air through this valve has been measured under differences of pressure of 200-700 mm. Hg. The values  $(p_1 - p_2)$  of  $G = V_1/(p_1 - p_2)$  agree with the theoretical ones derived from Poiseuille's formula. Poiseuille's formula obtains if the smallest dimension of the slit is at least 10 times the mean free path of the mols. passing through it. Data and curves are given. I. R. Scherz

New extraction apparatus Zoltán Silyom-Barna. *Magyar Chem. Folyóirat* 41, 94 (1935).—An Erlenmeyer flask is provided with a cooler ground into the stopper and a small extn. bottle suspended from the end of the cooler into the Erlenmeyer flask by means of a Pt wire. This bottle contains also a suction pipe to accelerate circulation of the extn. agent. Structural details and dimensions are given. S. S. de Finály

Phosphorus pipet for oxygen determination Zoltán Silyom-Barna. *Magyar Chem. Folyóirat* 41, 94-6 (1935).—Rods of P are kept under water within the lower part of the pipet and can be pushed up from outside by means of a glass rod. Structural details are given. S. S. de Finály

New applications of the expansion pyrometer. P. Chevenard. *Génie civil* 107, 424-7 (1935).—Construction improvements have made this type of pyrometer sufficiently robust for factory use, either as a control app. or as an automatic heat regulator. Drawings and photographs explain the construction and working principles of the pyrometers and their use in grain-growth studies. Ann. Nicholson Hurd

Maintenance of partial radiation pyrometers by standardization in practice J. Marwedel *Glastech. Ber.* 13, 416-20 (1935).—The use of a tungsten lamp is discussed in which the filament is a notched band. J. F. Hyde

Increasing sensitivity of bimetal thermometers J. Blair Dowden. *Product Eng.* 6, 389-9 (1935).—A multiple helix form of bimetal thermometer is described with photographs and the underlying principles are discussed. A single strip is wound in the form of concentric helices. The several coils are additive in rotational deflection, but the axial displacement is mutually counterbalanced between the coils. A thermal element no larger than the

bulb of a thermometer is accurate to fractions of a degree

Ann Nicholson *Hrd*  
Publications in the field of gas analysis in 1934 II  
Löffler *Z. osterr. Ver. Gas- u. Wasserfach* 75, 210-18 (1935) E. H. I.

Note on a routine gas analysis apparatus II K. Sen, Kanai Roy and Pankaj Roy *J. Indian Chem. Soc.* 12, 654-58 (1935)—The app. contains one novel feature, a vacuum-creating device for removing residual gases from the combustion chamber. Samples of 0.5-3 cc can be analyzed. Drawing of app. and data are given.

A simple air-analysis apparatus Tsun Chee Shen *Chinese J. Physiol.* 9, 363-5 (1935)—A simple app. for student or clinical use. L. A. Maynard

Prevention of foaming in crude fiber determinations H. W. Gerritz *Ind. Eng. Chem., Anal. Ed.* 8, 75 (1936)—A fine jet of air is blown against the surface of the liquid through a tube extending down through the condenser. J. H. Moore

An absorption apparatus for the microdetermination of certain volatile substances III The microdetermination of chloride with application to blood, urine and tissues Edward J. Conway *Biochem. J.* 29, 2221-35 (1935); cf. *C. A.* 27, 3537—A microdetn. of chloride is given; the absorption app. described by C., whereby the chloride is oxidized to Cl<sub>2</sub> with KMnO<sub>4</sub> at 20-30° and absorbed in 1 ml of 20% KI soln., is used. The liberated I<sub>2</sub> is titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> with a Conway buret (*C. A.* 28, 3941) or when the chloride content is between 7 and 35%, colorimetrically. For amts. between 7 and 0.7%, 0.5 ml of 0.2% starch is added to the KI soln. and the color is detd. colorimetrically. The coeffs. of variation for the 3 procedures are 0.5, 4-5 and 6-7%, resp. The "grey soln." of Thiel and a colorimeter provided with spectral filters were used instead of standards. The method is independent of the presence of iodide but bromide must be removed or detd. by a method to be described later. Protein up to 1 mg. in the soln. analyzed does not interfere. The blood chloride in 1 ml. of tungstate filtrate (0.1 ml. blood) can be detd. with a 0.5% variation; the urine chloride in 0.1 ml. or 1 ml. of dild. urine with a 0.7% variation and the tissue chloride in 0.2-g. samples (99% recovery of chloride added to ground ox. muscle). Protein in urine up to 1% does not interfere. E. W. S.

A sensitive check valve E. L. Green *Ind. Eng. Chem., Anal. Ed.* 8, 40 (1936)—A glass float seating within a ground joint in a gas delivery tube effectually seals the tube when a reverse pressure causes water to rise in the tube. Allen S. Smith

The development of the air-driven spinning top as transparent ultracentrifuge James W. McBain and Carroll M. O'Sullivan *J. Am. Chem. Soc.* 57, 2631-41 (1935)—The air-driven top-type centrifuge has been described at length. Speeds of 3000 r. p. m. have been obtained. Sedimentations of certain heavy mol. materials were made having mol. wt. as low as 208,000. Photographing is done by transmitted light. The present stage of development of the instrument is given. R. H. L.

A rapid portable stirrer for the manufacture of glues and adhesives Wollenberg *Deut. Wollen-Gewerbe* 67, 600-1 (1935)—Two of the stirrers shown and described are hand-driven; 2 others are electrically operated. The small stirrers are suitable for lab. purposes while the larger ones can be employed in large-scale operations. Leopold Scheffan

Calculation of condenser coolers I. G. Zak *Neft* 3, No. 22, 15-18 (1932)—Math. A. A. Boeltingk

Lessons on rectification A. M. Tregubov *Nefteprom. Khimika* 28, No. 7, 38-41 (1935)—A mathematical analysis of the performance of bubble towers is presented. A. A. Boeltingk

An apparatus for sugar and other titrations Edward S. West *Ind. Eng. Chem., Anal. Ed.* 8, 62 (1936)—The app. consists of a buret fitted with a syphon automatic leveling device and a hypodermic needle tip, and a stirring rod actuated by a vacuum windshield motor. Allen S. Smith

Decantation of crystalline suspensions I General theory II III G. Bozza *Atti accad. Lincei* 21, 697-700, 752-5, 809-13 (1935)—Math. A. W. C.

Automatic method for neutralization and registration of pH V. S. Ledneva, B. I. Petrov and M. A. Proskurnin *Zashchita Lab.* 4, 1007-13 (1935)—An app. for potentiometric regulation and recording of pH and neutralization of various liquids with the use of a W electrode is illustrated and described. Chas. Blanc

Air conditioning frees rotogravure plant from weather hazards II E. Jacobsen *Heating, Piping, Air Conditioning* 7, 423-4 (1935).—The most desirable dry-bulb temps. are: etching department 72°F., photogallery 74°F., retouching department 75°F., sensitizing of carbon printing 72°F., dark rooms 74°F. and drying room 75°F. A relative humidity of 50% is best for all departments. M. W. Schwarz

Piping and pumping process materials Leo P. Hynes and Charles D. Campbell, Jr. *Heating, Piping, Air Conditioning* 8, 3-6 (1936). M. W. Schwarz

Welding in the chem. engineering industry (Ingels, Andrews) 9. Fabrication of monel metal in chem. equipment (Nakamura) 9. Detn. of fixed points of low temp. with a H. thermometer (Anyama, Kanda) 2. Dry-cleaning (Brit. pat. 435,575) 23. App. for removing moisture from the air (Brit. pat. 434,666) 13.

Apparatus for obtaining pure distilled water for estimation of pH values Solomon F. Acree *Ger.* 619,616, Oct. 4, 1935 (Cl. 858) 4.

Apparatus for electrometric volumetric analysis I G. Farbenind, A.-G. (Udo Ehrhardt, inventor). *Ger.* 622,048, Nov. 18, 1935 (Cl. 421) 3 (04). This corresponds to Brit. 364,939 (*C. A.* 27, 1787).

Catalytic apparatus Soc. française de catalyse générale *Fr.* 787,896, Sept. 30, 1935. The space in which the gaseous reaction mixt. circulates is divided into a no. of narrow rectilinear conduits, e.g., the gas passes through a no. of long tubes and returns along the spaces between these tubes and outer surrounding tubes, the catalyst being in these spaces.

Apparatus for making chlorine solutions from liquid chlorine and water Georg OrNSTEN. *Ger.* 622,148, Nov. 21, 1935 (Cl. 12) 4.

Precipitating Arne J. Myhren and Byron Marquis (to The New Jersey Zinc Co.). *Brit.* 435,257, Sept. 18, 1935. The production of ppt. by reaction between a gas and a soln. is effected by causing the soln. repeatedly to entrain large vols. of the gas. The process may be carried out by passing the gas and soln. or slurry obtained by reaction through a series of tanks, withdrawing slurry from each tank and returning thereto a part thereof intimately mixed with a considerably larger vol. of gas withdrawn from the tank while conveying the remainder of the slurry to the next succeeding tank intimately admixed with gas and withdrawing from the last tank an amt. of slurry substantially equiv. to the amt. of soln. introduced into the 1st tank. App. is described.

Closures for double-walled vacuum flasks Hugo Scheider A.-G. *Brit.* 435,832, Sept. 30, 1935.

Vacuum mixing apparatus suitable for use with volatile substances Everts G. Loomis U. S. 2,027,185, Jan. 7

Rotary classifiers, applicable to ball-and-rng mills Ralph M. Hardgrove (to Babcock & Wilcox Ltd.). *Brit.* 434,923, Sept. 11, 1935.

Jacketed kettles or tanks with agitating and scraping devices for various heating and cooling operations Geo. H. Tay (to Lee Metal Products Co.). U. S. 2,027,756, Jan. 14. Various structural, mech. and operative details.

Box for holding carboys Allan W. James U. S. 2,019,890, Nov. 5. Structural features.

Rotary filter Wm. Rausch (to Municipal Sanitary Service Corp.). U. S. 2,027,652, Jan. 14. For removing scale from the drum of a rotary filter, a liquid spray is thrown on the surface of the drum in a direction opposite to the direction of movement of the surface and scale ma-

terial is removed in a continuous sheet by a rearward and outward brushing action on the surface in advance of the spraying action. App. is described.

Air filter. Lee Hand. U S 2,027,906, Jan 14 Structural details

Filter for air and gases Hundt & Weber G m. b H Fr 789,702, Oct 15, 1935

Filter belt for roller presses Adolph W Lissauer (to Louisville Drying Machinery Co.) U S 2,027,542, Jan 14 Structural details

Filtering material Società italiana Pirelli Ir 788,603, Oct 14, 1935 A filtering mass for aerosols comprises a fibrous material and inert powders, the fibrous material being in the form of porous felt The felt is immersed in a soln of the inert powder, dried and submitted to a mech treatment to break up the crusts and granulations of the powder Cf C A 30,909

Apparatus for filtering and thickening slimes or sludges from mining and other industrial processes Henry T Durant and Eric O Stubbings (to Blomfield Engineering Co, Ltd.) U S 2,027,681, Jan 14 Various structural and operative details

Apparatus for coagulating and filtering liquids Robert B. Morse Fr 789,548, Oct 11, 1935

Apparatus (with a centrifugal separator and filter) for separating solid matter entrained in air or other gases Ludwig F. Holty. U S 2,026,834, Jan 7 Various structural, mech and operative details

Rotary screen separator Geo H Fraser U S 2,027,375, Jan. 14. Structural and mech details

Gravity-separation apparatus Horace J Greaves Brit. 435,050, Sept 13, 1935 The outflow of H<sub>2</sub>O from a settling tank or pump sump to the circulating system of a coal washing plant or to waste is automatically controlled so that wastage does not occur on stoppage or failure in the washing app. or other part of the circulating system

Separating heaped materials by the action of gas streams Henry M. Chance. U S 2,022,585, Nov 20 App and various operative details are described

Apparatus for straining gasoline or other liquids or gases. John Wardle (to Lolo Strainers Ltd.) U S 2,022,016-17, Nov. 20 Various structural and operative details

Sifting-apparatus. Frank Pascall. Brit. 435,344, Sept. 19, 1935

Plansifters comprising a plurality of superposed sieve frames. John Speight and Henry Simon Ltd. Brit. 435,328, Sept. 19, 1935.

Centrifugal apparatus for separating dust from gases. François Jacques Berry Fr. 787,897, Sept. 30, 1935.

Apparatus for removing dust from gases Pierre Frémont. Fr. 788,115, Oct. 4, 1935 The gases are caused to impinge on the surface of walls down which water flows.

Apparatus for washing gases. Wm Learmonth, Gordon Nonhebel, John L. Pearson and Imperial Chemical Industries Ltd. Brit. 434,590, Sept. 5, 1935 Divided on 424,414 (C. A. 29, 49787). A gas-scrubber comprising a main body of packing consisting of transverse tiers of thin laths with vertical flow of gas and liquid has relatively deep plates arranged vertically side by side at the gas inlet in the path of the scrubbing liquid, the plates being spaced at a greater distance apart than the thin laths and having enlarged tops so that the plates receive a greater proportion of scrubbing liquor per unit area than the laths.

Apparatus for washing gases Axel Larsen Brit. 435,047, Sept. 13, 1935. Dust-laden gases are led through loose bodies, e. g., balls, rings, spirals or short pieces of Fe, resting on the inner wall of a stationary drum less than half filled therewith; fixed transverse positions prevent the gases passing through the free space in the container without coming into contact with the bodies, which are agitated by stirrers mounted on an axle above the level of the bodies and are kept moist by liquid added at the top and withdrawn by pipes.

Apparatus for washing gases Wm. H. Yardley. Brit. 435,550, Sept. 19, 1935. In app in which liquid falls

over several rows of helical blades disposed vertically in a casing, the blades in each row touch one another and the blades of 1 row are staggered with respect to and spaced apart from those in adjacent rows to form tortuous passages for the horizontal passage of the gas

Treating air with liquids Max Berlowitz Brit. 434,671, Sept 6, 1935. For moistening, cleansing, heating or cooling air, a motor-driven centrifugal pump dips into liquid and raises it over its top edge to deliver it over a perforated container packed with filling bodies through which air is forced by a fan co-axial with the motor

Gas analysis apparatus Matyan P Matuszak (to Fisher Scientific Co.) U S 2,026,842, Jan 7 Various structural and operative details

Automatic periodically operating gas-analysis apparatus H Mahak A-G (Richard Prantzs, inventor). Ger 622,137, Nov 21, 1935 (Cl 421 4 04)

Apparatus for dispensing gas such as oxygen from a liquefied gas supply Geo J Boshkoff (to Linde Air Products Co.) U S 2,028,110, Jan 14 Various details of app and operation

Apparatus for transferring liquefied gases William F Messenger (to Dominion Oxygen Co Ltd.) Can 353,938, Nov 5, 1935

Purifying liquids The Dorr Co Inc Fr 788,042, Oct 2, 1935 App is described for passing a current of the liquid through a flocculation or coagulation zone and bringing the liquid to a calm zone where the flakes are pptd. A slowing down of the flakes by gravitation in an ascending current is produced to obtain a zone concd with flakes and this zone is submitted to a supplementary agitation transverse to the direction of the current.

Centrifugal machine for separating liquids. Aktiebolaget Separator Brit 435,241, Sept 17, 1935

Drives for centrifugal machines The British Thomson-Houston Co Ltd Brit. 435,982, Oct. 2, 1935 The elec. motor of the short-circuited rotor type for driving the machine is slowed up during the filling operation by the automatic insertion of a single-phase stator resistance.

Centrifugal apparatus for disintegrating and dewatering wet raw materials. Alexander Dexter and Martin Ziegler. Ger 622,228, Nov. 22, 1935 (Cl 10c 7)

Centrifugal apparatus for removing oil from thick neutralization pulp Aktiebolaget Separator. Fr. 788,500, Oct 12, 1935.

Progressive evaporators. The Thermal Syndicate Ltd. and Burrows Moore Brit. 435,134, Sept. 16, 1935. Towers for the concn of liquids by evapn or absorption contain a no of transverse members, each having 1 or more openings provided with orifices or notches.

Multiple evaporator for sirup, etc Hermann Claassen. Ger. 619,822, Oct. 7, 1935 (Cl 12a. 5)

Evaporating foaming liquors such as black liquor from cellulose manufacture. Erik Oman and Sven H. Ledin (to Industriemiska Aktiebolaget). U S 2,027,115, Jan. 7. In passing a current of gas under pressure through a layer of the liquid, the gas is divided into streams substantially uniformly distributed with respect to the layer of liquid and which have an individual width of preferably at least 1.5 mm. and not more than 12 mm. and pitches at least 2 5 times the width of each stream. App. is described

Drying apparatus Bernard M. Carter (to General Chemical Co.). Can. 353,947, Nov 5, 1935 Operative and structural details are given.

Tube drier Zeitzer Eisengieserei und Maschinenbau-A-G Ger. 619,730, Oct. 10, 1935 (Cl. 82a. 23.01).

Conveyor-band apparatus for drying ammonium sulfate Elektra A-G für Elektrotechnik. Ger. 621,530, Nov. 8, 1935 (Cl 12a 2). Addn to 618,363 (C. A. 30, 74)

Kiln for drying granular and like substances, e. g., oats George S Hamilton. Brit. 435,077, Sept 13, 1935.

Muffle furnaces Henry W. Spencer and Leonard S Davis. Brit. 430,180, Oct. 7, 1935. In a furnace having its side walls and roof formed of refractory blocks suspended, resp., from angle beams and overhead girders,

the muffle itself is constituted by tiles keyed into and supported by the blocks

**Rotary tubular furnace** I G Farbenind. A-G Fr. 788,306, Oct 8, 1935 The furnace is surrounded by heat-insulating means which can be regulated by opening or closing apertures

**Regenerators** Soc anon d'Ougrée-Marhay Brit 435,162, Sept 16, 1935 An integral honeycomb element for Cowper type regenerators comprises at least 3 layers of superposed and intersecting bars

**Valve system for regenerative furnaces** Otto Reiner Ger 623,174, Nov 21, 1935 (Cl 24c 7 03)

**Closure device for a furnace observation opening** The British Thomson-Houston Co Ltd Brit 435,292, Sept 18, 1935

**Apparatus for furnace-combustion control** Shirley S Weeks U S 2,027,942, Jan 14 Various mech. and operative details

**Annealing furnace** Hans Cramer. Ger. 619,022, Oct 11, 1935 (Cl 18c 10 01) Details are given

**Apparatus for charging annealing furnaces** Homer Buck and Harvey W Ball (to Morgan Engineering Co.) U S 2,028,045, Jan 14 Mech features

**Apparatus for charging furnaces with articles in annealing boxes** Harvey W Ball (to Morgan Engineering Co.) U S 2,028,038, Jan 14 Various structural, mech and operative details

**Water heater for utilizing heat of waste gases** Peter W Kaiser U S 2,020,686, Nov 12 Various structural and operative details

**Heat-exchange system suitable for use with chemical solutions** Kurt V Nesselmann and Edmund Altenkirch (to Siemens-Schuckertwerke A-G) U S 2,027,610, Jan 14 Various details of app and operation are described

**Tube and fin heat exchange apparatus** Anthony F Hoessel U S 2,019,967, Nov 5 Structural details

**Tubular heat-exchange apparatus for use in sterilizing liquids** Geo J van Zijderveld, Jr, Geo J van Zijderveld, Sr, Johannes van't Hoff and Leonardus de Jonge (trading as Syndicate Jozydhoff) Brit 433,760, Aug 20, 1935

**Gilled tubes for heat-exchangers** Auguste Tummers Brit 435,895, Oct 1, 1935

**Crucibles** Alfred Kropf and Stahlwerke Röhring-Buderus A-G Ger 622,275, Nov 23, 1935 (Cl 31a. 3 70) Addn to 469,433 (C A 23, 1732) The method of prep crucibles of Ta carbide, described in Ger 469,433, is modified by adding to the initial mixt a metal in between 1100° and 3000°, e g, Cu, Fe, Ni, Cr, Ti, W or Mo The mech properties and the elec cond of the crucibles are thus improved Crucibles of Zr carbide or of mixed Ta and Zr carbides may be made in the same way

**Heating and cooking vessels** Wm H Hatfield and Joseph F. Bridge Brit 436,229, Oct 7, 1935 The undersurface of an austenitic stainless steel vessel is coated with a metal of high thermal cond, e g, Cu, Al, applied by spraying

**Ball mills divided by longitudinal partitions into chambers open toward the periphery of the tube** Mikael Vogel-Jørgensen Brit 435,046, Sept 13, 1935

**Impact pulverizers** Edmund E Bental Brit 435,925, Oct 1, 1935

**Gyrator crushers** Humboldt-Deutzmotoren A-G Brit 435,959, Oct 2, 1935

**Crushers with gyrator heads** Harvey H. Rumpel. Brit 435,237, Sept 17, 1935

**Rolling and crushing mills** Venesta Ltd and Angus Love Brit 435,367, Sept. 19, 1935

**Hygrometric and thermostatic apparatus suitable for recording humidity** Albert E Clawson (to Taylor Instrument Cos.) U S 2,027,959, Jan 14 Various structural and operative details

**Conditioning and dehumidifying air, as by the use of calcium chloride** Charles R Downs (to Weiss and Downs, Inc) U S 2,026,935-6, Jan 7. Various details of app and operation

**Use of calcium chloride for air conditioning and dehumidifying** Charles R Downs (to Weiss and Downs, Inc) U S 2,027,993, Jan 7 Lumps of CaCl<sub>2</sub> are used in which finely divided activated carbon is distributed and held for deodorizing the air treated U S 2,027,094 relates to a process in which air is treated successively with a soln of CaCl<sub>2</sub> and with solid CaCl<sub>2</sub> and describes app

**Air-conditioning apparatus** Pierre R. M Willoughby Brit 435,372, Sept 19, 1935 The air enters a conduit through sieves coated with resin to retain the dust, passes through a water spray, is cooled and finally passes through an ozonizer and is discharged by a fan

**Air conditioning system suitable for railway passenger cars** Milton E Hanson (to B. F. Sturtevant Co) U S 2,027,097, Jan 7. Various structural and operative details

**Air-conditioning and -cooling system (suitable for railway cars)** Charles R Neeson (to Baldwin Southwark Corp) U S 2,027,058, Jan 7. App. and various operative details are described.

**Air-moistening and -conditioning apparatus** John O Guffey U S 2,026,974, Jan 7. Structural, mech and operative details

**Apparatus and operative details for cooling and aerating comminuted solids such as ammoniated phosphate fertilizer** Augustus J Sackett. U S 2,028,413, Jan. 21 Various structural and operative details

**Bin with agitating apparatus and supplied with air under pressure for homogenizing pulverulent materials such as cement raw meal** Niels Nielsen (to F L Smidth & Co) U S 2,027,697, Jan 14 Various structural and operative details

**Apparatus for drying sheet material such as cloth** Frank B. Morrill (to James Hunter Machine Co) U S 2,027,317, Jan 7. Various structural, mech and operative details

**Apparatus for testing liquids such as lubricants by a rotor and closely fitted casing** Jean G Dintilhac (to "L'Huile des Records du Monde S A F.") U S 2,027,903, Jan 14 The liquid is fed into an adjustable space between a frusto-conical rotor and a surrounding casing with closely spaced walls of like shape and an atm of N may be used in the app. Various details of structure and operation are described

**Press (with a rotary drum and endless caterpillar chains) for dehydrating colloidal materials such as peat** Mathieu van Roggen and Leo Robin U S 2,027,657, Jan. 14 Various structural, mech and operative details

**Means for controlling the level of conducting liquid or flowing material in a container** Evershed & Vignoles Ltd and John C Needham. Brit. 435,426, Sept 20, 1935

**Apparatus and method for making foam** Komet Kompagnie für Optik, Mechanik und Electro-Technik G m b H Brit. 435,979, Oct 2, 1935 In prep. air foam, the foaming liquid and air are drawn into and forced through a mixing chamber by a steam injector Cf C A 29, 2984<sup>4</sup>

**Apparatus for converting solid carbon dioxide into liquid and gas** Eugene L Ragonnet (to Pure Carbonic Co of America) U S 2,026,799, Jan 7. Structural details

## 2—GENERAL AND PHYSICAL CHEMISTRY

FREDERICK L. BROWNE

Walter Ernest Adeney (1857-1935). A G G Leonard J. Chem. Soc. 1935, 1891-2.—Obituary. G G

Kenneth Frankland Armstrong (1909-1935). R. Robinson J. Chem Soc 1935, 1892.—Obituary. G G

Herbert Brereton Baker (1862-1935). J. C. Philip. *J. Chem. Soc.* 1935, 1893-6—Obituary with portrait. G. G.

Arthur Bramley (1878-1935). J. C. Philip. *J. Chem. Soc.* 1935, 1896-7—Obituary. G. G.

Samuel Francis Burford (1857-1935). Samuel B. Bratley. *J. Chem. Soc.* 1935, 1897-8—Obituary. G. G.

Harry Cooper (1888-1935). H. A. D. Jowett. *J. Chem. Soc.* 1935, 1898—Obituary. G. G.

Egbert Grant Hooper (1855-1935). J. J. Fox. *J. Chem. Soc.* 1935, 1899—Obituary. G. G.

Clara Thomas Kingzett (1852-1935). G. T. Morgan. *J. Chem. Soc.* 1935, 1899-1902—Obituary. G. G.

Charles Richet. C. Achard. *Compt. rend. soc. biol.* 120, 927-9 (1935)—Obituary. L. E. Gilson.

George Wm. Slater (1851-1935). F. W. Richardson. *J. Chem. Soc.* 1935, 1903-4—Obituary. G. G.

Christopher Calger Smith. F. G. Donnan. *J. Chem. Soc.* 1935, 1904—Obituary. G. G.

Andrew Jamieson Walker (1873-1935). Frederick Challenger. *J. Chem. Soc.* 1935, 1904-7—Obituary. G. G.

Brauner Memorial Lecture. S. I. Levy. *J. Chem. Soc.* 1935, 1876-90—Biography and portrait of Bohuslav Brauner. G. G.

An ancient Chinese alchemical classic. Ko Hung on the gold medicine and on the yellow and the white. The fourth and sixteenth chapters of Pao p'u tzu. Translated from the Chinese by Lu-Ch'ing Wu, with an introduction, etc. by Tenney L. Davis. *Proc. Am. Acad. Arts Sci.* 70, 221-84 (1935). I. H.

High-school science a foundation for science courses in college. Ralph L. Horton. *Science Education* 19, 163-9 (1935). W. H. Boynton.

German chemical books and journals in their cultural significance at home and abroad. P. Walden. *Chem.-Ztg.* 59, 873-5 (1935). E. H.

Chemical elements and the unity of matter. H. F. A. Paneth. *Scientia* 58, 272-9 (1935). cf. C. A. 30, 337<sup>1</sup>. L. E. Gilson.

Recent methods of determination of molecular weight. Karl Rast. *Chem.-Ztg.* 59, 853-7 (1935).—A review. E. H.

The ionization balance in the Arctic. Joachim Scholz. *Gerland's Beitr. Geophysik* 44, 306-20 (1935). J. F. S.

Deuterium as a research tool in the physical and biological sciences. Herrick L. Johnston. *Ohio J. Sci.* 35, 363-87 (1935).—A summary of the properties of D and some sp. problems to which the use of D has been applied. E. J. Rosenbaum.

Thermocouples from 2° to 90° absolute. J. Fliston. *Ahlberg and Walter O. Lundberg. J. Am. Chem. Soc.* 57, 2722-3 (1935).—Math. considerations lead to the equation

$E = A + BT + CT^2 + DT^3$ , where  $E$  is the thermocouple voltage in microvolts,  $T$  is the abs. temp. and  $A, B, C$  and  $D$  are consts. Comparisons of thermocouples show, to first approximation, that the differences in voltages of 2 different thermocouples are proportional to their av. voltage, the data suggest that only 4 isolated comparisons of voltage and temp. are necessary for the calibration of thermocouples for use below 90°K. C. J. West.

Resistance thermometry below 10°K. J. D. Babbitt and K. Mendelssohn. *Phil. Mag.* 20, 1025-40 (1935).—One sample of phosphor-bronze wire out of many tried has a temp. coeff. of resistance in the temp. range 2-7°K. which makes it suitable for a thermometer for that range. Heating and quenching this wire destroys the temp. dependence of the resistance. Other wires of the same compn. do not show the desired effect. The superconducting component of the phosphor-bronze might be the  $\alpha$ -phase of the Sn-P alloy, which becomes superconducting at 8°K. A Pb-Ag alloy with 5% Pb has a temp.-dependent resistance between 3° and 7°K. E. J. R.

Determination of fixed points of low temperature with a hydrogen thermometer. Shin'ichi Aoyama and Eizo Kanda. *Bull. Chem. Soc. Japan* 10, 472-81 (1935).—An

all-glass H<sub>2</sub>-thermometer of const. vol. with an accuracy of  $\pm 0.03^\circ$  at  $0^\circ$  and  $\pm 0.025^\circ$  at  $-200^\circ$  is described and discussed. The b. p. of N<sub>2</sub> is  $-195.83 \pm 0.03^\circ$ , that of O<sub>2</sub>  $-183.02 \pm 0.03^\circ$ , and the sublimation p. of CO<sub>2</sub>  $-78.52 \pm 0.009^\circ$ . Some Cu-constantan thermocouples were also calibrated in a special cryostat that could be kept const. within  $\pm 0.02^\circ$  even at  $-200^\circ$ .

John E. Milbery. Filtration of some mineral powders suspended in water or aqueous solutions. Paulette Berthier. *Compt. rend.* 201, 1368-70 (1935).—Various powders, C, Fe<sub>2</sub>O<sub>3</sub>, etc., were suspended in solns. of varying concn,  $c$ , of the Na salt of an organic acid,  $e$ , oleate, citrate, of surface tension,  $\tau$ . The amts.,  $A$ , passing through when filtered under identical conditions were detd. With Na oleate ( $\tau$  decreasing)  $A$  increases with  $c$ , with citrate ( $\tau$  independent of concn)  $A$  shows a max., but no definite connection between  $\tau$  and  $A$  was found. It is suggested that increase in filterability is due to fixation of anions by the particles and their consequent acquisition of a neg. electrification, which reduces their adsorbability by cellulose (cf. Spring, C. A. 4, 1011). C. A. Silberrad.

The odor of hydrocyanic acid. Julius Meyer. *Gasmasker* 7, 112 (1935).—The odor of HCN varies widely with the concn, duration of action on the olfactory nerve and other conditions. Liquid HCN or strong aq. solns. have a penetrating, pungent odor and cause a harsh, scratching sensation in the throat. In very dil. condition it has been described as aromatic, but is more "dull" in effect. HCN paralyzes the nerves of smell and of taste to an unusual degree so that in a few seconds it can no longer be distinguished by these senses. About 5-10% of persons cannot smell it at all in low concns. The odor is quite different from that of bitter almonds or its oil with which it is frequently incorrectly associated in descriptions. A. L. Kibler.

The atomic weight of neon (after experiments by R. Jungblut-Ficht and M. Hoeppefer). A. v. Antropoff. *Ber.* 68B, 2389-91 (1935).—Pure Ne was sep'd. from a mixt. of 82% Ne and 18% He by repeated absorption by ignited coconut shells of mixed grain size in long tubes kept at the temp. of liquid air and subsequent pumping off at the same temp. The at. wt. was found by d. measurements to be 20.183, it confirms the value obtained by Baxter and Starkweather (C. A. 22, 1504). J. E. A.

Relative atomic weights of oxygen in water and air. Malcolm Dole. *J. Am. Chem. Soc.* 57, 2731 (1935).—The difference in d. of H<sub>2</sub>O prep'd. from atm. O<sub>2</sub> and from O<sub>2</sub> prep'd. by electrolysis of H<sub>2</sub>O indicates that the at. wt. of O<sub>2</sub> in the air is 16.00008 if the at. wt. of O<sub>2</sub> in Lake Michigan H<sub>2</sub>O is 16.00000. The work is being repeated. C. J. West.

Abnormal density of water from the depths of Lake Baikal. I. Mendelev. *Compt. rend. acad. sci. U.R.S.S.* [N. S.], 3, 105-8 (1935).—A complete immersion chain aerometer was used to det. differences in d. of water from various sources, and that of water from Lake Baikal from the surface to the depths. The d. of the lake water at the surface was identical in the 7th decimal place with that of other water. The d. of distd. samples increased toward the depths to a value of 56 units of the 7th decimal place at 1650 m. The d. of a series of undistd. samples also indicate a collection of heavy isotopes in the depths after allowing for mineral content. It is thought that mols. contg. heavy O and to a lesser degree, heavy H are conc'd. in the depths by gravity. The investigation is being continued. C. E. P. Jeffreys.

The periodic system of energy coefficients. A. E. Fersman. *Compt. rend. acad. sci. U.R.S.S.* [N. S.], 3, 173-6 (1935) (in English), cf. C. A. 29, 5787<sup>1</sup>, 7719<sup>1</sup>.—The  $Ek$  values of elements are considered in relation to the positions of elements in the periodic table. Analysis of the results gives the following conclusions: (1) the energy contributed by unit valences increases with  $w$  (charge); (2) all the values of  $Vek$  of intermediate ions of a given valence are almost equal; (3) the values of  $Vek$  of ions of the noble-gas type are smaller than the corresponding values of  $Vek$  of ions of the Pd type; (4) the anions of noble-gas type are smaller than the corresponding cations.



Curves of hardness, at. cohesion, crystal energy, m. p., b p and sp gr, are similar to the  $\epsilon_k$  curves A. B. F. D.

A statistical perturbation theory. I. Perturbation calculation in the Thomas Fermi theory without exchange Paul Gombás *Z. Physik* 97, 633-54(1935)—A statistical perturbation theory on the lines of the Thomas-Fermi approximation is developed for a neutral atom. The first- and second-order perturbation energies are compared with the corresponding wave-mech. expressions, and agree satisfactorily. The method can be extended to ions. It is applied to the calcn of the induced dipole moment in an external field B Swirles

The solution of Dirac's equation for a centrally symmetrical field of force Fritz Sauter *Z. Physik* 97, 777-84(1935)—A new method of solution of Dirac's equation for a potential,  $\Phi = \Phi(r)$ , is given and its identity with previous forms of soln established B Swirles

Optical activity IV Racemization of the optically active oxalates Christopher Hollis Johnson. *Trans Faraday Soc* 31, 1612-21(1935), cf. C. A. 27, 3415—Properties of metallo-trioxalates are discussed with reference to optical activity and racemization. The ionization theory of racemization was shown to be unreliable. The newly discovered racemization of chromi- and cobalt-oxalates in the cryst state supports the intramol rearrangement theory. It is suggested that  $H_2O$  mols play an important part in detg the properties of these oxalates. A connection between optical isomerism and magnetic susceptibility is discussed. Some coordination compds of Al are described. All attempts to resolve  $[Al(C_2O_4)_3]^{3-}$  and  $[Fe(C_2O_4)_3]^{3-}$  failed V Racemization of the strychnine salts of dextro and levo chromoxalate in the crystalline state C. H. Johnson and A. Mead *Ibid.* 1621-32—The d salt  $K_2[Cr(C_2O_4)_3] \cdot 12H_2O$  does not racemize even after heating for 2 days at  $120^\circ$ . Partial racemization of the strychnine salt occurs at room temp, and the extent of racemization depends upon the temp though apparently it is never complete even after heating for a long period at  $130^\circ$ . The tri-strychnine d- and l-salts of chromoxalate were studied at a variety of temps. There is evidence that the loss of rotatory power occurs chiefly after dehydration and not contemporaneously. It was shown that racemization does not result from, nor is it accompanied by, decompn of the mol. ion. Tentative explanation regarding the mechanism is proposed. Both enantiomorphs were obtained for the first time in a state of almost abs optical purity. VI Racemization of potassium chromoxalate in aqueous solution—accelerating influence of ions N. W. D. Beese and C. H. Johnson. *Ibid.* 1632-42—The failure of  $C_2O_4^{2-}$  to depress the rate of racemization of  $K_2[Cr(C_2O_4)_3]$  in aq soln indicates that racemization is either a measure of ionization or it is not a factor at all. The latter is considered most probable. Racemization is markedly accelerated by pos ions, an effect that increases with valence. The Ag ion and Ti ion exhibit greater activity than alkali ions, but are weak in comparison with bivalent ions. Judging from the effect of mercurous ion  $Hg^{2+}$  is a doubly charged unit. Unionized  $HgCl_2$  is inert. No evidence for the existence of  $Ti^{++}$ ,  $Cr^{+++}$  or  $Al^{+++}$  in soln was obtained, but the two latter behave as  $[Al(OH)]^{++}$  and  $[Cr(OH)]^{++}$ .

The catalytic effects of  $CaCl_2$ ,  $MnCl_2$ , and  $CuCl_2$  were studied over a wide range of concn. It is hoped to apply this catalytic effect to a study of complex ions in soln, with a view of detg the degree of disson. C. E. P. Jeffreys

Degree of depolarization in the molecular scattering of light Harald Volkmann. *Ann Physik* 24, 457-84(1935)—The degree of depolarization of polarized, parallel sunlight scattered by  $CH_4$ ,  $H_2$ ,  $CO_2$  and  $N_2O$  was measured at pressures up to 45 atm. The values found are:  $CH_4$ , 0,  $H_2$  0.009,  $CO_2$  0.0724 and  $N_2O$  0.102. Sources of error are discussed E. J. Rosenbaum

Magnetic birefringence in solutions of paramagnetic salts of rare earths S. W. Chinchalkar. *Phil. Mag.* 20, 856-8(1935)—A regularity in the birefringences of the rare earth ions is found. If the rare earths in the order of at. no. are divided into 2 groups on the basis of Hund's

theory, the birefringences of these groups vary in the same way from a neg. value to a pos one E. J. Rosenbaum

Physical methods in chemistry. III. The dielectric constant, its measurement and application. P. Cohen Hennequiez and L. J. N. van der Hulst. *Chem. Weekblad* 32, 493-8(1935); cf. C. A. 29, 3203<sup>7</sup>—A review and description of methods and app. for accurate detn. of dielec const; a liquid filled condenser of 0.5 cc. content is described. Applications of  $\epsilon$  detn. to process control are described IV. Dipole measurement and its application in chemistry *Ibid.* 636-45—An elaborate review of dipole work and its application (C. A. 29, 972<sup>7</sup>). The discussion includes additivity of moments in mols, electron affinity, double-bond moments, neg and pos groups, structural study on the basis of dipole moment, pyramid structure, C, N, O and S moments in their compds, ring systems, intramol effects,  $\sigma$ -, m- and p-derivs, etc. There are 30 tables of  $\mu$  values for different types of mols and at. groups with conclusions as to structure.

B. J. C. van der Hoeven

Electric resistivity and magnetic susceptibility of sugar carbon after various thermal treatments Paul Corniez *Compt rend.* 201, 1489-7(1935); cf. C. A. 30, 920<sup>1</sup>—The resistivities and susceptibilities of the 6 samples of sugar C previously described show, resp, a steady decline and a steady increase as the temp to which they were heated was greater, approximating but not attaining, those for graphite (cf. C. A. 28, 5959<sup>9</sup>). C. A. S

The negative cohesion pressure V. Jacyna, S. Derev'yankin, A. Obnorski and T. Parfent'ew. *Z. Physik* 97, 774-6(1935)—The investigations of P. Weiss (C. A. 29, 4985<sup>7</sup>) and P. W. Bridgman (C. A. 29, 4982<sup>7</sup>) have shown that for sufficiently high temps neg values of  $\alpha$  occur in the van der Waals equation for all gases. This supports J.'s theory (Z. Physik 30, 372(1924)). B Swirles

The scattering of molecular rays in gases R. G. J. Fraser, H. S. W. Massey and C. B. O. Mohr. *Z. Physik* 97, 740-4(1935)—The geometry of the method of crossed mol. beams is discussed for the case of a primary beam of rectangular cross section and the cross section for collision of Na-Hg and K-Hg is newly evaluated. It is shown that the corrected values give reasonable expressions for the van der Waals potentials. B Swirles

Remarks on the work of M. Trautz and W. Müller. Definition and correction of the gas-diffusion constants Jarl Kuusinen. *Ann Physik* 24, 445-6(1935)—T and M.'s equation (cf. C. A. 29, 4228<sup>7</sup>) is similar to the one K. proposed, which was attacked. E. R. Schierz

Definition of (gas-) diffusion constants. Jarl Kuusinen. *Ann Physik* 24, 447-56(1935)—K. derives as the most useful and general definition of the diffusion const for tridimensional diffusion in a mixt. of several components the following expression.  $v_i \Delta \gamma_i = \sum_j \frac{v_i \gamma_j}{\gamma_i} \frac{c_i - c_j}{\gamma_i - \gamma_j}$  where  $\gamma_i, \gamma_j$  = abs. concn.;  $v_i, v_j$  = partial molar vol (Lewis);  $c_i, c_j$  are space vector for component  $s$  and  $j$ , resp.  $\Delta \gamma$  = concn. gradient. E. R. Schierz

New method of measuring the viscosity of gases André Fortier. *Compt rend* 201, 1330-2(1935)—The gas issues from a reservoir in which it is under approx const. pressure,  $P$ , through a capillary tube of length  $l$  and radius  $r$ , which causes it to arrive in a chamber,  $C$ , under pressure  $P - \pi$ , whence it passes by a small tube, cross section  $S$ , into a second chamber where the pressure is less than  $0.52(P - \pi)$ . The equation  $\pi = K(1 - K/2P)$  then holds, where  $K = \frac{8\mu T S^2}{12(1 + \gamma)} \frac{1}{1 + \gamma} \frac{1}{1 + \gamma} \frac{1}{1 + \gamma}$  and  $\mu$  is coeff. of viscosity,  $T$  and  $0$  are the abs. temps of the gas in the capillary and  $C$  resp.,  $\epsilon$  is the thickness of slip, and  $\gamma$  and  $R$  have their usual meanings  $P$  may be atm. pressure and  $\pi$  of the order of  $10^{-4}$  mm  $H_2SO_4$ . C. A. Silbertad

Equation for approximating heat capacities of gases calculated from spectroscopic data I. N. Godnev. *J. Am. Chem. Soc.* 58, 180-1(1936)—An equation is proposed for the approx. representation of the heat capacities of gases calcd. from spectroscopic data,  $C_p = C_{p0} + \sum \frac{E_i}{T^2} + aT + bT^2$ , the applicability of which was examd for  $CO$ .

$N_2$  and  $S$  in the range 100–5000°K. In these cases the approximation can be accomplished very well with the coeff.  $b$  equal to zero for the temp. interval 100–5000°K.

C. J. West

Flame speeds during the inflammation of moist carbon monoxide-oxygen mixtures. W. Payman and R. V. Wheeler. *Nature* 136, 1028(1935).—The increase in speed of mixts. richer in water vapor found by Flock and Roeder (C. A. 29, 7810) is explained by the simultaneous reaction  $CO + H_2O \rightarrow H_2 + CO_2$ .

R. E. DeRight

Color temperatures of the Hefner and acetylene flames A. R. Pearson and B. Pleasance. *Proc. Phys. Soc. (London)* 47, 1032–41(1935).—The color of the cylindrical acetylene flame depends upon the gas pressure, the rate of consumption and the height of the flame. The color temp. of the Jones flame is 2520°, and that of the Hefner flame is 1919°.

S. Bradford Stone

The new treatment of critical phenomena. A. J. Jauch. *Z. Physik* 97, 669–70(1935).—The attribution of the existence of a crit. region (as opposed to point) to the presence of impurities, by Natanson and others, is reconcilable with J.'s new theory.

B. Swirles

The viscosity and the constitution of organic liquids. S. G. Papkov. *Z. Physik Chem.* A174, 445–5(1935), cf. C. A. 29, 7758. By addition of the at. increment, the viscosity of org. liquids can be calcd. from the formula

$\eta = M/d \cdot \sqrt{v_m}$ . The validity of this function is proved by calcd. the value of  $\eta$  for 26 liquids. The degree of assoc. of strongly assoc. liquids was calcd. and compared with exptl. values from the literature.

R. H. Baechler

Surface tension by the ring method—applicability of the Du Nouy apparatus. Rudolph Macy. *J. Chem. Education* 12, 573–6(1935).—It is possible by the ring method to obtain surface-tension data uniform with those given as "correct" in "International Critical Tables" (I C T). Two methods are available: (1) to apply correction factors or (2) to employ a calibration curve in which detns. made by the ring method are plotted against correct I C T values. The latter gives almost as good results as the exact method of Harkins.

Philip D. Adams

Theory of surface tension of liquid metals. D. V. Gogate and Duleh Sinha Kothari. *Phil. Mag.* 20, 1136–44(1935).—Math. Agreement with observed values is obtained.

E. J. Rosenbaum

Surface tension near the critical point. Alfred W. Porter. *Phil. Mag.* 20, 1163–6(1935).—Uncertainty exists as to the behavior of liquids in capillary tubes near the crit. temp. because while the rise decreases, the surface becomes flattened as the crit. temp. is approached. The change in form of the surface is calcd.

E. J. Rosenbaum

Boiling points of normal paraffin series. Edwin R. Cox. *Ind. Eng. Chem.* 27, 1423–5(1935).—The b. pts. of the normal paraffin series are given by  $\log_{10} T_b(K^\circ) = 1.07575 + 0.94125 \log_{10} m - 0.101 \log_{10} m$ , where  $T_b$  is the normal b. pt. in temp. abs. and  $m$  is the approx. mol. wt. If  $CH_4$  and  $C_2H_6$ , which are not homologous with the rest of the series in regard to b. pts., are excluded the agreement is perfect from propane to octane, except in  $C_8H_{18}$ .

O. W. Wilcox

Isotopic shift of water by distillation. J. Horuchi and G. Okamoto. *Bull. Chem. Soc. Japan* 10, 503–5(1935).—Samples (20 g.) of heavy water contg. 2.60%  $D_2$  were distd. at const. low temps. so that 10 mg. distd. oil in 40–60 min. The  $D_2$  content of this first distillate was detd. by a microspectrometer. The shift was greater the lower the temp. of distn. Thus the percentage  $D_2$  in the distillate at 20.42° was 2.49, at 0.05°, 2.42 and at –6.42°, 2.38. The results are discussed thermodynamically and a few calcs. made.

John E. Milberry

Structure of anisotropic liquids. R. D. Shulvas-Sorokina and M. V. Posnova. *Physik. Z. Sowjetunion* 8, 319–23(1935).—The dielec. const. ( $DK$ ) of  $p$ -azobenzene ( $P$ ) in several phases was studied by the capacity bridge method as a function of frequency in the range 35–1000 Hertz. While the capacity of the condenser plus liquid ( $P$ ) falls off with decreasing frequency, the  $DK$  of ( $P$ ) is independent of frequency and dielec. losses are absent.

For the isotropic liquid at 139°, the  $DK$  is 5.2; for the anisotropic liquid at 126°, 5.4; for the same phase undercooled to 115–103° the  $DK$  is of the same order; for the solid at 20°, 3.24. The sp. resistance of the liquid is shown as a function of temp. in the range 140–100°.

C. D. West

Electric conductance in semiconductors. Wilfried Meyer. *Physik. Z.* 36, 749–55(1935).—The rule that the log of the cond.  $\sigma$  is proportional to  $-B$  in the cond.-temp. formula  $\sigma = a e^{B/T}$  holds only to a first approximation. Measurements on  $TiO_2$  contg. varying amts. of excess  $O_2$  show a relation between cond. and  $a$ . The no. of conducting electrons was measured and a graphic relation between cond. and electron concn. is presented.

C. E. P. Jeffreys

Formation of counter tensions in solid ionic conductors. Adolf Smekal. *Physik. Z.* 36, 742–9(1935).—Newer investigations of solid ionic conductors help explain the nature of dielec. anomalies of special conducting ionic crystals. The Maxwell-Wagner theory of dielectrically homogeneous solid substances and the Jaffé theory are in accord with the exptl. facts.

C. E. P. Jeffreys

Conductance of electricity in melts of alkali salts with a stoichiometric excess of metal. E. Mollwo. *Physik. Z.* 36, 740–2(1935).—Alkali salt melts dissolve up to 1 vol. % of alkali metal in the form of neutral atoms. No electronic cond. was observed in the melts. With equal no. of excess atoms absorbed the total cond. was at least  $1/2$  less than in the solid crystal at the m. p.

C. E. P. Jeffreys

Electric conductivity of cuprous oxide in equilibrium with its neighbor phases. F. Waibel. *Physik. Z.* 36, 760–4(1935).— $Cu_2O$  was brought into equil. with  $Cu$  over a temp. range of 100–1000°. The elec. cond. of the sample was measured after rapid cooling. A max. for cold cond. occurs at 400–500° analogous to the  $Cu_2O$ - $Cu$  equil. The formation of unidirectional plates for  $Cu_2O$  rectifiers is explained. The amt. of dissolved  $O_2$  was detd. by weighing and gas-volumetrically.

C. E. P. Jeffreys

The electric resistance of tantalum wires saturated with hydrogen. A. Sieverts and H. Brüning. *Physik. Chem.* A174, 365–9(1935); cf. C. A. 30, 923. The H absorption and elec. resistance of Ta wire were measured at temps. of 600°, 500° and 400° and pressure,  $P$ , up to 1 at. At 600° and 500° but not at 400° the amt. of dissolved H is exactly proportional to  $\sqrt{P}$ . The increase in resistance at 600° and 500° is proportional to the amt. of dissolved H and therefore to  $P$ . At 400° it is almost proportional. Only a few measurements were possible at 400° because the wire ruptured with an increase in absorbed  $H_2$ . The same regularities held for the system  $Pd-H_2$  at low  $H_2$  content as for the system  $Ta-H_2$ . In both cases the validity held only as long as  $H_2$  could be absorbed without the formation of a new phase.

R. H. Baechler

The solubility of deuterium and hydrogen in solid palladium. A. Sieverts and G. Zapf. *Physik. Chem.* A174, 359–64(1935).—The solubilities of  $H_2$  and  $D_2$  in solid Pd were detd. Former measurements of  $Pd-H_2$  were substantiated fairly well. The ratio of the solubilities  $L_D/L_H$  is 0.67 at 300°, increases to 0.91 at 1000° and at higher temps. approaches 1. The soly. of both isotopes in Pd at const. temp. is proportional to the 4th root of the gas pressure.

R. H. Baechler

Thermoelectric potential, Peltier heat and photoelectric potential in the element copper-copper oxide-copper. G. Monch. *Physik. Z.* 36, 755–7(1935).

C. E. P. Jeffreys

Thermal formation of color centers and their life period. R. Hilsch. *Physik. Z.* 36, 735–7(1935).—The degree of dissoc. of U-centers and the life period of F-centers in KBr were measured. Such measurements may lead to a quant. understanding of the properties of semiconductors.

C. E. P. Jeffreys

Plasticity of crystals. IV. Further foundation for the dynamic plasticity law. E. Orowan. *Z. Physik* 97, 573–95(1935); cf. *Z. Physik* 89, 634(1934).—The plastic limit is not noticeably affected by the recovery of the crystal during the expt. Hence its dependence on temp.

and flow velocity cannot be interpreted as a recovery effect in a statistical theory. In particular, its velocity dependence is directly connected with a dynamic plasticity law. Confirmation is derived from curves of flow of Zn crystals.

B. Swales

**Etching studies of concave bodies. II. Studies of hollow hemispheres and other concave forms of fluorite.** Hans Himmel and Willi Kleber. *Neues Jahrb. Mineral. Geol., Beilage-Bd. 70A*, 49-64 (1935), cf. *C. A.* 29, 5714<sup>1</sup>.

J. F. Schauer

**Surface optical properties of calcite.** René de Malle-mann and François Suhrer. *Compt. rend.* 201, 1344-6 (1935).—From observations on the result of reflecting rectilinearly polarized monochromatic light from a polished surface of calcite cut in definite directions it is inferred that the polishing produces an isotropic layer on such surface the precise nature of which depends on the method of polishing. There seems some reason to think that a similar surface to at least some extent exists on a natural (cleavage) surface.

C. A. S. J. S. J. S. J.

**Calculation of the magnitude and pressure dependence of ion-disarrangement energies and mobilities in crystals.** W. Jost. *Physik Z.* 36, 757-60 (1935).—By considering polarization effects, an approx. calcn. of disarrangement energies of ions in crystals is possible. The approximation depends upon whether the theory of Schottky (*C. A.* 30, 346<sup>1</sup>) or that of Frenkel is followed.

C. E. P. Jeffreys

**Some new features in habit modification shown by potassium perchlorate crystals.** H. E. Buckley. *Z. Krist.* 91, 375-491 (1935) (in English).—Effects of various dyes on the crystal habit of  $KClO_4$  are given and possible explanations discussed. The results are similar to those on  $KMnO_4$  (*C. A.* 25, 5811<sup>1</sup>).

L. S. Ramsdell

**A Röntgenographic study of the process of producing spongy iron from hematite by reduction with gases.** W. P. Kasanetz. *Z. physik. Chem.* A174, 370-83 (1935).—The products of reduction of hematite by H were studied by the Debye-Scherrer method. The findings on compn. of the products agreed with those obtained by chem. analysis. Mech. mixts. of the products were the rule, solid solns. being lacking. The length of the edge of the  $\alpha$ -Fe fine crystal was found to be of the order of magnitude given in the literature. Higher temp. produced longer edges. At 700° the change of  $Fe_2O_3$  to  $Fe_3O_4$  is faster than the formation of the next phase. In the next step  $FeO$  is formed which quickly changes to Fe. When the reduction is 82.5% complete  $\alpha$ -Fe and a small residue of  $Fe_3O_4$  are present, the latter rapidly changing to Fe. At 450° the reaction is much slower and  $FeO$  is lacking. Because of the sensitivity of the Debye method one can assert the lack of  $FeO$  in cases where it is indicated by other methods.

R. H. Baechler

**Examination of electrodeposited nickel coatings by x-ray diffraction.** W. A. Wood. *Phil. Mag.* 20, 964-71 (1935), cf. *C. A.* 29, 7924<sup>1</sup>.—Coatings formed under a variety of conditions were examined. Bright coatings formed with small c. d. s. are associated with a highly oriented structure, while dull coatings have the crystallites oriented at random. The harder deposits have broader spectrum lines than do the softer ones. Lattice distortion alone cannot account for the broadening, which is also due to the small size of the crystallites. This size is of the order  $10^{-3}$  cm. in the soft deposits while in the hard ones it is approx.  $10^{-4}$  cm.

E. J. Rosenbaum

**Simplified technique for lattice-parameter measurements.** J. T. Norton. *Metals & Alloys* 6, 342-4 (1935).—Measurements are described which can be made in a routine way without sacrifice of the accuracy necessary for good work.

Downs Schaeff

**Methods of determining structure.** Alfred Hettich. *Z. Krist.* 90, 473-92 (1935); cf. *C. A.* 29, 2943<sup>1</sup>.—The following are discussed: (1) assignment of indices to Weissenberg photographs, (2) use of x-ray goniometer for adjustment of specimens of unknown orientation, especially needles and plates, (3) the fact that in rotating-crystal methods, planes nearly parallel with rotation axis pass through the reflecting position more rapidly than

those at greater angles, a correction factor for this is derived; (4) the "Eigendiagram," a new method of treating atomic groups in Fourier analysis; (5) optical methods of representing the results of Fourier analysis in structure details.

L. S. Ramsdell

**Layer lattices and the base-exchange clays.** C. E. Marshall. *Z. Krist.* 91, 433-49 (1935) (in English).—Complete chem. analyses of montmorillonite and beidellite accord with the structure proposed by Hofmann, Endell and Wilm (*C. A.* 28, 2237<sup>1</sup>) with 2  $SiO_2$  layers and 1  $Al_2O_3$  layer. Montmorillonite, beidellite and some of the nontronites are members of a single series. In montmorillonite the chief lattice replacement is Mg for Al, in the beidellites that of Al for Si predominates; in many nontronites each of these is overshadowed by the substitution of Fe for Al. Other nontronites correspond more nearly to the kaolinite type. Two other replacements are probable, Ti<sup>3+</sup> for Al, and P-Al for 2 Si. Analyses of fractions of several clays have been interpreted and agree well with the results of mineralogical analysis. Cf. *C. A.* 29, 4701<sup>1</sup>.

L. S. Ramsdell

**Lattice constants of beryllium.** E. A. Owen and Llewellyn Pickup. *Phil. Mag.* 20, 1155-8 (1935).—The values for an annealed sample of high purity are:  $a = 2.2810$  and  $c = 1.5682$ .

E. J. Rosenbaum

**X-ray investigation of the thermal expansion of cadmium.** G. P. Kosolapov and A. K. Trapeznikov. *Z. Krist.* 91, 410-23 (1935).—The lattice constants  $a$  and  $c$  for Cd were measured in the range of 26° to 189°. Perpendicular to the hexagonal axis the linear coeff. of expansion varies from  $1.7$  to  $2.2 \times 10^{-5}$ , and parallel to the axis is  $4.8 \times 10^{-5}$ .

L. S. Ramsdell

**MgZn and MgZn<sub>2</sub>.** L. W. McKeen. *Z. Krist.* 91, 501-3 (1935).—It is pointed out that if the coordinates given by Tarschusch (*C. A.* 28, 2238<sup>1</sup>) for  $MgZn$  and  $MgZn_2$  are correct, the compds. must have lower symmetry than T. proposed. It is concluded that more expl. work is needed.

L. S. Ramsdell

**The crystal lattices of heteropoly acids and their salts. I. The lattice of silicotungstic acid, borotungstic acid and ammonium borotungstate.** O. Kraus. *Z. Krist.* 91, 402-9 (1935).—Rotation photographs show that the following compds. are tetragonal, space group  $D_{2d}^{10}$ , with 2 mols. in the unit cells, and with these dimensions: silicotungstic acid,  $H_4SiW_{12}O_{40} \cdot 31H_2O$ ,  $a = 12.93$  Å,  $c = 18.52$  Å; borotungstic acid,  $H_4BW_{12}O_{40} \cdot 31H_2O$ ,  $a = 12.83$  Å,  $c = 18.35$  Å; ammonium borotungstate,  $(NH_4)_2BW_{12}O_{40} \cdot 26H_2O$ ,  $a = 12.80$  Å,  $c = 18.40$  Å. There is apparent isomorphism between the borotungstic acid and its ammonium salt. The probable amt. of water in borotungstic acid is discussed. Methods of prep. these compds. are given.

L. S. Ramsdell

**Crystal structure of sodium uranyl acetate.** I. Fankuchen. *Z. Krist.* 91, 473-9 (1935) (in English); cf. *C. A.* 29, 4546<sup>1</sup>.— $NaUO_2(C_2H_3O_2)_4$  is cubic, space group  $T_h^1$ . The unit-cell size is  $10.670 \pm 0.001$  Å, with 4 mols. On the basis of packing considerations and symmetry, the acetate groups are located approximately and are assumed to be planar and sym. Parameters are as follows: (1) atoms in special positions, U, 0.425, 0.002; O, 0.55 and 0.31, 0.02; Na, 0.81, 0.03; (2) atoms in general positions, O, 0.26, 0.46, 0.84; O<sub>1</sub>, 0.32, 0.45, 0.62; C, 0.33, 0.45, 0.74. Me group 0.44, 0.35, 0.77. These values, obtained by the rotation method, differ somewhat from the powder-photograph results of De Jong (*C. A.* 24, 3149<sup>1</sup>).

L. S. Ramsdell

**Isomorphism of the double fluoroberyllates with the Tutton salts.** P. L. Mukherjee. *Z. Krist.* 91, 504 (1935) (in English).—Rotation photographs of  $(NH_4)_2Ni(BeF_4)_2 \cdot 6H_2O$  indicate a cell contg. 2 mols., with the dimensions  $a = 9.04$ ,  $b = 12.31$ ,  $c = 6.04$  Å, and  $\beta = 106^\circ 40'$ . The space group is  $C_{2h}$  with glide component  $a/2$ .  $(NH_4)_2Ni(SO_4)_2 \cdot 6H_2O$ , a typical Tutton salt, also belongs to  $C_{2h}$  with the dimensions  $a = 8.98$ ,  $b = 12.22$ ,  $c = 6.10$  Å, and  $\beta = 107^\circ 4'$ , indicating structural isomorphism with the former compd.

L. S. Ramsdell

**Crystal structures of hydrated compounds. II. Structure type  $M(ClO_4)_2 \cdot 6H_2O$ .** C. D. West. *Z. Krist.* 91,

480-93(1935)(in English)—Hydrated perchlorates and fluoroborates of some univalent and bivalent metals are described. The 12 isomorphous hydrates of Mg, Mn, Fe, Co, Ni and Zn have structures derived from hexagonal  $\text{LiClO}_4 \cdot 3\text{H}_2\text{O}$  (C. A. 28, 57337) by removing half of the Li atoms from octahedral positions and replacing the remainder with Mg. The crystals are twinned so that the water-perchlorate lattice is continuous by virtue of its high symmetry, while the metal lattice, with lower symmetry, is discontinuous at the twinning boundary.  $n_o$  and  $n_e$  and cell dimensions  $a$  and  $c$ , resp., are as follows (Li) 1.483, 1.448, 15.42 Å. (doubled), 5.42 Å. (Mg) 1.482, 1.458, 15.52 Å, 5.20 Å. (Mn) 1.492, 1.475, 15.70 Å, 5.30 Å. (Fe) 1.493, 1.478, 15.58 Å, 5.24 Å. (Co) 1.510, 1.490, 15.52 Å, 5.20 Å. (Ni) 1.518, 1.498, 15.46 Å, 5.17 Å. (Zn) 1.508, 1.487, 15.52 Å, 5.20 Å. (Cd) 1.489, 1.480, 15.92 Å (doubled), 5.30 Å. (Hg) 1.511, 1.509, trigonal, (Cu) monoclinic,  $\alpha = 1.495$ ,  $\beta = 1.505$ ,  $\gamma = 1.522$ .

L. S. Ramsdell

Crystal structure and composition of sodium dihydroxyphosphate. J. Bentma *Proc. Acad. Sci. Amsterdam* 33, 1015-20(1935)—Na dihydroxyphosphate crystallizes in the tetragonal bipyramidal class. The elementary cell contains a mass  $[\text{Na}_2\text{H}_2\text{Si}_2\text{O}_7 \cdot 5\text{H}_2\text{O}]$ , with  $c_0 = 8.00$  Å. and  $c_0 = 7.86$  Å. The cations and anions form a NaCl-like grating with each Sb surrounded by 6 O atoms in an almost octahedral configuration. The structure is in accord with Pauling's view that the substance has the compn NaSb(OH)<sub>2</sub>.

C. E. P. Jeffreys

Crystal structure of cerium tungstate J. Bentma *Proc. Acad. Sci. Amsterdam* 33, 1011-15(1935)— $\text{Ce}_2(\text{WO}_4)_3$  is tetragonal bipyramidal and possesses a scheelite-like structure, with  $c_0 = 5.33$  Å. and  $c_0 = 11.62$  Å. with a mass of  $\text{Ce}_2(\text{WO}_4)_3$  within its elementary cell. Only  $1/2$  of the positions of Pb atoms in  $\text{PbWO}_4$  which forms an uninterrupted mixing series with it are occupied by Ce atoms. The remaining places are void. The mixed crystals and  $\text{Ce}_2(\text{WO}_4)_3$  crystals are stable, however.

C. E. P. Jeffreys

The crystal form and chemical composition of lamprophyllite. B. Gossner and K. Drexler. *Z. Krist.* 91, 494-500(1935).—A crystal of lamprophyllite from Kola, with  $d = 3.35$ , is orthorhombic,  $V_h$ , with the dimensions  $a = 19.05$  Å,  $b = 7.05$  Å,  $c = 5.35$  Å. The basic formula is  $\text{TiSrNa}_2(\text{SiO}_3)_2$ . Ca and Ba may be present up to 25%. Further, SiO can be replaced by (AlF + FeF) and Sr by Mn.

L. S. Ramsdell

An x-ray examination of methylene blue W. H. Taylor. *Z. Krist.* 91, 450-65(1935)(in English), cf. C. A. 29, 7737.—The methylene blue sulfur chloride, bromide and iodide, and selenium bromide, were examined optically and by x-rays. The first 2 and the latter are isomorphous, forming monoclinic crystals, with symmetry of space group  $C_{2h} = P_2/c$ , and have the following dimensions sulfur chloride,  $a = 9.5$ ,  $b = 31.3$ ,  $c = 6.9$  Å.,  $\beta = 97^\circ$ , bromide,  $a = 9.7$ ,  $b = 31.5$ ,  $c = 6.9$  Å.,  $\beta = 97^\circ$ , selenium bromide,  $a = 9.65$ ,  $b = 31.5$ ,  $c = 7.0$  Å.,  $\beta = 97^\circ$ . Measured  $d_s$  are 1.29, 1.41 and 1.57, resp. These values, and cell vols., indicate that the chloride has 4 mols., and the 2 bromides 5 mols. of  $\text{H}_2\text{O}$ . The iodide has 2 forms, one triclinic, with  $a = 16.9$ ,  $b = 15.8$ ,  $c = 6.9$  Å.,  $\alpha = 98^\circ$ ,  $\beta = 95^\circ$  and  $\gamma = 89^\circ$ ; and the other triclinic, pseudomonoclinic, with  $a = 14.8$ ,  $b = 15.7$ ,  $c = 7.6$  Å.,  $\alpha = \gamma = 90^\circ$ ,  $\beta = 98^\circ$ . The former, while not isomorphous with the chloride and bromides, has a related structure. The space group of the latter form was not detd. The 2 iodides have 3 and 1 mol.  $\text{H}_2\text{O}$ , resp. The large scattering power of the I atoms makes possible a detn. of the structure. It is suggested that the C-N-S atoms form lath-shaped mols. which are stacked one above the other, and packed end to end, but adjacent stacks are sep'd. by layers of I atoms and  $\text{H}_2\text{O}$  mols.

L. S. Ramsdell

Theory of adsorption. H. Bradley. *Trans. Faraday Soc.* 31, 1652-5(1935).—The formula formerly proposed by B.,  $\alpha = ap^{\beta}/(1 + \beta p^{\beta})^{1/\beta}$ , where  $\alpha$  = gas vol. adsorbed in cc.,  $p$  = pressure in cm. Hg,  $T$  = abs. temp., and  $\alpha$ ,  $\beta$  are consts., was applied to a wide variety of published data with success. For the adsorption of air and

CO on coconut charcoal,  $\alpha = 1$ . In all cases of adsorption of  $\text{H}_2\text{O}$   $\alpha = 2$ . In the case of adsorption in  $\text{CaH}_2$ ,  $\alpha = 3$ . The value of  $\alpha$  may be of important significance in view of Fowler's formula (cf. C. A. 29, 49901), wherein  $1/\alpha$  is derived as the index of pressure under certain conditions. C. E. P. Jeffreys

Specific heat and binding conditions of adsorbed argon on charcoal. Hans M. Cassel. *J. Am. Chem. Soc.* 57, 2724(1935).—Measurements of sp. heat suggest that the majority of A atoms are adsorbed in the hollow edges or steps of the very cleavable material rather than on the smooth surfaces of the netplanes. C. J. West

Adsorption and oxidation of succinic acid by charcoals. B. Tamamushi and H. Umezawa. *Z. Elektrochem.* 41, 761-4(1935).—The adsorption of succinic acid (I) and fumaric acid (II) on Merck's animal charcoal and Kahlbaum's blood charcoal was detd. The adsorption of I by animal charcoal was less than by blood charcoal, but on the former catalytic oxidation was much greater. If it is more highly adsorbed by blood charcoal, and thus inhibits the oxidation of I. The adsorption and catalytic activity of a special hemin-sugar charcoal was about the same as the animal material. In neutral soln. I is oxidized partially to II as in acid soln. and partially to  $\text{CO}_2$ . The catalytic activity of the charcoal is diminished but not destroyed entirely when it is heated in  $\text{N}_2$  and the reaction effected in a stream of  $\text{N}_2$ , this shows that  $\text{O}_2$  carried by the charcoal takes part in the oxidation. C. E. P. J.

Sorption and diffusion of ammonia in analcam. Arne Tiselius. *Z. physik. Chem.* A174, 401-23(1935).—As a continuation of previous work (cf. C. A. 28, 2534\*, 5736\*) a study was made of the sorption and diffusion of  $\text{NH}_3$  in the zeolite analcam which seemed well suited because of its relatively simple structure. Various isotherms were taken, direct diffusion was measured on individual crystals by an optical method as well as measurements of absorption velocity on powders. R. H. Baechler

Emulsions. E. G. Thomssen. *Am. Perfumer* 31, 59-60, 96-8(1935).—Emulsions in general, emulsifying agents and app. are discussed with special reference to toilet articles. The use of triethanolamine in the manuf. of cosmetics like liquid, cold, vanishing and lanolin creams is considered. Among the app. illustrated are a push-pull mixer, paste-type mill, colloid mill and viscolizer.

W. O. E.

Collapse of unimolecular films of palmitic acid upon acid solutions. Robert J. Myers. *J. Am. Chem. Soc.* 57, 2734-5(1935).—In the study of the compression of unimol. films of palmitic acid on HCl or phosphate buffer solns. in a Langmuir film-pressure trough, the films collapsed at a pressure apparently connected with the H-ion concn. When the collapse pressures were plotted against  $p_H$  of the substrate a sigmoid curve resulted, the mid-point of which was about  $p_H 3$ , as compared to  $p_H 5$ , the mid-point of neutralization curves of fatty acids; this appears to be evidence of increased activity of palmitic acid when oriented on a  $\text{H}_2\text{O}$  surface. The collapse pressure was sp. for H ions alone, although increased spread at low pressures was noticed when Ca or Mg ions were present in the HCl soln.

C. J. West

Action of metallic copper on a film of cupric sulfide. Henri Devaux. *Compt. rend.* 201, 1305-7(1935).—If a small fragment of Cu is placed on the film of  $\text{CuS}$  formed by passing  $\text{H}_2\text{S}$  over aq.  $\text{CuSO}_4$ , the film near the Cu changes to steel blue in color and becomes more lustrous; the area so changed grows to a width of about 10 cm. in a few hrs. The Cu must touch the  $\text{CuS}$ , which must rest on the cupric soln. The change does not extend to any portion of the film not continuous with the part in contact with the Cu. The max. distance between the  $\text{CuS}$  mols. which will permit the change to pass on is 0.6-1 Å. Cu may be replaced by any metal that ppts Cu from the soln, e. g., Fe or Zn, but not by one that does not ppt. Cu, e. g., Au or Pt.

C. A. Suberrad

Comparison of the electrokinetic potentials at fused and unfused glass surfaces. Betty Monaghan and H. L. White. *J. Phys. Chem.* 39, 935-9(1935).—An app. and method are described for the prep. of microscopic glass

spheres Pyrex spheres and unfused Pyrex particles have the same electrophoretic velocity. The velocity of fused or unfused Pyrex particles is much less in dil. solns than the electroosmotic velocity at a fused Pyrex surface, the latter being measured by 2 independent methods.

A. E. Beitch  
Factors influencing electrophoretic mobilities and apparent critical potentials. H. L. White and Betty Monaghan. *J. Phys. Chem.* 39, 925-34 (1935).—The electrophoretic mobility of small glass particles in dil. electrolytes is less than the electroosmotic mobility at a glass surface. The two mobilities are identical in 0.01 M solns. Polarization of the double layer causes retardation of the particles in dil. solns because the mobilities of particles in crit. concns. of multivalent salts are abnormally low rather than because the crit. potentials with inorg. multivalent salts are abnormally high. The crit. mobility in inorg. multivalent salt solns is a true measure, while the true zeta potential in the crit. concns. of multivalent salts is higher than that indicated by the mobility and may be the same as that with inorg. univalent salts. A. E. Beitch

Colloid chemistry in 1935. Wm. Clayton. *Chem. Age* 33, 594-6 (1935).

Donnan effect in ultrafiltration of colloidal solutions. A. I. Rabinovich, P. Vasil'ev, and T. Gatovskaya. *Compt. rend. acad. sci. U.R.S.S.* [N.S.] 1, 3, 109-12 (1935).—By combining the relations  $A_s = (A_i - A_u) + A_u$ , and  $K_s = (K_i - K_u) + K_u$ , with the Donnan equations, where  $A_i$  and  $K_i$  represent activities of anion and cation and the indexes  $s$ ,  $r$  and  $u$ , represent initial, final and ultrafiltrate, resp., and  $n$  is the vol. reduction of the sol, a general expression is deduced for the concn. of ions of both signs in the ultrafiltrate at any vol. reduction. By solving the resulting equation,  $A_s = K_s = [(A_i + K_i)(n-1) + \sqrt{(A_i + K_i)^2(n-1)^2 - 4A_iK_i(n-1)}] / 2(n-2)$ , at given values of  $A_i$  and  $K_i$  with variable  $n$ , the curve for  $A_s = K_s$  against  $n$  shows a smooth curve following a linear trend parallel to the abscissa at high values of  $n$ , but dropping considerably at lower values of  $n$ . This type of function accounts for the apparent constancy of concn. of the ultrafiltrate previously observed. When solving from various ratios of  $A_i/K_i$ , it is seen that the drop of ionic concn. from initial sol to ultrafiltrate is more pronounced the higher the ratio  $A_i/K_i$ . The theory was verified by a series of measurements on Fe(OH)<sub>3</sub> sols dialyzed so as to obtain varying ratios of  $A_i/K_i$ .

C. E. P. Jeffreys  
Electric properties of colloidal silicic acid. Korgun, *et al.* *Sotsialist. Rekonstruktsiya i Nauka* 1935, No. 5, 112.—Perfectly neutral sols of silicic acid were obtained by two methods (1) SiCl<sub>4</sub> was hydrolyzed and the product freed of the traces of HCl by electrolysis and (2) SiH<sub>4</sub> was oxidized with O<sub>2</sub> by passing the gases into H<sub>2</sub>O. Cataphoretic measurements of the sols showed the insignificance of the charge on the particles and their immobility in the elec. field. On mixing sols of Ca salts with colloidal silicic acid adsorption compds. are formed instead of silicates. It is concluded that the acidity of the H<sub>2</sub>SiO<sub>3</sub> sols is due mainly to foreign strong acids. The stability of colloidal silicic acid in an acid medium depends upon the anions of the foreign acid and at pH 9 upon the anions formed by silicic acid itself. At pH 6-9 the particles are only slightly protected by the charges and min. stability occurs.

B. V. Shvartzberg  
Phosphatide autocomplex coacervates as ionic system and their relation to the protoplasmic membrane. H. G. Bungenberg de Jong and J. Bonner. *Protoplasma* 24, 198-218 (1935).—See C. A. 30, 354.

F. L. Dunlap  
Influence of neutral salts on the optical rotation of gelatin. III. Effect of the halides of lithium, sodium, rubidium and cesium. D. C. Carpenter and F. E. Lovelace. *J. Am. Chem. Soc.* 57, 2337-42 (1935), cf. C. A. 25, 5821.—The effect of the halides of Li, Na, Rb and Cs on the sp. rotation of gelatin at 0.5° and 40° is reported for the soly. range of the salts. The results confirm the earlier conclusion that there is a definite Hofmeister series of anions (I > Br > Cl) in their effects on the optical rotation of gelatin. There is also a definite Hofmeister series

for cations (Li > Cs > Rb > Na) but the effects of cations are much less in magnitude than those of the anions. In the absence of added salts, the optical rotation of the gelatinates of the alkali metals increases slightly with at no to a max. rotation with K, after which the rotation slightly decreases. IV. Rotatory dispersion of gelatin in sodium iodide solutions. *Ibid.* 2342-6.—The rotatory dispersion of gelatin is reported in various concns. of NaI at 0.5° and 40° at 5 different wave lengths in the visible spectrum. A single Drude equation expresses the expit. results, an absorption band at 2200 Å. governs the dispersion; the data show that gelatin has a high absorption in this region. The rotatory dispersion constants at 40° follow the linear equation  $k_{40} = 44.5170 - 6.220C_{NaI}$ , at 0.5° the constants are expressed by the sum of 2 equations  $C_{NaI} = (1/2.66) \log [a/(1-a)] - \log (1/K)$  and the linear equation  $K_{40} = 46.3300 - 6.7605C_{NaI}$ . The linear effect of salt concn. on the dispersion const. is probably due to a shift in the assocn. of the solvent (hydroly-polyhydroly equil.) or an assocn. between solvent and salt, the much greater effect of added salt at 0.5° is due to a dissocn. or assocn. of the gelatin mol. Based on the formulation of Lucas, it is shown that 2 and only 2 optically active species exist in gelatin solns or gels.

C. J. West  
Redetermination of the solubility of chloropentammine cobalt chloride. F. J. Garrick. *Nature* 136, 1027-8 (1935).—The soly. in H<sub>2</sub>O of this compd. purified by an improved method is 0.0211 and 0.00925 moles/l. at 25° and 0.20°, resp. This agrees with the results of Brønsted and Peterson, cf. C. A. 16, 670. Methods of pptg. the sample had no effect on the soly.

R. L. DeRicht  
Solubility of barium iodate in salt solutions. G. Macdougall and C. W. Davies. *J. Chem. Soc.* 1935, 1416-19.—The soly. of Ba iodate monohydrate in aq. solns of KCl, KNO<sub>3</sub>, KClO<sub>4</sub>, CaCl<sub>2</sub> and KClO<sub>4</sub> at 25° is reported. Corrections for ionic assocn. remove the specific deviations found in dil. soln. and the results are in accord with the Debye-Hückel limiting formula. The dissocn. const. of the BaIO<sub>3</sub><sup>+</sup> ion is 0.08 and that of BaClO<sub>4</sub><sup>+</sup> is approx. 0.2. William E. Vaughan

Some physicochemical properties of mercurous perchlorate solutions. Edgar Newbery. *Trans. Electrochem. Soc.* 69, 17 pp. (preprint) (1936).—The system Hg<sub>2</sub>(ClO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O was investigated and a soly. curve constructed. A simple quickly adjusted thermoregulator is found especially suitable for soly.-curve work. Small quantities of deliquescent crystals are rapidly dried in a tube centrifuge. Two hydrates, one with 4 H<sub>2</sub>O (orthorhombic) and one with 2 H<sub>2</sub>O (monoclinic) were recognized, the transition point being 36°. From strongly acid solns, the dihydrate is deposited at temp. considerably below the normal transition point. The presence of free acid lowers the soly. of the salt in such a manner that the total concn. of the common ion remains const., while the ionic product varies greatly. Hydrolysis occurs in 3 stages, at first Hg<sub>2</sub>O Hg<sub>2</sub>(ClO<sub>4</sub>)<sub>2</sub> is formed and this, on treatment with more water, is first hydrated to Hg<sub>2</sub>OH·ClO<sub>4</sub> and then hydrolyzed further to Hg<sub>2</sub>O. The first of these compds. is dissolved by a warm soln. of the normal salt, from which, on cooling, it is slowly deposited in the form of lenses, which very slowly change to tetragonal crystals. The second compd. crystallizes in the hexagonal system and is decomposed by water with formation of a soln. of the normal salt and a ppt. of Hg<sub>2</sub>O. The salt is hydrolyzed 2.5% in 0.1 M soln. In more concd. solns, the degree of hydrolysis is greater than would be expected, because of the formation of anhyd. basic salt, which is a weaker base than the hydrated form. The degree of dissocn. of the salt in dil. soln. appears to decrease with diln., either because of the formation of a soln. of unionized basic salt or the assocn. of single univalent to double bivalent ions. In highly concd. solns, abnormally high dissocn. is indicated both by cond. and by potentiometric methods. In such solns, the double mercurous ion is torn apart into single univalent ions by the powerful interionic forces present.

C. G. F.  
Diffraction of x-rays in some solutions. J. A. Prins and R. Fonteyne. *Physica* 2, 1016-28 (1935); cf. C. A. 29,

7182<sup>3</sup>, 7761<sup>4</sup>.—Previous work of the authors is confirmed and extended. In addn. to the cases already reported of  $\text{Th}(\text{NO}_3)_3$  and  $\text{UO}_2(\text{NO}_3)_2$  it appears that a "superarrangement" of the cations at least is present with  $\text{ThCl}_3$ ,  $\text{UO}_2\text{Cl}_3$ ,  $\text{La}(\text{NO}_3)_3$ ,  $\text{Ce}(\text{NO}_3)_3$ ,  $\text{Zn}(\text{NO}_3)_2$ ,  $\text{Ni}(\text{NO}_3)_2$ ,  $\text{ZnCl}_2$ ,  $\text{ZnI}_2$ ,  $\text{NiCl}_2$ . Moreover the multivalent anions of  $\text{Na}_2\text{WO}_4$ ,  $\text{Na}_2\text{MoO}_4$ ,  $\text{H}_2\text{P}_2\text{O}_7$ ,  $\text{HIO}_4$ ,  $\text{HClO}_4$  and  $\text{H}_2\text{SO}_4$  seem to show a superarrangement. With univalent cations or anions no superarrangement could be found. A rule summarizing the results is given. On the other hand,  $\text{HIO}_4$  was found to belong to the class with "gaseous distribution." This was also the case with  $\text{CdI}_2$  in water and  $\text{LiOH}$  and with  $\text{CdCl}_2$ ,  $\text{Hg}(\text{NO}_3)_2$ ,  $\text{Na}_4(\text{PW}_{12}\text{O}_{40})$ ,  $\text{Ce}_2(\text{SO}_4)_3$  and  $\text{Zr}(\text{NO}_3)_4$  in water. In these cases the diffraction pattern gives information on the inner structure of the mols.

Harold Gershinowitz

**Cryoscopic study of trichloroacetic acid and its hydrate in benzene and in dioxan solution** R. P. Bell and M. H. M. Arnold. *J. Chem. Soc.* 1935, 1432-5.—Phase-rule studies show that the solid phase in these cryoscopic measurements on  $\text{C}_2\text{H}_3\text{Cl}_3$  solns of trichloroacetic acid and its hydrate contains a little of the acid (probably in an absorbed state), the quantity is insufficient to interfere with a simple interpretation of the results. The dry acid in  $\text{C}_2\text{H}_3\text{Cl}_3$  solns exists principally as double mols. over the concn range studied ( $m = 0.01-1.6$ ). In dil. soln the hydrate exists as single mols. or forms double mols. at the higher concns. Several structures of the single and double hydrate mols. are proposed. In dioxan solns trichloroacetic acid is present as single mols., probably because of assocn. with the solvent. William F. Vaughan.

**Mixed melting points in eutropic series** H. D. K. Drew and J. K. Landquist. *J. Chem. Soc.* 1935, 1480-2.—Mixed m. p. of the tetraphenyl derivs of C, Si, Ge, Sn and Pb were detd., but no generalizations can be made. The data are tabulated. Mixed m. p. of the triphenyl derivs. in the series N, P, As, Sb and Bi show large depressions. The data are discussed. W. C. Vaughan.

**Surface tensions of ternary solutions** III. J. W. Belton. *Trans. Faraday Soc.* 31, 1648-52 (1935), cf. C. A. 30, 669<sup>7</sup>.—Surface tension of solns. of sucrose and  $\text{NaCl}$ , dil.  $\text{HCl}$  and  $\text{NaCl}$ , and dil.  $\text{HCl}$  and  $\text{KCl}$  were measured by the bubble pressure method and the surface adsorption of  $\text{H}_2\text{O}$  calcd. In the sucrose  $\text{NaCl}$  soln. the adsorptions are much less than the sum each solute would produce separately. An amt. of acid that has slight effect upon the surface tension of  $\text{H}_2\text{O}$  produces a strong effect on the adsorption of  $\text{H}_2\text{O}$  on a salt soln. C. E. P. J.

**Surface tensions of binary liquid mixtures containing benzene** J. W. Belton. *Trans. Faraday Soc.* 31, 1642-8 (1935).—Surface tensions of mixts. of  $\text{C}_6\text{H}_6$  with  $\text{AcOH}$ ,  $\text{CS}_2$  and  $\text{CCl}_4$  were measured by the bubble-pressure method and surface adsorptions calcd. by the Gibbs equation. Measurements with  $\text{AcOH}$  were made at 20° and 35°, with  $\text{CS}_2$  at 20° and 30° and with  $\text{CCl}_4$  at 50°. The form of the adsorption compn. curves and the application of the gas laws is discussed. C. E. P. Jeffreys.

**The surface tension of simple mixtures** V. K. Semenchko, A. F. Gracheva and E. A. Davydovskaya. *Sotzialist. Rekonstruktsiya v Nauka* 1935, No. 5, 159-60.—In triple mixts. the surface tension within the limits of 0° and 80° does not depend upon the concn. of any one of the components; therefore it cannot serve as an indication of the presence of certain substances in the mixt.

B. V. Shvartzberg

**The surface activity of ions** II. Per Ekwall. *Acta Acad. Aboensis, Math. Phys.* 8, No. 2, 13 pp. (1933), cf. C. A. 28, 6048<sup>7</sup>.—Alkyl-substituted  $\text{NH}_4$  picrates gave sigmoid surface tension-concn. curves, the curves being initially concave to the concn. axis, then passing through an inflection point and becoming convex to that axis. The lower the mol. wt. the less marked the sigmoid form. This is taken to indicate an attractive force between ions or mols. in the surface. Data obtained with diethyl- and dipropylammonium picrate at high concns. agreed fairly well with values calcd. by Szyskowski's equation (cf. C. A. 3, 133) but were appreciably lower than these values at low concns. The equation  $FA = RT$  (where  $F$  is the surface

tension lowering,  $A$  the surface area contg. 1 mol. of solute,  $R$  a const. and  $T$  the temp.), which should be valid if the solute in the surface behaves as a 2-dimensional gas (cf. Langmuir, C. A. 11, 2840), was tested by calcg. the value of  $(FA)/(RT)$  for diethyl, mono-, di-, tri- and tetrapropyl  $\text{NH}_4$  picrates. For the last salt the value remained unity for the whole range of concn. studied (up to a surface tension lowering of 3.5 dynes/cm.). For the other salts the value at first decreased, went through a min. and then increased rather steadily, the slope being greater the greater the mol. wt. of the salt. Thus strong attractive forces between the mols. were again indicated. This min. was lower the greater the no. of substituted alkyl groups or the longer the hydrocarbon chains. Comparison is made with similar calcns. by Schofield and Rideal (cf. C. A. 19, 3397, 20, 2005) whose work is briefly reviewed. Calcd. from data for the diethyl- and dipropyl-substituted salts, the coeff. of Traube's rule was smaller than the usually accepted value. M. G. Moore.

**Interfacial tension of sugar solutions at various liquid surfaces of separation** N. S. Stroganov. *Protoplasmata* 24, 431-52 (1935).—An investigation of interfacial tension of glucose, galactose, maltose, sucrose and lactose by contact with phases immiscible with  $\text{H}_2\text{O}$ : benzene, toluene, petr. ether, paraffin oil, castor oil and olive oil. The degree of lowering of the interfacial tension varies for the different types of sugars at various liquid surfaces of sepn. It depends on the nature of the phases and on the nature of the active substance dissolved in the  $\text{H}_2\text{O}$ . According to the degree of lowering, the sugars for interfacial tension with respect to benzene, toluene, petr. ether and paraffin oil may be placed in the following series: glucose < galactose < maltose < sucrose < lactose. At the surfaces of sepn. with respect to castor oil and olive oil, this series does not hold. Seventy-one references.

F. L. Dunlap

**Optical rotatory power of solutions in an electric field** J. Kunz and Andrew McLean. *Nature* 136, 705-6 (1935).—When an elec. field is impressed across a benzene soln. of the polar optically active compd. 2-menthyl hydrogen 3-nitrophthalate, the rotation changes instantly by as much as 0.59° but resumes its normal value on removal of the field. The change reaches its max. at a field strength of 4000 units abs. and remains const. for greater fields. Unexpected results were obtained with the inactive 2-ethyl hydrogen 3-nitrophthalate under the same conditions.

C. D. West

**The mobility of hydrogen and hydroxyl ions in aqueous solutions** G. Wanner. *Ann. Physik* 24, 645-68 (1935).—The high mobility and small temp. dependence are attributed to a proton exchanged by the hydrated  $\text{H}$  and  $\text{OH}$  ions with a  $\text{H}_2\text{O}$  mol. The result is the same as that of actual movement. Models and their quantum mechanics are discussed and the results compared with the Debye-Hückel theory. The explanation of the temp. coeff. is not completely satisfactory. R. E. DeRight.

**The sourness of acids** R. M. Beatty and L. H. Cragg. *J. Am. Chem. Soc.* 57, 2347-51 (1935).—Sourness is expressed as the normality of an equally sour  $\text{HCl}$  soln. Equally sour solns. of acetic, malic, tartaric and chloroacetic acids required equal amts. of a phosphate buffer to bring the  $p_n$  to 4.4. This amt. of buffer brought carbonic acid to 5.3, possibly because of an error in tasting. This relation holds for buffers of  $p_n$  6.9-7.4 and concns. of anhyd.  $\text{Na}$  phosphates from 0.04 to 0.35%. The sourness of acids too concn. or too dil. to taste can be detd. by the formula  $S = X/K$ ,  $X = \text{no. of buffer needed to adjust the } p_n \text{ of 1 cc. to a } p_n \text{ of 4.4}$  and  $K = \text{characteristic const. for the buffer}$ . R. L. DeRight.

**Equilibrium in solutions of tertiary electrolytes** K. Jablczynski and R. Legat. *Roczniki Chem.* 15, 351-8 (in German 353-9) (1935).—The equil. const. of dissoen. of  $\text{BaCl}_2$  was detd. from cryoscopic and elec. cond. data; it is 0.0875. M. Wojciechowski.

**The fourth ionization constant of ferrocyanic acid** I. M. Kolthoff and Wm. J. Tomiscek. *J. Phys. Chem.* 39, 955-8 (1935).—The fourth ionization const. of  $\text{H}_4\text{Fe}(\text{CN})_6$  is  $5.6 \times 10^{-11}$  at 25°. A. E. Beitch

The peculiar behavior of aqueous carbonic acid solutions Yrjö Kauko *Suomen Kemistilehti* 8B, 39-41 (1935)(in German)—The results and errors in the measurements of the first dissociation constant of carbonic acid by various researchers are discussed. The second dissociation constant was determined at 25° and found to have the value  $5.3 \pm 0.3 \times 10^{-11}$  MacInnes and Belcher (cf. C. A. 29, 7760) found  $5.61 \times 10^{-11}$  E. E. Jukkola

The Wien effect for electrolytes investigated with the cathode ray oscillograph Wilhelm Huter *Ann Physik* 24, 253-72 (1935), cf. C. A. 28, 2975—The current-p d relation at high field strengths was studied for solutions of NaCl, CuSO<sub>4</sub>, Ba<sub>2</sub>[Fe(CN)<sub>6</sub>], BaFe(CN)<sub>6</sub>, HCl, H<sub>2</sub>SO<sub>4</sub>, KOH, NH<sub>4</sub>OH and acetic, propionic, tartaric and citric acids. The results agree with those obtained previously by another method E. J. Rosenbaum

On the paper "The electrolysis of copper in potassium chloride" S. A. Artzibushev *Compt rend acad sci U R S S [N S]*, 3, 157-60 (1935)(in German), cf. C. A. 29, 2052—Some errors in the former paper are corrected. The mobility of the Cu ion is  $2.425 \times 10^{-11}$  m<sup>2</sup>/V sec (T = abs temp). An explanation of the diffusion of Cu into KCl based on a comparison of the ionic radii of the two ions is unsatisfactory A. B. F. Duncan

The stepwise titration of a dibasic acid S. Kilpi *Z physik Chem* A174, 441-4 (1935)—Conditions were derived for the stepwise titration of moderately strong and weak dibasic acids. The potential difference at the 1st stoichiometric equivalence point has a maximum when  $K_1/K_2 > 13.93$  and a minimum at  $K_1/K_2 < 13.93$ , whence it can be assumed that  $K_1 > K_2$ . This indicates a change in the buffer action of the equimolar mixture of an acid and base from a minimum to a maximum at  $K_{HA} K_{BOH} = 13.93 K_w$  or at  $K_{HA} K_{BOH} = K_w/13.93$ . The H ion concentration at maximum or minimum p d in the case of moderately strong or weak acids, like the H ion concentration at the first equivalence point, is independent of the value  $K_1/K_2$ . With high values for  $K_1/K_2$  the H-ion concentration at maximum p d, as well as the H-ion concentration at the 1st equivalence point, is independent of the strength of the acid R. H. Baechler

The potentials of cells in liquid-ammonia solutions The thermodynamic constants of the ammonium cadmium chlorides and of cadmium chloride Clifford S. Garner, Emerson W. Green and Don M. Yost *J Am Chem Soc* 57, 2055-8 (1935)—The e.m.f. of the cell Zn amalgam, ZnCl<sub>2</sub>·6NH<sub>3</sub> (solid), NH<sub>4</sub>Cl (solid in liquid NH<sub>3</sub>), CdCl<sub>2</sub>·6NH<sub>3</sub> (solid), Cd amalgam was measured at 25° at several concentrations of NH<sub>4</sub>Cl and of the amalgams. Calculated to pure Zn and Cd,  $E^\circ = 0.3605$  v. From the measured potential and its temperature coefficient, together with results of other investigators,  $\Delta F^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$  were calculated and tabulated for the solid ammonium cadmium chlorides and CdCl<sub>2</sub>. Provisional values for the activity coefficients of NH<sub>4</sub>Cl in liquid NH<sub>3</sub> at 25° are calculated and in combination with the free energy data yield the standard half-cell potentials, referred to H<sub>2</sub> in liquid NH<sub>3</sub>,  $Ti/TiCl_2 - 0.10$  v.,  $Zn/ZnCl_2 \cdot 6NH_3 + 0.7293$  v.,  $Cd/CdCl_2 \cdot 6NH_3 + 0.3688$  v. L. W. Elder

Application of the glass electrode to unbuffered systems Samuel B. Ellis and Samuel J. Kiehl *J Am Chem Soc* 57, 2139-44 (1935)—Coming off glass is too soft for glass electrode p<sub>H</sub> measurement in distilled water. Ordinary soft glass can be used in a flow method which flushes out soil impurities. Streaming potentials are avoided by proper placing of the liquid junction. Measurements of potential vs. rate of flow for pure water and dilute solutions of Na<sub>2</sub>HPO<sub>4</sub> and of KCl show that rates above 25 cc/min are necessary for unbuffered and weakly buffered systems L. W. Elder

The purification of water and its p<sub>H</sub> value Samuel B. Ellis and Samuel J. Kiehl *J Am Chem Soc* 57, 2145-9 (1935)—A still embodying the principles of Bourdillon (C. A. 8, 1889; 22, 7) and Wieland (C. A. 12, 560) removes CO<sub>2</sub> but not all NH<sub>3</sub>. Addition of alk. KMnO<sub>4</sub> or Nessler reagent to the stock distilled water used as feed did not remove NH<sub>3</sub> from the first portions of distillate. Intermediate fractions distilled from Nessler reagent were acid. H<sub>2</sub>PO<sub>4</sub> in the feed water yielded distillates of constant

p<sub>H</sub>. Measured by the authors' method (preceding abstract) the p<sub>H</sub> was  $7.01 \pm 0.01$  at 27.5° for samples collected in Ag vessels. L. W. Elder

Note on the paper on the antimony electrode for p<sub>H</sub> measurements by P. Wulff, W. Kordatzki and W. Ehrenberg *Arvid Holmqvist Z Elektrochem* 41, 807 (1935), cf. C. A. 29, 7762—Priority is claimed for some of the observations given. Reply. P. Wulff and W. Kordatzki *Ibid* C. E. P. Jeffreys

Junction potentials between solutions of sugars and potassium chloride John M. Ort and Martin H. Roepke *J Phys Chem* 39, 941-3 (1935)—For most potential measurements made on glucose solutions, the salt junction potential may be ignored when saturated KCl salt bridges are used and the p<sub>H</sub> of the glucose solution does not greatly exceed 10 and the sugar solution has not been heated or treated in any way that produces a large amount of ionized decomposition products A. E. Bettlich

Oxidation-reduction potential of reductone Rene Wurmser and Nélucia Mayer *Compt rend* 201, 1366-8 (1935); cf. C. A. 29, 1109—By means of solutions of reductone, CH(OH)C(OH)CHO, containing a leuco dye (e.g., leuco alizarin blue) suitably buffered, the oxidation-reduction potential was plotted against p<sub>H</sub> for p<sub>H</sub> 2-8, whence if the dissociation constants of reduced and oxidized reductone are taken as  $10^{-10}$  and  $10^{-11}$ , respectively, which gives a curve agreeing well with experiment up to p<sub>H</sub> 6, the normal potential at p<sub>H</sub> 0° and 38° is 0.283 v. Reductone reduced in this way is regenerated unchanged by I C. A. Silberrad

Thermal reaction between gaseous iodine monochloride and hydrogen W. D. Bonner, W. L. Gore and Don M. Yost *J Am Chem Soc* 57, 2723-4 (1935)—For the reaction of ICl and H<sub>2</sub> in the gaseous state values of  $k$  are  $205^\circ, 0.031 \pm 0.006$ ,  $230^\circ, 0.17 \pm 0.06$ ,  $240^\circ, 0.35 \pm 0.06$ . The tentative mechanism suggested is  $H_2 + ICl = HI + HCl$  (slow);  $HI + ICl = HCl + I_2$  (rapid). When the average values of the specific reaction rate constants are plotted against  $1/T$  a straight line results. The energy of activation for the assumed slow reaction is 33,900 cal C. J. West

Equilibrium studies on the exchange reaction between acetylene and heavy water L. H. Reyerson and Bruce Gillespie *J Am Chem Soc* 57, 2250-1 (1935); cf. C. A. 28, 4373—An isotopic exchange occurs between C<sub>2</sub>H<sub>2</sub> (I) and alk. solutions of D<sub>2</sub>O (II). The reaction takes place because of the ionization of I, but the details of the change in d of II makes it possible to calculate  $K_2$ . The equilibrium between I and aq. solutions containing 9.77 and 18.97% of II was studied at 0°, 25° and 100°. Average values of  $K_2$  for the reaction  $I + II \rightleftharpoons C_2D_2 + H_2O$  were found to be 0.365, 0.45 and 0.51 at 0°, 25° and 100°, respectively. In contradistinction to the findings of Bell (C. A. 29, 3506) positive proof of the existence of C<sub>2</sub>D<sub>2</sub> in the equilibrium mixture was obtained by mass-spectroscopic investigation and the amount found compared favorably with that determined from the equilibrium study. A Raman spectrum determined of a sample of gas taken from the equilibrium reaction showed lines for the C<sub>2</sub>D<sub>2</sub> molecule. It is felt that these equilibrium studies represent real equilibrium values within the limit of error of the experimental technique employed C. R. Addinall

A reaction of atomic iodine with quartz glass Gerhard Brauer *Z physik Chem* A174, 435-40 (1935)—Thermally dissociated I vapor on coming into contact with quartz glass produces a brownish coating which can be identified as SiI<sub>4</sub>. If the O<sub>2</sub> is not conducted away, the coating consists of SiO<sub>2</sub>. Optically dissociated I produces the same effect R. H. Baechler

The velocity of alkaline saponification of several crotonic esters E. Schjånberg *Z physik Chem* A174, 465-71 (1935)—The velocities of alkali saponification of several crotonic and butyric esters and its dependence upon temperature were determined. Esters of  $\alpha, \beta$ -unsaturated acids saponify more slowly than their saturated analogs. There is a difference in the saponification velocities of primary and secondary alcohols. The activation energy and action constants were determined from the temperature coefficients. Secondary alcohols have smaller velocity constants because of their smaller action constants

Crotonic esters have smaller velocity constants than butyric esters because of their higher energy of activation.

R. H. Baechler

The velocity of thermal decomposition of ammonia on a quartz surface. J. A. Christiansen and Eggert Knuth. *Kgl. Danske Vidensk. Selskab Math.-fys. Medd.* 13, No. 12, 20 pp (1935) (English).—The authors detd the velocity of decompn of  $\text{NH}_3$  in a quartz bulb of 200 cc vol and 210 sq cm surface heated in a manually regulated elec. furnace at temps of 106.2–1132°K with final pressures of 36.50–59.42 mm. The pressure changes were read on a Hg manometer to within 0.01 mm. They suggest the mechanism  $\text{wall} + \text{NH}_3 \rightleftharpoons \text{NH} + \text{H}_2$  + wall,  $\text{NH}^* \rightleftharpoons \text{NH}$ ,  $\text{NH}^* + \text{NH}_3 \rightleftharpoons \text{N}_2 + 2\text{H}_2$ ,  $\text{NH}$  and  $\text{NH}^*$  are 2 different forms of the radical. The presence of  $\text{NH}$ , although in very small amts, was established by its conversion into  $\text{NH}_4\text{OH}$  and detection of the latter by the method of Blom (cf. C. A. 20, 1368). Numerical data and curves are given.

E. R. Schierz

The kinetics of the autoxidation of catechol in the presence of several foreign substances. G. E. K. Branch and M. A. Joslyn. *J. Am. Chem. Soc.* 57, 2388–94 (1935).—The oxidation is definitely a chain reaction because small amts of cysteine,  $\text{K}_2\text{SO}_4$  and *o*-aminophenol inhibit during the initial period while hydroquinone, resorcinol, pyrogallol, gallic acid and *p*-aminophenol accelerate the reaction after an induction period. Large concns. of hydroquinone or pyrogallol increase the rate because of a process of chain exchange and there is no induction period. Initial rates in the presence of resorcinol are decreased but later become greater than those of catechol alone on account of the formation of a rapidly oxidized intermediate that acts as an accelerator.

R. E. DeRicht

Kinetics of the permanganate-oxalate reaction. I. Effect of various salts on the rate of reaction. R. W. Fessenden and Bryan C. Redmon. *J. Am. Chem. Soc.* 57, 2246–9 (1935).—This reaction was studied in the presence of small oxalate concns. and no manganous ion. A neg. kinetic salt effect was found with the sulfates of Na, K and Mg, while a pos. effect was found with Al, Zn and Cd sulfates. The cations of the salts that showed a pos. effect are known to form oxalate complexes. The rate varies inversely as the oxalate concn. A mechanism is proposed.

E. J. Rosenbaum

Allotropy of very pure calcium. Alfred Schulze. *Physik. Z.* 36, 595–8 (1935).—The changes from  $\alpha$  to  $\beta$  and  $\beta$  to  $\gamma$  phases are shown very precisely with Ca 99.9% pure. The changes were followed by measuring 1 ps, elec. cond. of pure Ca wires and by dilatometric methods.

A. B. F. Duncan

Allotropy of calcium. M. C. Neuburger. *Z. Elektrochem.* 41, 790 (1935).—Analysis of published data show that Ca exhibits 3 and not 4 allotropes. Below 300° a cubic, face-centered ( $A_1$ -type) is stable, at 300–450°, type 2 and above 450° a hexagonal (type 3) are the stable forms. The other form reported to exist above 450° was due to measurements on Ca contg 4–5%  $\text{N}_2$ . Ca, 99.9% pure, exists only in the hexagonal lattice form.

C. E. P. Jeffreys

Isotherms of methane between 0° and 150° and densities 19 and 53 Amagat (pressures between 20 and 80 atmospheres). A. Michels and G. W. Nederbragt. *Physica* 2, 1000–2 (1935).

Harold Gershinowitz

The phase diagram of low-melting mixtures. II. The melting diagram of oxygen-nitrogen and the phase diagram of nitrogen-carbon monoxide. M. Rubeman, A. Likhter and P. Komarov. *Physik. Z. Sowjetunion* 8, 326–36 (1935); cf. C. A. 29, 321.—Measurements of sp. heat show that CO-N mixts. form 2 complete series of mixed crystals. The system O-N has a eutectic at 50°K. and 23% by vol. of N. The O lattice dissolves 16% N and the N lattice 7% O. The heats of fusion of the N-O mixt. show a min. near the concn. of the satd. N crystal.

Morris Muskat

Layer formation in a two liquid system. R. V. Mertzlin and V. I. Ust-Kachintzev. *J. Gen. Chem. (U. S. S. R.)* 5, 771–8 (1935).—From a study of the isotherms of the

systems  $\text{Et}_3\text{N}-\text{H}_2\text{O}-\text{KCl}$  at 0°, 10° and 15°,  $\text{C}_4\text{H}_9\text{N}-\text{H}_2\text{O}-\text{KCl}$  at 0°, 20°, 50° and 80° and  $\text{C}_6\text{H}_{11}\text{N}-\text{H}_2\text{O}-\text{KCl}$  at 20°, 40°, 60° and 80° it is concluded that the binary systems  $\text{Et}_3\text{N}-\text{H}_2\text{O}$ ,  $\text{C}_4\text{H}_9\text{N}-\text{H}_2\text{O}$  and  $\text{C}_6\text{H}_{11}\text{N}-\text{H}_2\text{O}$  should have at low temps a min. crit. temp. of layer sepn. S. L. M.

Equilibrium diagrams of the system magnesium-lithium. P. Sal'dau and F. Shamrai. *Z. anorg. allgem. Chem.* 224, 388–98 (1935).—Mg forms 3 solid phases with Li, not 2 as has been previously claimed. The limits of the  $\alpha$ -phase proceed from 10 atoms % Li at 15° to 12.5 atoms % Li at 465° and 19 atoms % Li at 500° until they cut the eutectic line at 588.2° at the point which corresponds approx. to 21% Li. Mg forms a fixed compd. with Li,  $\text{Li}_2\text{Mg}$ , which melts without decompn. and forms on crystn. of solid solns ( $\beta$ -phase) with both its components in the region 29–95 atoms % Li. The  $\gamma$ -phase which forms a solid soln of Mg in Li occurs between 98 and 100% Li.

M. McMahon

Intermetallic compounds. Introduction. W. Klemm. *Angew. Chem.* 48, 713 (1935). Recent methods and results on the detection of intermetallic compounds. G. Grube. *Ibid.* 714–19.—The following methods are discussed: x-ray investigation, dilatometric method (thermal expansion), detn. of elec. resistance, as a function of temp. and the detn. of magnetic properties as a function of the concn. and temp. Many graphs and diagrams are shown. Equil. diagrams are presented for the amalgams of Cs, Rb, K, Na and Li. Compds. of Mg and Li with various other metals are discussed. Twelve references. The structures of intermetallic phases. Gunnar Hägg. *Ibid.* 720–3.—The literature findings are discussed and the properties of metallic phases are summarized. Energy and space problems in the formation of intermetallic compounds. Wilhelm Biltz. *Ibid.* 729–34, cf. C. A. 29, 3273.—The methods available for measuring affinity in alloys are discussed and a general summary of the most recent results is given. The space chemistry of intermetallic compds. is reviewed. Ten references. Magnetism and chemical bond in intermetallic phases. Eckhart Vogt. *Ibid.* 734–40.—An extensive discussion of magnetic phenomena in their relation to chem. bonds. Twenty-three references.

Karl Kammermeyer

System stannic chloride-hydrochloric acid. André Chrétien and Georges Varga. *Compt. rend.* 201, 1491–3 (1935).—The constitutional diagram shows, besides  $\text{SnCl}_4$  and  $\text{HCl}$ , 2 compds:  $\text{SnCl}_4 \cdot \text{HCl}$  (I) and  $\text{SnCl}_4 \cdot 2\text{HCl}$  (II), melting congruently at –94.7° and –85.4°, resp. The eutectics are  $\text{HCl}$  and I, m. –121.4°, 6.8%  $\text{SnCl}_4$ ; I and II, m. –96.2°, 30.0%  $\text{SnCl}_4$ ; and II and  $\text{SnCl}_4$ , m. –93°, 45.8%  $\text{SnCl}_4$ .

C. A. Silberrad

System antimony iodide-sodium iodide-water. Félix François. *Compt. rend.* 201, 1489–91 (1935).—Solr. isotherms are traced for 15°, 35° and 60° for solns. of such compn. that  $\text{SbI}_3$  is not hydrolyzed. Only 2 double salts,  $\text{SbI}_3 \cdot 2\text{NaI} \cdot \text{H}_2\text{O}$  (I) and  $\text{SbI}_3 \cdot \text{NaI} \cdot 6\text{H}_2\text{O}$  (II) were found, with no trace of  $\text{SbI}_3 \cdot \text{NaI} \cdot \text{H}_2\text{O}$  or  $2\text{SbI}_3 \cdot 3\text{NaI} \cdot 12\text{H}_2\text{O}$ . By slow evapn. of suitable solns. I was obtained in orange prisms, II in long needles.

C. A. Silberrad

Binary addition compounds in ternary systems. IX. System phenacetin-sulfonal-resorcinol. Franciszek Adamus. *Roczniki Chem.* 15, 383–9 (in French 389–90) (1935); cf. C. A. 29, 6826.—By thermal analysis there were found a peritectic point 59.0° at 29% phenacetin, 37% sulfonal and 34% resorcinol, and a eutectic point 50.0° at 15.5% phenacetin, 43.5% sulfonal and 41% resorcinol.

M. Wojciechowski

Homogeneous catalysis—decomposition of acrolein catalyzed by iodine. H. W. Thompson and J. J. Frewing. *Trans. Faraday Soc.* 31, 1660–8 (1935); cf. C. A. 29, 1703.—The kinetics of the decompn. of acrolein catalyzed by  $\text{I}_2$  are complicated and unsuitable for detailed analysis, but certain conclusions may be drawn. In the presence of  $\text{I}_2$  vapor the temp. at which acrolein begins to decompose is more than 100° lower than in the absence of catalyst. There are indications that the decompn. is unimol. and yields more  $\text{C}_2\text{H}_4$  than without  $\text{I}_2$ . Acrolein appears to retard its decompn. presumably by some chain-breaking



process. The velocity is approx proportional to  $[I_2]^{1/2}$ . It is not possible to decide whether this implies catalysis by I atoms or molecules. The catalyzed reaction has an activation energy of about 32,000 cal. C. E. P. Jeffreys

Catalysis of oxidation at bounding surfaces D. B. Kroon *Acta Brevis Neerland Physiol. Pharmacol. Microbiol.* 5, 127-9 (1935), cf C A 29, 49031—A discussion

Catalytic oxidation of carbon Jesse E Day, Richard F Robey and Hypp J Dauben *J. Am. Chem. Soc.* 57, 2725-6 (1935)—The catalytic effect of NaCl on the oxidation of soot C, demonstrated by Taylor (C. A. 24, 4209), is exhibited by other salts (LiCl, NaCl, KCl, CaCl<sub>2</sub>, SrCl<sub>2</sub>, BaCl<sub>2</sub>, Ca(OH)<sub>2</sub>, Ba(OH)<sub>2</sub>, Na<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>SO<sub>4</sub>). Numerical values are given, at 575° the C burned off bare glass in about 180 sec, which was cut down to 20-30 sec by all the salts, at 515°, the oxidation from the bare surface required 630 sec, this was cut down by Ba and Sr salts to 300, by Ca salts and NaCl to 230 and by KCl, LiCl, Na<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>SO<sub>4</sub> to about 170 sec. C. J. West

Application of Polanyi's potential theory to the van der Waals adsorption of gases on iron synthetic ammonia catalysts P H Emmett and Stephen Brunauer. *J. Am. Chem. Soc.* 57, 2732-3 (1935)—Potential-vol curves for the adsorption of N and A on a single promoted Fe synthetic-NH<sub>3</sub> catalyst are given, which seem to represent the isotherms very closely throughout the entire pressure range covered, including the region concave to the pressure axis where probably less than a mol. layer of gas exists, the linear portion that has been postulated to represent the building up of a second layer of gas on the catalyst surface and the higher-pressure portion, which is convex to the pressure axis and which, it is generally believed, represents the condensation of the gas to liquid in the capillaries of the adsorbent. The potential curves exhibit no variation with temp, the -183° and -195.8° points falling on the same curve. C. J. West

Effect of adsorbed water on the catalytic decomposition of hydrocarbons by a molecular beam method Otto Beecck. *Nature* 136, 1028-9 (1935)—Enough H<sub>2</sub>O is necessary in a gas to maintain a unimol layer on the filament (Pt or C) if dehydrogenation is to take place. Excess H<sub>2</sub>O inhibits at lower temps. Above 1550° all hydrocarbons except CH<sub>4</sub> decompose completely, indicating the formation of free radicals, by the rupture of a C-C, which are adsorbed on the walls. H<sub>2</sub>O can be substituted by mols contg H and an electroneg element. H<sub>2</sub>O is also necessary for hydrogenation. R. E. DeR.

Catalysis by fusions. I. E Adadurov and F D Didenko *J. Am. Chem. Soc.* 57, 2718-22 (1935)—Although orientation of a metal surface may be retained at the m. p., the decompn of CH<sub>3</sub>OH on Zn surfaces is now found to be catalyzed by ZnO, hence the const. rise in activity from solid to liquid Zn found by Steacie and Ekin, C. A. 28, 9604, is not conclusive proof of the nonexistence of active centers. Further evidence is that the catalytic oxidation of NH<sub>3</sub> by Sn is due to the oxide; hence the rate increases upon fusion but the activity of Ag falls off abruptly at the m. p. and the nature of the reaction changes because the oxide is not formed. Mechanisms are cited for the formation of ZnO and expts show Zn chips to be coated with oxide. R. E. DeRicht

A promoter effect of gases upon catalysts of hydrolysis

K. Ablezova and S. Rogunskii. *Z. physik. Chem.* A174, 449-64 (1935).—The promoter effect of H upon Ni was studied. A well-defined max in the curve  $k = f(p/m)$ , which represents the catalytic activity as a function of the gas content of the layer formed by condensation, was demonstrated, the optimum corresponds to 1 H<sub>2</sub> mol. to 100 Ni atoms. The curve explains a series of contradictions obtained in a study of the effect of the gas upon catalytic activity. R. H. Baechler

Heat capacity, entropy, and free energy of rubber hydrocarbon Norman Bekkedahl and Harry Matheson *J. Research Natl Bur Standards* 15, 503-15 (1935) (Research Paper No. 844)—Values are given for the heat capacities at const. pressure ( $C_p$ ) of the rubber hydrocarbon in its different forms from 14 to 320°K. The  $C_p$  value for both the metastable amorphous and crystalline forms at 14°K. is 0.064 joules/g./degree. With increase in temp the  $C_p$  increases gradually up to a transition at about 199°K, the amorphous form having a little the greater value. At 199°K. both the forms undergo a transition of the second order, the  $C_p$  rising sharply. For the amorphous form above this transition the  $C_p$  rises gradually without discontinuity to the highest temp. of the measurements. The crystalline form undergoes fusion (a transition of the first order) at 284°K., the heat of fusion being 16.7 joules/g. At 298.1°K. (25°C.) the  $C_p$  of the rubber is 1.880 ± 0.002 joules/g./degree C., its entropy 1.881 ± 0.010 joules/g./degree C., and its standard free energy of formation from the elements 1.35 ± 0.10 kilojoules/g. N. Bekkedahl

Phase-boundary energies in phase systems of pure electrically neutral phases H. Hohn and E. Lange. *Physik. Z.* 36, 603-21 (1935).—A discussion of thermodynamic relations between surface energy, work of adhesion, surface tension, work of cohesion, surface cohesion, immersion energy and free energy, interfacial tension and similar quantities. A table of formulas with 23 references is included. A. E. F. Duncan

Temperatures and heat tones of genotypical transformations of the alkali salts of long-chain fatty acids. Peter A. Thiessen, Jürgen v. Kleck, Hilde Gockowiack and Joachim Stauff. *Z. physik. Chem.* A174, 335-58 (1935).—The temps at which the heat tone corresponding to the genotypical transformation sets in were detd. by a simple differential process. The temp.-dielec. const. curve shows a break at the transformation. The optical refraction in a crystal of Na stearate changes sharply at the start of genotypical transformation and at 49° a monotropic transformation is indicated. The heat of transformation increases with length of the chain and depends upon the nature of the cation. A relation exists between the heat tone and the radius of the cation. The sp. heat-temp curve shows a break at the genotypical point, in agreement with the prevailing idea of the structure of long-chain fatty acids. App and technic are described. R. H. Baechler

X-ray structure and elec. resistance of Pd-Ag-H alloys (Rosenhall) 9. Chem. action caused by neutrons and  $\gamma$ -rays and the effects of these agents on colloids (Hopwood, Phillips) 3. Detn. of ionization by ultraviolet spectrophotometry, its application to the measurement of strength of weak bases (Flexser, et al) 3.

### 3—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

W. ALBERT NOYES, JR.

The neutrino theory of light O. Scherzer. *Z. Physik* 97, 725-39 (1935)—A quant. application of the neutrino theory is made which gives the main optical phenomena correctly but does not avoid the problem of infinite proper energy of the electron. B. Swirles

Ionic generator for high potentials Marcel Pauthemer and Marguerite Moreau-Hanot. *Compt. rend.* 201, 1332-4 (1935); cf C. A. 26, 1184, 29, 684—A dust-laden gas is aspirated round a circuit passing first through an

ionizing tube in which the axial wire is maintained at a potential of  $\approx 10$ -15 kv., then through a U-tube of conducting material enclosed in the sphere to be charged (30 cm diam.), the middle portion of the U-tube being flattened so as to ensure contact therewith of the charged particles. A charge of 450 kv. was thus readily obtained on the sphere. Calcn. shows that there should be little difficulty in attaining much higher potentials. C. A. Silberrad

Thermal diffusion of color centers. O. Stasiv. *Physik.*

Z. 36, 737-40(1935); cf. C. A. 29, 3236<sup>6</sup>.—Thermal diffusion consts. of color centers in KCl were measured and found to lie between 0.057 and  $0.4 \times 10^{-10}$  sq. m./sec. at a temp. of 490-755°. The consts. were compared with older measurements of velocity of migration of pos. ions in an elec. field. Above 700° the electrons move at about the same rate as pos. ions. Below 700° there is some slowing down of the electrons by the pos. ions.

C. L. P. Jeffreys  
The theory of electron motion in nonmetallic crystal lattices. I. Hund. *Physik Z.* 36, 725-9(1935).

C. L. P. Jeffreys  
Electronic conduction in alkali halide crystals. R. W. Pohl. *Physik Z.* 36, 732-5(1935); cf. C. A. 29, 7801<sup>1</sup>.—Color centers in alkali halide crystals permit optical measurements of electron velocity and the detn. of the no. of migrating electrons. The crystals with U-centers are semiconductors and may be studied as photoelec. conductors.

C. L. P. Jeffreys  
Disarrangement phenomena in ionic lattices as basis for ionic and electronic conduction. Carl Wagner. *Physik Z.* 36, 721-5(1935).—Examples of 3 types of disarrangement in ionic lattices: in-between-lattice type, vacant-space type and substitution type, are discussed in relation to ionic and electronic conduction. Three types of oxides whose conductances vary with O<sub>2</sub> pressure are discussed from the point of view of disarrangement of electron distribution.

C. L. P. Jeffreys  
The problem of ionic and electronic conduction in non-metallic solid substances. B. Gudden and W. Schottky. *Physik Z.* 36, 717-21(1935).—A general discussion.

C. E. P. Jeffreys  
Glow electron emission and electronic conduction of solid substances. A. Gührts. *Physik Z.* 36, 704-7(1935).—Glow electron emission from cathodes covered with monatomic adsorption layers is a purely surface phenomenon, as in the case of homogeneous cathodes. The variance of the proportionality const. in the formula  $I = ATe^{-T}$  from the theoretical value, 60.2 amp./cm.<sup>2</sup>/deg.<sup>3</sup> is due to special properties of the adsorption layer and not to a relation between electronic conduction and glow emission.

C. E. P. Jeffreys  
Concentration of sodium vapor in the electric arc. Thadée Peculski and Nicolas Szule. *Compt. rend.* 201, 1335-7(1935).—The spectrum of Na was observed by means of a current of 10-80 amp. passed between C electrodes embedded in NaCl. From the width of the lines D<sub>2</sub> and D<sub>1</sub> and the relation  $D = 1.9 \times 10^{-7} \sqrt{n}$ , where  $n$  is the concn. of the vapor (cf. Trumphy, C. A. 21, 1061) the concn. of Na atoms producing lines of emission (i. e., in the interior of the arc) is approx. 10 times that of atoms in the exterior portion, producing absorption,  $7 \times 10^{13}$  and  $0.8 \times 10^{14}$ , resp.

C. A. Sjöstrand  
Negative glow discharge. Investigations of hindered discharges in hydrogen. W. Weizel and H. Fischer. *Ann. Physik* 24, 201-30(1935).—An auxiliary electrode was placed in the neg. glow of a H discharge tube and the currents resulting from various combinations of potentials were measured. The diffusion of the charge carriers was important. Rogowski's theory does not apply to these results.

E. J. Rosenbaum  
The "counting" phenomenon as a space-charge problem. A. v. Hippel. *Z. Physik* 97, 455-81(1935); cf. C. A. 27, 2099<sup>1</sup>.—The current-voltage characteristic for rarefied gases is considered and it is shown how a discharge becomes unstable because of a pos. space-charge and how stabilization occurs because of diminution in the no. of collisions. The region of validity of Paschen's law and an exact definition of sparking potential are given. The behavior of the characteristics for spark and steady discharge in a homogeneous field are given and a "counting region" and "counting conditions" are found. Further the pos. and neg. forms of discharge in an inhomogeneous field are explained, with applications to the actual form of the Geiger counter.

B. Swirles  
Excitation function of mercury in atomic collisions. K. Gailer. *Ann. Physik* 24, 421-39(1935).—The excitation of Hg atoms by H atoms with energies in the range

2-40 e. kv. is at least 20 times as great as that by protons. The excitation function for the 2537 Å. line has a max. at 20 e. kv. In these collisions the Hg atoms only are excited. Excitation functions for H atoms and electrons are compared. In a collision, the effect of a fast particle does not depend on either its velocity or its kinetic energy alone but on the energy which can be transferred. This is a function of the excitation and ionization energies and of the masses of both collision partners. E. J. Rosenbaum

E. J. Rosenbaum  
Energy losses of electrons in helium, neon and argon. R. Whiddington and F. G. Woodroffe. *Phil. Mag.* 20, 1109-20(1935), cf. C. A. 28, 4978<sup>8</sup>.—The accuracy of the method described previously has been increased. The electron energy used was around 100 e. v. The energy losses in He were 21.11, 23.06 and 23.70 e. v. with relative probabilities of 10, 2 and 1, resp. In Ne the prominent losses were 10.76, 18.62 and 10.08 e. v. with relative probabilities of 10, 4 and 8, resp., while in Ar the prominent losses were 11.77 and 14.23 e. v. Most of the energy losses could be correlated with particular excited states of these atoms. E. J. Rosenbaum

E. J. Rosenbaum  
Collisions of slow electrons with methane molecules. Henry L. Brose and John E. Keiston. *Phil. Mag.* 20, 902-12(1935), cf. C. A. 24, 5218<sup>1</sup>.—The min. effective collision cross section of CH<sub>4</sub> for slow electrons detd. by the Townsend method agrees with the value found by the Ramsauer method. The electron velocities for min. cross section are also in agreement. The similarities and differences of the interaction of CH<sub>4</sub> mols. and Ar atoms with electrons are discussed. The av. fraction of an electron's energy which is lost in a collision with a CH<sub>4</sub> mol. is several thousand times that for a similar collision with an Ar atom. The value of the electron energy for which the av. loss of energy is a max. corresponds to the wave length of the main infrared absorption bands of CH<sub>4</sub>. E. J. Rosenbaum

E. J. Rosenbaum  
Quantized velocity losses of slow electrons and effective cross sections in molecular gases. Helmut Löhner. *Ann. Physik* 24, 319-60(1935).—The velocity losses of slow electrons in H<sub>2</sub>, N<sub>2</sub>, CO, NO, N<sub>2</sub>O and CO<sub>2</sub> were studied by the Lenard method. Particular attention was paid to the smallest velocity at which inelastic collisions began. The smallest velocities are in v. H<sub>2</sub> 4.25, N<sub>2</sub> 2.45, CO 2.1, NO 3.65, N<sub>2</sub>O 7.2 and CO<sub>2</sub> 7.1. The losses in v-osteric collisions are also discussed. The sepn. of the velocity-effective cross-section curve into parts due to elastic and inelastic impact and the relation of this curve to the velocity losses of electrons are discussed. E. J. Rosenbaum

E. J. Rosenbaum  
Quantitative and qualitative observations on the electron diffraction pattern from the natural (111) face of diamond. R. Beeching. *Phil. Mag.* 20, 541-55(1935).—Intensity measurements were made on this pattern and details of the pattern are discussed. The inner potential detd. by the rotation and the Kikuchi line methods is 20.8 ± 0.7 v.

E. J. Rosenbaum  
The oxygen afterglow. Eric M. Stoddart. *Proc. Roy. Soc. (London)* A153, 152-7(1935), cf. C. A. 29, 1323<sup>1</sup>.—S. shows that the criticism of Rayleigh (C. A. 29, 5021<sup>1</sup>) is not valid. The yellow O afterglow is obtained best with a min. amt. of H<sub>2</sub>O vapor in the gas phase and a max. amt. adsorbed on the walls.

A. B. F. Duncan  
Air afterglow. F. H. Newman. *Phil. Mag.* 20, 777-81(1935).—The air afterglow is very faint in a clean tube but increases in brightness after a discharge is passed. Its spectrum is continuous in the range 4200-6700 Å. with a no. of diffuse bands superimposed. It is quenched by the vapors of water, acetone and benzene. CS<sub>2</sub> forms a blue glow where it enters the airglow stream and S is deposited on the walls. The afterglow is attributed to an oxidation of NO by O<sub>2</sub>, both of which are formed in the discharge. The O afterglow is similar. E. J. Rosenbaum

E. J. Rosenbaum  
Paramagnetism. III. Light absorption in paramagnetic crystals and solutions. S. Datta and Manindramohan Deb. *Phil. Mag.* 20, 1121-30(1935); cf. C. A. 28, 5300<sup>1</sup>.—The absorption spectra were studied for solns. of CrCl<sub>3</sub>, CoCl<sub>2</sub> and NiCl<sub>2</sub> in H<sub>2</sub>O, F<sub>2</sub>OH and concd. HCl soln. at temps. in the range -115° to 60°. For all 3 solns. the absorption max. shifts from its position for aq. solns. to longer wave lengths if the dielec. const. is

less than that of  $H_2O$  or if an excess of  $HCl$  is present. The absorption max. of  $EtOH$  and  $HCl$  solns. of  $CoCl_2$  and  $NiCl_2$  shift to shorter wave lengths as the temp. is lowered to  $-115^\circ$ . At this temp. the values agree with those in aq. soln. Whenever the values from magnetic data are large the absorption max. is shifted to longer wave lengths and conversely. An explanation is discussed.

I. J. Rosenbaum

Adsorption of alkali metals on metal surfaces. VI. Selective photoelectric effect. J. H. de Boer and C. F. Veemans. *Physica* 2, 915-22 (1935), cf. *C. A.* 29, 7150\*—The adsorbed atoms can give rise to a selective photoelectric effect for wave lengths relatively far in the ultraviolet. The spectral max. is situated at a wave length shorter than that of the ionization limit of the atom concerned. For large values of  $\theta$  other atoms are adsorbed which are polarized in the opposite direction. This gives rise to a selective photoelectric effect with a spectral max. farther toward the long wave lengths. The results of I-IV are summarized.

Harold Gershonowitz

Neutrino theory of light III. R. de L. Kronig. *Physica* 2, 968-80 (1935), cf. *C. A.* 29, 7175\* H. G. The cosmic radiation problem. P. M. S. Blackett. *Physik Z.* 36, 773-6 (1935).

C. E. P. Jeffreys

The passage of cosmic radiation through matter. H. Kulenkampff. *Physik Z.* 36, 785-7 (1935).—Coincidence measurements were made with counter tubes perpendicularly over each other with absorption sheets between and over the tubes. A secondary radiation of range 35 cm. of Fe was released in the absorber by  $\gamma$ -radiation with an absorption coeff. of  $0.009 \text{ cm}^{-1}$  Fe. C. E. P. Jeffreys

Nuclear phenomena and cosmic rays. W. F. G. Swann. *Ohio J. Sci.* 35, 311-42 (1935); cf. *C. A.* 29, 4667\*—A review.

E. J. Rosenbaum

Forces which determine the structure of the atomic nucleus. C. F. v. Weizsäcker. *Physik Z.* 36, 779-85 (1935).—The theoretical derivations of nuclear forces are discussed.

C. E. P. Jeffreys

Artificial radioactivity. E. O. Lawrence. *Ohio J. Sci.* 35, 388-405 (1935).—A review, particularly of L.'s recent work.

E. J. Rosenbaum

Nuclear transformations and the origin of the chemical elements. G. Gamow. *Ohio J. Sci.* 35, 400-13 (1935).—A review, mainly of recent theories of the state of stellar interiors.

E. J. Rosenbaum

Ways and kinds of artificial atomic transformations. W. Bothe. *Physik Z.* 36, 776-9 (1935).—Nuclear reactions, the different types of transformations and the stability of the products are considered.

C. E. P. J.

Artificial radioactivity. II. D. van der Veen. *Chem. Weekblad* 32, 667-71 (1935).—Continuation of a previous article (*C. A.* 29, 2073\*).

A review. N. Belledahl

Destruction of lithium, boron and deuterium. Kessar. D. Alexopoulos. *Helv. Phys. Acta* 8, 601-36 (1935).—An app. for bombarding with 140-kv. protons and deuterons is described. These are generated as canal rays and then accelerated in an elec. field. If any 1.8- and 4.0-cm. particles were obtained from Li bombarded with protons their intensities were less than 3% of that of the 7-cm. range. Li bombarded with deuterons gave less than one quantum for 8 disintegrations. Deuterium bombarded with deuterons gave neutrons and protons according to the schemes  $2D^+ \rightarrow {}^3He^+ + {}^1H^+$  and  $2D^+ \rightarrow {}^3He^+ + {}^1H^+$ .

Gregg M. Evans

Isotopy of the elements. J. Mattauich. *Z. Ver. deut. Ing.* 79, 1140-4 (1935).—A review of recent developments.

Harold Gershonowitz

Agreement between nuclear energy differences and experimental masses of light elements. Louis Cartan. *Compt. rend.* 201, 1363-6 (1935).—From considerations of the work of Aston, and of Oliphant, et al. (*C. A.* 29, 4225\*, 4665\*), and results deduced from band spectra the following revised at. masses are deduced.  ${}^1H$  1.0083  $\pm$  0.0005;  ${}^2H$  1.0081  $\pm$  0.0001;  ${}^3H$  2.0143  $\pm$  0.0002;  ${}^4He$  4.0034  $\pm$  0.0003;  ${}^6Li$  6.0166  $\pm$  0.0010;  ${}^7Li$  7.0167  $\pm$  0.0012;  ${}^{9Be}$  9.0152  $\pm$  0.0010;  ${}^{10B}$  10.0141  $\pm$  0.0009;  ${}^{11B}$  11.0109  $\pm$  0.0010 and  ${}^{12C}$  12.0037  $\pm$  0.0010.

C. A. Silberrad

Energies and products involved in nuclear disintegration and synthesis. M. L. Pool. *Ohio J. Sci.* 35, 343-41 (1935).—A review contg. tabulated data and excellent diagrams of potential barriers and nuclear energy levels.

E. J. Rosenbaum

Influence of the velocity of slow neutrons on their capture by certain nuclei. Peter Preiswerk and Hans von Halban, Jr. *Nature* 136, 1027 (1935).—The cross section for the capture of neutrons by Ag is inversely proportional to their velocity. An increased absorption by I was found for slow neutrons. The effect of temp. on velocity is discussed.

R. E. DeRight

Artificial radioactivity of scandium. G. Hevesy. *Kgl. Danske Vidensk. Selskab Math.-fys. Medd.* 13, No. 3, 17 pp. (1935) (English).—A sealed tube 5 cm. long and 3 mm. in diam. filled with a mixt. of Be powder and 200-300 millicurie Rn serving as the source of neutrons was placed in the center of a tube 12 mm. in diam. filled with  $Sc_2O_3$ . The whole was imbedded in a block of paraffin 15 cm. on edge and left for a few days. A Geiger-Müller tube was used to measure the activity of the  $Sc_2O_3$  after the removal of the neutron source. The  $Sc_2O_3$  was dissolved in dil.  $HCl$ , 100-150 mg. NaCl and CaO added, then pptd. with  $NH_4$ -free carbonate. The filtrate was treated with  $H_2C_2O_4$ ; the ppt. of  $CaC_2O_4$  was removed. The NaCl was recovered from the last filtrate. The  $CaC_2O_4$  was inactive, the NaCl was very active owing to  ${}^{11}K^+$  (half life time 16 hrs.) and the oxide owing to  ${}^{48}Sc^+$  (half life time some years).  ${}^{48}K^+$  emits  $\beta$ -rays having an Al. absorption coeff. 10 cm. $^{-1}$ . Those of  ${}^{48}Sc^+$  have a coeff. of 63 cm. $^{-1}$ . It is probable that the formation of  ${}^{48}K^+$  to which is due the natural radioactivity of K in geological times takes place in a way similar to that of  ${}^{48}K^+$  in the lab.

E. R. Scherz

Deuterium. I. D. MacGillivray. *Chem. Weekblad* 32, 490-3 (1935).—A review of the discovery of D and a description of a method of sepn. of  $D_2$  and  $H_2$  by diffusion. II. *Ibid.* 650-5.—A review of several phases of the D literature: concn. of D by fractionated freezing and crystn., phys. properties of  $H_2$ ,  $H_2O$  and the isotopic compds.

B. J. C. van der Hoeven

Isotopic constitution of iron. J. de Gier and P. Zeeman. *Proc. Acad. Sci. Amsterdam* 38, 959-61 (1935).—By use of a very pure sample of carbonyl with properly chosen canals the line of isotope 68 of Fe was identified. The abundance relative to 57 was obtained by varying the time of exposure. The relative abundance of the isotopes is:  ${}^{54}$ , 6.5%;  ${}^{56}$ , 90.2%;  ${}^{57}$ , 2.8%;  ${}^{58}$ , 0.5%.

C. E. P. Jeffreys

A simple apparatus for the demonstration of the diffusion of  $\alpha$ -rays. R. Mercier and P. Scherrer. *Helv. Phys. Acta* 8, 589-90 (1935).—On a brass base sealed within a glass tube the Po source of the rays is fixed. The radiation is confined in direction by a narrow tube fixed at  $45^\circ$  to the center line of the base and enclosing tube. Over the end of the tube rests the foil which the rays are to penetrate. This whole assembly is fitted by a ground joint to a glass base having 2 arms at  $45^\circ$  to the center line above. One of these connects through a stopcock to the vacuum pump. The other contains the counter. By turning the ground joint the angle between the primary direction of the rays and the counter can be varied up to  $90^\circ$ .

Gregg M. Evans

Some properties of the radioactive series. E. Lopochnik. *Compt. rend.* 201, 1361-3 (1935).—On the basis of Perrin's models of at. nuclei extended to the heavier elements (cf. *C. A.* 26, 4238) certain regularities in the manner in which radioactive disintegrations occur in the elements subject thereto are traced.

C. A. Silberrad

Determination of small amounts of radium in rocks. J. A. Boettner. *Chem. Weekblad* 32, 482-6 (1935).—The Maché differential method (Hallebauer, *C. A.* 21, 3820) was used to det. small amts. of Ra, in it are used 2 ionization chambers (brass,  $10 \times 17$  cm., cylindrical), one contg. the unknown emanation, and a Wolf double-wire electrometer. The emanation was obtained by bubbling dry air, free of activity, through the unknown soln., and drying the gas over soda-lime and Na. Details are given on standardization of the app., natural ionization,

etc. The accuracy is  $10^{-14}$  g. Ra, giving 0.8% of the natural discharge rate. In Norwegian eschvite after proper chem. treatment a Ra content of  $6.8\% \times 10^{-14}$  g. per g. was found.

B. J. C. van der Hoeven  
Physical methods in the chemical laboratory XXVII  
The application of x-ray methods to chemical problems  
R. Brill and F. Halle *Angew. Chem.* 43, 785-95 (1934),  
cf. C. A. 29, 6477.—A review Karl Kammermeyer

A back-reflection Laue method for determining crystal orientation. Alden B. Greminger *Z. Krist.* 91, 424-32 (1935) (in English). cf. C. A. 29, 1002.—In unsym. back-reflection Laue photographs (cf. C. A. 26, 5005) a quasi-sym. arrangement of spots is maintained to an extent which permits ready identification of important spots; this identification is accomplished from visual inspection coupled with the use of a "hyperbolic polar-coordinate plot." The plot is illustrated, and data for its drawing are given. The method is for cubic crystals, and can be extended to hexagonal, tetragonal and orthorhombic if the axial ratio is known. L. S. Ramsdell

New technique for obtaining x-ray powder patterns. R. A. Stephen and R. J. Barnes *Nature* 136, 793-4 (1935).—For obtaining powder lines that are sharp at large angles of diffraction from flat specimens such as are met with in metallurgy, the authors recommend that 2 apertures be used in front of the circular film and rotated with the specimen, to permit only such diffracted beams to reach the film as make small angles with the surface of the specimen. This arrangement is used to advantage with a wide beam of x-rays collimated by a multiple diaphragm system. C. D. West

Effect of the slit on distribution of intensities of lines in an x-ray powder diagram. Anatole Rogozinski *Compt. rend.* 201, 1354-6 (1935).—The error in the spacing,  $d$ , resulting from error in detg. the Bragg angle,  $\theta$ , by reason of shifting of the max. of intensity in a line due to the breadth of the slit,  $2e$ , being finite is given by  $\Delta d/d = 1/\cot\theta \cos 2\theta e/D$ , where  $D$  is the distance of the substance under examn. (arranged in a band of width  $2e$  and indefinite length) from the photographic plate, or  $d$  is known the same expression gives the value of  $\Delta\lambda/\lambda$ . The ratio of the intensity obtained by means of a slit as above to that with a circular aperture of radius  $e$  is  $(4R/e)^{1/2}$ , and the optimum length for the band of substance under examn. is  $4\sqrt{Re}$ , where  $R = D \tan 2\theta$ . C. A. Silberrad

Absorption of short-wave x-rays. W. V. Mayneord and J. E. Roberts *Nature* 136, 793 (1935).—A narrow band of x-rays of mean wave length  $59 \text{ X}$  U was isolated from a 250-kv source by a filter of Pb 3 mm., Sn 2 mm. and Al 3 mm. This was used to measure, for 18 elements between Li and Pb, the mass absorption coeffs.  $\mu/\rho$  over a corresponding range 0.107-0.847. The mean absorption per electron for elements up to Ca is  $\mu_e = 4.6 \times 10^{-28}$ , in agreement with theory. The photoelectric absorption coeff. per electron  $\mu_p = \mu_e - 4.06 \times 10^{-28}$  is found to be proportional to the cube of the atomic no. For  $\text{EtOH}$ ,  $\mu/\rho = 0.142$ , for  $\text{H}_2\text{O}$  0.133, for 99% D O 0.118, in agreement with the expectation that  $\mu/\rho$  for H should be about twice that for other light elements, notably D. C. D. West

Fine structure of the K edges of magnesium and aluminum. J. Veldkamp. *Physica* 2, 932-4 (1935).—It is shown that Sandstrom's explanation of the fine structure found at the K edges of Mg and Al (Dissertation, Uppsala 1935) cannot be correct. By comparing these fine structures with those of Ti and Ca it appears that they agree with Kromg's theory. H. G.

New data on K spectrum of copper. Yvette Cauchois. *Compt. rend.* 201, 1379-81 (1935), cf. C. A. 29, 41.—With the increased dispersion, (4.7 X U./mm.) available several lines are shown to be complex. C. A. S.

New data on K spectra of molybdenum and rhodium. Horia Hulubei. *Compt. rend.* 201, 1378-9 (1935); cf. C. A. 29, 41.—These spectra have been reexamined with H.'s app. and several lines, in particular  $\beta_2$  and  $\beta_3$ , have been resolved. C. A. Silberrad

Scattering of x-rays by silver. Alexander A. Rusterholz. *Helv. Phys. Acta* 6, 365 (1933); cf. C. A. 25, 1153.—Scattering of Cu K radiation by the (220) plane of Ag has been studied. The K electrons of Ag do not contribute to the scattering. B. C. A.

Contribution of x-ray analysis to the question of electron terms in ionic lattices. R. d. L. Kronig. *Physik Z.* 36, 729-32 (1935).—The factors governing the course of fine structure of ionic crystals are discussed. It is emphasized that a more accurate knowledge of the wave functions, and more exptl. work in the soft and ultra-soft x-ray region are necessary before electron energy levels in ionic crystals can be detd. C. E. P. Jeffreys

Atomic scattering factors of nickel, copper and zinc. G. W. Brindley and F. W. Spiers *Phil. Mag.* 20, 865-81 (1935).—The intensity of Cu K $\alpha$  x-radiation scattered by Ni, Cu and Zn powders was detd. by a photographic method. Relative scattering factors were converted to absolutes by a comparison with results on KCl powder. A correction was applied to get the scattering factors for the atoms at rest. E. J. Rosenbaum

X-ray examination of lattice distortion in copper and nickel powders. G. W. Brindley and F. W. Spiers *Phil. Mag.* 20, 882-93 (1935), cf. preceding abstr.—The at. scattering factors were detd. for Cu and Ni from measurements on both pptd. and filed powders. The values for the filed powders were lower than the others. Conclusion: Filing results in a random displacement of the atoms from their position in the undistorted lattice. The amt. of this displacement is 0.106 Å for Cu and 0.083 Å for Ni. Some preferential displacement affecting the (100) planes is indicated. E. J. Rosenbaum

X-ray investigation of lattice distortion in a copper-beryllium alloy. G. W. Brindley and F. W. Spiers. *Phil. Mag.* 20, 893-902 (1935), cf. preceding abstr. and C. A. 29, 5794.—The av. at. scattering factor was detd. for the filed powder of an  $\alpha$ -phase Cu-Be alloy (0.72% Be). The exptl. values are less than those calcd. for an ideal lattice. The deviations are greater than those introduced by the filing process. It is suggested that the addnl. deviations are due to the presence of some atoms in the  $\gamma$ -phase. E. J. Rosenbaum

X-ray and magnetic measurements of potassium chloride powders in relation to lattice distortion and photochemical coloration. G. W. Brindley, F. W. Spiers and F. E. Hoare. *Phil. Mag.* 20, 1041-54 (1935), cf. preceding abstr.—X-ray measurements show that grinding a KCl powder produces very little, if any, lattice distortion. The fact that ground KCl powder is more rapidly colored by x-rays than a pptd. powder cannot be attributed to this effect. The diamagnetic mass susceptibility of ground KCl is approx. 1% less than that of the pptd. powder. This difference is attributed to the effect of grinding. E. J. Rosenbaum

Determination of the distribution of electron density and potential in a crystal lattice from x-ray data. V. E. Lashkarev. *Physik Z. Sowjetunion* 8, 227-39 (1935).—The use of the "equiv." atom, having spherical symmetry and an at. factor equal to the exptl. value, offers a new and simpler way of making the calcs. The equations are developed for cubic crystals. Calculation of potential distribution in certain crystal lattices. V. E. Lashkarev and A. S. Chaban. *Ibid.* 240-54.—The potential distribution is calcd. by the foregoing method and represented by topographic maps for the following cubic crystals: Li on (110) plane, Al on (100) and (111), NaCl on (100), diamond on (110). The potential at each point is detd. as the sum of potentials due to equiv. atoms situated at points of the actual lattice, the mean potential of the lattice being taken equal to zero. C. D. West

Spectrographic investigation of the thermoluminescence of feldspar. Satoyasu Imuro and Ei-ichi Iwase. *Sci. Papers Inst. Phys. Chem. Research (Tokyo)* 23, 147-51 (1935).—Irradiation with x-rays increased the intensity of the thermoluminescence without changing the wavelengths of the bands. Feldspar samples were irradiated, heated to 400° and the thermoluminescence spectra were photographed. These consist mostly of blue and yellow

bands whose intensity varies greatly from sample to sample. L. J. Rosenbaum

Line groups and fine structures. H. F. Paschen. *Sitzber preuss akad Wiss Physik-math Klasse* 1935, 430-43, cf C A 27, 1822.—The effect of the magnetic field of a nucleus on a group of lines, described previously, is here investigated for the narrow multiplets that occur in In II. It is shown that the no. distribution and intensities of the lines that make up a complex group can be quantitatively accounted for by certain simplifying consequences of the theory of Goudsmit and Bacher (C A 27, 4166).

C. C. Kiess  
The intensity of  $H\beta$  in the chromospheric spectrum. Philip C. Keenan. *Astrophys J* 83, 47-54 (1936).—From the measured  $d$  of  $H\beta$  in the light of the chromosphere 2000 km above the photosphere the energy emitted at  $H\beta$  over a hemisphere by 1 sq. cm. of the sun's surface was found to be  $5 \times 10^{10}$  ergs/sec. From this the no. of H atoms in the 4th quantum state at the same height is calculated as 7 atoms/cc. These results agree in order of magnitude with those found from eclipse observations.

C. C. Kiess  
Second spark spectrum of mercury. Raymond Ricard. *Compt rend* 201, 1342-4 (1935).—By means of the electrodeless discharge 30 new terms of group  $E_2$  (part of the Hg III spectrum), of which the most characteristic line at 4797.01 Å, excitation potential about 44 v., has been previously observed (Dejardin, C A 19, 1815).

C. A. Silberrad  
The spectra of early type stars in the near ultraviolet regions. P. Swings and M. Désirant. *Astrophys J* 83, 31-46 (1936).—The lines that appear between 3570 Å and 3930 Å in the spectra of 14 early-type stars have been measured and, so far as possible, have had their chem. origins assigned.

C. C. Kiess  
Nebular spectrum of Nova Herculis. Marie Bloch and Jean Dufay. *Compt rend* 201, 1463-5 (1935).—The spectrum of Nova Herculis from Oct. 18 to Nov. 14, 1935 showed permitted lines of H I, He I, He II, O II, N II, C II, O III, N III and probably C III, and forbidden lines of O I, N II, O II, S II, O III and Ne III and perhaps of Ne IV and F IV, while the origins of 4656 (perhaps identical with 4658.2 of Nova Pictoris and the nebula of Orion) and 4603 (N V) are uncertain. The spectrum closely resembles that of the planetary nebulae (cf Bowen C A 29, 1711).

C. A. Silberrad  
Reproducibility of the relative energy distribution of the continuous hydrogen spectrum emitted by a hydrogen discharge tube. V. E. Gonsalves. *Physica* 2, 1003-15 (1935).—Expts. are described showing the dependence of the relative energy distribution on the pressure of H in the tube and the current. The intensity of the total ultraviolet between 1850 and 3200 Å is practically const. for  $p = 3.0-3.4$  mm. if the current is const. The relative energy distribution is independent of the current, measured between 150 and 250 mμ, if  $p$  is const. The relative energy distribution does not change in the region of 1-3.5 mm. pressure for currents of 150-250 ma. These results indicate that the H tube can in principle be used as an ultraviolet standard lamp. Harold Gershonowitz

Predissociation in the  $A''$  level of CO and the dissociation energy of this molecule. F. Brons. *Physica* 2, 1108-13 (1935).—A rotational analysis of the bands  $10 \rightarrow 19$ ,  $10 \rightarrow 20$  and  $10 \rightarrow 21$  of the fourth pos. group of CO is given. From this analysis the rotational consts.  $B$  and  $\alpha$  were calculated, and compared with those from other investigations. For the  $R$ -branch  $B' = 1.359$  cm $^{-1}$ . For the rotational consts. of the lower level  $B''_{10} = 1.585$ ,  $B''_{19} = 1.572$  and  $B''_{21} = 1.553$  cm $^{-1}$ ,  $\alpha' = 0.024$  cm $^{-1}$  and  $\alpha'' = 0.018$  cm $^{-1}$ . The agreement is good. The height of the predissoc. in the upper level  $A''$  was calculated and ascribed to the dissociation state  $^1P(\text{oxygen}) + ^1D(\text{carbon})$  of the mol., all other possibilities being excluded. This makes the value of 8.41 v. for the dissociation energy of CO perfectly certain. H. A. Smith

Dissociation energy of carbon monoxide. D. Coster and F. Brons. *Proc. Acad. Sci. Amsterdam* 38, 961-4 (1935).—On analyzing the rotational structure of the 4th

pos. group of the CO bands, a sudden disappearance of lines which combine with the vibrational level  $v' = 10$  of the upper  $A''$  level, which is 9.67 v. above the  $X'^2\Sigma^+(v = 0, J = 0)$  state of the mol., was discovered. The difference  $11.08 - 9.67 = 1.41$  v. gives the energy of dissociation of the Angstrom bands. This is exactly the difference  $15.5D$  of the C-atom. This agreement proves the level diagram developed to be correct, and gives  $8.41$  v. =  $193.9$  kg.-cal. for the dissociation energy of CO. With this value the heat of sublimation of solid C is calculated to be 107.6 kg.-cal.

C. E. P. Jeffreys  
Dissociation energy of carbon monoxide. F. Brons. *Nature* 136, 766 (1935).—The structure of the fourth positive group of bands in the CO spectrum affords, in connection with other data, a certain explanation of the observed predissociations in the  $A''$  and  $B''\Sigma$  levels, and confirm the previous suggestion of Coster and Brons (C A 28, 6063) that the dissociation energy of CO is 8.41 v.

C. D. West  
SiBr bands. E. Miescher. *Helv. Phys. Acta* 8, 587-8 (1935).—A photographic reproduction of the band structure of SiBr is given with the various bands graphically identified, with tables of frequencies and intensities.

G. M. Evans  
Ultraviolet fluorescence spectra of iodine vapor. Influence of foreign gases. F. Duschinsky and Peter Pringsheim. *Physica* 2, 923-32 (1935); cf. C. A. 29, 7184, 30, 3807.—The intensive bands in the region of 3400 Å found in the fluorescence of  $I_2$  with the admixt. of  $N_2$  were further resolved and are tentatively classified. The influence of the noble gases in the place of  $N_2$  was investigated. Helium gives a spectrum in the same position but much changed.  $N_2$  also gives this spectrum but at higher temps. In the presence of traces of Hg the bands can be excited as sensitized fluorescence; HgI bands are also found. Energy relations are discussed. In an appendix the HgI band at 4400 Å is described. H. G.

The absorption spectrum of solid oxygen. H. A. Prinkothko and M. Ruheman. *Physik Z. Sowjetunion* 8, 294-9 (1935), cf. C. A. 29, 6141.—A study of the absorption spectrum of solid  $\alpha$ -O between 3000 and 4000 Å. revealed the existence of a series of 5 bands similar to the 2 series in the visible region. Preliminary results were obtained concerning the band system below 2000 Å. Morris Muskat

Temperature of atmospheric ozone. J. Devaux. *Compt rend* 201, 1500-1 (1935), cf. C. A. 26, 3727.—With a similar arrangement the infrared spectrum (4-15  $\mu$ ) of the atm. was automatically recorded at hourly intervals at the Pic-du-Midi.  $O_3$  shows a strong band at about 10  $\mu$ . The temp. of the  $O_3$  is much below 0°, and neither it, nor the height of the  $O_3$  band, nor the amt. of  $O_3$  shows any marked variation. C. A. Silberrad

Absorption spectra of nitrophenylhydrazines. A. Killen Macbeth and J. R. Price. *J. Chem. Soc.* 1935, 1503-7.—Data are given for  $o$ -,  $m$ - and  $p$ - $O_2NCH_2NHNH_2$ , 2,4-( $O_2N$ ) $_2C_6H_3NHNH_2$ , 3,4,6-Me( $O_2N$ ) $_3C_6H_2NHNH_2$  and 2,4-( $O_2N$ ) $_2C_6H_3NHNH_2$ . The values of the  $\lambda_{max}$  of the band of shorter wave length common to all the curves of the  $O_2NCH_2NHNH_2$  in aq. EtOH are nearly all the same; it seems reasonable to attribute this absorption to the electronic transition of the C-C system of the nucleus, the small differences being due to vibrational effects. The other  $\lambda_{max}$  recorded for the  $o$ -compd. differs markedly in location from the corresponding bands shown by the  $m$ - and  $p$ -isomers. This is true for both EtOH and aq. EtOH solns. of the  $O_2NCH_2NHNH_2$ , and is in agreement with the spectra of the  $O_2NCH_2NHNH_2$  in EtOH and of the Me( $O_2N$ ) $_3C_6H_2NHNH_2$  in ether EtOH or  $H_2O$ . Similar differences in the intensities of these bands of the  $m$ - and  $p$ -compds. are observed in all cases. The results can be accounted for on the assumption that the long-wave absorption is due to electronic excitation in the  $NO_2$  group influenced by the nuclear  $NH_2$  or  $NHNH_2$  group. The view that the maxima of longer wave lengths in the nitroamines and nitrophenylhydrazines are due to an electron controlled by the N atom of the  $NO_2$  group is supported by the spectra of 2,4-( $O_2N$ ) $_2C_6H_3NHNH_2$  (I),

3,4,6-Me(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHNH<sub>2</sub> (II) and 2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>2</sub> (III). Both the *o*- and *p*-nitrohydrazine effects persist in I tOH and aq I tOH solns, the former being characterized by the inflection at 392 mμ (I tOH), 403 mμ (H<sub>2</sub>O) and 395 mμ (I tOH), 410 mμ (H<sub>2</sub>O), and the latter by the bands at 372 mμ (I tOH), 376 mμ (H<sub>2</sub>O) and 350 mμ (I tOH), 365 mμ (H<sub>2</sub>O) in I and II, resp. The positions of the maxima do not coincide exactly with those of the simple *o*- and *p*-O<sub>2</sub>N<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHNH<sub>2</sub>, but this is to be expected as being the effect of introducing an addnl NO<sub>2</sub> group into the nucleus. Although the curves of the O<sub>2</sub>N<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHNH<sub>2</sub> in I tOH and H<sub>2</sub>O are similar in character, the positions of the maxima are appreciably altered. The displacements are in either direction, and although a strict comparison cannot be made, aq I tOH solns having been used instead of H<sub>2</sub>O for the hydrazines owing to solubilities, yet the changes in frequency were, with 2 exceptions, generally of the same order as those recorded by the previous workers. C. J. West

The dispersion of carbon disulfide and carbon tetrachloride in the infrared A. H. Pfund. *J. Optical Soc. Am.* 25, 351-4 (1935).—The following values of  $n_D^{20}$  were detd for various wave lengths in an app consisting of 2 mirror spectrometers in series with a hollow prism with rock-salt sides (angle 15°23'24") for the liquids CS<sub>2</sub>: 0.5893,  $n_D^{20}$  1.6275, 0.740, 1.6085, 0.910, 1.5097, 1.32, 1.5904, 1.69, 1.5862, 2.41, 1.5815, 3.21, 1.5754, 3.92, 1.5666, 4.22, 1.5575, 4.67, 1.5540, 4.84, 1.5476, 5.14, 1.5411; 6.47, 1.5112, 5.80, 1.4812, 7.39, 1.7247, 7.62, 1.6975, 7.80, 1.6849, 8.06, 1.6711, 8.74, 1.6486, 9.32, 1.6381; 9.90, 1.6316, 10.45, 1.6277, 11.06, 1.6244, CCl<sub>4</sub>: 0.5893, 1.4007, 0.740, 1.4541, 0.91, 1.4517, 1.32, 1.4489, 2.41, 1.4466, 3.92, 1.4434, 5.14, 1.4387, 5.47, 1.4374, 5.90, 1.4351; 6.11, 1.4356, 6.22, 1.4361, 6.41, 1.4360, 6.60, 1.4370, 6.80, 1.4315, 7.20, 1.4295, 7.46, 1.4274; 7.80, 1.4254, 7.98, 1.4248, 8.06, 1.4223, 8.45, 1.4183; 8.80, 1.4219, 9.02, 1.4090, 9.21, 1.4036. Anomalous dispersion occurs in each case at wave lengths corresponding to the absorption bands of the compd. By use of a specially cut cylindrical quartz cell to obtain very thin layers, the transmission of pure CS<sub>2</sub> was measured between 1949 and 5401 Å. A strong absorption band at 1965 and a weak band at 3276 were found. All the absorption bands will have to be considered in any calcn of dispersion. Janet F. Austin

The luminescence of frozen solutions of certain dyes. Frances G. Wick and Charlotte G. Throop. *J. Optical Soc. Am.* 25, 368-74 (1935).—The intensity of luminescence of 0.0004 M solns of uranine (I), rhodamine B-extra (II) and naphthasulfam (III) in H<sub>2</sub>O (1), Me<sub>2</sub>CO (2), I tOH (3) and glycerol (4) at temps from 50° to the temp of liquid air were measured with a special app and an optical pyrometer with various color filters in the eyepiece. All solns. in (2), (3) and (4) and III in (1) showed luminescence at all temps. The luminescence of II in (1) ceased at 0° and reappeared in a different color with max intensity at -125°. I showed no luminescence below 0°. I frozen solns. of each in (2), (3) and (4) showed phosphorescence. The 2 regions of excitation and the approx limits of the fluorescence bands were detd for each soln. Marked changes in the brightness of luminescence-temp curves occurred at temps at which the phys condition of the solvnt changes, such as, at the f p, change to a viscous liquid, and from a viscous liquid to a glass. Other points of inflection may correspond to changes in states of assocn. of the solvents. Janet E. Austin

Absorption spectrum of nitrogen peroxide. Maurice Lambrey and Jean Corbière. *Compt. rend.* 201, 1334-5 (1935); cf. C. A. 25, 458.—The absorption coeffs.,  $\beta$ , of N<sub>2</sub>O<sub>4</sub> and NO<sub>2</sub> have been detd. for  $\lambda$  3200-4105. Within the limits of observation (26-96°, and 580-730 mμ) there was no variation for temp. or pressure. For N<sub>2</sub>O<sub>4</sub>  $\beta \times 10^3$  rose from 0.70 for 3200 Å to a max. of 1.59 for 3400 Å. and then fell to 0.53 for 4105 Å.; for NO<sub>2</sub> the rise was from 0.50 for 3200 Å. to 0.52 for 3800 Å. and then a fall to 0.78 for 4105 Å. The accuracy is about  $\pm 10\%$ . C. A. Suberrad

First discharge spectrum across nitrogen peroxide.

Léon Grillet and Michel Duffieux. *Compt. rend.* 201, 1338-40 (1935).—The spectrum (3350-10,200 Å) was obtained by passing a rectified discharge through a very short tube through which a rapid current of NO<sub>2</sub> was passed. In addn to spectral O I, N I, the 2nd pos. group of N<sub>2</sub> with traces of the 1st, and the  $\beta$  bands of NO, there were observed a vibration-rotation band (nul line 5996, width 21.4 Å, corresponding to a moment of inertia of the mol of  $17 \times 10^{-40}$ ), and several bands and lines (10 and 9, resp., measurable with the app used) of unknown origin. C. A. Suberrad

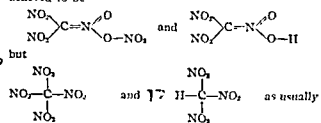
Absorption spectra of tautomeric selenazoles. R. F. Hunter. *Nature* 136, 1030 (1935).—Qual results for the shift of the absorption toward shorter wave lengths with tautomeric change is given. R. E. DeRight

Infrared absorption spectra of halogen derivatives of nuclear hydrocarbons. Jean Lecomte. *Compt. rend.* 201, 1340-2 (1935) cf. C. A. 28, 1926.—While for halogen substituted aliphatic derivs the vibration C-X corresponds to a definite absorption band, approx. the same so long as X is the same, with the halogen in the nucleus of a ring compd the vibrations and bands depend also on the nucleus and position of the halogen. Thus for the F, Cl, Br and I derivs of C<sub>6</sub>H<sub>5</sub> the corresponding bands are at 802, 705, 677 and 659 cm<sup>-1</sup>, but for the *o*-derivs of toluene at 749, 680, 655 and 644, resp., with similar differences for the *m* and *p* isomers, and also for other bands and those of X<sub>2</sub>, X<sub>3</sub> and X<sub>4</sub> derivs. The results are confirmed by Raman spectra, but in some cases modes of vibration indicated by one method are not by the other. Similar results are obtained with cyclohexane and C<sub>6</sub>H<sub>5</sub> derivs. C. A. Suberrad

Infrared spectra of amino acids and polypeptides. Irwin Heintz. *Compt. rend.* 201, 1478-80 (1935).—The infrared spectra ( $\lambda$  0.8-8  $\mu$ ) are plotted for glycocoll, *dl*-alanine, *d*-arginine, *l*-cystine, cysteine, *l* leucine, *d*-glutamic acid, *dl*-proline, tyrosine, *l*-histidine, phenylalanine, 3 peptides and albumose from casein. The  $\lambda$  of the absorption bands agree with those deduced from Vles's formula  $\lambda \rho K = 21.2$  (C. A. 29, 2440). C. A. Suberrad

Influence of intermolecular forces on the Raman effect of liquids. Wolfgang Buchheim. *Physik Z.* 36, 694-711 (1935).—The influence of homogeneous elec fields on Raman scattering is theoretically evaluated. Conclusions: Raman lines should broaden with increasing field strength; this broadening should be most marked for vibrations connected with a 2nd-order change of the dipole moment of a mol; the lines should undergo a shifting of the center of gravity by the field smaller than the broadening; lines forbidden by mol symmetry may appear. These effects should be sufficiently large to be observed from fields arising in dipole liquids or electrolytes. Line broadening by infrared vibrations is theoretically of the same order of magnitude as the electrostatic effect. The change in the order of magnitude of the intensity distribution in the Raman spectrum on mixing scattering compds with others is expected to be a few %. This conclusion is verified in expts. with benzene, CCl<sub>4</sub> and CHCl<sub>3</sub>. Allen S. Smith

The Raman effect of nitromethanes. Louis Médard. *J. chim phys* 32, 136-41 (1935).—The last sentence of the abstr in C. A. 29, 5350<sup>1</sup> should read "The resp structural formulas of C(NO<sub>2</sub>)<sub>3</sub> and CH(NO<sub>2</sub>)<sub>3</sub> are not believed to be



written." Harry D. Weiser  
Ultraviolet spectrograph of large aperture. Albert Arnulf and Bernard Lyot. *Compt. rend.* 201, 1490-2

(1935)—The essential point of the spectrograph is the replacement of the object glass by a spherical mirror with an arrangement (preferably of 2 lenses) to correct spherical aberration. An instrument in quartz on these lines, with aperture  $f/1$ , mirror of radius 10 cm., and corrected for  $\lambda$  2900, works well for  $\lambda\lambda$  2900–4000, and with 5 hrs. exposure showed  $\lambda$  3022 in the night sky spectrum.

C. A. Silbernad

Separation of the mercury isotopes by a chemical method. K. Zuber. *Nature* 136, 790 (1935), cf. *C. A.* 29, 6140.—When a mixt. of Hg vapor and  $O_2$  is irradiated with the Hg line 2537, the Hg is oxidized, presumably after excitation, according to the scheme  $Hg^* + O_2 = HgO + O$ . Following a proposal of Mrozowski (*C. A.* 27, 899), Z. irradiated such mixts with radiation which had passed through a filter transmitting only the hyperfine-structure components capable of exciting the isotopes 200 and 202. On examg. the isotopic compns. of the resulting Hg and HgO by his method of absorption of single hyperfine-structure components, Z. found samples in which the pressure ratio  $P_{202}/P_{200}$  and  $P_{202}/P_{200}$  differed by a factor of 4 from that of the starting material. The sepn., although not complete, is favored by the presence of  $N_2$ .

C. D. West

Continuous spectrum of the night sky and diffusion of light in space. Jean Dufay. *Compt. rend.* 201, 1323–4 (1935), cf. *C. A.* 29, 5016.—The theory that part of the light of the night sky is due to diffusion of starlight by particles small compared with the wave length is discussed, and a formula for testing is deduced. *C. A.* S.

The photochemical decomposition of nitric oxide. Paul J. Flory and Herck L. Johnston. *J. Am. Chem. Soc.* 57, 2041–51 (1935).—Products of the decompn. are N and O. The latter is quantitatively removed by compression in the McLeod gage with production of  $HgNO_2$ , which liberates NO ( $k = 1.5 \times 10^{-4}$  hr.<sup>-1</sup>). This rapid removal of O makes pressure measurements more sensitive. In the pressure range 0.02–7 mm. Hg decompn. is directly proportional to the rate of light absorption and to the pressure. The absorption obeys Beer's Law (extinction coeff. =  $2.9 \times 10^{-4}$  per mm., per mm. of pressure). Primary disocn. is the 1st step, and the concn. of N has no effect. The use of filters indicates the Hg line  $\lambda$  1832 which is absorbed by the ( $1,0'$ ) band of the  $\gamma$  system as the effective radiation. Results with various metal sparks support this. The primary process is predissocn. occurring in the banded region, which agrees with the emission spectrum. Secondary processes are ruled out. The same mechanism may be applied to MacDonald's data at higher pressures (*C. A.* 22, 2112), but predissocn. from the  $\beta$  bands is the primary step. R. E. DeRight

Photobromination of tetrachloroethylene and of chloroform, with special reference to the effects of oxygen. John Willard and Farrington Daniels. *J. Am. Chem. Soc.* 57, 2340–5 (1935).—The rate of photobromination of liquid  $C_2Cl_4$  with light of wave length 4360 Å. is increased by small amts. of O. When 1 atm. of O is used, the photobromination is almost completely inhibited, probably because of competing Br-sensitized oxidation. The products of bromination inhibit the reaction. The temp. coeff. of the reaction is 1.12, the concn. of  $C_2Cl_4$  is not important. The dark reaction is slow. The quantum yield varies from 24 to 0.009 mols. Br disappearing per quantum, with values of 1 to 5 most frequent. A mechanism for this reaction is suggested which involves the free radical  $C_2Cl_4Br$ . It is assumed that the life period of this radical is much shorter at higher temps. and that the radical is stabilized by O. The photobromination of  $CHCl_3$  does not occur at 25° with light of wave length 2650 Å. in the absence of O but a reaction occurs in the presence of small amts. of O. B. J. Rosenbaum

Reaction between ethylene and chlorine in the presence of chlorine acceptors. The photochlorination of ethylene. T. D. Stewart and Bernhard Weidenbaum. *J. Am. Chem. Soc.* 57, 2036–40 (1935).—The rate of the initial stage of the photochlorination of  $C_2H_4$  in the gas phase is proportional to the light intensity for a wave length of approx. 4360 Å. The quantum yield is probably large

1 Possible mechanisms are suggested. If H is present, none of it reacts in the dark and very little reacts under illumination. In  $C_2H_4Cl_2$  and  $C_2H_5Cl$  soln., the reaction is rapid. Of the Cl used up in the  $C_2H_4Cl_2$  soln. less than 10% is involved in a substitution reaction. In  $C_2H_5Cl$  between 37 and 73% of the Cl substitutes, and this fraction increases with the  $C_2H_4/Cl$  ratio. An explanation for these facts is discussed. E. J. Rosenbaum

2 The effect of wave length on the iodine sensitized decomposition of ethylene iodide in carbon tetrachloride solutions. Roscoe G. Dickinson and Nelson P. Nies. *J. Am. Chem. Soc.* 57, 2382–6 (1935).—Since the rate of decompn. is proportional to  $I^{1/2}$ , absorption coeffs. were detd. and the distribution of the intensity in the soln. was calcd. The relative values of the rate consts. were 1.0, 0.91, 0.867 at wave lengths 4358, 5461 and 5770 Å., resp. This difference is less than that found by Schumacher and Steger (*C. A.* 25, 3917) and is due to differences in absorption coeffs. The abs. values for the rate consts. at 76.6° in einsteins  $^{-1/2} \times cm^2 \times sec^{-1/2}$  were 0.42, 0.39 and 0.37, resp. Thus, light corresponding to continuous absorption of I in the gas phase is slightly more effective than that in the discontinuous.

R. E. DeRight

Photochemical investigations. II. The photochemical decomposition of ethylene iodide in solutions of carbon tetrachloride. Robert E. DeRight and Edwin O. Wig. *J. Am. Chem. Soc.* 57, 2411–15 (1935); cf. *C. A.* 29, 7899.—A mechanism and rate equation involving primary disocn. into  $C_2H_2I$  and at. I is suggested. Absorption begins at 360 m $\mu$ . The apparent quantum yield was 0.76 for the Hg lines at 303 and 313 m $\mu$ . This includes the photochem. and subsequent thermal reactions. The rate reaches a definite max. after a certain amt. of decompn. 5 Increased initial concns. of  $C_2H_2I$  increase the rate slightly, ethylene has no effect and I decreases the rate. The results substantiate the theory of Franck and Rabinowitch (*C. A.* 28, 2269) for reactions in soln.

R. E. DeRight

Spectrum, fluorescence and photochemical decomposition of acetaldehyde. H. W. Thompson and J. W. Linnett. *J. Chem. Soc.* 1935, 1452–9.—Two regions of absorption for acetaldehyde in the range 2000–7000 Å. were found. 6 (a) from about 3800 to about 2900 Å. and (b) from 2320 Å. to shorter wave lengths. A preliminary analysis of the band system for (a) has been made. An attempt to excite fluorescence by irradiation with light corresponding to this region of absorption was unsuccessful. The quantum efficiencies of the photochem. decompn. and polymerization have been studied with light of 3665 and 3135 Å. The efficiencies of both reactions rise with increasing wave length; for the decompn. it is of the order of  $10^{-4}$  at 3665 Å. and 0.15 at 3135, for the polymerization, 0.39 at 3665 Å. and 2.3 at 3135. The data are discussed.

William E. Vaughan

Primary photochemical reactions. VII. Photochemical decomposition of isovaleraldehyde and dipropyl ketone. C. H. Bamford and R. G. W. Norrish. *J. Chem. Soc.* 1935, 1504–11, cf. *C. A.* 29, 5022.—The decompn. of isovaleraldehyde and  $Pr_2CO$  by Hg-vapor irradiation follows N's previous findings on  $MeEtCO$  (*C. A.* 28, 5758). The proportions of the 2 types of chance in isovaleraldehyde are: type 1 forming CO and isobutane, 47%; type 2 forming propylene and  $CH_3CHO$ , 53%. With  $Pr_2CO$  the proportions are: type 1 forming CO and hexane 37%; type 2 forming  $C_3H_6$  and  $MePrCO$ , 63%. Elliott J. Roberts

Photochemical decomposition of nitrophenols. Jean Molnar. *Compt. rend.* 201, 1482–4 (1935), cf. *C. A.* 29, 5725.—An alk. soln. of picric acid ( $pK$  greater than 13) exposed to light develops a red-brown tint, slowly in diffused light, rapidly in sunlight and still more rapidly close to a Hg lamp, followed in the last case by a violet tint and then decoloration. Chem. analysis shows the formation of picramic and isopurpuric acids,  $HNO_2$ ,  $NH_3$  and  $HCN$ . 2,5-Dinitrophenol behaves similarly. alk. solns. of other nitro- and dinitro-phenols darken in color with formation of an amino group. There is no evi-

dence that the photolysis is selective as regards  $\lambda$ .

C. A. Silberrad

Fluorescence of some pure substances (alcohols). Étienne Canals, Pierre Peyrot and Roger Noël. *Compt. rend.* 201, 1488-9(1935); cf. *C. A.* 29, 6144<sup>1</sup>—The depolarization factor, 100  $\rho$ , and the value of  $2f/(I + i)$  were detd. for several alcohols. The factors of the normal series (C 1-4) are 6.0, 6.7, 6.9 and 8.6, resp., of the iso series (C 3-5), 5.0, 6.6 and 8.0, and of  $\text{PhCH}_2\text{OH}$  56.8. All are slightly fluorescent,  $2f/(I + i)$  for the aliphatic alcohols being 0.03-0.06, and for  $\text{PhCH}_2\text{OH}$  0.18.

C. A. Silberrad

Determination of ionization by ultraviolet spectrophotometry, its validity and its application to the measurement of the strength of very weak bases. Leo A. Flexser, Louis P. Hammett and Andrew Dingwall. *J. Am. Chem. Soc.* 57, 2103-15(1935)—The ionization constants of weak org. electrolytes were detd. from intensity changes in the ultraviolet absorption spectra in going from the acid to the basic forms of these electrolytes. The method was tested with dil. aq. solns. of  $\text{BzOH}$ , 2,4-dinitrophenol and aniline and was found satisfactory. A method for considering the medium effect with very weak electrolytes is presented. The effect on indicators of a change of medium is discussed. The base strengths of acetophenone,  $\text{BzOH}$ , anthraquinone and phenylacetic acid were detd. The method is not suitable for *p*-nitrobenzoic acid.

E. J. Rosenbaum

Photochemical reactions connected with the quenching of fluorescence of dyestuffs by ferrous ions in solution. Joseph Weiss. *Nature* 136, 794-5(1935), cf. *C. A.* 29, 3901<sup>1</sup>—The photochem. oxidation of ferrous ion in soln. contg.  $\text{H}_2\text{SO}_4$  in the absence of O takes place in ultraviolet light with evolution of  $\text{H}_2$  or in visible light if certain dye mols. are present, with reduction of the latter to the leuco form. The mechanism of these reactions is discussed and implications in connection with *biol. problems* are pointed out.

C. D. West

Phosphorescence and heterogeneity in crystals. Maurice Curie. *Compt. rend.* 201, 1352-3(1935)—Phosphorescence is associated with the presence of impurities, which produce abnormal levels of heterogeneity (and hence of energy) in their neighborhood. The electron responsible for the phosphorescence is driven by light to the level of appropriate energy and on its return therefrom produces the phosphorescence. This theory makes more definite C.'s view attributing phosphorescence of long duration

<sup>1</sup> (Lenard's  $d$  band) to collisions of the 2nd order. (Cf. *C. A.* 26, 3993, Tomaschek, *C. A.* 23, 5109.)

C. A. Silberrad

Chemical action caused by neutrons and gamma rays and the effects of these agents on colloids. F. L. Hopwood and J. T. Phillips. *Nature* 136, 1026(1935)—The effect of  $\gamma$ -rays on chem. reactions is enhanced by neutrons. The stability of neg. colloids was increased and of pos. colloids decreased by a source of neutrons. The effect of pure  $\gamma$ -rays was similar but only half as effective despite the fact that the sources produced  $10^8$  quanta of  $\gamma$ -rays per neutron. Anomalous results are obtained in the presence of foreign electrolytes.

R. E. DeRight

Heterogeneous reaction kinetics. Effect of light exposure on the kinetics of thermal decomposition of silver oxalate. Arthur F. Benton and Geo. L. Cunningham. *J. Am. Chem. Soc.* 57, 2227-34(1935)—The rate of the thermal decompn. of  $\text{Ag}_2\text{C}_2\text{O}_4$  was studied by measuring the amt. of  $\text{CO}_2$  formed. The rate at  $100^\circ$  is not greatly affected by drying or the presence of  $\text{CO}_2$  or free Ag but is greatly increased by previous exposure to light of wave length less than 520  $\text{m}\mu$ . Contact with O during exposure retards the initial decompn. rate. The effect of exposure to light is less at higher decompn. temps. A quantum theory is developed which is in fair agreement with the data. Light of wave length 370  $\text{m}\mu$  produces about 0.1 mol. of  $\text{CO}_2$  per quantum absorbed. The activation energy of nucleation is about 64  $\text{kg.-cal}$  while that for the growth of the nuclei is 8.5  $\text{kg.-cal}$ .

E. J. Rosenbaum

Relations between electrochemical constants, infrared spectra and reactivities. Fred Vles. *Compt. rend.* 201, 1475-7(1935), cf. *C. A.* 29, 7798<sup>1</sup>—The atom or group responsible for the absorption band giving the relation  $\lambda\rho K = 21.2$  is identified in several cases, thus in  $\text{Ph-COOH}$  it is the CO group, in  $\text{PhNH}_2$ ,  $\text{N}^{\text{III}}$ . In anthranilic acid the acid function  $\text{p}K_a = 4.95$  and the basic  $\text{p}K_b = 2.10$ , correspond to  $\lambda = 4.28$  and  $10.1 \mu$ , resp., while other values correspond to CO at 4.3 and  $\text{N}^{\text{III}}$  at  $10.0 \mu$ . Other examples are drawn from the values of  $21.2/\text{p}K (= \lambda)$  for the half decompn. of urea by urease (indicating the presence of  $\text{N}^{\text{III}}$  and  $\text{N}^{\text{V}}$  and so supporting Werner's cyclic formula), and sucrose by saccharase (indicating attack of the aldehyde group).

C. A. Silberrad

Perturbation theory. I. Perturbation calcn. in the Thomas-Fermi theory without exchange (Gombás). 2. Raman effect and org. chemistry (Gredy) 10.

## 4—ELECTROCHEMISTRY

COLIN G. PINK

Progress in design and application of electric furnaces. H. Knight. *Metallurgia* 13, 53-6(1935)—Melting and heat-treating furnaces recently installed in England are described.

J. L. Gregg

The power factor in spark-type high-frequency induction furnaces. R. Dufour. *Recherches et inventions* 16, 457-79(1935)—The elec. characteristics are investigated and discussed.

Leopold Pessel

Nitrogen in electric steel. N. Chuiko. *Domes* 1935, No. 8, 27-39—Absorption of N in steel was studied in connection with a no. of elec. steel furnaces. The atm. in the furnaces was found to contain a max. of 0.436% by vol. of oxides of N. Depending on length of melting operation a max. of 0.03-0.04% N was found. The rate of N absorption increased during the carburization period, and also with increase in voltage. The type of elec. arc, whether stationary or rotating, had no effect on the rate of absorption of N.

S. L. Madorsky

The preparation of artificial graphite electrodes. N. P. Bozhlo. *J. Chem. Ind. (Moscow)* 12, 1161-7(1935)—Various kinds of coke and anthracite were ground, mixed with pitch, shaped into electrodes and graphitized. Electrodes of lowest elec. resistance were obtained by heating them to  $500^\circ$  in 4 hrs., then to  $1200^\circ$  in 3.5 hrs. and keeping them at  $1200^\circ$  for 3 hrs. The temp. was then

finally raised very slowly to  $2300-2400^\circ$ . The greatest changes occurred at  $1200^\circ$  and at  $2000-2300^\circ$ . The resistance and ash content of the electrodes were greatly reduced by this treatment. Anthracite gave the best electrodes, although some kinds were better than others. When anthracite mixts were used, the properties of the resulting electrodes were the sum of the properties of the individual components. Fine-ground materials gave better results than coarser ones. A high ash content in the electrodes had a harmful effect. Repeated graphitization was of no value.

H. M. Leicester

Metallization of electrodes for electric furnaces. G. F. Morenko. *Domes* 1935, No. 8, 39-45; cf. *C. A.* 29, 4267<sup>1</sup>—Graphite electrodes coated with Cu and Al were compared with non-coated electrodes in actual operation of smelting steel in an elec. furnace. Cu stood up better than Al under these conditions, but neither of these was as good as Fe or steel coatings.

S. L. Madorsky

Electrolytic cell for aluminum production. Evaluation of its design. A new automatic closed cell. Ferrand. *Procs. verb. séances soc. ing. civils France* 1935, No. 6, 130-8(March 22); *Chimie & industrie* 34, 848—To avoid having to open the cell at frequent intervals, it is necessary to: (1) regulate the anode feed automatically in order to distribute the c. d. evenly, (2) feed the  $\text{Al}_2\text{O}_3$  under the



roof continuously and uniformly, (3) prevent anode effect. By oscillation of the anodes a slow stirring of the cryolite bath is produced and this, with the continuous feed of  $Al_2O_3$ , prevents the anodes from burning. The new cell shows a reduction of 12500 kw-hr per ton Al. A. P. C.

The manufacture, properties and uses of refined aluminum. R. Gadeau. *Chimie & Industrie* 34, 1021-6 (1935).—Though Hoopes succeeded (cf. Frary, C. A. 19, 1665) in producing refined Al of 99.9% purity by a process derived from that of A. G. Betts (Fr. pat. 304,521, March 23, 1906) and even attained a purity of 99.99%, commercial application of the process has so far been found impossible. The Cie de Produits Chimiques & Electro-metallurgiques d'Alsais, Froges et Camargue has modified the process. It is producing regularly Al of 99.99% purity and even 99.9980%. The anode consists of a Cu-Al alloy containing about 33% Cu, the bottom of the cell is of C and the walls are lined with ordinary MgO bricks (containing 90% MgO, besides  $Fe_2O_3$  and  $SiO_2$ ); the electrolyte consists of  $AlF_3$ , 23, NaF 17 and  $BaCl_2$  60%. Electrolysis is carried out at about 750°, at 6 v. and 10,000 amp (or c d. of 40 amp/sq dm), anodic current efficiency, 100%, cathodic current efficiency, 97%. There is some volatilization of Al as  $AlCl_3$ , so that the composition of the fluoride portion of the bath approaches  $AlF_3$ -3NaF. When this composition is reached, the refined Al may contain up to 3% Ba. Accordingly it is then necessary to add a little  $AlF_3$  or NaF to the bath, when the Ba in the refined Al passes back into the electrolyte and the phenomenon does not recur. Highly refined Al is appreciably softer than ordinary Al, the difference between the conditions of the cold-hardened and annealed metal decreases as the degree of purity increases, highly refined Al (99.99%) exhibits an exceptionally high resistance to the action of acids. Possible applications are briefly outlined. Also in *Rev. met.* 32, 503-8 (1935). A. P. C.

Direct electrolytic manufacture of aluminum-magnesium alloys. I-IV. Ichitaro Namari and Tashio Ishino. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 583-91 (1935).—The direct electrolytic production of Al-Mg alloys by deposition of Mg on Al cathodes was investigated. The effects of NaCl or KCl added to the  $MgCl_2$  electrolyte were the same, both lowering the temp. at which electrolysis could be carried out. The results were not affected by change in c d. and temp. of bath above 600°. An almost constant current efficiency of 94% was obtained. With  $MgCl_2$  only, as electrolyte, the best results were obtained and the mean current efficiency was 95%, the only disadvantage being that electrolysis had to be performed at temps. about 700°. The alloys produced corroded in the air more readily if made in the KCl or NaCl-diluted bath, if  $MgCl_2$  alone was used, the alloys were very resistant. However, the most suitable electrolyte for practical operation was  $MgCl_2$  + 5-10% NaCl, temp. of electrolysis, about 670°. The time of electrolysis did not affect the current efficiency very much. The composition of the final alloys varied from the wt. increase of the cathode agreed with the analytical results. Fe and Si are possible impurities. Metallographic exams. showed that the produced alloys were of quite uniform composition and compact structure. Karl Kammermeyer.

Electrolytic production of boron and its alloys. J. L. Andreux. *Rev. met.* 32, 487-93 (1935).—In the electrolytic production of B, a graphite crucible was used as anode and a water-cooled Fe rod as cathode. The fused bath was composed of  $B_2O_3$ , to which were added oxides and fluorides of metals (preferably Mg) which are able to reduce B. The bath is electrolyzed at 1100°. Mg concentrates around the cathode and reacts with the bath. Mg borates more basic and less fusible than the bath and metallic B are formed and deposit on the cathode. The deposit is subsequently treated with HCl, and a metal containing about 85% B is obtained. Electrolyzing borates in the presence of Ca, Ba, Sr, Ce and Mg halides results in the production of corresponding borides. Many alloys may be prepared by using cathodes made of the metal in question. The bath is heated to a temp. at which the cathodes are still solid but at which the eutectics with B melt and run down

For example Ni eutectic m. 900° and Fe-B eutectic m. 1174°. The composition of the alloys or borides so produced depends, within certain limits, on the temp. and duration of electrolysis. J. D. Gat.

Decomposition of barium sulfate by chlorine. A. Ya. Zvorukin. *Chimie & Industrie* 34, 760-4 (1935).—Experiments were carried out on the treatment of finely pulverized  $BaSO_4$  by  $Cl_2$  in a furnace heated electrically to 850-1030°. The proportion of  $BaSO_4$  converted into  $BaCl_2$  increases, under the conditions of the experiments, with temp., with the velocity of the  $Cl_2$  current and with the fineness of  $BaSO_4$ , grinding. The unreacted  $Cl_2$  may be reintroduced into the cycle. The presence of NaCl increases the yield of  $BaCl_2$ , the presence of  $SiO_2$  or silicates decreases it. By combining addition of 5% NaCl and reutilization of the unreacted  $Cl_2$ , a yield up to 92.8%  $BaCl_2$  can be obtained. A considerable amount of decomposition can be obtained with dilute  $Cl_2$ , even 1 part  $Cl_2$  dilute with 6.5 parts of air will give a satisfactory yield of  $BaCl_2$ . The process seems capable of commercial application. A. Papineau-Couture.

The behavior of oxygen electrodes in alkali carbonate melts. Emil Baur and Roland Brunner. *Z. Elektrochem.* 41, 794-6 (1935).—An electrode consisting of a thermoelement and tube of Pythagoras material (Al silicate) with a ball of 2.2 g. fine Pt wire at its lower end was placed in a graphite crucible filled with a mixture of  $K_2CO_3$ ,  $Na_2CO_3$ , NaCl and borax. When the electrode is fed with  $O_2$  at 500-600°, the voltage is insufficient and strong polarization occurs, likewise with balls of Ag wire, of silvered Cu or steel wool. But if a mixture of  $O_2$  or of air with  $CO_2$  is used, the current-potential curves immediately show the influence. The reason for the improvement of the electrode with the addition of  $CO_2$  is that oxides (NaO) form on the cathode which incrust the electrode.  $CO_2$  must dissolve these. M. McMahon.

Polarization of electrodes during the decomposition of metals from solutions of complex cyanides. O. A. Esin and A. Matanzev. *Z. physik. Chem.* A174, 344-94 (1935).—Analysis of data by S. Glasstone (cf. C. A. 24, 4208) shows that the concentration polarization conditioned by accumulation of CN on the cathode in most cases indicates a relation between polarization and current density which is analogous to the equation for H overvoltage. This permits the assumption of chem. polarization in the separation of Hg from cyanide solutions, which polarization is conditioned by the overvoltage through retarded discharge in the sense of Volmer's theory (C. A. 28, 4164). The data on the deposition of Ag and Cd give no consistent indications. In Cu-plating no chem. polarization appears, but a depolarization is suspected. R. H. Baechler.

Electrodeposition of zinc and cadmium on aluminum and aluminum alloys. B. K. Braund and H. Sutton. *Trans. Faraday Soc.* 31, 1595-1611 (1935).—The effect of plating Al and Al alloys with Zn and Cd as a protection against corrosion under marine conditions was studied. None of the previous plating methods was found satisfactory. The most promising cleaning method was found to be anodic cleaning in 10%  $NH_4OH$  solution, but electrodeposits were usually satisfactory only on part of the article. In a bath prepared by dissolving Zn in 7% NaOH some Al was dissolved and a coating of Zn deposited which acted as a good undercoating for electrolytic Zn deposition. In some cases the zincate bath gave dark noncoherent deposits. Addition of a small amount of  $Na_2SO_4$  to the bath improved the results. Surfaces of Al-rich casting alloys can be prepped and plated easily by this method, but Al and duralumin sheets require more care. Lower pH in  $ZnSO_4$  baths was possible when the Al was prepped in this way than with sand blasted material.  $ZnCl_2$  baths are also satisfactory at pH 4.0. Gas pitting was rarer than with the  $ZnSO_4$  bath, and the deposits were superior to those obtained with cyanide baths. If Pb occurred even in small amounts in the  $HNO_3$  used for cleaning, all Zn and Cd deposits were blistered and poorly adherent. Current distribution in the  $ZnSO_4$  bath was studied. C. E. P. Jeffery.

Electrodeposition of nickel from nickel chloride solutions. N. I. edot'ev and R. Kinkulskii. *Z. anorg. allgem.*

*Chem.* 224, 337-50(1935).—Compact deposits of Ni can be obtained from NiCl<sub>2</sub> solns. without the addition of other substances; purer metal is obtained than with the sulfate, since contamination by S is eliminated. The optimum acidity of the electrolyte lies from pH 6.8 to 6.2. The Ni concn. of the electrolyte must not fall below 40 g/l.; otherwise the cathode ppt. is damaged. The temp. optimum is between 70° and 80°. At 60°, pH = 5.0, and for a concn. of 40 g Ni/l. the energy consumption at a c.d. of 1.5 amp/sq dm is about 2.0 kw hrs., with a c.d. of 4.5 amp/sq dm the consumption rises to 3.5 kw. Working of Ni ores by chlorination and subsequent electrolysis of the chloride is worthwhile. When ores are worked which contain rare metals as well as Ni, the Pt group metals will be obtained. M. M.

**The effect of oxidizing agents on nickel deposition.** II. Chromic acid. A. W. Hotherhall and R. A. I. Hammond. *Trans. Faraday Soc.* 31, 1574-82(1935). cf. *C. A.* 29, 10177.—Previous investigations (cf. *C. A.* 24, 4611) on the effect of H<sub>2</sub>CrO<sub>4</sub> on the cathode efficiency of Ni deposition and on the appearance of the deposit have been extended to explain the results and to compare them with the action of H<sub>2</sub>O<sub>2</sub> and Ni(NO<sub>3</sub>)<sub>2</sub>. The H<sub>2</sub>CrO<sub>4</sub> seems to have a two fold function: (1) with low concn. initial depolarization results in pptn. of basic Ni compds. contg. Cr at the cathode, the Ni ion concn. being thereby depleted. H-ion discharge and reduced cathode efficiency are thus favored, (2) with higher concn. H<sub>2</sub>CrO<sub>4</sub> interacts chemically with the cathode metal forming a selectively permeable film on its surface. Ni deposition is consequently completely inhibited. With intermediate concns. both of these effects may occur simultaneously. Contrary to results with Ni nitrate and H<sub>2</sub>O<sub>2</sub>, the effects produced by H<sub>2</sub>CrO<sub>4</sub> can only be connected indirectly and to a smaller degree with depolarization of H. M. McMahon.

**Electric currents flowing over rusting iron.** U. R. Evans. *Nature* 136, 792-3(1935).—Currents flowing over a flat Fe surface covered by filter paper wet with NaHCO<sub>3</sub> soln. are measured by a microammeter, contact with the filter paper being made through nonpolarizing electrodes of superficially oxidized Cu. A fresh scratch ruled on a clean salt Fe surface rusts intensively under these conditions. The scratch and the surface for 2 mm. on either side of it are found to be an anodic area, while areas further removed from the scratch, which do not rust, are cathodic. This confirms current ideas on the electrochemical mechanism of corrosion. C. D. West.

**Hydrogen peroxide theory of electrolytic oxidation and the influence of the electrode surface on anodic processes.** S. Glasstone and A. Hickling. *Trans. Faraday Soc.* 31, 1656-60(1935), cf. *C. A.* 29, 1721<sup>1</sup>.—Anodic overvoltages were dealt, at smooth Pt, platinumized Pt, untreated and treated in various ways, Au, Mn, PbO<sub>2</sub>, are gas C and Ni electrodes. The results show that overvoltages cannot be correlated in any simple manner with the efficiencies in the oxidation of acetate, thiosulfate and sulfite ions. It is believed that the rate-determining step is OH<sup>-</sup> = O<sub>2</sub> + e (electrode) and H<sub>2</sub>O<sub>2</sub> is formed by union of the OH radicals. The conclusions of Walker and Weiss (cf. *C. A.* 29, 7822<sup>2</sup>) are criticized. No one mechanism is likely to account for the various types of anodic oxidations. C. E. P. Jeffreys.

**The electrolytic preparation of deuterium from heavy water.** I. L. Norling. *Physik. Z.* 36, 711-13(1935).—The app. described permits the prepn. of high-purity D from small quantities of D<sub>2</sub>O under conditions excluding exchange reactions. It is cheap, requires little attention, and produces a yield of D but a few % under the theoretical. Enrichment in the last fraction is attained if the D<sub>2</sub>O is not 100%. A V-shaped electrolytic cell is used with 3 electrodes. The third electrode, on the O side, is used to maintain const. pressure on both sides by an auxiliary electrolysis which occurs when the level varies from a pre-determined point. Allen S. Smith.

**Efficiency of separation of hydrogen and deuterium by electrolysis.** T. H. Odde. *Proc. Phys. Soc. (London)* 47, 1040-7(1935).—After correcting for losses due to escape and spraying, the efficiency (α) is shown to in-

crease with increasing current density. α is independent of pH and of the nature of the electrodes. The observed values agree with the theoretical values of Urey and Teal. S. Bradford Stone.

**The oxidation potential of the system potassium ferrocyanide/potassium ferricyanide at various ionic strengths.** I. M. Kolthoff and Wm. J. Tompkins. *J. Phys. Chem.* 39, 1045-54(1935).—The normal potential of the K<sub>4</sub>Fe(CN)<sub>6</sub>/K<sub>3</sub>Fe(CN)<sub>6</sub> electrode is 0.3560 v at 25° and not 0.41 v as generally accepted. The change of the potential in a very dil. soln. with increasing ionic strength is greater than calculated on the basis of the simple Debye-Hückel expression because of incomplete dissociation of alkali and alk. earth ferrocyanides. While the anion effect for the same valence types of salts upon the potential is the same at the same ionic strength, the effect of cations decreases in the order Cs, Rb, K = NH<sub>4</sub>, Na = Li for alkali ions and is of about the same order for alk. earth ions, the latter having much greater effect than the univalent cations especially at smaller ionic strengths. A. T. Beilich.

**New method of preparing perfectly polished metallic surfaces.** Pierre A. Jacquet. *Compt. rend.* 201, 1473-5(1935), cf. *C. A.* 29, 5749<sup>1</sup>.—The process has been slightly modified. The area of the cathode plate should be slightly greater than the area to be polished (anode). The soln. contains not less than 400 g/l. H<sub>2</sub>PO<sub>4</sub> or H<sub>2</sub>P<sub>2</sub>O<sub>7</sub> (H<sub>2</sub>PO<sub>4</sub> is useless), and temp. is 15-25°. The current required depends on the concn. of the soln. and position of the anode. With 530 g/l. H<sub>2</sub>PO<sub>4</sub> and anode vertical 10 amp/sq dm was required, while with anode horizontal 6 sufficed in about 15 min. The p.d. should be such as does not quite cause bubbles to form on the anode. C. A. Silberrad.

**The oxidation of cobalt amalgam.** I. P. Dwyer and J. W. Hogarth. *J. Proc. Roy. Soc. N. S. Wales* 69, 105-10(reprint)(1935).—By electrolyzing CoSO<sub>4</sub> and Co(SO<sub>4</sub>)<sub>2</sub> solns. with a pure Hg cathode D and H prep. Co<sub>2</sub>Hg, which is a brittle, cryst., magnetic solid decomp. without melting. It is stable in vacuum, dry air, H<sub>2</sub>, N<sub>2</sub>, CO, and under acids and in water free from air. On exposure to air it decomposes with the sep'n. of a black powder Co<sub>2</sub>O. This oxide decomposes into Co and CoO in the ratio 3:1. The CoO, because of its fine state of division, is a powerful reducing agent and on treatment with NH<sub>4</sub> salts gives a pyrophoric form of Co. E. R. Schierr.

**The nature of the oxidation at a graphite anode and comparative electrolysis of β-tartaric and mesotartaric acid in a solution of sodium hydroxide.** V. Sihvonen and M. Pyklänen. *Suomen Kemistilehti* 8B, 37-9(1935)(in German).—One hundred cc. of 0.5 M Na tartrate and 3 N NaOH soln. was electrolyzed with Pt, Ni, Fe and C anodes and various c. ds. Analyses were made for O<sub>2</sub>, glyoxylic acid, formic acid and CO<sub>2</sub>. The results for the dextro- and mesotartaric acids were very similar. Oxidations at the graphite and Ni anodes were much alike contrary to expectation. I. E. Jukkola.

**Electrolytic reduction potential of organic compounds.** XXII. The mechanism of the electrolytic reduction. Isamu Tachi. *J. Agr. Chem. Soc. Japan* 11, 734-40(1935); cf. *C. A.* 28, 4085<sup>1</sup>.—The deformation of a small drop of difficultly sol. org. compd. such as nitrobenzene, camphor oil and CCl<sub>4</sub> on a Hg cathode of small dimension was observed photographically. The drops gradually flatten on the cathode surface because of polarization but tend to restore to their original drop shape on further polarization. This phenomenon agreed with the electrocapillary phenomenon of polarized Hg. The contact angles of the various org. compd. globules to the plane of the Hg and the changes of the angles with polarization differ one from the other because of different properties of the compds. and their action on Hg. The contact angle of the camphor oil droplet was the largest. The nitrobenzene droplet was flatter in NaOH soln. than in HCl soln. The mechanisms of the reduction of nitrobenzene and of camphor are discussed. Y. Kishara.

**Electrolytic conduction of a solid insulator at high voltages—formation of the anodic oxide film on aluminum**

E J W Verwey *Physica* 2, 1059-63 (1935).—The structure of  $\gamma$ - $\text{Al}_2\text{O}_3$  formed at the anode by electrolytic action at room temp is a solid soln of  $\text{Al}^{+++}$  in a face-centered cubic lattice of O atoms. The  $\text{Al}^{+++}$  atoms are distributed interstitially among the O atoms with the restriction that 70% of the cations have a coordination no of 6 while 30% have one of 4. The  $\gamma$ - $\text{Al}_2\text{O}_3$  is formed by the deposition of O atoms at the anode surface with highest symmetry packing. At high field strengths  $\text{Al}^{+++}$  atoms are liberated from the metal and drawn into the O lattice. Thus the O lattice is built up from the outside and the  $\gamma$ - $\text{Al}_2\text{O}_3$  lattice is built up from the metallic side. The building up process stops when the  $\text{Al}^{+++}$  atoms no longer have sufficient mobility or energy to cross the barrier set up by the O atoms. The current through such films may be expressed as  $i = \alpha e^{e\phi}$  where  $\alpha = (0.24ekT/a^2v_0) e^{-e\phi_0/kT}$  and  $\beta = 3oe/8kT$ . Here  $v_0$  is the min potential barrier,  $\tau$  the time of vibration,  $a$  the lattice parameter and  $F$  the field strength in v. The order of magnitude of  $\alpha$  and  $\beta$  is checked with values previously published.

H. A. Smith

Spectroscopic observation of recurrent phenomena in discharge tubes R H Sloane and C M Minnis *Proc Phys Soc (London)* 47, 1019-28 (1935).—Methods are given for the synchronous and for the direct photography of irregular moving striations. Pure A shows no appreciable recombination in the dark phase. A-Hg mixts show excitation of only the Hg lines in the dark phase.

S Bradford Stone

Sliding contacts—electrical characteristics R M Baker. *Elec Eng* 55, 94-100 (1936).—Expts with oxidized and oxide free materials indicate that the elec. characteristics of the ordinary sliding contact are dependent upon the oxide film on the surface ring. Exptl results are shown and an explanation is made of the breakdown of the oxide film by the increase of current through the contact. Tests to det. the effects of liquid films on the contact voltage drop and the variation of thermal voltage in a sliding contact are discussed.

W. H. Boynton

Advances in the chemistry of the manufacture of dry batteries C. Drottschmann *Chem-Ztg* 59, 881-4 (1935).

E. H.

Relative temperatures and pressures of gases in an electric arc G Righini *Physica* 2, 585-90 (1935).—The temps and pressures of different parts of a C arc were detd by the intensity of the CN bands. The (0,0) and (1,1) bands of the violet group  $\Sigma^--\Sigma^--$  were used; the arc was formed between coiled C electrodes, 9 mm diam, 5 mm apart (anode above), 5 amp. The pressures of C were calcd from CN. The temps varied from 3800° to 4800° abs.

B. J. C. van der Hoeven

Manuf. and applications of Mg and Mg alloys (Deveraux) 9 Examn of electrodeposited Ni coatings by x-ray diffraction (Wood) 2. Industrial heating app [elec] (Can pat 354,059) 21. Fe alloys [for elec heating resistance] (Brit. pat 434,849) 9

Storage battery Edgar W. Allen U S 2,027,262, Jan. 7. Structural features.

Storage battery Edward W. Smith (to Electric Storage Battery Co.) U S 2,027,310, Jan. 7. Structural features.

Vent plug for storage batteries, etc Joseph L Woodbridge (to Elec Storage Battery Co.) U S 2,028,113, Jan. 14. Various details.

Battery terminals of hard and soft metals Thomas H McGrath U S 2,027,646, Jan. 14. Structural details. Lids for electric batteries The Ever Ready Co. (Great Britain) Ltd and Edward Noel Rowbotham Brit 435,235, Sept. 17, 1935.

Condensers Porzellanfabrik Kahla Brit 436,056, Oct. 3, 1935. In arrangements for obtaining condensers of const capacity or circuits with const. characteristics with variations of temp., having condensers with pos and neg temp.-capacity characteristics assembled together, the elements with neg. characteristics are constructed with a dielectric of a sintered ceramic material contg  $\text{TiO}_2$ .

Condensers. Francis C. Stephan, Frederick L. G. Betttridge and The Telegraph Condenser Co Ltd Brit. 436,195, Oct. 7, 1935. Tubular condensers are made by applying directly to the glass or ceramic dielectric a layer of adherent graphite and electroplating a metal onto the graphite layer.

Electrodeposition of metals of the platinum group Alan R. Powell and Emory C. Davies (to Johnson Matthey & Co Ltd) U S 2,027,358, Jan. 7. A plating bath for deposition of metals such as Pt, Pd or Rh comprises a soln of a sol double Na nitrite of the metal rendered slightly acid by the addn. of a small proportion of an inorg acid such as  $\text{H}_2\text{SO}_4$  or  $\text{H}_3\text{PO}_4$ , the anion of which is neither oxidized at the anode nor reduced at the cathode during plating.

Electrodeposition of nickel, etc Louis Weisberg and Wm B. Stoddard, Jr (to Weisberg & Greenwald, Inc.) U S 2,026,718, Jan. 7. A soln contg. Ni sulfate or chloride is electrolyzed in the presence also of an  $\text{NH}_4$  salt such as the sulfate, a formate such as that of Ni or Na and formaldehyde, which also may be used with a Co salt.

Apparatus for electrodeposition of precious metals on metal bands Albert Oberhauser. Fr. 788,417, Oct. 10, 1935.

Electroplating aluminum and its alloys with other metals such as nickel, iron, chromium, etc Oscar Bornhauser (to Société d'électrochimie, d'électrometallurgie des aciéries électriques d'Ugine) U S 2,028,312, Jan. 21. The article to be plated is preliminarily heated to above 100°, plunged into a coned bath of basic Al chloride brought to the same temp and then withdrawn from the bath when a violent reaction with attack of the metal is produced, this preliminary treatment serves to produce a film of Al hydride which facilitates plating.

Removing iron from chromium electrolytes. Siemens & Halske A.-G. (Johannes Fischer, inventor). Ger. 619,883, Oct. 9, 1935 (Cl. 48a 6 04). Fe is removed from Cr electrolytes by addn of compds of metals with a lower potential than Fe in solid powd form. To produce a readily filtered sludge, carbonates of alk. earth or alkali metals, in amts sufficient to ppt completely the  $\text{SO}_4$  ions present, are added. Thus, a Cr bath contg 31.4%  $\text{Cr}_2\text{O}_3$  and 1.7% Fe is given an addn of powd  $\text{MgCO}_3$  to ppt the Fe as  $\text{Fe}(\text{OH})_3$ . The  $\text{Fe}(\text{OH})_3$  is then filtered off. Cf. C. A. 29, 5028<sup>3</sup>.

Producing oxide coatings on aluminum and its alloys Siemens & Halske A.-G. (Nikolai Budiloff, inventor) Ger. 622,480, Nov. 29, 1935 (Cl. 48a. 16). Addn to 607,012 (C. A. 29, 1766<sup>4</sup>). The metal or alloy is subjected to anodic oxidation in a  $\text{CrO}_3$  soln to which a low const voltage, e. g., 20 v., is applied at a temp. of at least 65°. The coatings so obtained are absorbent and are particularly suitable for use in the photographic marking or ornamenting process described in Ger. 607,012. Cf. C. A. 29, 2460<sup>1</sup>.

Electrolytic production of oxide coatings on aluminum and its alloys Aluminum Colors Inc. Ger. 622,451, Nov. 28, 1935 (Cl. 48a. 16). Addn to 600,397 (C. A. 28, 6375<sup>1</sup>). The process is effected at atm temp and an initial voltage of 6-12 in  $\text{H}_2\text{SO}_4$  of 20-35% concn by wt.

Electrolytic metallization John Kronsheim and Charles P. Neale. Fr. 787,992, Oct. 2, 1935. Means for supporting the articles treated is described.

Electrolytic decomposition apparatus operated under pressure Ludwig Schürmer. Ger. 622,121, Nov. 25, 1935 (Cl. 12a. 13).

Apparatus for electrically heating metal blanks John R. Blakeslee (to Ajax Mfg Co.) U S 2,027,416, Jan. 14. Various structural, mech and operative details.

Carbon black John J. Jakosky (to Electroblacks, Inc.) U S 2,027,732, Jan. 14. An electrode is specified for use in the electrothermal disson of org liquids to form C black that is easily dispersed in a rubber mixt by an ordinary rubber milling procedure.

Electrolytic furnaces Louis Ferrand. Brit. 434,001, Aug. 23, 1935. The electrodes have their major axes horizontal and are so mounted as to be subjectable to a

slight longitudinal reciprocatory movement superimposed upon an oscillatory movement

**Induction furnaces** Siemens & Halske A.-G. Brit. 431,909, Sept 11, 1935 In a polyphase furnace with an open core, the hearth is angular in cross section with plane walls and a pole of the core is arranged at the geometrical center of each of the walls

**Induction furnace** Siemens-Schuckertwerke A.-G. Ger 619,807, Oct 9, 1935 (Cl 21k 18 01)

**Induction furnace for metals** Allgemeine Elektricitäts-Ges. Fr 788,006, Oct 2, 1935

**Electrolytic reduction furnaces** Det Norske Aktieselskab for Elektrokemisk Industri Fr 788,063, Oct 3, 1935 Electrodes in furnaces for the production of Al and like metals are protected against corrosion by air by metal plates extending too near the surface of the bath

**Apparatus for heating surfaces by induced currents, e. g., metal bodies for surface hardening, glass sheets for cutting** Edwin F. Northrup (to Electric Furnace Co. Ltd.) Brit 435,313, Sept 19, 1935

**Electric heating units** Allgemeine Elektricitäts-Ges. (Walter Dressler, inventor) Ger 622,172, Nov 22, 1935 (Cl 21k 2 01) Elec heating units comprising oxide-coated conductors embedded in an insulating compn are improved by adding gypsum to the insulating compn

**Nonmetallic electric heating unit** Globar Corp. Ger 619,501, Oct 2, 1935 (Cl 21k 1) The prepn of a heating unit by recrystallization of SiC is described

**Electric resistance units** Paul G. Andres (to P. R. Mallory & Co.) U S 2,027,413, Jan 14, 1935 A metallic contact member of high thermal and elec cond is placed adjacent to an elongated resistance unit including a metallic conductive oxide such as oxides of Cu, Fe, Mn, Pb or Cd so as partly to cover the resistance unit but leaving another part uncovered, and part of the unit is heated to reduce its resistance

**Negative resistance units suitable for electric circuits** Frich Habann U S 2,027,277, Jan 7, 1935 Discrete particles of materials such as  $TiO_2$ ,  $Mn_2O_3$ ,  $ZnO$ ,  $Cr_2O_3$ ,  $Fe_2O_3$ ,  $WO_3$ ,  $CuO$  or  $SnO_2$  are compressed into a body and a high-voltage elec current is passed through the compressed body, of low initial but slowly increasing intensity, until puncturing of the body occurs and it is given a desired cond Various operative details are described

**Metal anode plates** Heinrich Leiser Fr 788,528, Oct 11, 1935 A metal such as Ni is deposited on a supporting plate and the layers of Ni are sepd from the support by passage between 2 rolls traveling at a slightly different rate. The sepn is made more easy by a slight oxidation or greasing of the support

**Oxide cathodes** Telefunken Ges für drahtlose Telegraphie m b H. (Horst Rothe, inventor) Ger 619,503, Oct 2, 1935 (Cl 21g 17 02) The cathode layer carrier is coated with a heavy-metal oxide and binding agent. This layer is coated with a second layer of an alk.-earth metal compd. which, on heating, decomposes to the metal and a gaseous or solid residue not affected by the electron emission. The second layer may be  $BaNi$  or  $Ba(CN)_2$

**Cathode for photoelectric cells** The General Electric Co. Ltd. Fr 788,097, Oct 14, 1935 The interior surface of the vitreous envelope is exposed to attack by an alkali metal, e. g., Cs, or by a compd. of an alkali metal,

1 before the formation of the sensitive cathode with which the cell is finally provided

**Photoelectric cells** N. V. Philips' Gloeilampenfabrieken Brit. 435,109, Sept 11, 1935. In mfg. photoelec. electrodes by reducing a metallic oxide by a photoelec. metal, the oxide is interspersed or coated with metallic particles prior to exposure to the photoelec. metal. In 1 method, the oxide is partly reduced by H at  $150^\circ$ , less than 50% being so reduced if  $Ag_2O$  is used, Cs is then admitted and the tube filled with A, Xe or Kr at a few hundredths mm pressure

**Photoelectric cells** The General Electric Co. Ltd. and Charles H. Summs Brit 436,023, Oct 3, 1935 In the manuf. of cells, where Cs vapor acts upon a cathode of oxidized metal, e. g., Ag, the variability of the emission is reduced by exposing the interior of the vitreous envelope to attack by Cs or a Cs compd. before the final formation of the sensitive cathode is begun

**Photoelectric device** Jan H. de Boer and Marten C. Teves (to Radio Corp. of America) U S 2,027,025, Jan 7, 1935 A device is described comprising an envelope with an interior partition on one side of which are an anode such as one of W wire and an alkali metal cathode such as Cs and on the other side of which an alk. earth metal such as Ca is placed with means for vaporizing it

**Removing phenolic catalysis inhibitors from aqueous solutions such as those used for treating flue gases** Henry F. Johnstone (to Board of Trustees of the Univ. of Ill.) U S 2,027,982, Jan 14, 1935 The soln is subjected to d.-c. electrolysis and simultaneous aeration, the anodic c. d. being controlled in accord with the character of the phenolic compds. to remove them completely by oxidation from the soln and the rate of flow of the soln being controlled in accord with the concn. of such compds.

**Electric discharge tubes** N. V. Philips' Gloeilampenfabrieken Fr 788,250, Oct 7, 1935 The electrode (of W or Mo) is coated, at least in part, with  $ZrO_2$

**Incandescent cathode-discharge apparatus** The British Thomson-Houston Co. Ltd. Brit 435,442, Sept 20, 1935 Thermionic valves, rectifiers, etc., are protected during starting by the provision of starting resistances with neg temp coeff in the anode circuit, e. g.,  $UO_2$

**Electric lamps** Emile P. Hirtz. Fr 787,877, Sept 30, 1935 The life of elec lamps is prolonged by using ether, particularly sulfuric ether, as a constituent of the gas therein

**Flash light lamps** N. V. Philips' Gloeilampenfabrieken Brit 434,253, Aug 28, 1935 Mg-Al alloys, excluding the series having a Mg content of 13-85% are used. Salts influencing the photographic properties of the light, e. g., of Sr, Ca, Na and Hg, and O-yielding salts, e. g.,  $KClO_4$ , may be provided on the alloy, which may contain small proportions of Zn or Mn

**Leading-in wires for electric incandescent lamps** Clemens A. Lave and Jacob Kurtz Brit 434,192, Aug 23, 1935 A composite bar, rod or wire having a core of Cu or Cu alloy, e. g., Cu-Be, surrounded by and welded to a covering of Ni or Ni alloyed with Ta, Ti, Pt, Au, Ag, etc., which may contain Mn and preferably has a thin film of hydride on the surface, is welded to a member of a refractory metal, e. g., W, Mo, Re or Ta, by bringing it into contact therewith and heating, the alloy formed by the constituents of the wire serving as the brazing material.

## 5—PHOTOGRAPHY

E. R. BULLOCK

**New emulsions for special fields in motion-picture photography.** W. Leahy. *J. Soc. Motion Picture Engrs* 25, 214-53 (1935).—Characteristics of the Agfa Infrared, Linopan, and Superpan Reversible are described.

C. M. Tuttle  
**Speedy finishing methods for rush photographs.** J. I. Waters and S. B. Clason. *Camera* (Phila.) 51, 169-70 (1935).—Working instructions of the U. S. Army Air Corps are given for extremely rapid processing. With this

7 method, the exposure can be made, the negative processed, printed, and a dry print obtained in 5 min. The negative is developed for 20 sec. in a concd. caustic M-Q developer, rinsed in a stop bath for 15 sec., and fixed in a "speed" fixing bath for 1 min. Without washing, it is placed in the printer and a sheet of Kodaloid is squeezed to the emulsion. Three exposures are made with varying exposure times and developed by inspection in full-strength, Kodak D-72 developer for 30 sec. The best print is fixed in a

standard acid hardening fixing bath for 1 min., rinsed in H<sub>2</sub>O for 15 sec., and soaked in alk. for 15 sec. The print is suspended by one corner and the alc. is lighted and allowed to burn off. H. A. Kurtzner

**Blackening law of the photographic plate** H. Kienle *Naturwissenschaften* 23, 7, 2 (1935).—The subject of photographic photometry is discussed in the light of the no. of papers which have been published recently on the subject of reciprocity-law failure and the intermittency effect. These papers are criticized and their bearing upon photographic photometry is summarized. J. H. Webb

**Action of various elements and compounds on photographic plates** H. Shinichi Aoyama and T. Fukuroi *Bull. Inst. Phys. Chem. Research (Tokyo)* 14, 481-96 (1935). cf. C. 4 29, 60<sup>2</sup>.—Direct chem. action of activated gases on the photographic plate is a more plausible view than emission of radiation when the gases associate. B. C. A.

**Effect of soaking a plate in sodium salicylate solution as regards contrast for light of 4000-2400 Å** Tin Kiu *Compt. rend.* 201, 1348-50 (1935).—Soaking a plate in soln. of Na salicylate (cf. Thovort, C. A. 27, 506<sup>2</sup>) not only increases the sensitivity in the ultraviolet, but also stabilizes and increases the  $\gamma$ . C. A. Silberrad

**Recent fine grain and compensating developers** Adolf Lux *Photofrend* 15, 200-11, 232-3 (1935).—A comprehensive bibliographic summary. L. E. Muehler

**Reducing the contrast and grain of small negatives by redeveloping with p phenylenediamine** Gerd Heymer *Veroffentlich. v. Zentral-Lab. phot. Abt. Agfa* 4, 198-200 (1935).—Bleach in CuSO<sub>4</sub>, 5H<sub>2</sub>O 100 g., NaCl 100 g., concd. H<sub>2</sub>SO<sub>4</sub> 25 cc. and H<sub>2</sub>O 1 l. Redvelop, until the image ceases to darken as viewed from the back, in p phenylenediamine base 3 g., Na<sub>2</sub>SO<sub>3</sub> 20 g., H<sub>2</sub>O 1 l. The resulting negative, which is brownish in tone, is both notably softer than the original and improved as regards graininess. E. R. Bullock

**Brown tones by development** Paul Hancke *Atelier Phot.* 42, 158 (1935).—Tones ranging from warm black through sepia to reddish tones can be obtained on gaslight papers with the following developer: aduril 4 g., hydroquinone 4 g., Na<sub>2</sub>SO<sub>3</sub> 30 g., Na<sub>2</sub>CO<sub>3</sub> 25 g., KBr 1/2 g., and H<sub>2</sub>O 600 cc. With normal exposure and 15 min. development, this gives a warm black. By dilg the developer, adding KBr, and increasing the exposure and development times, the tone can be varied through sepia to reddish brown or red. H. Parker

**Traces of hypo decrease blue black tones with developers containing nitrobenzimidazole** Edith Weyde *Lichtbild* 11, 26-7 (1935).—Low concns (0.05 to 0.1 g. per l.) of nitrobenzimidazole, benzotriazole, etc., in developers for AgCl papers give blue-black tones but the addn. of small amts. of hypo (0.3 to 0.5 g. per l.) to such solns. decreases or eliminates the blue-black tones, giving a warm brown instead. L. E. Muehler

**Chrome alum and warm developing conditions** H. P. *Photofrend* 15, 236 (1935).—Alkali or alkali metal salt adds to chrome alum fixing baths remove the hardening properties. To insure adequate hardening, a stop bath of 20% chrome alum contg. Na<sub>2</sub>SO<sub>4</sub> is suggested. Treatment in the bath should be for not more than 5 min. So-called alkali free developers, such as the following, are also suggested with chrome alum fixing and stop baths: Na<sub>2</sub>SO<sub>4</sub> 30 g., chrome alum 20 g., H<sub>2</sub>O 1 l., for use, add 5 g. of amidol. L. E. Muehler

**Developers for papers** Anon. *Phot. Chronik* 42, 341-2 (1935).—Thirteen formulas for developers suggested by various firms for producing blue black and brown tones on papers are given. L. E. Muehler

**p Phenylenediamine research** F. W. Lowe *Leica Photographic No.* 36, 10 (1935).—It is claimed that the 2 factors that exert the greatest influence in improving the fine-grain characteristic of a p phenylenediamine developer are (1) a chem. reaction between the developer constituents that occurs on aging the soln. before use, and (2) the accumulation of Ag or Ag salts in the soln. The exact nature of the 1st phenomenon is not known. Several

methods of adding Ag salts to the soln. with different formulas are described. G. E. Matthews

**Thirty-five mm. film development in a Kodak tank** Lynn S. Wells *Camera (Phila.)* 51, 91-3 (1935).—A method of processing miniature 35 mm. film in a Kodak film tank. H. A. Kurtzner

**Sensitometric studies of processing conditions for motion picture films** H. Meyer. *J. Soc. Motion Picture Engrs.* 25, 230-47 (1935).—Test-object negatives were sent to 8 Hollywood labs. and later prints from these negatives were obtained. M's data indicate an approx. const. printing factor of 11%. Selected prints use one half the toe region and one half the straight-line portion of the over-all reproduction curve, and the over-all reproduction curve has an av. slope of 1. C. M. T.

**Double-coated Schumann films** H. P. Knaus and R. V. Zumstein *Phys. Rev.* 45, 124 (1934).—K. and Z. have found that it is possible to ppt. a thin Schumann emulsion directly onto the sensitive surface of Eastman 33 plates, thereby obtaining plates that are not only as fast in the S. Schumann region as 3 other ultraviolet-sensitive materials with which they were compared but are equally as fast as the Eastman 33 plates themselves for the longer wave lengths. E. R. Bullock

**Determination of the color sensitivity of negative materials for pictorial exposures** Martin Biltz and John Eggert *Veroffentlich. v. Zentral-Lab. phot. Abt. Agfa* 4, 35-41 (1935); cf. C. A. 30, 390<sup>1</sup>.—A somewhat more detailed account of the work already described. E. R. Bullock

**Recent characteristic surfaces** Hans Arens *Veroffentlich. v. Zentral-Lab. phot. Abt. Agfa* 4, 15-25 (1935), cf. C. A. 29, 783<sup>2</sup>; 30, 35<sup>1</sup>.—Addnl. illustrations are given of surfaces representing the variation of the photographic d. with the logarithm of the intensity and with the logarithm of the time of exposure, for normal and solarizing exposures, the Villard effect, and for phys. development. E. R. Bullock

**Making harmonious enlargements from small negatives** Arpad v. Biehler *Veroffentlich. v. Zentral-Lab. phot. Abt. Agfa* 4, 193-7 (1935).—A method which is described for obtaining harmonious enlargements from negatives having a great range of d. consists in first toning the negative blue [with Fe] and then enlarging it on a contrasty paper. Author

**Oxide coatings on Al and its alloys [used in photographic marking]** (Ger. pat. 622,480) 4

**Color photography** Béla Gáspár. *Fr.* 787,937, Oct. 1, 1935. Into light-sensitive layers or solns. or emulsions used for their production, the dye is introduced in coarse dispersion in a noncoloring or only slightly coloring state, a state in which it is almost nonabsorptive. After exposure the dye is brought, at any point of the treatment of the image, to an active coloring state. This may be realized by treatment with solvents or chem. agents.

**Photographic development** I. G. Farbernd A-G *Fr.* 783,472, Oct. 10, 1935. Films may be developed in a bright light if the exposed films are treated before development with solns. of dinitro or polynitro derivs. of imidazole, triazole or pyrazole, or if these are added to the developer.

**Photographic development** Gevaert Photo-Produeten, N. V. *Fr.* 788,511, Oct. 11, 1935. Emulsions are developed without fogging by adding to the developer substances obtained by condensing thiothiazolidine or its homologs and derivs. with nonbasic cyclic aldehydes. The prepn. is given of 5-benzal-2-thiothiazolidine-4-ketothiazolidine from 2-thiothiazolidine-4-ketothiazolidine and BzH and 5-piperonylidene-2-selenoketo-4-ketothiazolidine from 2-selenoketo-4-ketothiazolidine and hehtopropine.

**Photographic emulsions** I. G. Farbernd A-G *Fr.* 788,043, Oct. 2, 1935. A Ag halide emulsion has, in addn. to orthochromatic sensitization (I), a sensitization (II) for the deep red and infrared region of the spectrum and a sensitization gap between 5,900 and 6,200 Å. For I dyes of the group benzothio-, benzocleno-, thio-

pseudo-, selenopseudo-, seleno- and oxocarbo-cyanines, and for II, thiopentacarbo-, selenopentacarbo-, thioheptacarbo- and selenoheptacarbo-cyanines are appropriate.

Sensitizing photographic emulsions I G. Farbenindustrie A.-G. Brit 434,235, Aug 28, 1935 Ag halide emulsions are sensitized by incorporation therein of unsym. pentacarbocyanine dyes of formula  $YCR^1CR^2CR^3CR^4$ , wherein Y and Z

are S, Se or -CH-CH-,  $R^1, R^2, R^3$  and  $R^4$  are H, alkyl or aryl or  $R^1, R^2$  and (or)  $R^3, R^4$  together represent a substituted or unsubstituted phenylene, naphthylene or other polynuclear arylene group,  $R^1$  and  $R^2$  are alkyl and X is an acid radical such as Cl, Br, I, ClO, MeCfH<sub>2</sub>SO<sub>3</sub>, MeSO<sub>3</sub>, EtSO<sub>3</sub>, lack of symmetry being fulfilled by either Y and Z or  $R^1, R^2$  and  $R^3, R^4$  differing, or both. The H atoms of the polymethylene chain may be replaced by alkyl and, in the case that Y or Z is -CH-CH-, the polymethylene chain may be linked to the heterocyclic ring in the 4-position instead of the 2-position. The dyes are made by the process of Brit 434,234 (C. A. 30, 6909) Examples of 28 dyes with sensitizing data are given. 1,1'-Diethyl-2-(6-methylquino)-benzothioheptacarbo-cyanine iodide is made by condensing p-toluenediamine-EtI and  $\alpha$ -phenylamino- $\gamma$ -phenyliminopropene-HCl and condensing the product with 2-methylbenzothiazole EtI Cf C. A. 29, 3249.

Photographic sensitizers I G. Farbenindustrie A.-G. (Martin Dablow and Alfred Philips, inventors) Ger 622,211, Nov 22, 1935 (Cl. 22c. 3) See Brit 378,239 (C. A. 27, 2896)

Photographic diazotype layers N V Philips Gloeilampenfabriek Brit. 433,999, Aug 23, 1935 Light-sensitive layers contg. a diazonium compd. and a coloring component, especially 1 having a self-developing property, i. e., of producing a color after exposure, by treatment with H<sub>2</sub>O vapor, are prepd. by applying to the carrier of the diazonium compd., in pulverulent form, 1 substance assisting in the production of color and 1 substance that absorbs moisture from the atm. while retaining its pulverulent form and not assisting in the production of the coloring material. In examples, (1) paper is coated with p-diphenylaminobenzenediazonium chloride in the form of the ZnCl<sub>2</sub> double salt, dried, rubbed with rice meal and powd.  $\alpha$ -naphthol (I) is dusted thereon, or the rice meal and I may be mixed and rubbed in, and (2) 4-dimethylaminobenzenediazonium borofluoride is applied to paper which is then rubbed with a mixt. of rice meal, phloroglucinol and anhyd. Na<sub>2</sub>CO<sub>3</sub>. In 434,761, Sept. 9, 1935, prints are produced by giving to a layer sensitized with a diazonium compd. that will combine with its light-decomp. product to form a colored substance an exposure such that the diazonium compd. is only partly decompd. and subsequently bringing about the decompn. of the diazonium compd. in the parts not exposed to light by means other than light action, e. g., by heat and (or) moisture. A Cu salt may be included in the layers, which should be stored in a dry atm. before use. Among examples, 2-hydroxy-5-sulfobenzenediazonium salt and Cu(NO<sub>3</sub>)<sub>2</sub> are developed either by keeping the layer in an atm. satd. with H<sub>2</sub>O vapor for 3 days or 30 min., the

material in the latter case being ironed under a damp cloth at 100°.

Photographic layers for accelerated diazo printing Walker M. Hinman (to Frederick Post Co.) U. S. 2,027,229, Jan. 7. A carrier such as paper is provided with a thin layer of material contg. a light-sensitive diazonium compd. and the essential component parts of a light-sensitive salt such as a higher oxide compd., e. g., ferric oxalate and an org. reducing agent. Numerous examples are given.

Photographic negatives Arthur E. Field U. S. 2,027,271, Jan. 7. For treating a negative to produce an improved tone-printing image, as for use in photo-mech. printing work, a fixed negative is subjected to the action of a first soln. comprising a bleaching reagent such as I and KI soln. which produces on the plate a Ag halide photographic image of positive appearance, then treating the plate with a second soln. that is a solvent for the Ag halide so as to effect the requisite retouching on the positive image without redeveloping it to a negative image, and finally with a third soln. which blackens the Ag halide image and reconverts the image of positive appearance to a negative image. Various details are given.

Colloid reliefs Kodak A.-G. Ger. 619,605, Oct. 4, 1935 (Cl. 57d. 2/2). In making metal molds for printing copies by the aid of colloid reliefs, colloid layers contg. metal salts are used. These are exposed under a copying app., heated and developed. The layers are coated with lacquer before developing. The lacquer may contain for example, cellulose nitrate, tritoly phosphate and AcOEt.

Film for color photography John G. Capstaff (to Eastman Kodak Co.) U. S. 2,026,964, Jan. 7. A light-transmitting support of cellulose compn. contains a neutral dye and has minute lenticulations on one surface, and on the other surface carries a sensitive, panchromatic layer and an outer antihalation layer contg. a material such as MnO<sub>2</sub> or Ag which is absorptive of light of all colors and is readily removable in the baths of a photographic reversal process.

Laminated sheet material suitable for photographic films Max Hagedorn (to Agfa Anso Corp.) U. S. 2,027,688, Jan. 14. An internal stratum contg. an org. cellulose ester such as cellulose acetate carries on both surfaces external strata of regenerated cellulose carrying outer adhering layers of a colloid less sensitive to water than cellulose hydrate, such as a specified nitrocellulose compn.

Television films Fernseh A.-G. Brit. 434,873, Sept. 10, 1935. A film for television is exposed with the light-sensitive layer in a coagulated condition without being completely dried. The thickness of the coagulated layer is about that of the dimension of a picture point. The emulsion contains a larger Ag salt content and a smaller colloid content than normal.

Retouching photographic films or plates Karl Kipphan (to Agfa Anso Corp.) U. S. 2,026,899, Jan. 7. For applying brightening retouches to a photographic material, there is finely distributed in a light-sensitive layer a compd. such as Fe(OH)<sub>3</sub>, resisting photographic baths and diminishing the transparency to light of the layer, and the layer is partly cleared by a chem. reagent such as 5% oxalic acid soln. in conformity with parts of a picture to receive a greater transparency.

## 6—INORGANIC CHEMISTRY

A. R. MIDDLETON

Chemistry of bromine. I. Preparation of bromine dipyrindine perchlorate and bromine dipyrindine nitrate H. Carlsohn Ber. 68B, 2209-11(1935)—Br in CHCl<sub>3</sub> added to a cooled soln. of AgNO<sub>3</sub> in CH<sub>3</sub>OH gave bromine dipyrindine nitrate, [Br(pyr)]NO<sub>3</sub>. AgNO<sub>3</sub> in CH<sub>3</sub>OH soln. treated with NaClO<sub>4</sub> gave silver dipyrindine perchlorate, [Ag(pyr)]ClO<sub>4</sub> (A). Treating A with Br in CHCl<sub>3</sub> soln. gave bromine dipyrindine perchlorate [Br(pyr)]ClO<sub>4</sub>. II. Preparation, properties and constitution of some

water-pyridine compounds of bromine oxides Ibid. 2212-14.—A preliminary discussion R. C. Roberts

Amphoteric oxide hydrates, the solutions of their hydrolyzed salts and their high-molecular compounds XXX. Iso- and heteropolypyridine acids in particular relation to the formation mechanism of arseno- and phosphotungstic acids. Gerhart Jander and Hans Banthien Z. anorg. allgem. Chem. 225, 162-72(1935); cf. C. A. 29, 5371\*—In P- and As-free as well as in tungstate solns

contg. P and As the monotungstate with increasing H-ion concn is converted first into *hexatungstic acid*, therefore a polyacid. This is stable in acid soln. This hexatungstic acid combines with phosphoric acid, again with the aid of  $H^+$ , to form *1-phospho-1-hexatungstic acid* (I), a new compd. The tendency of I to secondary disson depends upon the  $H^+$  and tungstic acid concn of the soln. Increase in both concns suppresses it. With higher H-ion or phosphate concn I can again combine with other hexatungstic or phosphoric acid mols to form various *x-phospho-y-hexatungstic acids*.

**H. Stoertz**  
A study of the dehydration of hydrated cobaltic oxide. H. Armin Pagel, Wm K. Noyce and Myron T. Kelley. *J. Am. Chem. Soc.* 57, 2552-3 (1935).—A careful study of hydrated cobaltic oxide at temps. between 155° and 260° has been made. The percentages of  $Co_2O_3$  and  $H_2O$  found are tabulated.  $Co_2O_3$  could not be oxidized to  $Co_3O_4$  at 100 atms and 200° to 400°. More drastic treatment shows the O disson pressure of  $Co_2O_3$  is greater than 127 atms at 315°. Particle size and porosity are believed to be important factors in rate of dehydration. Presence of the wet monohydrate during drying at these temps for short periods of time prevents much decompn into  $Co_2O_3$ .

**Raymond H. Lambert**  
Constituents of nitrous vapors. A Sanfourche and Jean Bureau. *Compt. rend.* 202, 66-8 (1936).—Contrary to the usual mode of reaction (cf. C. A. 15, 3352) "nitrous anhydride" when passed into a soln. of  $PhNH_2$ , HCl reacts mainly as a mixt. of  $NO$  and  $NO_2$ ,  $2(NO + NO_2) + PhNH_2 = PhN(OH) + HNO_2 + 2NO$ . This is demonstrated by dividing a stream of " $N_2O_3$ " into 2 equal parts and passing one into such a soln. and the other into  $H_2SO_4$  (1.833), and detg. the amts. of azo, nitrous and nitric N resulting in each (cf. C. A. 18, 1552). C. A. S.

The formation and composition of lithium aluminate. Harold A. Horan and John B. Damiano. *J. Am. Chem. Soc.* 57, 2434-6 (1935), cf. C. A. 26, 936.—An insol. Li aluminate is pptd., practically quantitatively, when a soln. of an Al salt contg. sufficient Li salt is made alk. with  $NH_3$ . Analysis shows that the ppt. contains Li and Al in the at. ratio 1:2. Upon ignition at a high temp. this substance becomes const. in wt. The wt. of the residue, obtained from a ppt. const. a known amt. of Al, suggests the formula  $Li_2O \cdot 2Al_2O_3$ . W. C. Fernelius

Anhydrous sulfates of the magnesian series. Fritz Hammet. *Compt. rend.* 202, 57-9 (1936).—X-ray examn of the anhyd. sulfates of Mg, Mn,  $Fe^{++}$ , Co, Ni, Cu and Zn show them all to be orthorhombic, the values of  $a$ ,  $b$  and  $c$  being (Mg) 4.8, 6.7, 8.3, (Mn) 4.8, 6.8, 8.5, ( $Fe^{++}$ ) 4.8, 6.8, 8.6, (Co) 4.6, 6.6, 8.4, (Ni) 4.6, 6.5, 8.4, (Cu) 4.8, 6.6, 8.3, (Zn) 4.7, 6.7, 8.5. C. A. S.

Cesium iodomercurate. Fernand Gallais. *Compt. rend.* 202, 54-6 (1936).—Cond. and magnetic rotation detos of solns. contg. varying amts. of  $HgCl_2$  and CsI indicate  $Cs_2HgI_4$  to be the only double iodide. C. A. S.

Ortho- and meta tellurates. Salts of benzenide. Marcel Patry. *Compt. rend.* 202, 64-6 (1936), cf. C. A. 29, 5724.—Neutral orthotellurate of benzenide,  $C_6H_5N_2 \cdot Te(OH)_4$ , is obtained by adding an alc. soln. of benzenide to an aq. soln. of orthotelluric acid, of such strengths that the mixt. contains less than 30% EtOH, if stronger in EtOH a mixt. of neutral and acid orthotellurates results. The orthotellurate forms amber-colored leaflets, slightly sol. in  $H_2O$  (1.1%), and EtOH (1.46%). At 80° it loses 4  $H_2O$ , forming  $TeO_3 \cdot C_6H_5N_2$ . In similar fashion polymetalluric acid in aq. or alc. soln. forms the neutral metatellurate  $C_6H_5N_2 \cdot H_2TeO_4$ , if the mixt. solns. contain less than 30% EtOH, if more the product contains more or less acid metatellurate, which is obtained pure ( $C_6H_5N_2 \cdot 2H_2TeO_4$ , 44.3% Te) with pure EtOH as solvent. Both are gray amorphous powders, sensitive to the action of light, but permanent in moist air, and rapidly blackening above 60°. Kretov's failure (C. A. 27, 42) to prepare a tellurate was due to his use of benzenide HCl. With mineral alkalis both telluric acids yield the same product. C. A. Silberrad

Rubidium and cesium fulminates. Louis Hacksplund and Willy Schumacher. *Compt. rend.* 202, 60-71 (1936).—

These fulminates were prepd. by Wöhler's method (Ber 38, 1355 (1905)), i. e., by the action of an amalgam of Cs or Rb on Hg fulminate suspended in MeOH at -15° for  $\frac{1}{2}$  hr., the Cs or Rb fulminate then being pptd. by  $Et_2O$ . As thus prepd., they always contain some Hg double salt from which the pure Cs or Rb salt is obtained by resolu. in MeOH and reprecip. by  $Et_2O$ . Both closely resemble the corresponding Na and K salts, save that they are very deliquescent, and detonate more violently the higher the mol. wt. The double Hg salts are still more sensitive, the RbHg fulminate exploding at 45°. C. A. Silberrad

Reactions in the action of hydrogen sulfide upon bisulfite solutions. J. Janickis. *Z. anorg. allgem. Chem.* 225, 177-203 (1935).—In the action of  $H_2S$  upon bisulfite solns. whose  $pH$  lies between 5.1 and 2.3, thiosulfate, trithionate, tetrathionate, pentathionate, S and  $H^+$  are produced. At 1st the main products are  $S_2O_3^{--}$  and  $S_4O_6^{--}$ , but when sufficient  $S_2O_3^{--}$  and  $H^+$  ion concns. have been attained,  $S_2O_3^{--}$  and  $S_4O_6^{--}$  are formed. The latter is assumed to be produced as follows:  $H_2SO_3 + 2HSO_3^{--} \rightarrow S_2O_3^{--} + 2H_2O$ . Addn. of  $S_2O_3^{--}$  to initial soln. increases the yield of all polysulfonates, this increase being greater with greater initial  $H^+$  ion concn.  $S_2O_3^{--}$  is formed as follows:  $S_2O_3^{--} + HSO_3^{--} \rightarrow S_2O_3^{--} + S_2O_3^{--} + H^+$  and trithionate partially by the reaction  $S_2O_3^{--} + HSO_3^{--} \rightarrow S_2O_3^{--} + S_2O_3^{--} + H^+$  and also by  $H_2SO_3 + 2HSO_3^{--} \rightarrow S_2O_3^{--} + 2H_2O$ . H. Stoertz

Permanent electric moment and structure of phosphorus pentachloride. Pierre Trunel. *Compt. rend.* 202, 37-9 (1936).—Extrapolated to infinite diln. the polarization and mol. refraction of  $PCl_5$  at 25° in soln. in either  $CS_2$  or  $CCl_4$  are, resp., 51 and 35.5, giving an elec. moment,  $\mu = 0.8$ , which indicates a nonsym. structure, explicable either by nonequivalence of all 5 Cl atoms or the nonplanar character of the mol. (cf. Simons and Jessop, C. A. 25, 2837). C. A. Silberrad

Remarks on the red and green form of Magnus's salt. H. D. K. Drew and H. J. Tress. *J. Chem. Soc.* 1935, 1580-8.—The red form is not identical with the empirically isomeric triamine platino-salt which is much more sol. in  $H_2O$ . They are clearly distinguishable by chem. tests. In prep. according to Jørgensen and Sørensen (*Z. anorg. Chem.* 48, 441 (1906)) occasionally gray or gray-green mixts. of the 2 forms were obtained. The 2 forms are similar in cryst. form, soly and chem. reaction. They probably have the same mol. wt. and dichroism cannot explain their formation. The red form can be prepd. from certain chloroplatinate solns., even in concd. HCl. Some samples of the red form are stable to considerable boiling in  $H_2O$ , to hot HCl and to seeding with the green form. The condition of the chloroplatinate ion det. whether the red or green form will be produced. It is suggested that the 2 forms are electroisomers. Foster Dee Snell

Chemical formula of malachite. Marcel Guillot and Georges Geneslay. *Compt. rend.* 202, 136-7 (1936).—The hydrated basic Cu carbonates prepd. by the interaction of satd. solns. of  $K_2CO_3$ ,  $Na_2CO_3$ ,  $KHCO_3$  or  $NaHCO_3$  and  $CuSO_4$ , or by hydrolysis of various alkali cupricarbonates all give products which x ray examn. shows to be identical, and also with Auger's "hydrated malachite" (C. A. 8, 2126), and with natural malachite, to which the formula  $8CuO \cdot 4CO_2 \cdot 5H_2O$  is assigned. C. A. Silberrad

Complex dipyrrolyl salts of nickel and copper. F. M. Jaeger and J. A. van Dyk. *Proc. Acad. Sci. Amsterdam* 38, 972-7 (1935).— $[Ni(Dipyr)](NO_3) \cdot 5H_2O$  is formed when 1, 2 or 3 equivs. of dipyrrolyl (I) are added to a soln. of  $Ni(NO_3)_2$  and the soln. is evapd. at room temp. The red crystals are hexagonal bipyramidal. The dimensions of a unit cell are  $a_0 = 13.5 \text{ \AA}$ ,  $c_0 = 10.8 \text{ \AA}$ . The cell contains 2 mols. After deprotonation of this salt a ppt. of violet needles is obtained which has the compn.  $[Ni(Dipyr)](NO_3) \cdot 3H_2O$ . The crystals are monoclinic prismatic,  $a \cdot b \cdot c = 0.3912 \cdot 1.0 \cdot 0.3825$ ,  $\beta = 73^\circ 43'$ . They are dichroic. For vibrations parallel to the  $c$  axis they are pink, and perpendicular blue. From a soln. treated with  $\frac{1}{2}$  equiv. of I a blue salt of the compn.  $[Ni(Dipyr)](H_2O)_2 \cdot (NO_3)_2 \cdot H_2O$  is formed. No measurable crystals were ob-

tained. From  $\text{NiCl}_2$  solns. treated with 2 or 3 equivs of I the salt  $[\text{Ni}(\text{Dipyridyl})_2]\text{Cl}_2 \cdot 7\text{H}_2\text{O}$  was obtained. The crystals are monoclinic prismatic,  $a:b:c = 0.6113:1:2.0941$ ,  $\beta = 72^\circ 55'15''$ . The crystals were isomorphous with corresponding  $\text{Zn}$  and  $\text{Cu}$  salts. On heating, the anhyd. dipyrilidyl salt is formed. When 1 equiv of I is added, and after deposition of a tripyridyl salt, some crystals with the compn.  $[\text{Ni}(\text{Dipyridyl})(\text{H}_2\text{O})_2]\text{Cl}_2$  were pptd. Reexamination of the crystals of  $[\text{Cu}(\text{Dipyridyl})_2]\text{Cl}_2 \cdot 7\text{H}_2\text{O}$  shows it to be monoclinic prismatic with  $a:b:c = 0.6111:1:2.1278$ ,  $\beta = 72^\circ 54'15''$ . C. E. P. Jeffreys

Osmotic complex with two stationary liquids. F. A. H. Schreinemakers and J. P. Werre. *Proc. Acad. Sci. Amsterdam* 38, 952-8 (1935).—An osmotic complex,  $\pi_1(W) \cdot L_1(z) \cdot L_2(u) \cdot L_3(v)$ , 20.37%  $\text{NaCl}$ , where the invariant liquids  $z$  and  $u$  were initially varying mixts. of  $\text{NaCl}$  and  $\text{Na}_2\text{CO}_3$ . The paths of concn. of  $\text{NaCl}$  in the variable liquids were followed during osmosis and depicted graphically. When a complex contains 1 variable liquid only, the liquid will, during transition toward a stationary state, proceed along a path, the shape of which does not depend upon the quantity of liquid. In a complex with 2 or more variable liquids the path depends upon the quantity of each of the liquids. C. E. P. Jeffreys

Constitution of thioether compounds of platinum. K. A. Jensen. *Z. anorg. allgem. Chem.* 225, 115-41 (1935).—

1 Cond. of aq. solns. of  $\beta\text{-}[\text{PtCl}_2(\text{Et}_3\text{S})_2]$  depend upon an extremely rapid hydration— $[\text{PtCl}_2(\text{Et}_3\text{S})_2] + 2\text{H}_2\text{O} \rightarrow [\text{Pt}(\text{H}_2\text{O})_2(\text{Et}_3\text{S})_2]^{2+} + 2\text{Cl}^-$ . In  $\text{CH}_3\text{OH}$  a similar "alcoholation" occurs which can be followed by cond. measurements. An important difference between  $\alpha$ - and  $\beta$ -thioether compds. exists only in the velocity of, but not the magnitude of, alcoholation. The chlorides, bromides and nitrites are only slightly, the nitrate and sulfate on the other hand are almost completely, alcoholated. By mol. wt. determ. it is shown that formulae of compds. of the type  $[\text{PtCl}_2(\text{R}_3\text{S})_2]$  should be doubted. There are only 2 isomers of the type  $[\text{PtX}_2(\text{R}_3\text{S})_2]$ — $\alpha\text{-}[\text{PtCl}_2(\text{Pr}_3\text{S})_2]$  and the  $\alpha$ - and  $\delta\text{-}[\text{PtCl}_2(\text{Bu}_3\text{S})_2]$ , mentioned by several workers, are claimed by J. not to exist. H. Stortz

The preparation of potassium molybdo- and molybdicyanides. Holmström H. Willard and R. C. Thielke. *J. Am. Chem. Soc.* 57, 2609-11 (1935), cf. Sand and Burger, *Ber.* 38, 3344 (1905).—Tri- and quadrivalent Mo thiocyanate complexes are only partially extd. by ether. A 70-78% yield of  $\text{K}_2\text{Mo}(\text{CN})_2 \cdot 2\text{H}_2\text{O}$  (I) is obtained by first pptg. a quadrivalent Mo pyridine thiocyanate (thick oil) in  $\text{NH}_3$  soln. and then treating with  $\text{KCN}$ . Quant. yields of unstable  $\text{K}_2\text{Mo}(\text{CN})_2$  in soln. are obtained by oxidizing I with  $\text{MnO}_2$  in acid soln. and removing Mn by oxidation to  $\text{MnO}_2$  with  $\text{MnO}_2$  after making the soln. alk. Quadrivalent Mo is best prepd. by electrolytic reduction of  $\text{MoO}_3$  in 8 N  $\text{HCl}$  to Mo followed by oxidation with  $\text{MoO}_3$ . W. C. Ierrehus

## 7—ANALYTICAL CHEMISTRY

W. T. HALL

The limitations of colorimetric analyses by present methods. E. L. Armstrong and M. L. Kuder. *J. Lab. Clin. Med.* 21, 181-7 (1935).—Errors in the colorimetric methods for analysis of sugar, nonprotein N, urea N and uric acid are discussed. A new type of photoelec. colorimeter is described. E. R. Main

Aromatic analysis. A useful technic in estimating small amounts of heavy precipitates. V. R. Damerell and M. Axelrod. *J. Am. Chem. Soc.* 57, 2724-5 (1935).—Details are given of a simple technic whereby compact circular (or square) piles of ppt. of reproducible dimensions can be obtained in the center of the flask bottom. The apparent areas of these "spots" can be readily measured and compared with the areas produced by known amts. of ppt. C. J. West

Problems and methods in industrial spectroscopy. S. Judd Lewis. *J. Soc. Chem. Ind.* 54, 427-32T (1935).

Qualitative and quantitative spectrographic analysis. W. Gerlach. *IX Congr. intern. quim. pura applicada Madrid, 1934; Neues Jahrb. Mineral. Geol., Referate I*, 1935, 13; cf. C. A. 29, 1736<sup>1</sup>, 3225<sup>1</sup>.—A review of app. methods and results. J. F. Schaefer

Spectral analytical studies. Walther Gerlach. *Z. anal. Chem.* 103, 356 (1935).—W. Kramer. *Ibid.*—Polemical concerning previous papers by Kramer, C. A. 28, 2641<sup>1</sup>, 4312<sup>1</sup>, 5771<sup>1</sup>. W. T. H.

The use of spectral analysis for the determination of alkalis and alkaline earths. V. The direct photoelectric estimation of alkalis. W. H. Jansen, J. Heyes and C. Richter. *Z. physik. Chem.* A174, 291-300 (1935); cf. C. A. 29, 2171<sup>1</sup>.—The quant. spectral analysis for alkali metals is simplified. The air-C<sub>2</sub>H<sub>2</sub> flame sprayed with the liquid to be tested stands before a monochromator which permits the principal line of the elements being detd. to reach the photocell. The liberated electron stream is measured by a charge process with a Lindemann electrometer. The linear relation between the measurements and concn. of elements makes possible the calcn. of alkali by linear interpolation of the measurements. The method is accurate to 5%. Tables contain measurements of the individual alkali ions and the size of error. R. H. Baechler

The practicability of conductometric methods with

visual observation for titration in the presence of many different foreign electrolytes. G. Jander and A. Thiert. *Z. Elektrochem.* 41, 700-4 (1935).—The app. and expl. arrangements for conductometric titrations in the presence of extraordinarily large amts. of foreign electrolytes in the soln. are described. A sufficiently concd. reagent soln. and a calibrated microburet must be used, and care must be taken that in the addn. of the reagent soln. a too decided decrease in cond. does not occur because of diln. Polarization influence is prevented or reduced to a min. by polarizing the electrode, and temp. must be kept const. M. McMahon

Detection and separation of difficultly soluble compounds by concentrated hydriodic acid. F. A. R. Caley and M. Gilbert Burford. *Ind. Eng. Chem., Anal. Ed.* 8, 63-7 (1936).—Substances such as  $\text{SnO}_2$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{BaSO}_4$ ,  $\text{SrSO}_4$ ,  $\text{PbSO}_4$ ,  $\text{Cr}_2\text{H}_2(\text{SO}_4)_3$ ,  $\text{AgI}$ ,  $\text{AgBr}$ ,  $\text{AgCl}$ ,  $\text{CaCl}_2$  and anhyd.  $\text{CrCl}_3$  can be decomposed by heating with HI and it is recommended in qual. analysis to test any residue insol. in  $\text{HCl}$  or aqua regia with 6 ml. of HI, d. 1.7, per g. of sample. The residue from this treatment should be tested for  $\text{CaI}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  in the usual way. W. T. H.

Calculating the blank. Bartholow Park. *Ind. Eng. Chem., Anal. Ed.* 8, 32 (1936).—Instead of actually running a blank it is recommended to det. the end point for portions of 10, 20, 30 and 40 cc. and assume that the blank is the same in each titration. Then by simple computations it is easy to compute the numerical value. W. T. H.

Critical studies concerning organic compounds as analytical reagents. IV. p-Nitrobenzenesulfonate orcinol as a reagent for magnesium. I. Laird Newell, Nathan R. Pike and Joseph B. Icklen. *Z. anorg. allgem. Chem.* 225, 241-4 (1935).—In spite of encouraging reports from Saito and Okuma, C. A. 20, 3000<sup>1</sup>, Pegriew, C. A. 23, 2903 and Ruigh, C. A. 23, 2905, this reagent is not as sp. as might be desired, although it may be useful in helping to identify Mg. The test is reliable, however, only in the absence of  $\text{NH}_4$  salts and ions other than those of  $\text{As}^{+++}$ ,  $\text{Be}^{++}$ ,  $\text{Ce}^{+++}$ ,  $\text{I}^+$ ,  $\text{Na}^+$ ,  $\text{Ag}^+$ ,  $\text{Fe}^{++}$ ,  $\text{Ti}^+$ ,  $\text{AcO}^-$ ,  $\text{Cl}^-$ ,  $\text{MnO}_4^-$ ,  $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{SO}_4^{--}$  and  $\text{VO}_4^{--}$ . W. T. H.

New organic reagent for cadmium. Alfred W. Scott and Eleanor G. Adams. *J. Am. Chem. Soc.* 57, 2541-2 (1935).—1-(2-Quinoly)l-4-allyl thiosemicarbazide,  $\text{C}_{12}\text{H}_{11}\text{N}_3\text{S}$



NHCSNHNHC<sub>4</sub>H<sub>9</sub>N (0.5 g. in 100 cc. of 50% alc.) is a sensitive precipitant for Cd<sup>++</sup> and can be used for detecting Cd in the presence of Cu. W. T. H.

Standardization of permanganate solutions with sodium oxalate. Robert M. Fowler and Harry A. Bright. *J. Research Natl. Bur. Standards* 15, 493-501 (1935) (Research Paper No. 843).—The usual procedure for standardizing KMnO<sub>4</sub> consists in slowly titrating the hot, acid soln. with the temp. at least 60° at the finish. The results agree better with those obtained by other methods of standardization, if the following procedure is used, because there is less danger of decomg. the oxalate by the hot, dil. acid. To 250 ml. of 1.9 N H<sub>2</sub>SO<sub>4</sub>, which has been recently boiled for 10-15 min. and then cooled to 27-30°, add the accurately weighed Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> (0.3 g.) and when, with the aid of stirring, the sample has all dissolved, add 39-40 ml. of approx. 0.1 N KMnO<sub>4</sub> in a steady stream during about 90 sec. while stirring slowly. Let stand until the pink color of the KMnO<sub>4</sub> has disappeared (45 sec.), heat to 55-60° and finish the titration by the dropwise addn. of the KMnO<sub>4</sub>. Toward the last be careful not to add a new drop of the KMnO<sub>4</sub> soln. until the color of the previous drop has disappeared. The end point should persist for 30 sec. W. T. H.

Procedure for the separation of the six platinum metals from one another and for their gravimetric determination. Raleigh Gulchurst and Edward Wichers. *J. Am. Chem. Soc.* 57, 2565-73 (1935).—A new and reliable procedure is given for the sepn. and detn. of Os, Ru, Pt, Pd, Rh and Ir which is much simpler than methods hitherto employed. The Os is first distd. off as OsO<sub>4</sub> from HNO<sub>3</sub> soln. through which a current of air is passing. The OsO<sub>4</sub> is absorbed in HCl which is satd. with SO<sub>2</sub>, from the resulting soln. the Os is eventually pptd. by hydrolysis and weighed as metal after reduction in H<sub>2</sub>. After the removal of the Os, the soln. is heated with HCl and fumed with H<sub>2</sub>SO<sub>4</sub>. NaBrO<sub>3</sub> is added and RuO<sub>4</sub> distd. off and absorbed in HCl which is satd. with SO<sub>2</sub>. This element is pptd. in much the same way as Os by adding NaHCO<sub>3</sub> and boiling; the hydrated ppt. is ignited in H<sub>2</sub> and weighed as Ru. Next, the Pd, Rh and Ir are sepd. from Pt by a controlled hydrolytic pptn. from which the Pt alone remains in soln. It is pptd. as sulfide and weighed as metal. The hydrated dioxides of Pd, Rh and Ir are dissolved in HCl, the Pd is pptd. with dimethylglyoxime and the ppt. is dried and weighed. After the removal of Pd, the soln. is treated with H<sub>2</sub>SO<sub>4</sub> + HNO<sub>3</sub> to destroy the excess org. reagent and the Rh is pptd. by means of TiCl<sub>4</sub>. The pptd. metal is dissolved in H<sub>2</sub>SO<sub>4</sub> and the pptn. repeated. The last ppt. is again dissolved in acid and the Rh is thrown down as sulfide, ignited in H<sub>2</sub> and weighed as metal. Finally, the Ti is removed with cupferron, the soln. freed from org. matter by treatment with H<sub>2</sub>SO<sub>4</sub> + HNO<sub>3</sub> and the Ir pptd. by hydrolysis as hydrated dioxide which is ignited in H<sub>2</sub>. W. T. H.

Determination of aluminum and alumina in steels. G. T. Motok and E. O. Walte. *Iron Age* 135, No. 26, 23-5 (1935).—The Al is detd. in a soln. of 10 g. drillings in 100 cc. of 4 N HCl, and the Al<sub>2</sub>O<sub>3</sub> in the residue. The latter is washed successively with 0.6 N HCl, hot water, 3% Na<sub>2</sub>CO<sub>3</sub>, 1.2 N HCl and hot water. It is then ignited, dissolved in strong acid and the soln. made strongly basic with NaOH. After filtering, the Al is pptd. by adding HCl till acid and NH<sub>4</sub>OH till neutral. In the Al detn., the Al is first pptd. as AlPO<sub>4</sub> in the usual way and the ppt. treated the same as the original residue was. The method is not applicable to stainless steels. Thirty-three references. F. G. Norris.

Estimation of minute amounts of arsenic in organic material. K. Winterfeld, E. Dorle and C. Rauch. *Arch. Pharm.* 273, 457-67 (1935).—App. and procedures are illustrated and described for the convenient and accurate mineralization of As in org. material, whereby the As is distd. off as AsCl<sub>3</sub> from the reduced material. For the titrimetric estn. (up to 15 mg. As) a procedure is suggested, which involves a simple specially constructed app. designed to convert the AsCl<sub>3</sub> into AsH<sub>3</sub> and absorb the latter in Hg-Cl<sub>2</sub>. Smith's method for the iodometric titration of AsH<sub>3</sub>,

absorbed by HgCl<sub>2</sub>, has been developed into a dependable procedure. The results attained in the present study indicate that by means of the simplest possible app. a micro method for extg. As is now available. W. O. E.

Analysis of beryllium, aluminum and magnesium in ferrous metals. R. Gadeau. *Rev. met.* 32, 398-400 (1935).—One-half g. of metal is dissolved in HCl or H<sub>2</sub>SO<sub>4</sub>, baked to dryness, didd. and neutralized with NH<sub>4</sub>OH after addn. of 5 g. of tartaric acid. Fe is pptd. with H<sub>2</sub>S, filtered off and washed with H<sub>2</sub>S water. After the H<sub>2</sub>S is boiled off, the soln. is made slightly acid with acetic acid and Al pptd. with 8-hydroxyquinoline. Beryllium is pptd. in the filtrate with Na H<sub>2</sub>NH<sub>4</sub> phosphate. The filtrate is then made basic with NH<sub>4</sub>OH and Mg is pptd. with 8-hydroxyquinoline. J. D. Gat.

Determination of chromium and nickel in ferrous alloys containing manganese and more than one percent of carbon. Tadeusz Cichocki. *Przemysł Chem.* 19, 12 (1935).—From 0.1 to 0.5 g. of alloy is heated for 20 min. with 30 cc. of HClO<sub>4</sub> (d. 1.12), 40 cc. of H<sub>2</sub>O is added to the orange-red soln., contg. CrO<sub>3</sub>, and it is titrated with standard aq. Mohr salt (NH<sub>4</sub>Fe(SO<sub>4</sub>)<sub>2</sub> indicator); the mean error is 0.01-0.03%. Ni is detd. in a similarly prepd. soln. by Chugaev's method. Si, if present, is pptd. in a readily filterable form from the soln. B. C. A.

Iodometric determination of copper. Adjustment of hydrogen ion concentration. Wm. R. Crowell, Thomas B. Hillis, Sidney C. Rittenberg and Raymond F. Evenson. *Ind. Eng. Chem., Anal. Ed.* 8, 9-11 (1936).—In the Park method for detg. Cu, C. A. 25, 660, the use of K biphthalate is unnecessary. The p<sub>H</sub> at the end point is nearer 3.3 than 4.0 and yet the end point is practically permanent. Completely to ext. all Cu from an org. contg. S or As, a double treatment with HNO<sub>3</sub> + HCl or a single treatment followed by the addn. of satd. Br<sub>2</sub> soln. is advisable. W. T. H.

Inorganic complex compounds in analytical chemistry. III. Detection and determination of copper. C. Mahz. *Z. anorg. allgem. Chem.* 225, 386-92 (1935). *cf. C. A.* 27, 5023.—Reinecke's salt, NH<sub>4</sub>[Cr(CNS)<sub>3</sub>](NH<sub>4</sub>)<sub>2</sub> H<sub>2</sub>O, is an excellent reagent for the detection or detn. of Cu. The desired ppt. forms in solns. which are strongly acidic in mineral acid, and ions other than those of Hg, Ti and Ag do not interfere. As little as 3.2 γ of Cu can be detected in the presence of considerably more Bi, Cd, Zn or Pb. To carry out the test dissolve 0.1 g. of K<sub>2</sub>SnCl<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>, by heating with 1 cc. of N HCl, and add 1-2 cc. of the HCl soln. to be tested. After mixing, add 1 drop of a freshly prepd. and filtered soln. of 0.2 g. Reinecke's salt in 10 cc. of hot N HCl. A silky, lustrous, yellow ppt. soon forms or, when very little Cu is present, a yellow color is visible. To det. Cu, take a soln. contg. about 25 mg. Cu, and not over twice this quantity, in 100 cc. and add HCl until the soln. is at least N but not over 3 N in this reagent. If a ppt. of PbCl<sub>2</sub> forms, filter it off and wash with N HCl. Heat to boiling, introduce solid K<sub>2</sub>SnCl<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub> until the green soln. is colorless and add 0.2-0.5 g. of the salt in excess. Meanwhile dissolve for each 10 mg. Cu, 0.1 g. of Reinecke's salt in 10 cc. of 1-2 N HCl at 50°, filter and allow this soln. to drop into the soln. of cuprous salt. Boil for a short time, allow to stand 5 min. and make sure that the pptn. is complete by adding more reagent. Filter through a sintered glass filter, wash the voluminous yellow ppt. with cold 2 N HCl until the filtrate is free from Sn and then with hot water. Dry at 110°. The ppt. contains 16.26% Cu when weighed in this form and not 16.67% Cu according to the formula Cu[Cr(CNS)<sub>3</sub>](NH<sub>4</sub>)<sub>2</sub>. The ppt. can also be dried by washing with alc. and ether. A more rapid method of finishing the analysis consists in dissolving the Cu ppt. in concd. HNO<sub>3</sub>, oxidizing the Cr to chromic acid by heating the didd. soln. with HCl and sufficient KBrO<sub>3</sub>, decomg. the excess bromate by evapg. with (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and then detg. the CrO<sub>3</sub> iodometrically. One cc. 0.1 N Na<sub>2</sub>SO<sub>3</sub> = 1.589 mg. Cu. W. T. H.

Determination of the total oxidizing power, the nitrite, the ozone and the total chlorine content of ordinary and poisoned air. H. Cauer. *Z. anal. Chem.* 103, 321-34 (1935).—The method of detg. the total oxidizing power of

the air is described and the expts. performed to show the accuracy and reliability of the procedure are summarized. In brief, the method consists in passing air through a suitable wash bottle con'd. dil. KI soln with sufficient added acid to make the pH below 2.8. The I content of the KI soln is det'd colorimetrically by comparing the color of the  $\text{CHCl}_3$  ext. after treating with a little nitrite, shaking and centrifuging, both before and after the air has passed through the KI soln. Any  $\text{I}_2$  developed as a result of oxidation of KI by the air is carried away mechanically and is det'd by difference. Under the prescribed precautions a good indication of the quantity of oxidizing gas present in the air is obtained. The removal of such gas is practically complete but, to some extent at least, the values obtained are only relative and do not give the actual content of oxidizer present. The procedure, however, appears to be capable of practical application. W. T. H.

Field method of determining gold content of ore. K. Pozharitzkii. *Soviet Zolotoprom* 1935, No. 6, 20-2.—A review. S. L. Madorsky

Rapid method for the volumetric determination of indium. Henry B. Hope, Madeline Ross and J. E. Skelly. *Ind. Eng. Chem., Anal. Ed.* 8, 512 (1936).—The method is based on the titration of In acetate soln with  $\text{K}_2\text{Fe}(\text{CN})_6$  to an end point with diphenylbenzidine which changes from green to blue. W. T. H.

Colorimetric determination of iron by means of ferric thiocyanate. M. Bertaux. *Documentation scientifique* 4, No. 32, 49-52 (1935). *Chimie & Industrie* 34, 1058-9.—The usual method has been modified with a view to eliminating the errors due to differences in color resulting from differences in the concn. of alkali thiocyanate and in the nature and concn. of the acid used for dissolving  $\text{Fe}(\text{OH})_3$ . Dissolve the sep'd  $\text{Fe}(\text{OH})_3$  (contg. not more than 0.04 g. Fe) in 30 cc. of 50%  $\text{HCl}$ , add 5 cc. of a mixt. of 2 parts of 36%  $\text{Be HNO}_3$ , 2 parts of 3%  $\text{H}_2\text{O}_2$  and 1 part  $\text{H}_2\text{O}$ , dil. to 500 cc. with dist'd or Fe-free tap water, add 25 cc.  $\text{NH}_4\text{CNS}$  (exactly 175 g. per l.), and dil. to 900 cc. To each of 2 other flasks add 30 cc. of 50%  $\text{HCl}$  and 5 cc. of the  $\text{HNO}_3\text{-H}_2\text{O}_2\text{-H}_2\text{O}$  mixt., dil. to 500 cc., add 25 cc.  $\text{NH}_4\text{CNS}$ , dil. to 900 cc., to 1 of the flasks add a 0.1 g. per l. soln. of  $\text{FeCl}_3$  till the color is just slightly less than that of the sample, and in the other sufficient of the soln. to give a slightly deeper color, and such that the difference in the amts. added to the standards does not exceed 0.001 g.  $\text{FeCl}_3$ . A. Papineau-Couture

Accurate separation of precipitated mercuric sulfide and sulfur in the gravimetric determination of mercury. Earle R. Caley and M. Gilbert Burford. *Ind. Eng. Chem., Anal. Ed.* 8, 43 (1936).—Instead of attempting to dissolve out the S it is recommended to weigh the ppt. of  $\text{HgS} + \text{S}$ , dissolve the  $\text{HgS}$  in cold, conc'd.  $\text{HI}$  and weigh again. The results obtained with samples of com.  $\text{HgS}$  apparently justify this procedure. W. T. H.

Potentiometric determination of quadrivalent osmium with chromous sulfate. Wm. R. Crowell and Harlan L. Baumbach. *J. Am. Chem. Soc.* 57, 2007-9 (1935).—App. and procedure for storing and handling  $\text{CrSO}_4$  solns for standardizing with  $\text{K}_2\text{Fe}(\text{CN})_6$  and  $\text{K}_2\text{Cr}_2\text{O}_7$  and for detg. Os in  $\text{K}_2\text{OsBr}_6$  and  $\text{K}_2\text{OsCl}_6$  by potentiometric titration. In the reaction, quadrivalent Os is reduced to the trivalent state and bivalent Cr is oxidized to the trivalent state. The titer of the  $\text{CrSO}_4$  soln. remained const. for 1 month. The error in the titration with 0.01 N  $\text{CrSO}_4$  was less than 0.2%. W. T. H.

Determination of radium in carnotite and pitchblende. L. D. Roberts. *Ind. Eng. Chem., Anal. Ed.* 8, 5 (1936).—Ra is det'd electroscopically by means of radon introduced into an electroscope chamber. With a Lind electroscope, accurate values can be obtained. Fuse the ore with  $\text{Na}_2\text{CO}_3$ , chill the melt suddenly by immersing the Pt boat in cold water, dissolve the melt in  $\text{HNO}_3$  and collect the radon. W. T. H.

Determination of rhenium. I. Qualitative. Loren C. Hurd. *Ind. Eng. Chem., Anal. Ed.* 8, 11-15 (1936).—The bibliography of Re is given and most all of the tests that have been proposed are considered critically. The colorimetric detn. of Re as developed by Geilmann,

Wrigge and Weibke, *C. A.* 27, 42, is the most convenient lab. test for the rapid detection of heptavalent Rh. In the Noyes-Bray scheme of analysis, Re was found in the Te group by Kao and Chang, *C. A.* 28, 3686<sup>1</sup>; in the Prescott and Johnson scheme, the element will appear in the test for As. The spectroscopic identification is easy when much Re is present but considerable trouble in identifying 4 of the lines is encountered when much Fe, Mn or Mo is present. If solns. contg. Mo and Re are treated with Ft xanthate, a Mo complex is formed which can be removed by shaking with  $\text{CHCl}_3$  and the Re can then be detected easily. W. T. H.

Colorimetric determination of silver in minerals. C. F. Miller. *Chemist Analyst* 25, No. 1, 8-10 (1936).—From 1 to 20 mg. per l. of Ag can be det'd by reducing the  $\text{Ag}^+$  to a clear yellow sol with  $\text{Na}_2\text{S}_2\text{O}_4$  in the presence of a stabilizer such as gelatin. The Ag content is det'd by comparison with standard solns. treated similarly. The Ag is first ppt. as  $\text{AgBr}$ , the small ppt. is dissolved by long digestion with strong  $\text{NH}_4\text{OH}$ , 0.03-0.06 g. of the powder reagent is added and the sample is heated to 50°. W. T. H.

Rapid method for the determination of titanium. Henry B. Hope, Raymond F. Moran and Arthur O. Ploetz. *Ind. Eng. Chem., Anal. Ed.* 8, 48-9 (1936).—Instead of reducing the Ti in a Jones reductor and titrating with an oxidizing agent such as  $\text{KMnO}_4$ , it is recommended to reduce with liquid Zn-amalgam and titrate the soln. with ferric alum to an end point with KCNS soln. (cf. Knecht and Hibbert, *Ber.* 36, 146 (1903)). W. T. H.

The iodometric titration of zinc according to Rudolf Lang. Chr. W. Raadsvelde. *Chem. Weekblad* 32, 653-7 (1935).—The theoretical factor, rather than the empirical factor given by Lang (*C. A.* 24, 1055), should be used in computing the results, but the result of many analyses indicate that the method is useful only as an approximation method. N. Bekkedahl

The determination of phosphorus by the x-ray spectrograph. A. Merkel. *Ber. Naturf. Ges. Freiburg* 34, 249-52 (1934). *Neues Jahrb. Mineral. Geol.*, Referate I, 1935, 131. Spectrographic analysis of an eruptive rock for  $\text{P}_2\text{O}_5$  gave 0.27%, chem. analysis 0.21%. An intensity factor is necessary. J. F. Schaerer

The determination of sulfur in soluble sulfides by photometric titration. Shiro Hirano. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 598-601 (1935), cf. *C. A.* 29, 5379<sup>2</sup>.—As standard titrating solns. (1)  $\text{Pb}(\text{NO}_3)_2$ , (2)  $\text{HgCl}_2$  and (3)  $\text{BiCl}_3$  were used. Data show that methods (1) and (2) can be used in the presence of reducing agents such as sulfites, thiosulfates, etc. All 3 methods are suitable for detg. S in sol. sulfides in solns. of deep color or contg. a protective colloid. Karl Kammermeyer

Analysis of calcium phosphate. Hans Trapp. *J. prakt. Chem.* 144, 93-105 (1935).—In the analysis of a sample of  $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$  good results can be obtained in detg. Ca by dissolving the sample in 30%  $\text{AcOH} + 1$  cc. of conc'd.  $\text{HCl}$ , dilg. to 400 cc. and adding 4 g. of  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . Weighing as  $\text{CaC}_2\text{O}_4$  after moistening the ignited ppt. with  $(\text{NH}_4)_2\text{CO}_3$  soln. and heating again is advocated. Fairly good results are also obtained by pptg. and weighing the Ca as  $\text{Ca}_3(\text{PO}_4)_2$ . To accomplish this, dissolve the sample in dil.  $\text{HCl}$ , dil. with hot water to 250 cc. and add  $\text{NH}_4\text{OH}$  dropwise until a neutral reaction to phenolphthalein is obtained, then add 25 cc. of 10%  $\text{NH}_3$  soln. in excess. After standing overnight, filter and wash the ppt. with hot water. The sample can also be analyzed volumetrically by adding a measured vol. of  $\text{NaOH}$ , filtering after 12 hrs., titrating the excess  $\text{NaOH}$  with acid and detg. thereby the quantity of base required to convert  $\text{CaHPO}_4$  into less sol.  $\text{Ca}_3(\text{PO}_4)_2$ . Some studies were carried out with respect to weighing the yellow phosphomolybdate ppt. The difficulty in obtaining a const. wt. after drying is not due to the variability of the Mo/P ratio but rather to a reduction and formation of a blue comp'd. insol. in  $\text{NH}_4\text{OH}$ . By dissolving out the unchanged  $\text{NH}_4$  salt, it is fairly easy to oxidize the blue comp'd. but no recommendation is made with respect to preventing its formation. W. T. H.

Determination of water in glycerol. C. P. Spaeth and

G F Hutchison *Ind Eng Chem, Anal. Ed* 8, 29-32 (1936) —A crit study of the modified Rojahn method, C A 14, 2602, shows that it only approximates the true  $H_2O$  content of glycerol. A new method is described which is based on the removal of water at  $100^\circ$  under 2-3 mm pressure. The volatility of glycerol and low-boiling impurities is controlled by a condenser. The error of the method is not over 3% when the water content is 0.5 to 5% W T H

Source of loss of ammonia in Kjeldahl distillations. Method of eliminating this loss. Høke S Miller. *Ind Eng Chem, Anal. Ed* 8, 50-1 (1936) —There is danger of some  $NH_3$  being lost during the first few min of a Kjeldahl distn. This loss can be prevented to a large extent by using a delivery tube contg small holes which cause the first air bubbles that come over to be so broken up that small quantities of  $NH_3$  are more readily absorbed in the acid W T H

An x-ray investigation of the solid-solution nature of some nitrate contaminated barium sulfate precipitates. Geo H Walden, Jr., and M U Cohen *J Am Chem Soc* 57, 2591-7 (1935) —The app used is described. A series of ppts contaminated with nitrate were prepd and examd. The compn of the ppts with respect to nitrate appears to be a function of the  $Ba^{++}$  concn from which the ppt is formed and the nitrate concn has little effect. Apparently the contaminant enters the  $BaSO_4$  lattice with the formation of a solid soln. The role of such a solid soln in the general problem of ppt contamination is discussed W T H

Method of analysis for fluoride. Application to determination of spray residue on food products. W M Hoskins and C A Ferris *Ind Eng Chem, Anal. Ed* 8, 6-9 (1936) —The method of Armstrong, C A 23, 2397, 5273, which involves the titration of the  $F^-$  in 50% alc with  $Th^{+++}$  soln in the presence of Na alizarinsulfonate was studied and modified. In the revised form the method is capable of detg 0.006 to 0.09 mg of F in 5 cc of soln, within 1% of the truth. In a vol of 50 cc equally accurate results were obtained with 0.057 to 0.76 g of F W T H

Iodometric determination of sulfates. S Rivas Goday and A Calatayod *Bol Farm Militar* 13, 361-4 (Dec. 1934), *Chimie et Industrie* 34, 792 —A modification of Hummer's method (*Ann J. Sci and Arts* 114, 478 (1877)) in which the soln is made basic to phenolphthalein with NaOH after adding  $BaCl_2$  and before adding  $K_2Cr_2O_7$  A Papineau Couture

Rapid gravimetric determination of silicic acid. N A Tananaev and M K Buichkov *Z anal Chem* 103, 349-53 (1935) —The results of 20 analyses for detg the  $SiO_2$  content of limestone, quartzite, glass, clay and loam prove that good results can be obtained in about 2.5 hrs by a treatment which consists in fusing the sample with a considerable excess of  $Na_2CO_3$ , pouring the melt in a thin layer on to a flat piece of porcelain. Ni, polished steel or Pt, treating with concd  $HNO_3$ , eventually dilg with at least 4 times as much water, adding 6-15 cc of hot 0.1% gelatin soln, filtering, washing with 3%  $HNO_3$ , igniting and weighing W T H

A new test for tartaric acid. Maurice Pettez *J pharm chim* 21, 542-6 (1935), cf C A 30, 1517 —The reagent (A) is a soln of 2 g resorcinol and 10 g KBr in 100 cc  $H_2O$ , with 1 cc  $H_2SO_4$  added when soln is complete. To apply the test, add 0.1 cc of A to 2 cc  $H_2SO_4$  (d 1.84), then 0.1 cc (2 drops) of 2% tartaric acid (B) soln and heat on a steam bath. A pale blue color appears in 1 min, it becomes deeper in 5 min, and intense when cautiously heated over a direct flame. When neutralized by NaOH, a violet tint is obtained. Addn of 1 cc  $H_2O$  to the blue soln changes it to current-red, addn of 10 cc  $H_2O$  causes it slowly to fade. The blue color is formed with a 1% soln of B in 2 mm, with a 0.1% soln in 5 mm. The reaction is sp for resorcinol, also for B except in the case of chloral, with which the same tints are produced (cf Ware, C A 30, 953) but which can previously be removed by heat. Hypochlorites, chlorates, nitrates and permanganates can be eliminated by

previous reduction with a Zn-Cu couple (5 cc of a soln contg Zn and 4.5 drops of aq. satd  $CuSO_4$  soln). The test permits detection of B in presence of bromides, bromates, nitrites and ferric salts S Waldbott

Eliminating the effects of phosphate radical in qualitative analysis. I-V Saburo Ichimaru *Science Repts Tohoku Imp Univ, First Ser* 24, 426-80 (1935) —See C A 27, 3806, 5029, 5670, 28, 1950<sup>4</sup>, 29, 2884<sup>4</sup> G G

The separation and determination of the metal ions and phosphate radical in the presence of one another. I, II Saburo Ichimaru *Science Repts Tohoku Imp Univ, First Ser* 24, 481-92, 493-511 (1935) —See C A 28, 6392<sup>4</sup>, 29, 2848<sup>4</sup> G G

Detection of the oxalate ion. N A Tananaev and A A Budkevich *Z anal Chem* 103, 353-5 (1935) —Under the conditions given, neither indigo nor oxalic acid is oxidized by  $Cr_2O_7^{--}$  but when both these substances are present, both are oxidized. By the resulting decolorization of the indigo soln, very little oxalic acid can be detected. If  $S^{--}$ ,  $SO_3^{--}$  or  $S_2O_3^{--}$  is present there is interference because these anions react with  $Cr_2O_7^{--}$ . When they are present proceed as follows: Take 2-3 cc of the soln to be tested and add an excess of 7 N  $H_2SO_4$ . Boil until there is no more odor of  $H_2S$  or  $SO_2$ . Filter if there is much S pptd. To the clear soln add some 0.01 N  $K_2Cr_2O_7$  soln. In another test tube heat 0.01 N  $K_2Cr_2O_7$  with enough indigo soln to give a distinct blue color. Now mix the 2 solns and if the soln is 0.01 N in  $C_2O_4^{--}$  decolorization results within 2 min. As little as 0.03 mg of  $H_2C_2O_4 \cdot 2H_2O$  can be detected W T H

Destruction of organic matter prior to the determination of mercury in organic compounds. C V Bordenave *Ann sci univ Jassy* 20, 129-31 (1935) —Treat 0.3-0.5 g of the compd in a 250-cc Erlenmeyer flask with 1-1.5 g of powd  $KMnO_4$  + 10 cc. of concd  $HNO_3$ . After the mixt stands 30 min at room temp add 1-2 cc of concd  $H_2SO_4$  and allow to stand 15-20 min longer. Then dil with 50-60 cc of water and heat on the water bath. Decolorize the soln with 3%  $H_2O_2$  and det. Hg by the thiocyanate titration. If halide is present it is necessary to heat with  $H_2SO_4$  more strongly, in order to remove all halide. The results obtained in the analysis of several Hg org compds were satisfactory. W T H

Determination of organic sulfur by the liquid ammonia-sodium method. F J Sowa, V G Aracchi and J. A. Newland *Ind. Eng Chem, Anal. Ed* 8, 49-50 (1936) —Compds. sol in liquid  $NH_3$  which do not form intermediate products that cannot be reduced easily can be detd accurately by reducing with Na whereby either  $Na_2S$  or  $Na_2SO_3$  is formed. By adding  $Na_2O_2$  and evap., the S is oxidized to sulfate and can be detd. as  $BaSO_4$ . Excellent results were obtained with 16 typical org compds W T H

Determination of formic acid in pyrolytic liquors. Herman D Weihe and P. Burke Jacobs *Ind Eng. Chem, Anal. Ed* 8, 44-7 (1936) —Neutralize with NaOH and add 20% in excess. After 24 hrs. add 5 cc of syrupy  $H_3PO_4$  and distil by heating to  $150^\circ$  in a glycerol bath. Neutralize a suitable aliquot with  $Ba(OH)_2$  to a phenolphthalein end point and evap to dryness on the water bath. Dissolve the residue in 50 cc of warm water and transfer to a reaction flask. Add a small piece of paraffin to reduce foaming and 5 cc of NAOH. Dil to about 100 cc and boil 10 min while passing  $CO_2$ -free air through the app to remove any dissolved  $CO_2$ . Then add 20 cc of  $Hg(OAc)_2$  oxidizing soln (100 g  $H_2O$  dissolved in 11 of 0.5 N NAOH and boiled 1 hr to remove  $CO_2$ ). By means of the Hg salt, the formic acid is oxidized to  $CO_2$  which is absorbed in a known vol of standard  $Ba(OH)_2$  soln and the excess is detd by titration. Small quantities of formic acid can be detd within 3% of the truth and large quantities within about 0.5% W T H

The use of the original Hanau lamp in qualitative analysis. I. Photoanalysis in the pyridine and indole series. Hans Freytag *Z anal Chem* 103, 334-40 (1935) —If Schleicher & Schüll filter paper No. 589 is dipped in an approx 0.2% soln of pyrazolone and is then dried without exposure to daylight, a very sensitive test paper for pyri-

dune and its derivs. is obtained. Under the ultraviolet rays of the quartz lamp, a spot produced with 0.01 cc. of a soln. contg. pyridine will soon show a red or gray coloration, which will become changed to an ultramarine blue in less than 10 min. The test paper, however, must be kept in the dark as it turns brown when exposed to light and thereby loses its ability to react with pyridine. If 0.01 cc. of a soln. in alc. or in aq. alc. of indole is dropped upon a Schleicher & Schüll filter paper and then treated with a drop of the same size of dil.  $H_2SO_4$ , exposure to the rays of the quartz lamp will soon cause the appearance of a pink or flesh-colored spot. The reaction takes 250 times as long in diffuse sunlight as it does under the quartz lamp.

**W. T. H.**  
Potentiometric determination of mercaptans. Miroslav W. Tamele and Lloyd B. Ryland. *Ind. Eng. Chem., Anal. Ed.* 8, 16-19 (1936).—Use enough 0.1 N NaOAc dissolved in 96% EtOH to dissolve the sample. Titrate with 0.01 N  $AgNO_3$  dissolved in isopropyl alc. contg.

about 9% water. Det. the end point potentiometrically with an electrode of Ag as indicator and a Hg half cell as the other electrode. The results are good. W. T. H.

Colorimetric determination of salicylic acid. P. Karsten. *Chem. Weekblad* 32, 526 (1935).—Heestermans observation was confirmed (C. A. 30, 702). For detn. of salicylic acid colorimetrically in 50 cc. of soln. in is used 2 cc. of a soln. contg. 54 mg.  $FeCl_3 \cdot 6H_2O$  and 10 cc. AcOH in 100 cc.

**B. J. C. van der Hoeven**  
Color reaction for detection of cyclopentadiene. Boris N. Alanas'ev. *Ind. Ing. Chem., Anal. Ed.* 8, 15 (1936).—Mix 1 drop of the liquid with 1 cc. each of  $CHCl_3$  and glacial AcOH and treat cautiously with 2-3 drops of concd.  $H_2SO_4$ . As little as 0.1 mg. of the compd. suffices to give a distinct violet coloration. W. T. H.

Detn. of phosgene in gases from expls. fires extinguished with  $CCl_4$  fire-extinguisher liquid (Yant, et al.) 13.  
Stepwise titration of a dibasic acid (Kilpi) 2

## 8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND J. F. SCHAUER

**Aurosiridum** O. Zvyagintsev. *Sotzislst. Rekonstruktsiya*: Nauka 1935, No. 3, 176.—A new mineral has been found composed of Ir 52, Os 25, Au 19 and Ru 3.5%, it represents a natural alloy in the form of grains 0.2-2 mm. in diam. B. V. Shvartzberg

**Todorokite**, a new manganese mineral from the Todoroki mine, Hokkaido, Japan. Toyotomi Yoshimura. *J. Faculty Sci., Hokkaido Imp. Univ. Ser. IV* 2, 279-37 (1934). *Neues Jahrb. Mineral. Geol., Referate I*, 1935, 147.—Todorokite is monoclinic with cleavages on 100 and 010. It is sol. in HCl and in hot concd.  $H_2SO_4$ , and analysis gave  $K_2O$  0.54,  $Na_2O$  0.21,  $MgO$  1.01,  $CaO$  3.28,  $BaO$  2.05,  $Al_2O_3$  0.28,  $Fe_2O_3$  0.20,  $MnO$  65.89,  $O$  12.07,  $H_2O$  +9.72,  $H_2O$  -1.56,  $SiO_2$  0.45,  $TiO_2$  and  $CO_2$  traces,  $P_2O_5$  0.42,  $SO_3$  0.28, invol. 1.28, sum 93.24%. Formula  $3(Mn_2O_3 \cdot 3MnO_2 \cdot 2H_2O) \cdot 2RO \cdot MnO_2 \cdot 2H_2O$ , with  $R = Ca, Mg, Ba$  and  $Mn^{4+}$ . J. F. Schauer

**Mineral formation in the system,  $Al_2O_3$ - $SiO_2$ - $H_2O$**  W. Noll. *Neues Jahrb. Mineral. Geol., Beilage-Bd. 70A*, 65-115 (1935).—Böhmite ( $Al_2O_3 \cdot H_2O$ ) is the stable hydrothermal hydrate of alumina. The following cryst. phases were encountered: bayerite ( $Al_2O_3 \cdot 3H_2O$ ), corundum ( $Al_2O_3$ ), kaolin ( $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ ), a "pyrophyllite mineral" ( $Al_2O_3 \cdot 4SiO_2 \cdot H_2O$ ), montmorillonite ( $Al_2O_3 \cdot 4SiO_2 \cdot H_2O \cdot nH_2O$ ).  $SiO_2$  remained amorphous. J. F. Schauer  
**Almandine** from some deposits of North Caucasus and the Ukraine. D. Serdyuchenko. *Mem. Soc. russe Mineral.* 62, 97-101 (1933). *Neues Jahrb. Mineral. Geol., Referate I*, 1935, 167.—Chem. analyses of almandine are given. J. F. Schauer

**X-ray examination of phyllite from clay-with-flints of Vernon (Eure).** Boris Branjnikov and Pierre Urban. *Compt. rend. soc. géol. France* 1934, 203-5.—A red-brown phyllite occurs in the clay-with-flints, filling cavities in the chalk near Vernon. Samples of (1) the crude clay, (2) the phyllite dried and powdered and (3) as (2) but completely decolorized by treatment with HCl, were examd. by x-rays. All gave identical spectra. The color is due to leaflets of  $Fe_2O_3 \cdot nH_2O$ . The spectra are also identical with that of typical montmorillonite, which is decolorized by HCl exactly as this phyllite. This makes it doubtful whether montmorillonite is, as hitherto supposed, a hydrated ferric silicate. C. A. Silberrad

**X-ray investigation of turquoise** Fritz K. Mayer. *Chem. Erde* 9, 311-17 (1935).—X-ray examn. of 12 natural (d. 2.687-2.816) and 3 synthetic (d. 2.46-3.173) turquoises and of wavelite (d. 2.416) and chalcocyanite (d. 3.211) showed all to give similar diagrams with numerous lines, slight differences in those of the turquoise being due to slight differences in compn. (mainly varying proportions of Fe, turquoise and chalcocyanite being connected by an isomorphous series). Removal of  $H_2O$  (17.69-20.86% in the natural, 11.96-12.20 in the artificial) by heating

turquoise in a flame caused no distortion in the lattice, but a diminution in grain size. C. A. Silberrad

**X-ray investigation of turquoise and other phosphates.** Hermann Jung. *Chem. Erde* 9, 318-20 (1935), cf. C. A. 26, 3754.—J. doubts Mayer's conclusion (preceding abstr.) as to the similarity of the Debye-Scherrer diagrams of turquoise and wavelite, finding that of  $FePO_4$  distinctly different from that of  $AlPO_4$ , which again differs from that of turquoise. Moreover the great difference in d. and of turquoise (2.81 to 1.62) and wavelite (2.33 and 1.53) points to divarication, while the diagram of harborite (cf. Brandt, C. A. 27, 1795) of compn. similar to that of turquoise differs from that of wavelite. The suggestion that the apparent absence of distorting effect on the lattice of the removal of  $H_2O$  from turquoise points to the  $H_2O$  being zeolitic is inconsistent with the absorption spectrum, which points to the presence of (OH) and not of  $H_2O$ . C. A. Silberrad

The indices of refraction of scheelite from Japan Junpei Harada. *J. Faculty Sci., Hokkaido Imp. Univ. Ser. IV* 2, 279-87 (1934); *Neues Jahrb. Mineral. Geol., Referate I*, 1935, 163.—Precise optical constns. are given for scheelite from Gyōzōyōmen, Japan. J. F. Schauer

**Critical observations of mineral behavior when associated with superheated water.** Arthur J. Crowley. *Eng. Mining J.* 137, 27-30 (1936).—The phenomenon discussed is believed to function importantly in the processes of mineral depositions in nature. The S-luqueifer described and illustrated permits the production of 99.5% S from a 80% S flotation concentrate from Nevada ores. It seems likely that similar conditions may have caused the formation of certain S deposits. W. H. Boynton

**Geology and mines of environs of Giromang (Belfort).** Ch. Thurion. *Bull. soc. géol. France* [5], 5, 33-63 (1935).—An account is given of mines 15 km. north of Belfort, which in early times were an important source of Ag, Cu and Pb. The veins contg. galena, chalcocyanite, tetrahedrite and sphalerite, with occasionally pyrite, malachite, pyromorphite, cerussite, arsenopyrite with some danite, chalcocite, zinkenite and cinnabar, with quartz and fluorite and less commonly barite and siderite as gang. The country rock is mainly volcanic—dacite, albized rocks, tufts and breccias and the ores have resulted from hydrothermal action. Many of the mines were abandoned early in the 17th century, and all by the middle of the 18th, save for unsuccessful attempts at reopening since. C. A. S.

A tin occurrence in the limestone at Arandis, German Southwest Africa. Paul Ramdohr. *Neues Jahrb. Mineral. Geol., Beilage-Bd. 70A*, 1-48 (1935).—The Sn occurrence is characterized by a large suite of contact-metamorphic minerals, particularly those contg. Cl, F and B. An unknown mineral believed to be  $Sn_2S_3$  is reported. J. F. S.

Radrum ores V A Zilbermintz *Sotnislal Rekonsstruktsiya i Nauka* 1935, No 4, 117-20 —A review of the world's production of Ra and a description of 2 beds of yellow U-V minerals discovered in U S S R

B V Shvartzberg  
Peculiar deposits of bauxite in Provence Jacques de Lapparent *Compt rend soc géol France* 1934, 64-6 —The bauxite at le Montagne de Regaigues (Bouches-du-Rhône) occurs in 3 veins in Jurassic dolomite, 0.5-4 m. thick and 1.5-3.5 m. apart East of Auroul on the Bouches-du-Rhône Var border it occurs in large pockets contig. alternate layers of bauxite contig lignite, lignite fragments cemented by bauxite and psilolitic bauxite. In both cases it is thought that preexisting bauxite has been washed into subsequently formed cavities in the dolomite

C A Silberrad  
Bauxites of the Lika, Yugoslavia Georges Emethanoff *Compt rend* 201, 1405-7 (1935) —The bauxite deposits ("pockets of decalcification") east of the Velebit Range in the valley of the Lika are described and shown to be the oldest in the Balkan peninsula, probably Triassic

C A Silberrad  
Gypsum deposits of central Pyrenees J Durand *Compt rend soc géol France* 1934, 165-8 Léon Bertrand *Ibid* 181 3 —Deposits of gypsum at Arnavé, Milhas and (with anhydrite) at Bedeilhac are described C A S

The origin and composition of clays D G Runner *Roads and Streets* 79, 53-6 (1936) —A discussion of origin, compn and classification of the various types of clay common to the U S, with 24 references K K

The sand from Viareggio—physical, chemical and mineralogical properties S Bonatti *Atti Congr Naz Idrol Chim e Terapia Fisica, Viareggio-Montecatini* 1932, 13 pp, *Neues Jahrb Mineral Geol*, Referate II, 1934, 871 —A complete chem analysis is given showing  $\text{SiO}_2 = 70.02\%$  J F Scharrer

A French deposit of diatomite Jacques de Lapparent and R Hovasse *Compt rend soc géol France* 1934, 205-6 —A deposit some 3 m. thick, at Saint-Laurent la-Vernède, Gard, is described C A Silberrad

Sinaia strata of Introrsura Buzau, Romania Constantin Zaharesco *Compt rend soc géol France* 1934, 74-6 —Coal of excellent quality occurs in these strata in the marl. It has a conchoidal fracture and appears to be of sapropelic origin, consisting of pollen and spores in a carbonaceous coagulum Analysis shows 1.77% S, 4.48%  $\text{H}_2\text{O}$  and 7.365-7.635 cal As a rule it occurs in unworkably small deposits, but in larger amts near the river Sita and perhaps elsewhere C A Silberrad

Probable origin of bitumen found on Kerguelen Is E Aubert de La Rüe *Compt rend soc géol France* 1934, 251-2 —The block of bitumen found on Kerguelen Is, thought at first to have been left there by some boat, is, in view of similar occurrences on the coasts of Australia, New Zealand and Chili, now considered to be probably derived from some submarine deposit The block in question is impregnated with sand and yielded 46% of oil,  $d_{44} 0.883$  C A Silberrad

Bituminous shales of Saulx-de-Vesoul Louis Longchambon *Compt rend* 201, 1417-19 (1935) —In the neighborhood (mainly S W. and S E) of Saulx-de-Vesoul, Haute Saône 18 bores show the presence of bituminous Toarcian shales in thicknesses of 2.5-33 m at depths varying from 0 to 98 m (usually less than 40 m) The content of oil averages 4.9% (3.6-6.3). Workable shale is proved over an area of about 3350 hectares, contg an estd amt of 25,000,000 metric tons of crude oil C A Silberrad

Geology of Texas Panhandle oil and gas field Henry Rogatz *Bull Am Assoc. Petroleum Geol.* 19, 1089-1109 (1935) —The gas is divided into 2 distinct zones, "sweet" and "sour" The "sour" gas contains 2-500 grams of  $\text{H}_2\text{S}$  per 100 cu ft. The sourdest gas is located furthest west and low in the structure Alden H Emery

The heavy minerals of the "Ernpura" granite and microgranite of the Danta State (N. Guyrat) N. L. Sharma and S. Purkayastha *Proc. Indian Acad Sci.* 2B, 369-76 (1935), cf C A. 29, 4701 —A number of

1 samples of rock from various locations in the district were ground and the minerals with sp. gr. greater than 2.85 were sepd with CHBr<sub>3</sub>. These heavy minerals were zircon, sphene, fluorite, epidote, zoisite, biotite, hornblende and occasionally a trace of some other mineral Descriptions of slides made from these minerals are given The heavy minerals composed 0.4-15% of the granites and 0.4-2% of the microgranites John B. Milbery

Brazilian rocks (collected by B. von Greyberg) Paula Schneiderhohn *Neues Jahrb Mineral Geol., Beilage-Bd* 70A, 151-201 (1935) —Twenty new rock analyses J F Scharrer

Eruptive rocks of Cape Mazagan B Yovanovitch and R Frey *Compt rend soc géol France* 1934, 20-1 —Monzonitic microgranite, rhyolite and (probably) dacite have been found, the last (at the base of the dolomite) contains veinlets of spherulitic and axiolitic structure C A Silberrad

Pre Cambrian rocks of Jebel Sagho L Clariond and St Ghika-Budesti *Compt rend soc géol France* 1934, 208-10 —These rocks consist (from below upward) of gabbros, granites (pyroxene and dioritic), monzonite and rhyolites (probably rhyodacite and dellemites) All of them are probably derived from the same basic magma which has been subjected to differentiation *in situ* C A Silberrad

Studies in the region of the Drosendorf map sheet 4 II The gabbro of Nonndorf and Kurlupp Otto Hackl and Leo Waldmann *Verh Geol Bundesanst Wien* 1931, 160-5, *Neues Jahrb. Mineral Geol., Referate II*, 1934, 885 —An analysis of gabbro from Nonndorf is given J F Scharrer

Alkaline rocks from the watershed of the river Chodschatschan (Ferghana). A Moskvyn *Abh Pamir Expedition* 1930, IV, *Petrog Mineral Akad Wiss Leningrad* 1932, 1-98; *Neues Jahrb Mineral Geol., Referate II*, 1934, 896-8 —An analysis of nephelitesyenite is given J F Scharrer

Petrography of the region between the Dsungarian Desert and the Kaschfluss (Irenkhaburghun group of the Tianshan Mts) K Willmann *Neues Jahrb Mineral Geol., Beilage-Bd* 70A, 116-50 (1935) —Analyses of 2 types of keratophytic eruptive rocks are given J F Scharrer

6 Phonolites in the neighborhood of Mazagan B Yovanovitch and R Frey. *Compt rend soc géol France* 1934, 163-4 —Phonolite occurs east of Am Tiourghet as isolated fragments of vesicular lava consisting of a feldspathic paste contg minute crystals of eugite augite, feldspar often in skeleton crystals and nephelite, with phenocrysts of augite and much hauynite, this last forming large pinkish gray crystals with numerous inclusions C A Silberrad

7 Post-Triassic granite of Cambodia and its contact rocks Y. and J Gubler *Bull soc géol France* [5], 4, 801-25 (1935) —The intrusions of granite into Permian and Triassic rocks (mostly sandstones, but some limestone) are described, with the resulting metamorphism and formation of biotite, amphibole, pyroxene- and tourmaline-hornfels, cryst limestone and at one place a rock analogous to diagenite resulting from fumarole action on a marl and contg (besides quartz and orthoclase) diopside, wollastonite, sphene and prehnite C A Silberrad

Sericite schists of southern Cevennes and grano-gneissic massif of Segala and Levezou A Demay *Bull soc géol France* [5], 5, 3-18 (1935) —Included is a detailed account of the metamorphism caused by the intrusion of porphyroid granite into sericite and mica schists and quartzite C A Silberrad

9 Weathering of rocks and the composition of clays Antti Salminen *Maatalouskoulantoksen Maatutkimusosasto Agrgeol Julkaisu* (Soil Div Central Agr Expt Sta Finland) No 40, 1-149 (1935) (available in English) —Phenomena influencing the compn of clay sediments were studied and the chem, mineralogical and mech compn of Finnish clays at various places are presented A sample of Rapakivi (a diabase) was found to have lost  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$  and Fe in the process of weathering and to

have gained in  $\text{Fe}^{+++}$ ,  $\text{SiO}_2$  and  $\text{K}_2\text{O}$ . In a sample of granite  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{FeO}$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$ ,  $\text{P}_2\text{O}_5$  and  $\text{TiO}_2$  decreased while  $\text{SiO}_2$  and  $\text{K}_2\text{O}$  increased. Changes in this granite were shown by microscopic exam. to be due mainly to weathering of biotite and plagioclase. New analyses of a peridotite boulder showed little change in chem. compn on weathering, indicating that the minerals concerned, olivine and augite, are resistant. A rather strongly weathered mica-schist, consisting of quartz, biotite and feldspar, was found to have lost  $\frac{1}{4}$  of its  $\text{Fe}$ ,  $\frac{1}{4}$  of its  $\text{MgO}$ ,  $\frac{1}{2}$  of the  $\text{K}_2\text{O}$  and some of the  $\text{Al}_2\text{O}_3$ , while  $\text{SiO}_2$ ,  $\text{CaO}$  and  $\text{Na}_2\text{O}$  increased.  $\text{H}_2\text{SO}_4$  present in this rock accelerated the weathering which occurred mainly by decompos. of the biotite. Samples of serpentine, always contg. dolomite or magnesite and tremolite, talc or sometimes olivine, lost, on weathering, 50% of the  $\text{Al}_2\text{O}_3$ , 90% of the  $\text{CaO}$ , 50% of the  $\text{CO}_2$  and some  $\text{MgO}$ , while  $\text{SiO}_2$ , S and  $\text{H}_2\text{O}$  increased. Atm. weathering in Finland is negligible, but considerable weathering occurs beneath the surface of the soil soln. contains  $\text{H}_2\text{SO}_4$ . Differences in the reaction of the soil soln. are the fundamental cause of the variations in the trend of weathering in different climatic zones. When the reaction is acid, weathering causes loss of Fe and Al, i. alk.,  $\text{SiO}_2$  decreases, and Fe and Al gain. The  $\text{pH}$  of spring waters in the eastern Rapakivi area varied from 4.6 to 5.93. The wear of angular and rounded rocks on sandy and stony beaches was studied by placing rock fragments of known weight on the beach and recording the loss in weight at intervals. Both angular and round fragments show rapid wear. A moderate wind velocity grinds the rock material to a product resembling clay. The wear during an equal no. of speed-hrs. (miles per sec. multiplied by time in hours) is independent of the velocity of the wind. Wave action is discussed. The reaction of the water that comes into contact with the finely ground material during the sedimentation process is important. The  $\text{pH}$  of sea water around Finland varies from 7.70 to 8.48. The surface layers of Finnish lakes are nearly neutral but the deeper water has a  $\text{pH}$  of about 5. Rocks and minerals are more sol. in sea water than in distd.  $\text{H}_2\text{O}$ . The trend of weathering in sea water is opposite to that on land. The av. compn. of bedrock in Nivala and Ylivieska was calcd. from analyses and the area occupied by each variety. The results were  $\text{SiO}_2$  63.22,  $\text{TiO}_2$  0.57,  $\text{Al}_2\text{O}_3$  16.67,  $\text{Fe}_2\text{O}_3$  0.97,  $\text{FeO}$  4.47,  $\text{MnO}$  0.4,  $\text{MgO}$  3.37,  $\text{CaO}$  4.68,  $\text{Na}_2\text{O}$  3.22,  $\text{K}_2\text{O}$  2.67 and  $\text{P}_2\text{O}_5$  0.14%. The chem. and mineralogical compn. of Finnish clays from the same regions was investigated by sampling, with a borer, clays of different ages from the same vertical profiles, subjecting them to mech. and complete chem. analysis, and fixing the age by diatom detn. The mineralogical compn. was calcd. from the results. The chem. compn. of a clay is dependent mainly on the amt. of the finest grain-size group. With increasing amts. of the finest grain-size group the bases increase and  $\text{SiO}_2$  decreases. Neither the age of the clay nor the underlying bedrock had much effect on its compn. The chem. compn. of the clays can be explained by the enrichments in micas without much effect due to chem. weathering. Differences in chem. and mech. compn. are caused mainly by heterogeneity of the original rock. The clays of the eastern Rapakivi granite region were investigated in the same manner as those above but not as completely. Bedrock (diabase) and clays from the island of Valama were studied. Because of the sorting action of  $\text{H}_2\text{O}$  and the well-mixed parent moraine material, the clays have a similar compn. over a wide variety of rocks. The chem. compn. of artificially sepd. clay fractions from various countries is discussed, as is also the enrichment of  $\text{TiO}_2$ , Fe oxides and  $\text{Al}_2\text{O}_3$  in clays. Variations in layers of laminated late-glacial clays are explainable in the same manner as in other clays. Investigations of Finnish clays also indicate that chem. weathering was unimportant and that the kaolin content is very small. One hundred and two references. Colin W. Whitaker.

Gneissic complex of the Darjeeling District, Bengal. S. K. Ray. *Quart. J. Geol. Mining Met. Soc. India* 7, 25-44 (1935).—Analyses of sillimanite-gneiss, feldspathic

mica-schist and the Kalupang gneiss are given.

J. F. Schairer

Alteration in quartz in sedimentary rocks. Jacques Boucart. *Compt. rend. soc. géol. France* 1934, 55-6, cf. *Ibid.* 1933, 197.—The corroded quartz found in some Tertiary and Quaternary limestones in Morocco and Spain, and thought to be due to the action of  $\text{CaCO}_3$  on  $\text{SiO}_2$  at a high temp., is always assoc. with phosphate. On the analogy of the assoc. of the disease of "mottled enamel" with F in the  $\text{H}_2\text{O}$  B suggests that the corrosion may be due to the combined action of F in soln. and phosphate on  $\text{SiO}_2$ . C. A. Silberrad.

The composition of "burnt-rock" from Kusnezsk-Becken. B. Behlov. *Trav. Inst. petrog. Acad. Sci. U. R. S. S.* 4, 91-101 (1933). *Neues Jahrb. Mineral. Geol.*, Referate II, 1934, 865.—Chem. and mineralogical data support the conclusion that the rock was formed by the melting of a clay-bearing sandstone. J. F. S.

Studies of two samples of sediments from the Mugelsee. W. Benade. *Mitt. Lab. Preuss. Geol. Landesanstalt* 19, 31-43 (1933).—Data are given on the phys. properties and complete chem. analyses of 2 sludges. The ash content and solvent effect of alc.,  $\text{CCl}_4$  and  $\text{Na}_2\text{SO}_4$  were detd. as well as the C and N contents of the material extd. by the solvents. For methods used cf. C. A. 29, 4113<sup>1</sup>. J. F. Schairer.

Studies of four samples of sediments from the Sakrower See. W. Benade. *Mitt. Lab. Preuss. Geol. Landesanstalt* 19, 45-58 (1933).—cf. preceding abstr. J. F. S.

Spongillae and silicified gypsum in the upper Eocene of the Berry. André Vatan. *Compt. rend.* 201, 1402-4 (1935).—At various places in western Cher there occur pebbles and buhrstones of skeleton crystals of gypsum more or less silicified, and in places lenticular masses of similar compn. approximating to opalite, all pointing to lagoon or lacustrine origin in an arid climate. C. A. Silberrad.

The gases, sublimation and encrustation products and thermal waters of the "lades" of Merapi. M. Hartmann. *Vulkanol. seismol. Mededel.* No. 12, *Dienst van der Mynd. van Nederlandisch Indie* 117-31 (1933); *Rei. Geol.* 15, 242.—The sublimation products found around the "lades" of the volcano Merapi, after the eruption in 1930, consisted of chlorides and sulfates. Secondary fumaroles showed temperatures between 90° and 110°. The escaping gas contained  $\text{CO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{SO}_2$  and  $\text{COS}$ . The last gas is not primary but is formed from org. material and primary S at a temp. over 400°. Analyses of the thermal waters show large quantities of dissolved material. J. F. Schairer.

Content of less common elements in eruptive rocks. E. Tröger. *Chem. Erde* 9, 286-310 (1935).—The occurrence in eruptive rocks of the chief rock-forming minerals contg. the less common elements (P, Ti, Zr, Hf, Th, Ge, Sc, Ga, B, V, Cr, W, U, Mn, Ni, Co, Cu, Zn, Pb, Be, Sr, Ba, Li, Rb, Cs, F, Cl, S and the rare earths) and their mutual relations, with special reference to the at. radii of the elements, are discussed. Thence with the aid of some 1000 analyses, the av. content of these elements in each of 28 classes of eruptive rocks is deduced. C. A. Silberrad.

Geochemistry of the alkali metals. II. V. M. Goldschmidt, H. Bauer and H. Witte. *Nachr. Ges. Wiss. Göttingen, Math.-physik. Klasse* 1, 39-55 (1935); cf. C. A. 27, 4757<sup>1</sup>, and Tolmachev and Filipov, C. A. 29, 300<sup>1</sup>, 4295<sup>1</sup>.—The qual. spectrographic method of Tolmachev (C. A. 28, 4676<sup>1</sup>) for Rb and Cs is quant. if the mineral is mixed with an equal wt. of NaCl to insure complete volatilization of the alkalis, and the photographs are compared with standards. The most suitable lines are 7800.30 and 7947.63 for Rb, 8521.15 and 8943.60 for Cs. The Rb detn. can also be made by comparison with added Rb. As expected, Rb and Cs are especially high in the K silicates of pegmatites. The at. ratio Rb/Cs is somewhat variable, but approximates 45. Green amazonites are found to be much higher in Rb and Cs than intimately assoc. red microclines. Michael Fleischer.

The geochemistry of selenium. II. V. M. Goldschmidt and L. W. Strock. *Nachr. Ges. Wiss. Göttingen, Math.-*

*physik Klasse 1*, 123-42(1935); cf. *C. A.* 27, 4757—The Se content of sulfides was detd by soln in  $\text{HNO}_3$  and Br, evapn with  $\text{H}_2\text{SO}_4$ , distn by a modification of the method of Robinson, Dudley, Williams and Byers (*C. A.* 28, 5763), and colorimetric detn by the Cousen method (*C. A.* 18, 1735). The Se content of sea water was detd by pptg  $\text{Fe}(\text{OH})_3$ , which adsorbs the Se, and analyzing the ppt as usual. The Se content of primary, magmatic sulfides was higher than that of other materials, but the Se S ratio was highest in sedimentary Fe and Mn oxide ores. Sea water, salt beds and sedimentary rocks are very low in Se. North Sea water contained  $3.8 \times 10^{-7}$  g/l. This is due to the fact that Se reaching the ocean is not oxidized to selenate, but is adsorbed by pptg. Fe and Mn hydroxides. The only salt deposit high in Se is the Chulcan caliche, formed under conditions sufficiently oxidizing to convert the Se to selenate.

Michael Fleischer  
Selenium content of European and Japanese clay shales  
I. Minami. *Nachr. Ges. Wiss. Göttingen, Math.-physik. Klasse 1*, 143-5(1935)—The Se content of European and Japanese paleozoic and Japanese mesozoic clays was detd by the method of Goldschmidt and Strook (cf. preceding abstr.). The Se content varied somewhat, but the at. ratio S Se was approx. 10,000 for all 3. M. F.  
Occurrence of vanadium in fossil coals V. A. Zilber-

mintz. *Compt. rend. acad. sci. U. R. S. S. [N. S.]*, 3, 117-20(1935)—Over 500 samples of coals from Russian fields were analyzed for V. In most of them only insignificant amts. (0.02-0.3%  $\text{V}_2\text{O}_5$ ) were found. A no. of samples of definite vegetable origin, of Jurassic age, in the Ural region showed 0.56-8.79%  $\text{V}_2\text{O}_5$ . Most of these coals belong to the clareno-vitrene type and often have a lignite structure. Most of the V content is ascribed to infiltration of V-bearing material during the laying down of the coal beds. C. E. P. Jeffreys

The composition and amount of contraction of the earth  
I. I. Zaslavskii. *Mineralog. petrog. Mitt.* 46, 331-9(1935); cf. *C. A.* 26, 2147, 2001; 27, 3168, 29, 4710—Calcs. based on more recent data on at. vols.

J. F. Schauer  
The need of revising the apparatus employed in the measurement of an electrochemical effect of the ground.  
A. Dellugi. *Beitr. angew. Geophys.* 5, 169-77(1935)—A discussion of app. used in the measurement of the variation of a polarization current at the contact between 2 bodies of different chem. compn. J. F. Schauer

Layer lattices and the base-exchange clays (Marshall)  
2 Surface optical properties of calcite (de Malleman, Sohier) 2.

## 9—METALLURGY AND METALLOGRAPHY

D. J. DEMOREST, OSCAR E. HARDER AND RICHARD KIMBACH

Application of chemistry to metallurgy E. I. Orlov. *Trans. VI Mendeleev Congr.* 1932, II, 1, 378-95(1935).—A progress report on the iron and steel industry. Among new developments in the U. S. S. R. are to be noted investigations on the direct production of iron from ore without the blast furnace process, on the utilization of complex iron ores and on the use of O. E. E. S.

Rapid determination of solid phase in flotation pulp L. M. Iol'son and E. I. Dubovitskaya. *Zavodskaya Lab.* 4, 1106-8(1935)—With the aid of prep. comparative tables, the percentage of solids in a flotation pulp is detd from the d. of a sample of the pulp obtained by weighing and measuring it. A long handle porcelain scoop and a buret are illustrated and described. Chas. Blanc

Practical examples of ore preparation in Bolivia István Szegedy. *Bányász Kohász Lapok* 68, 422-8(1935)—Difficulties encountered after modernization and rebuilding of the plant of the Sociedad Española Morococala, where pyritous cassiterite ores are prep. and enriched, are discussed. A special lab. pulsation sedimenting app. is described. S. S. de Finály

Beneficiation of low-grade ores will prolong life of Mesabi Range E. W. Davis. *Iron Age* 136, No. 20, 30-1, 41(1935), cf. *C. A.* 29, 3952<sup>4</sup> F. G. Norris

Roasting and sintering dusts and powdery ores in a Saint-Jacques turbulent-flow furnace Camille Saint-Jacques and Louis Poupet. *Rev. met.* 32, 581-8(1935)—The pulverulent nature of most materials to be sintered interferes with heat transfer, increasing both the time and heat consumption of the operation. In the present furnace, roasting is carried out when particles are suspended in hot gases. The operation takes only seconds and the material reaches the bottom of the furnace in an incandescent state so that it sinters easily. Mech. details of the furnace, which was in com. operation for more than a year, are given together with a description of its application for calcining phosphate rocks, clinkering cement mixts or roasting and sintering blast furnace dust and ores. The furnace is small and inexpensive both to build and operate. J. D. Cat

Manganese ores of Urkut István Vitéz Bányász Kohász Lapok 68, 346-58, Roy Hung. Palatin-Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met. 7, 54-74(1935)—At Ujakra a new deposit has been found of ores contg. in certain portions of the deposit 46-52% Mn. A special enriching process was worked out to produce a

concentrate contg. 40% Mn from ores contg. 23-27% Mn S. S. de Finály

Low-temperature reduction of magnetite ore. Effect of catalytic compounds G. C. Williams and R. A. Ragatz. *Ind. Eng. Chem.* 28, 130-3(1936)—Certain Na and K compds greatly accelerated the rate of reduction of magnetite ore at 600°. No other materials, except certain Ba compds, showed a similar effect. Hardwood charcoal was a much more active reducing agent than metallurgical coke and responded to the action of Na carbonate as a catalyst. The catalytic effect of various compds was a function of their concn in the reducing mixt. The presence of  $\text{SiO}_2$  tended to inhibit the catalytic effect of Na carbonate. A considerable loss of Na occurred in using a reducing mixt. contg. Na carbonate. Leopold Pessel

Itogon—a Philippine gold producer, Ralph Keeler. *Eng. Mining J.* 137, 3-8(1936)—A description of the mining operations of the Itogon Mining Co. where 10,000 tons of ore is treated per month by all chmng, counter-current cyanidation, with close to 92% recovery. A flow sheet and numerous illustrations are included. W. H. Boynton

Continuous determining of the specific gravity of a cyanide solution A. A. Linevskii. *Tekhnika* 1935, No. 32—A description of a continuously acting areometer in which the funnel is counterbalanced with wts. on the balance principle. B. V. Shvartzberg

Flux and reduction experiments with iron-rich bauxites J. Székely and A. Romwalter. *Roy. Hung. Palatin-Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met.* 7, 42-51(1935)—Expts. were made with a homogeneous colloidal, deep red Hungarian ore mixt. contg.  $\text{SiO}_2$  0.9,  $\text{TiO}_2$  1.8,  $\text{Fe}_2\text{O}_3$  24.3 (= 18.99% Fe) and  $\text{Al}_2\text{O}_3$  47.75%, loss on ignition was 25.38%. Magnetic sep. of reduced samples of this ore was unsuccessful. The reduction product probably contained both magnetic and  $\text{Al}_2\text{O}_3$  mols. within its colloidal particles. Economical working up of such ores seems possible by means of a special reducing soda flux by which both  $\text{Al}_2\text{O}_3$  and a spongy metallic iron can be produced. S. S. de Finály

Treatment of pulverized materials M. Gutman and G. Leizerovich. *Trans. VI Mendeleev Congr.* 1932, II, 409-20(1935)—A review of existing technical trends in the treatment of pulverized raw materials in the non-ferrous metal industry and of recent progress in the industry of the U. S. S. R. E. E. Stefanowsky

**Economics of the aluminum industry.** Francis C. Trary. *Ind. Eng. Chem.* 28, 146-52 (1936). G. G.

The thermal processes in the blast furnace. Alfonz Nahoczky. *Bányász Kohász. Lapok* 68, 449-57 (1935). Thermal processes are shown by curves of heat in kg-cal plotted against temp. Heat consumption in the blast furnace depends on the indirect reduction processes, the more richer the ore. Above 80%  $Fe_2O_3$  the blast furnace is much more sensitive to thermic influences.

S. S. de Findly

Distribution of burden at the top of a blast furnace. H. L. Goldstein. *Domes* 1935, No. 8, 1-10. A math discussion of the distribution of burden, with regard to lump size, as it drops from the hopper into the blast furnace.

S. L. Madorsky

Operating conditions in the melting stage of an open-hearth furnace. I. D. Semkin. *Domes* 1935, No. 8, 10-25. A discussion of the relation between the life of a furnace roof and conditions prevailing in the combustion zone of an open-hearth furnace.

S. L. Madorsky

Relation between angle of incline of the flame and horizontal cross section of an open hearth furnace. M. S. Sokolov. *Domes* 1935, No. 8, 25-7. A study of data from successfully operating open hearth furnaces leads to the conclusion that the best results are obtained when  $E/L = \sin \varphi$ , where  $E$  and  $L$  are width and length of the hearth and  $\varphi$  is angle of incline of the flame to the horizontal, also, when  $\alpha + \beta = 2\varphi$ , where  $\alpha$  and  $\beta$  are angles of incline of gas and air channels of the furnace.

S. L. Madorsky

Further notes on open-hearth boils. Henry D. Hubbard. *Iron Age* 136, No. 13, 98, 104, 108 (1935). Cf. C. A. 20, 566. The results of early investigations of the gases exd from pig iron and Bessemer steel are cited. Certain relations between these results and the requirements for making running steel are inferred.

F. G. Norris

Slag systems. R. Hay, James White and A. B. McIntosh. *J. West of Scotland Iron and Steel Inst.* 42, Part 6, 90-104 (1935). Cf. C. A. 28, 6407. The nonexistence of grunerite,  $FeO \cdot SiO_2$ , is established by thermal and optical evidence. A previously unidentified substance with  $n = 1.74$  is a eutectic of fayalite and silica. In the ternary system  $FeO-MnO-SiO_2$  a deep trough extends from 1155°, the m. p. of the fayalite- $SiO_2$  eutectic, with a gradual rise in m. p. to 1200° at the rhodonite-terephate eutectic. No ternary eutectic was found. The binary  $MnO-Al_2O_3$  was studied thermally and optically. The addn of  $Al_2O_3$  lowers the m. p. of  $MnO$  from 1785° to the eutectic at 21%  $Al_2O_3$  and 1520°. With further addns of  $Al_2O_3$  the liquidus rises continuously to 2050°, the m. p. of  $Al_2O_3$ . The compd.  $MnO \cdot Al_2O_3$  m. 1560° incongruently, is hard and brittle, is amber but appears white under the microscope, is isotropic, and has a  $n$  between 1.70 and 2.00. All melts in this system have low surface tension.  $Al_2O_3$  is sol. in solid  $MnO$ . Thermal data and absence of eutectic at 60%  $Al_2O_3$  indicate  $MnO$  is sol. in the compd. The soln of  $MnO$  changes the color from amber to bright emerald green. The reaction of  $FeO \cdot MnO \cdot SiO_2$  slags with C was studied at 1310°. The oxides were melted and then crushed and remelted with C rod and the gas pressure was measured. The order of activity is  $FeO$ , knebelite, fayalite and Mn silicate. Use of high C steel as a source of C indicates the same order of activity. With excess  $MnO$ , Fe is a reducing agent. The resulting Mn alloys with the Fe and C. Work is in progress with white cast Fe as a source of C. FeO is the most active oxidizer in this ternary system. At 1310° fayalite is only partially dissociated. In the presence of  $MnO$ , stable Mn silicates are formed lowering the concn of free  $SiO_2$  and thus increasing the free FeO. Mn silicates are less active toward C because they are less dissociated, and because  $MnO$  is less reducible.

I. G. Norris

Distribution of phosphorus between metal and slag in puddled iron. Nello Collari. *Ann chim applicata* 25, 432-47 (1935). In detg. P in Fe it is necessary to det the partition of P between metal and slag. The P in the slag (occlusions) is present as phosphate, while that in the

metal is present as phosphides. Therefore, detn. of the total P and of phosphate P is sufficient. A. W. C.

Foundry coke. W. E. Mordecai. *Colliery Guardian* 151, 1079-81 (1935); C. A. 29, 2337. A discussion of the influence of the phys. and chem. properties of coke on cupola operation. Also in *Gas World* 104, No. 2083 Coking Sect., 3-5, 11 (1935).

Joseph H. Wells

Circulation of gas in mold. The formation of scums and their classification. A. Collin. *Bull assoc tech fonderie* 9, 305-8 (1935). Scums on the casting surface are due to the passage of gas through the metal and the mixing of sand particles with the metal. G. T. Motok.

Nonmetallic impurities in open-hearth steel. Folke W. Sundblad. *Iron Age* 136, No. 20, 21, 76-7 (1935). Various impurities can be eliminated from the bath by the boil on a rod or pole. This method is recommended especially in case of a large bath which is stated to be inferior to a small bath in respect to cleanliness. F. G. N.

Comparative investigations of the effect of special deoxidizers with soft steel. P. Zimmermann. *Mitt Kohle- und Eisen Forschung G. m. b. H.* 1, No. 3, 29-49 (1935). The various deoxidizers for steel have a decided influence on segregations, no. size and distribution of non-metallic inclusions, pipe formation and tendency to aging. The deoxidizers tested and their percentage compns are:

(1) ferrosilicon, Si 70.12, Fe 23.88, (2) calcium silicon, Si 55.84, Ca 29.25, Fe 14.91, (3) ferrosilite, Si 30.50, Al 19.15, Ti 1.32, Fe 43.13, (4) Sumanal, Mn 19.44, Si 20.95, Al 16.16, Ti 1.50, Fe 31.95, (5) Alsilme, Si 34.10, Al 41.74, Ti 2.88, Fe 18.28, (6) Aluminum, A. 98.50, Fe 1.50 and (7) ferrosilicon plus aluminum. The most effective agent was (6), which produced a melt with high Si, and the steel showed few inclusions and the least pipes, there were, however, some alumina inclusions. A similar effect was obtained with (4). All agents reduced segregations, inclusions were higher for (1) and (2) than for the other agents, (2) gave only small and uniformly distributed inclusions in the ingot, and killed the steel completely without addn of Al. The S content was not reduced with this deoxidizer. The least effective was pure Al, it produced the most and coarsest inclusions in very irregular distribution and the greatest segregations. The action of (7) was unfavorable but less so than that of pure Al.

Compared with not-killed soft-steel melts, all melts treated with addl. deoxidizers showed deoxidation products in the solidified steel and tendency to pipe formation, but no difference could be found in mech. properties and deep-drawability between killed and not-killed soft steel. It is concluded that for many purposes soft steel without addl. deoxidizers is superior from several angles. A table gives test results of phys. properties and deep-drawability for steels deoxidized with the above-mentioned agents.

Twenty-two references. M. Hartenhiem

Survey of the testing methods for the determination of the mechanical strength of aluminum castings. A. v. Zeidler. *Aluminum* 17, 570-4 (1935). The manner in which a specimen has been made for tensile or hardness tests is of utmost importance. Separately cast and specimens cast with or taken from the casting are discussed and the tests described. The best results are obtained with horizontal casting.

M. Hartenhiem

Control of grain size and structure in large forgings by heat treatment. G. J. Horvitz and G. Brinton Jack, Jr. *Iron Age* 136, No. 13, 48-50 (1935).

Brass and copper forgings—their manufacture, properties and uses. E. F. Cone. *Metals & Alloys* 6, 337-41 (1935).

Dwons Schaal

Tensile testing of bearing metals at elevated temperatures. Jean Galibourg, Leon Guillet, Jr., and Popoff. *Riv. met.* 32, 3/3 7 (1935). A series of tests made on bearing metals, the analysis of which was not specified, indicated that with the same loads and with const. and changing temps the appearance of the time-elongation curves is approx. the same. At the beginning elongation is slow, the curve being nearly a horizontal line; then the rate increases and for a short time the curve makes an angle of about 45° with the axes, in the final period the curve is nearly vertical.

J. D. Gat



**Heat-treating spring leaves.** H. J. Langley, *Ind. Age* 14, No. 7, 7, 8, 16(1930).—For the heat treatment of automobile springs, a special gas fired furnace is used which insures a reducing atm. and the avoidance of oxide or scale, except mill scale, and the maintenance of the required soaking temp. prior to quenching. The steel is then removed from the furnace, automatically clamped, formed, and quenched in oil by a specially designed machine. The temp. of the quenching oil is maintained below 100°F by cooling.

**Temper hardening of commercial nickel-coppers containing phosphorus.** E. C. Rollason and T. G. Bamford *Metals & Alloys* 6, 345-6, 340(1935).—An investigation of the temper hardening of 3 Cu alloys contg. (1) 1.72 Ni, 0.03 P, trace Fe, (2) 1.73 Ni, 0.11 P, 0.01 Fe, (3) 2.33 Ni, 0.03 P, 0.03 Fe.

**Temperature measurement of liquid and solid metals as represented by irons.** Rudolf Hase *Z. Ver. deut. Ing.* 79, 1351 (1935).—Temp. of molten metals is usually measured by radiation methods, either total radiation or partial radiation. Rapid temp. changes can be observed with an optical pyrometer used with an oscillograph. Fundamentals of emissivity and black body conditions are reviewed. The detn. of the relative radiation of molten metals is difficult because temps. are measured in the runner or metal streams and variations are caused by the formation of oxide coatings which depend on temp. and metal compn. W-Mo thermocouple in a sillimanite protecting tube can also be used for detg. the temp. of molten metals. A recent development is the color pyrometer. Use of an optical pyrometer and a partially closed graphite dip tube overcomes some of the disadvantages of variations in radiation and sudden temp. changes. Temp. measurements in solid metals are best made by thermocouples. The use of melting bodies, change in color, metallographic evidences of temp. change, and measurement of expansion are discussed.

**A malleable cast iron from an ordinary cupola furnace.** E. Rappoport, E. Kogan, N. Poplavko and P. Tokarevskii *Tekhnika* 1935, No. 11.—The complicated duplex process for pig iron for malleable castings was replaced by a system of casting from a cupola. The rate of casting and the difficulties surmounted are described. A metal is obtained contg. C 2.3-2.5, Si 0.9-1.1, S 0.1 and Mn 0.13%.

**Influence of the accompanying and addition elements Si, P, Ni, Cr, Mo, W and Cu on the sensitivity of gray cast iron to variations in wall thickness.** E. Hugo, E. Piwowarsky and H. Nipper *Giesserei* 22, 421-8, 452-8 (1935), cf. *C. A.* 30, 637.—Alloy adds were made to 31 casts of cupola I.e., each contg. approx. 3.25% C, 0.60% Mn and 0.12% S. The effect of variations in each element separately and of Ni and Cr in combination was studied. In addn. to regular test bars, a hollow block was cast with each wall of a different thickness, viz., 20, 40, 60 and 80 mm. The tensile strength, bending strength, shear resistance, Brinell hardness, carbide content and structure were detd. for each wall thickness. In a new method of measuring shear resistance a sample 1.6 mm thick is held down on an anvil contg. a slot 3.15 mm wide. The shearing force is exerted by a plunger 3 mm wide and 19.8 mm long centered over the slot. One advantage of the new method is that it can be used to measure the strength of the entire cross section. The results are compared with those of the Sipp-Rudeloff and of the Fremont method. Both tensile strength and bending strength are proportional to the shear resistance. Although the cooling conditions of the 20-mm. wall and of the 30-mm. diam. rod are similar, the worked 30-mm. rod has higher tensile strength and less scattering of the values. The tensile strength, hardness and carbide content are logarithmic functions of the wall thickness. Si decreases the value of all phys. properties, particularly the shear resistance. P decreases the phys. properties, though not as markedly as does Si. Ni and Mo increase the properties to about an equal extent. W increases the properties. The effect of Ni, in contrast to that of Cr, is beneficial especially if it replaces part of the Si. Cu has a favorable effect. J. G. Norris

**Nitricastiron cylinder sleeves.** A. T. Colwell *Iron Age* 136, No. 25, 31-3(1935).—A survey of the causes of wear in cylinders indicates that in addn. to high hardness the cylinder must have strength, soundness and corrosion resistance in order to be wear resistant. A hardness of 800 to 1000 Brinell for a depth of 0.004 to 0.006 in. results from nitriding centrifugally cast Fe liners contg. total C 2.93, graphitic C 2.31, combined C 0.62, P 0.058, S 0.029, Mn 0.76, Si 2.69, Cr 1.28, V 0.18, Mo 0.24 and Al 1.01%. The liner will serve for 300,000 to 400,000 miles after which it must be replaced because it cannot be reconditioned as can gray Fe blocks and alloy sleeves. Nitrided cylinder liners are recommended particularly for truck service.

**Cementation of iron and ferrous alloys with beryllium.** J. Laissus, *Rev. mét.* 32, 293-301, 351-60, 401-22(1935), cf. *C. A.* 29, 1046.—Cementation expts. previously made by Feszczenko-Chopinski (cf. *C. A.* 22, 2535) and by Cazaud are described. In the expts. described in the paper, specimens were placed in powd. 98% Be or in 80% Fe-Be contained in a receptacle closed with threaded plugs. This container was then placed in a closed box filled with charcoal and cast iron chips so as to prevent O<sub>2</sub> attack on the cementation powder. Cementation formed on electrolytic Fe a case consisting of a zone of solid soln., of solid soln. with some eutectoid and an outside zone of hypereutectoid alloy contg. much FeBe. Diffusion in C steels is similar to the diffusion in electrolytic Fe, with the difference that the presence of C results in more complex structures. The constituents of the case produced in C steels in the outward order are an area with gradually decreasing pearlite content, a practically decarburized layer, a solid soln. of Fe-Be-C, a sharply defined hypereutectoid layer of solid soln. Fe-Be-C and a triple eutectoid, eutectoid zone and hypereutectoid zone. Cementation with 80% Fe-Be gives the same results as with 98% Be. For electrolytic Fe the thickness of case is directly proportional to time and temp. of cementation. In higher-C steels the width of the decarburized layer passes through a max. with increasing temps. and grows with cementation time. C content has a retarding action on the speed of penetration. Hardness of case increases with C content but is not a function of temp. Case produced on electrolytic Fe reads 268 Vickers, on 0.9% C steel 1596 Vickers. Cementation with Be reduces resistance of steel to scaling and to chem. attack by most of the reagents. It increases the resistance to atm. corrosion and to fresh and salt water attack, to HNO<sub>3</sub> (provided the case does not have any cracks) and to bleaching liquor.

**Steel scrap addition in chilled casting melting.** Hugo Patsch *Giesserei* 22, 584-6(1935).—The literature is meager on steel addn. to chilled castings. The effect of the addn. of steel to gray iron and the mechanics of graphite formation in cast iron are discussed. Addn. of steel to chilled iron increases the mech. properties and the depth of chill.

**Cast iron and steel differentiated.** A. L. Norbury *Iron Age* 136, No. 19, 25, 104-12(1935).—The recommendation published by the Brit. Cast Fe Research Assn. is based on the presence or absence of carbide or graphite eutectic in the microstructure. Many previous definitions are critically reviewed.

**Effect of mixed acids on irons and steels.** Justice Eddy and F. A. Rohrman *Ind. Eng. Chem.* 28, 30-1(1936).—Loss of wt. tests showed that the tendency of plain C steels to resist mixed acids and to retain their passivity with increasing adds of water increased with their C content. Samples high in S and Mn showed greater losses of wt. and less tendency to retain passivity in dil. solns. than those low in these 2 constituents. Other alloying constituents improved the resistance. Quenched steels showed greater resistance than those cooled in the furnace.

**Some trends in steel metallurgy during 1935.** J. H. G. Monypenny *Metallurgia* 13, 50-2(1935).—New and cheap low-alloy steels are mentioned. Grain size control is discussed and it is concluded that it is too often specified

Many specifications also limit visible nonmetallic particles. The invisible particles may be just as harmful. For many purposes acid-open-hearth steel is just as good or even better than elec.-furnace steel. Highly polished corrosion resistant sheets are now being produced.

**Causes and effects of brittleness in steel.** C. L. Shapero. *Heat Treating and Forging* 24, 467-72, 517-21, 560-74, 576 (1935).—See C. A. 29, 3861.

**Soft spots on hardened steel.** H. H. Bleakney. *Iron Age* 136, No. 24, 32-3, 94-6 (1935).—Soft spots either may penetrate to the core or, as is more common, may be superficial layers overlying fully hardened steel. The superficial type is attributed to the faster critical cooling speed of the hypereutectoid surface layer relative to that of the eutectoid zone beneath. The C content of the case should be as nearly eutectoid as possible. This condition is easier to attain with gas than with pack carburization. If a higher C case is used, the quenching temp. should be high enough completely to dissolve the carbide. If low quenching temps. must be used with excess C, a normal steel and a drastic quench with adequate circulation is required.

**Heterogeneity of steel ingots.** W. H. Hatfield. *Iron and Steel Ind.* 9, 57-65 (1935).—Work to date of the Ingot Comm. (6 reports) is summarized. Shape and form of an ingot are frequently determined by the subsequent use of the ingot and consequently the question of heterogeneity becomes secondary. Ingots are classified into the following types: (1) solid ingot with pipe restricted to that portion of the mold which was lined by the refractory liner, (2) rimmed-steel ingot with a solid exterior but a large no. of blowholes in the interior, (3) ingot contg. a deep pipe and exterior blowholes, (4) ingot contg. a shallow pipe and numerous blowholes. It is partial to the use of type 1 ingots. Heterogeneity assumes different aspects with large ingots and small ingots, in a very large ingot a steel is poured in the bottom different in compn. from that added to the top. Influence of casting temp. on crystal size and therefore heterogeneity is traced, when freezing begins at a vast no. of nuclei, a mass of small crystals results and heterogeneity is at a min. Sprints and macro etchings of the various types of ingots are given. In rimmed ingots the run is shown to be pure metal. It postulates that this is indicative that the rimming may be explained by a consideration of the allotropic modifications of Fe which is deposited from the liquid solns. under these conditions. A centrifugally cast ingot shows a "bacon" effect, which may indicate a heterogeneity in the liquid phase. The detn. of O in steel, effect of N in steel, discussion of rimming steel, and pyrometry are but mentioned since they are covered in detail in the 6th report recently issued.

**Tool steels.** H. André Michel. *Métallurgie* 10, 180-7 (1935), cf. C. A. 30, 64.

**Application of alloy steels.** H. W. McQuaid. *Iron Age* 136, No. 26, 18-22 (1935).—A nontechnical review of the effect of alloy additions.

**Color-producing reactions for microscopic examination of carbon steels.** Joseph Maletic. *Compt. rend.* 201, 1353-7 (1935).—To prep. the reagent, "M(Mo) normal," 15 g.  $\text{NiH}_2\text{molybdate}$  is dissolved in  $\text{H}_2\text{O}$  and the soln. made up to 100 cc.; this is poured into 100 cc.  $\text{HNO}_3$  (1.2), allowed to stand 4 days and filtered, of it 2 cc. is added to 100 cc.  $\text{LiOH}$ , and the polished sample is placed therein for 30-45 min. The various constituents (except C which remains black) then assume different colors, violet, blue, green, yellow, etc., while ferrite shows different colors according to the orientation of the crystals. A more delicate reagent "M(Mo) slow" is obtained by using 2 to 4 times as much  $\text{LiOH}$ . Similar but different colors are obtained if  $\text{NiH}_2\text{vanadate}$  or  $\text{vanadate}$  replaces the  $\text{molybdate}$ . The method is applicable to steels, irons and some cast irons.

**Hardening characteristics and other properties of commercial 0.6% carbon tool steels.** T. G. Digges and Louis Jordan. *J. Research Natl. Bur. Standards* 15, 385-407 (1933) (Research Paper No. 837).—The effect of the

initial structure prior to heating for hardening upon the austenitic grain size and grain growth was studied on 2 selected brands of 1% C tool steel having widely differing hardening characteristics. Comparison of the steels was also made with respect to their behavior in the transformation temp. range during thermal analysis, relative rate of transformation of the cementite, Charpy impact strength, susceptibility to grinding cracks, Rockwell hardness on quenching at different rates, structural normality, performance as lathe tools, and the effect of rate of heating to the hardening temps. on the depth of hardening. The relation between austenitic grain size and the critical cooling rates over a range of quenching temps. of 1425° to 1775°F. was influenced by the initial structure of the steel at quenching temps. below that at which all the C is completely dissolved in the austenite. Above that temp., each steel approached both a grain size and critical cooling rate characteristic of the steel regardless of its initial structure. At the highest temp., i. e., 1775°F., the characteristic grain size and cooling rate was higher in the so-called "controlled" steel than in the "non-controlled" steel, the effect of the grain size on the critical cooling rates was marked through the entire range of quenching temps. The critical cooling rate of the controlled steel may change considerably with little or no change in the austenitic grain size, the controlling factors at lower quenching temps. being differences in the C content and C distribution in the austenite, and at higher temps. oxides or carbides introduced for grain control. The initial structure of the controlled steel exerts an influence on the grain size-critical cooling rate relations at both low and high quenching temps. A non-controlled steel contg. C above eutectoid compn. in a given austenitic grain size showed a lower critical cooling rate than the same steel with eutectoid compn. Differences in the transformation of the steels on slow heating to the temp. range of austenite are believed to be due to variations in the rates of C diffusion, the controlled steel having a higher rate than the non-controlled one. On slow cooling from the austenitic temp. range, the controlled steel was more abnormal in structure. The depth of hardening of the 2 steels was not influenced by the rate of heating to the hardening temp., and no marked differences of the 2 steels were observed as to tool performance, Charpy impact values, Rockwell hardness and susceptibility to grinding cracks.

**Hardness testing of nitrided steels.** John H. Ruska. *Iron Age* 136, No. 13, 22-3, 20-8, 30, 58 (1935).—A critical review. The Herbert pendulum is the only app. that gives satisfactory results on the hardness of nitrided steels at elevated temps. Superficial Rockwell tests are satisfactory at room temps.

**Nitrided tool steels.** Horace C. Knerr. *Iron Age* 136, No. 13, 44, 5, 96 (1935).—The compn., heat treatment and application of 2 typical nitriding steels are described.

**The science of metals grows apace—many new alloys and methods of treatment.** Nonferrous physical metallurgy. Introduction. Robert F. Mehl. *Mining Met.* 17, 42 (1936). Secondary metals more closely examined. W. Romanoff. *Ibid.* 42-3. New lead and tin alloys seen in batteries, bearings and bullets. Geo. O. Hiers. *Ibid.* 43-4.—The feasibility of making grids for some types of storage batteries from an alloy contg. Ca 0.1% and Pb 99.9% is indicated. Dispersion hardening, accompanied by heat treatment, appears necessary to obtain strength or hardness = to Sb-Pb. Batteries using Ca-Pb grids show less self discharge than when employing Sb-Pb, and there is better elec. cond. Sn-base Babbitt metals have been replaced to some extent in automobile engines, but with the trend toward greater cooling capacity in engines Sn-alloys should regain some ground. The Pb-Cu alloys (30-50% Pb) used as thin linings on steel backs require the Cu to exist in dendritic crystals branching away from the steel. Diesel engines in new high-speed trains use bearing linings of a Pb alloy contg. a few % of several elements including Ca and Sn. Small amts. of Zn markedly increase the surface tension of molten monotype metal; Na decreases it, small amts. of As, Bi, Cd, Mg, K,

and Fe have little effect. Transformation of Sn to gray Sn is inhibited by 0.0035% Bi and not by 0.001%. New and old aluminum alloys meet a rapidly expanding demand. L. W. Kempf *Ibid* 44 5.—Two new types of Al alloys were put on the market in 1935—(1) free cutting of duralumin type for screw-machine stock and (2) an alloy for die-casting burner heads for gas ranges. The use of Al alloy pistons in motor-vehicle engines is increasing; most are of Si type alloy with an electrolytically produced oxide coating. Al alloy cylinder heads are used where max engine performance is desired. The recently developed wrought Al alloys of the Al-Mg type are increasing in popularity because of their high mech. properties and excellent workability. Brass and copper mills continue efforts to secure highest quality in products. John L. Christie *Ibid* 45-6.—Progress has been made in com. application of controlled furnace atms. in bright annealing. Fluxes and deoxidizers used in making castings for wrought Cu, brass and bronze are getting more attention. Two processes—the Eldred and the Hazlet—produce semi-finished material direct from molten metal. Extrusion of tubing is increasing. Advances have been made in hot working of alloys that formerly were not considered hot-workable. Uniformity in annealing is increasing. Wide diversification in use leads to biggest year in nickel. Robert C. Stanley *Ibid* 46-7.—The field of use of Ni alloys is being extended. Use of corrosion- and erosion-resisting materials and of alloy cast Fe is increasing. New alloys are being developed for weight reduction. Thickness of coatings has been increased. The outstanding development in this field has been the perfection of the process for bright Ni plating. Ni is being used with Sn in bronzes to improve mech. properties. Ni bearing Al alloys are increasing in popularity, especially for casting. Uses of Ni in the mining industry are listed. Die castings and electrogalvanizing boost use of purest zinc. W. M. Pierce *Ibid* 48.—Several new developments in use of silver and precious metal alloys. Robert H. Leach *Ibid* 48.—Developments in the chem., food and elec. industries have caused the principal increase in demand. Use of Ag solders has been extended. Prepn of Ag-Ni, Ag-Mo, Ag-W and Ag-graphite for elec. contacts by pressing and sintering metal powders has increased. Use of small amts of Ag in alloys is being pushed. Magnesium alloy products improve in quality and size. John A. Gann *Ibid* 49.—Alden H. Emery.

**Permanent magnet materials.** C. S. Williams. *Elec. Eng.* 55, 19-23 (1936).—The essential properties of some new materials are enumerated and comparison is made with the characteristics of the more familiar C, Co and Cr steels. The new materials listed are: Ni-Al-Fe alloys, Co-Mo-Fe alloys and Co-W-Fe alloys, a non-metallic material composed of Co and Fe oxides and a Co-Ni-Ti-Fe alloy. Magnetic and phys. properties of the alloy types are discussed, also their economic status. W. H. B. X-ray study of aluminum zinc alloys at elevated temperatures. E. A. Owen and Llewelyn Pickard. *Phil. Mag.* 20, 761-771 (1935).—Powder photographs were taken of a series of alloys which were kept at high temps. in a vacuum. The phase structure found previously was confirmed. The comps. of the phase boundaries at 315°, 340° and 360°, resp., are in wt % Al between  $\alpha + \beta$  and  $\beta$ , 17.4, 18.0 and 19.0, between  $\beta$  and  $\beta + \gamma$ , 29.5, 28.0 and 23.5, and between  $\beta + \gamma$  and  $\gamma$ , 32.0, 47.6 and 58.5. The temp. of the eutectoid transformation  $\beta$  to  $\alpha + \gamma$  is 272°. As the temp. increases, the Zn content of the phase increases and the parameter value decreases. The compn. range of the  $\beta + \gamma$  region at 360° is about 3%.

E. J. Rosenbaum. Scientific work on the production of iron alloys. B. Siseev. *Sotetsialist. Rekonstruktsiya i Nauka* 1935, No. 4, 154 v.—Work on iron alloys for the past 2 yrs. is summarized. Ferrochrome with 0.02-0.05% C is obtained by refining at 1650° and a lowered pressure of about 25 mm. Ferrochrome was refined from 6 to 1% C by blowing air through the molten metal in an arc furnace and a subsequent reduction with ferrosilicon. Better results from blowing were obtained in an induction furnace, in shorter

time. The original content of C, Si and Cr changed from 6.8, 2.02 and 59.9% to 1.5-0.6, 0.61-0.15 and 60-62%. Decarbonizing and dephosphorizing of ferromanganese by treating with an ore was not successful. By silico-thermic reduction a noncorrosive chrome steel was produced directly from the ore. Work in a closed furnace and the use of magnesite slags of higher basicity increased the output of Cr and lowered the amt. of ferrosilicon used from 750 to 450 kg. per ton of ferrochrome. Magnesite proved to be a stable lining material. B. V. S.

**Metals and alloys.** XV. X-ray analysis of lithium zinc alloys. E. Zintl and A. Schneider. *Z. Elektrochem.* 41, 764-7 (1935), cf. *C. A.* 29, 5400<sup>7</sup>.—X-ray examn of Li-Zn alloys gave results essentially the same as those obtained by Grube and Vosskuhl (cf. *C. A.* 28, 859<sup>9</sup>), namely, that 5 intermediary kinds of crystals occur at room temp. XVI. Structure of platinum-thallium alloys. E. Zintl and A. Harder. *Ibid* 707-71.—Pt takes up little Tl and conversely the soly of Pt in Tl seems to be very slight. Both metals only form an intermediary kind of crystal of the formula PtTl which has hexagonal lattice  $a = 5.605 \text{ \AA}$ ,  $c = 4.639 \text{ \AA}$ ,  $c/a = 0.828$  with 8 atoms per cell in the following positions: 000,  $1/2, 1/2, 0$ ,  $1/2, 0, 0$ ,  $1/2, 1/2, 1/2$ ,  $1/2, 1/2, 1/2$ . Tl forms no compd with Pt which has a Ni arsenide structure. XVII. X-ray analysis of lithium amalgams. E. Zintl and A. Schneider. *Ibid* 771-4.—X-ray analysis gave results in agreement with thermo-analytical results. In the system LiHg at room temp. in the region from 0 to 78% Li, 5 intermediary phases of the following approx compn occurred: LiHg, Li<sub>2</sub>Hg, Li<sub>3</sub>Hg, Li<sub>4</sub>Hg and Li<sub>5</sub>Hg. LiHg, and Li<sub>4</sub>Hg cannot yet be structurally explained. From Li<sub>3</sub>Hg, a hexagonal elementary body with  $a = 6.240 \text{ \AA}$ ,  $c = 4.794 \text{ \AA}$ ,  $c/a = 0.768$  is derived. Li<sub>3</sub>Hg has the structure of  $\beta$ -brass with  $a = 3.287 \text{ \AA}$ . Li<sub>4</sub>Hg has the same structure as Cu<sub>3</sub>Al, the  $\beta$ -phase of the system Cu-Al. M. McMahon.

Lattice distortion and coercive force in single crystals of nickel-iron-aluminum. W. G. Burgers and J. L. Snoek. *Physica* 2, 1064-74 (1935).—A report on preliminary results obtained in a correlated investigation of the aging characteristics of alloys of the Ni-Fe-Al system wherein a simultaneous study of the ferromagnetic and x-ray diffraction properties is being made. Single crystals 6 mm. square and 10-20 mm. long have been made of the alloy 28.3% Ni, 12.6% Al and 59.1% Fe. Three methods of heat treatment were used: (a) the sample is quenched and reheated to the desired temp., (b) the sample is quenched to the desired temp. and held there, and (c) the sample is quenched at varying cooling rates. In agreement with Glocker (*C. A.* 29, 5794<sup>9</sup>), lines of the  $\gamma$ -phase are only visible after cooling rates which are considerably slower than those required to produce the max. coercive force (590 gauss) after cooling from 1200-700° (in 45 sec.) and superlattice lines of the FeAl type were always observed in almost constant relative intensity. Line-broadening results do not agree with those of Glocker. Marked line broadening (1310) Co  $K_{\alpha}$  radiation occurs wherever the coercive force differs much from zero. When the coercive force has reached its max. value and is decreasing the usual type of line broadening occurs. Previous to this value, the photometer curves show an increasing blackening of background with no loss in definition of the doublet. This latter type of broadening is thought to be due to the formation of regions of slightly larger and slightly smaller spacing in the matrix lattice which has not lost its coherence. Tentatively, a no. of possible explanations are given for the behavior of coercive force and line broadening. H. A. Smith.

Recent developments in the manufacture and applications of magnesium and magnesium alloys. W. C. Devereux. *Metallurgia* 13, 47-9 (1935).—Mg is now produced in England and Austria by reduction of MgO with C in an arc furnace in a H atm. Crude metal is purified by distn. in inert atm. Properties and uses of Mg alloys are discussed. J. L. Gregg.

Magnesium alloys in airplanes. E. R. Gadd. *Aircraft Eng.* 7, 299-302 (1935).—The resistance to corrosion is increased by immersing the part in a hot aq. soln. contg

$K_2Cr_2O_7$ ,  $(NH_4)_2SO_4$ ,  $(NH_4)_2Cr_2O_7$  and  $NH_4$ . Another method employs an aq. soln. of  $SeO_2$  and  $NaCl$  at room temp. M. W. Schwarz

X-ray structure and electrical resistance of palladium-silver-hydrogen alloys. Gunnar Rosenhall. *Ann. Physik* 24, 297-325 (1935); cf. C. A. 28, 167. —Methods heretofore used for fixing compns. of alloys in this system are shown to be faulty. The system is represented on a triangular equilateral diagram for the components Ag-PdH-Pd. The system Ag-Pd is one-phase, the system Pd-PdH is two-phase, but the gap of immiscibility is narrowed by the introduction of Ag, all alloys contg. 25 atoms % or more being one-phase. Pd alone takes up 0.8 H per Pd, but 70Ag-30Pd is unable to take up any H. Lattice consts. and sp. resistances are given for over 100 preps. The expansion of the lattice following introduction of H, as shown by x-rays, is confirmed by direct linear measurements. In the one-phase region the lattice expands regularly with increasing H-concn., but the resistance passes through a sharp min. as the H-concn. increases. Hypotheses as to the position of the H in the crystal lattice are discussed in the light of these observations. C. D. West

Sodium alloy lamps. Gorton R. Fonda. *J. Optical Soc. Am.* 23, 312-18 (1935). —Results of expts. with alloys in the standard type of a c. Na lamp filled with Ne at 1.5 mm. pressure are given, with 2 tables and 4 diagrams. The closest approach to white light was given by alloys contg. Na 40%, Cd 30-65% and Hg 30-65%, the most pleasing light being from Na 40%, Cd 45% and Hg 48%. Alloys gave better results than phos. mixts. of the elements, and they were prepd. by Vanstone's method (*Trans. Faraday Soc.* 7, 42 (1911)). J. H. Moore

Modern uses of monel metal. James A. Rabbitt. *Japan Nickel Rev.* 3, 513-84 (1935). E. H.

Fabrication of monel metal in chemical equipment. Sakai Nakamura. *Japan Nickel Rev.* 3, 602-10 (1935). E. H.

Monel metal in the chemical industry of Japan. Takashi Okamoto. *Japan Nickel Rev.* 3, 594-601 (1935). E. H.

Monel metal in the rayon industry of Japan. Shin-ichiro Anita. *Japan Nickel Rev.* 3, 611-12 (1935). E. H.

Monel metal chemical screens. Motoy. Tsunekawa. *Japan Nickel Rev.* 3, 627-31 (1935). E. H.

Monel metal in chlorine-handling valves. Kimutoshi Nishino. *Japan Nickel Rev.* 3, 620-3 (1935). E. H.

K monel. W. A. Mudge and Paul D. Merica. *Japan Nickel Rev.* 3, 506-12 (1935). E. H.

Modern development of high-strength corrosion-resistant alloys. Masaharu Gotō. *Japan Nickel Rev.* 3, 504-5 (1935). E. H.

Melting points of eutectics. Lipowitz alloy and Wood's metal. Sidney J. French. *Ind. Eng. Chem.* 28, 111-13 (1936). —Melting and cooling curves of Lipowitz alloy and Wood's metal were detd. To eliminate the confusion existing between the compn. and m. p. of these alloys, it is suggested to drop both names and to call the eutectic alloy a quaternary eutectic, stating its compn. and melting-freezing range. Leopold Pessel

X-ray studies on the systems tin-antimony and tin-arsenic. G. Hägg and A. G. Hybnette. *Phil. Mag.* 20, 913-29 (1935). —With the Sn-Sb system at room temp., the SnSb phase has a homogeneity range between 45 and 55 atoms % Sb. At the Sn-rich limit it has a deformed NaCl structure with rhombohedral unit cell,  $r = 6.117 \text{ \AA}$ ,  $\alpha = 89.70^\circ$  and the vol. is 238.9 cu. Å. The deformation increases with increasing Sb content. At the Sb-rich limit,  $r = 6.183 \text{ \AA}$ ,  $\alpha = 89.18^\circ$  and the vol. is 231.2 cu. Å. The change is attributed to the fact that Sb atoms are larger than Sn atoms. In the Sn-Sb system As is not sol in Sn. Around a concn. of 40 atoms % As, lines due to  $Sn_{1/2}As_{1/2}$  were found. The Laue symmetry of this phase is  $D_{2h}$ , with a rhombohedral cell contg. 7 atoms  $\%$ ,  $r = 12.23 \text{ \AA}$  and  $\alpha = 19.22^\circ$ . SnAs has a NaCl structure with a lattice const. of 5.716 Å. which does not

change with compn. Arsenic can dissolve 21 atoms % Sn. With increasing Sn concn. the rhombohedral edge of the As phase increases and the rhombohedral angle decreases. These results disagree with those of Bowen and Jones (C. A. 25, 5885) and Willott and Evans (C. A. 28, 6680). E. J. Rosenbaum

Metal cleaning here and abroad. R. W. Mitchell. *Iron Age* 136, No. 13, 112-13, 116-17, 122, 126, 132, 134, 141-3 (1935), cf. C. A. 29, 4307. —Various types of equipment are discussed. Methods are classified as phys. and chem., which include the use of solvents, saponifiers, and emulsifiers. F. G. Norris

The preparation of light-metal surfaces for painting. G. O. Taylor. *Paint Manuf.* 6, 19-21 (1936). —A review of methods of prep. light-metal surfaces to secure max. adhesion of paint under conditions of vibration, stress and corrosion. Mech. and chem. methods of roughening are noted. The water-EtOH-H<sub>2</sub>O<sub>2</sub> soln. degrades the surface, roughens and etches it and renders it passive by the formation of a thin film of insol.  $AlPO_4$ . Al alloys such as duralumin are protected by (1) electrooxidation or (2) by chem. oxidation. Special treatment for Mg alloys is touched upon. The patented alkali-chromate treatment for these alloys and Se processes are briefly discussed. W. H. Boynton

Galvanizing defects on steel sheets due to defective base material. M. von Schwarz and H. Fromm. *Korrosion u. Metallschutz* 11, 241-5 (1935). —The connection between carbide structure and appearance of the Zn coating is shown by photomicrographs of 4 types of sheet steel contg. 0.12, 0.07, 0.06 and 0.016% C. The more complete the formation of cementite, the more regular and attractive the appearance of the coating. Degenerated pearlite has a depreciating effect. This is explained by the assumption that normal pearlite as well as free boundary cementite constitute normal equi. in which the carbide is densely embedded in the ferrite, while in intermediate stages microcapillary cavities are present which cause H<sub>2</sub> absorption during pickling and thus affect the Zn coating. Leopold Pessel

The mercurous nitrate test. H. C. Jennison. *Metals & Alloys* 6, 348-9 (1935). —Specifications are cited showing the proper manner of using the  $HgNO_3$  test in evaluating Cu alloys. The quant. application of this method is shown to be very misleading. Downs Schaaf

Progress in corrosion- and heat-resisting steels. W. H. Hatfield. *Metallurgia* 13, 41-4 (1935). —A review of the development and use of these steels. J. L. G.

Corrosion of lead-alloy-coated steel cylinders used as fire extinguishers. W. L. Hewlett. *Chemistry & Industry* 54, 1094-5 (1935). —Immersion expts. with partly stripped steel plates coated with Pb-Sn alloys in a  $NaHCO_3$  soln. showed increasing corrosion of the exposed steel surfaces with decreasing Sn content of the coating. The potentials of the cells likely to occur, i. e., of Fe-Cu, Pb-Cu, Pb-Fe, Sn-Fe and Sn-Pb, in satd.  $NaHCO_3$ , were observed for 35 days. The alloy layer is subjected to preferential attack, while a protective basic carbonate film forms on the steel. This film was broken down, and corrosion initiated, by the presence of NaCl, phthalic, malic or citric acid. Use of water free from excessive amts. of chlorides, sulfates,  $NH_4$  salts and org. acids is recommended. Saponin, due to the formation of acidic sapogenins, may cause corrosion. The addn. of 1 oz. of trisac. Na phosphate per gallon of soln. is recommended. Greasy protective layers, such as lanolin, should be avoided. Leopold Pessel

The corrosion of Elektron Alloy AM 503 by leaded fuels. I. Mechanism of attack. G. D. Bengough and L. Whitby. *J. Roy. Aeronaut. Soc.* 39, 1144-53 (1935). —Specimens of the wrought sheet alloy (Mn 2.5, Al, Zn and Cu 0.2 each, Si 0.4, impurities 0.5%, Mg remainder) were tested. After pickling in 10%  $HNO_3$ , the samples were placed in various fuel mixts. contained in glass vessels held in a mech. shaker. Expts. were conducted in an open shed, and daily max. and min. temps. were recorded. The fuels studied were a 40% tech. benzene mixt., a straight-run paraffin type aviation gasoline, and a naph-

themic-type fuel. Summary. Liquid  $H_2O$  is necessary for corrosion. Gasoline and  $H_2O$  mixed with  $PbEt_4$  or with  $n-C_4H_9Br$  alone do not cause appreciable corrosion, but when mixed with both products, intense attack may occur.

M. W. Schwarz

Welding in the chemical engineering industry. N. P. Inglis and W. Andrews. *Iron & Steel Inst., Symposium on the Welding of Iron and Steel* 1, 259-65 (1935). E. H.

Electric welding of heat resistant steels. N. Kreshchanovskii. *Novosti Tekhniki, Mashinostroenie* 1935, No. 30, 12-13. In elec welding of heat-resistant steels contg Cr, Ni and Si the electrodes must be coated. The first coating of the electrode consists of fluorspar 50,  $K_2CrO_4$  10, ferromanganese (Mn 75%) 5, ferrosilicon (Si 75%) 5, water glass 15, and  $H_2O$  10 parts. The electrode, 5 mm. thick, is dipped into the coating material and then dried at 150-60°. On the first coating, 0.5-0.7 mm thick, a second coating is formed consisting of marble 25, fluorspar 15, ferromanganese 5, KOH 6, water glass 30, and  $H_2O$  10 parts. B. V. Shvartzberg

Safety and sanitary measures in the welding and oxygen-cutting of metals. R. Granjon. *Rev soudure autogene* 27, No. 255, 8-10 (May, 1935). *Chimie & industrie* 34, 1082. In  $CaH_2$  welding the combustion gases have no injurious effects on the operators, and special ventilation is required only when the operation is carried out in confined spaces. In elec-arc welding great care is required to avoid eye-injury through fatigue, ultraviolet rays, and the even more dangerous infrared rays. The present-day practice of using, in arc welding, electrodes with thick coatings contg volatile or noxious substances necessitates not only adequate ventilation of the premises, but also direct aspiration of the fumes, vapors and dust. Welding of painted or galvanized parts necessitates adequate ventilation or the wearing of a mask. The same precautions are required in the blast-cutting of structures covered with Pb paints or anti-fouling coatings, which should be completely scraped off or otherwise destroyed.

A. Papineau-Couture

Manuf., properties and uses of refined Al (Gadeau) 4. Underground gasification of coal as a basis for direct reduction of Fe ore (Trautman) 21. Refractories for the electrothermic Zn industry (MacBrude) 19. Influence of the temp of drying of blast-furnace slag on the hydraulic properties of slag cement (Eremenko) 20. Examn of electrodeposited Ni coatings by x-ray diffraction (Wood) 2. Intermetallic compds (Klemm) 2. Technic for lattice-parameter measurements (Norton) 2. Semiactive refractory materials [for open-hearth furnaces] (Zhikharevich, Kogan) 19. Chemistry of corrosion (Crook) 14. App for heating metal bodies for surface hardening (Brit. pat. 435,343) 4. Agglomerating ores (Ger. pat. 622,146) 20.

Mineral flotations. Frederick G. Moses, Raymond W. Hess and Robert L. Perkins (to Barrett Co.) U. S. 2,027,357, Jan. 7. A diaryl disulfide such as ditolyl disulfide and a thiophenol or a mercaptan are used together for treating Cu sulfide ore materials or the like.

Setting apparatus for concentrating ores, etc. Wilhelm Oberjohann. Ger. 622,023, Nov. 18, 1935 (Cl. 1a, 4). An automatic device for regulating the discharge of the gang is described.

Setting apparatus for concentrating ores, etc. Schuchtermann & Kremer Baum A-G. *für Aufbereitung*, Ger. 622,024, Nov. 18, 1935 (Cl. 1a, 4). An automatic device for regulating the discharge of the gang is described.

Extraction of ore. George J. MacKay. Can. 354,049, Nov. 12, 1935. Refractory ores are prepd. for extrn. by mixing the concentrate with a carbonate or nitrate of Na or K and the mixt is heated to 950-1250°F, but below the temp. of fusion. During this treatment Na polysulfides are formed by removal of one atom of S from the  $FeS_2$ , and Na thioarsenate is formed by action on the arsenopyrite particles. The heated mixt is quenched in water to form sol. salts of the precious metals, and is then treated with lime and filtered.

Wet separation of ores, coal, etc. Léon Hoyois. *Fr.* 788,688, Oct. 14, 1935. Details of app and operating currents of liquid.

Apparatus for milling and separating ores such as those of precious metals. John H. Dequer. U. S. 2,026,825, Jan. 7. Various structural, mech. and operative details.

Recovering values from ore pulp. Max Kraut. Can. 354,045, Nov. 12, 1935. A part of the pulp is drawn off, aerated under pressure and returned below the surface of the pulp body in widely dispersed jets. Structural details are described.

Ores of beryllium. Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. *Fr.* 788,243, Oct. 7, 1935. Ores of Be, particularly beryl, are heated to about 400° with alk. compds., particularly caustic alkalis, in the presence of water and the disaggregation product is extd. with water, aq. alkalis, etc., and the residue is worked up in known manner with acid. For 1 mol. of beryl, 16 mol. of caustic alkali are used.

Reducing ores. Victor Defays. *Ger.* 622,125, Nov. 21, 1935 (Cl. 18a, 1.10). See *Fr.* 737,840 (C. A. 27, 1603).

Sulfating nonferrous metals in ferruginous sulfide ores or roasts. Carl P. Debuch (to American Lurgi Corp.) U. S. 2,026,775, Jan. 7. Material contg Fe sulfide together with sulfides of other metals, such as Meggen pyrites, is passed through a rotary tubular furnace. At an early stage of its passage the ore is subjected to roasting conditions, and the temp. of the ore is maintained at a sulfating temp. for a substantial part of its passage while simultaneously introducing into the furnace gases of the sulfating zone a dust of a catalyst such as oxidized pyrites dust for conversion of  $SO_2$  into  $SO_3$ , and the ore and resulting gases are caused to move through the furnace countercurrent to each other, at least a part of the air admitted into the furnace being introduced into the furnace in portions at points intermediate the charging and discharging ends.

Plant for roasting ores. Metallgesellschaft A-G. (Carl Paul Debuch and Ernst Markworth, inventors). *Ger.* 619,581, Oct. 4, 1935 (Cl. 40a, 2.01).

Apparatus for introducing materials such as sulfide ore fines into furnaces. Charles A. Gallagher and Carl M. Allen (to General Chemical Co.). U. S. 2,027,817, Jan. 14. Structural and operative details.

Furnace with helioidal hearth for roasting ores. Paul Flajollet-Bracq. *Fr.* 788,185, Oct. 6, 1935.

Rotary furnace for roasting sulfidic substances, etc. Metallgesellschaft A-G. (Carl P. Debuch, inventor). *Ger.* 619,857, Oct. 8, 1935 (Cl. 40a, 2.01).

Blast furnaces. Dortmund-Hoerder Hüttenverein A-G. *Fr.* 788,333, Oct. 8, 1935. Means for closing breaches in the wall is described.

Blast-furnace throat. Franz Suck. *Ger.* 619,908, Oct. 11, 1935 (Cl. 18a, 6.05).

Open hearth furnace for steel manufacture. Martin J. Conway. U. S. 2,027,151, Jan. 7. Various structural and operative details, including means for balancing the pressure of preheated air entering the furnace and products of combustion or waste gases leaving the furnace.

Nonreversing open-hearth furnace. Fred H. Loftus. U. S. 2,028,290, Jan. 21. Various structural and operative details.

Muffle furnaces. Brayshaw Furnaces and Tools Ltd., Shipley. N. Brayshaw and Frederick C. Newman. *Brit.* 434,593, Sept. 2, 1935. In a gas- or oil-fired muffle furnace for the heat-treatment of steel, etc., the muffle atm. is circulated by a fan or air propeller.

Annular muffle furnace for the continuous distillation of zinc, cadmium, mercury and like metals. Fried. Krupp Grusonwerk A-G. *Ger.* 622,472, Nov. 29, 1935 (Cl. 40a, 5.01).

Bell-type annealing furnace suitable for annealing metal articles. Albert N. Otis (to General Elec. Co.). U. S. 2,028,106, Jan. 14. Structural details.

Metal-heating furnaces. Henry W. Spencer and Leonard S. Davis. *Brit.* 436,181, Oct. 7, 1935. The suspended roof is enclosed in an outer insulating housing to provide

a jacket through which air, in regulatable quantity, is forced by a blower, the air thus heated being supplied to the burners

Method and apparatus for hardening the surfaces of metal articles, e. g., gear-wheels, by means of a burner Gleason Works Brit 435,831, Sept 30, 1935

Apparatus for hardening tools by treatment with a blow-pipe flame followed by chilling I G Farbenind A -G Ger. 622,126, Nov 21, 1935 (Cl 18c 234)

Case-hardening E I du Pont de Nemours & Co Brit. 434,961, Sept 12, 1935 Fe and steel articles are case-hardened by immersion in a bath comprising a fused nonoxidizing salt having a suitable m. p., or a mixt of such salts, to which has been added a suitable org N compd., the articles thereafter, if desired, being quenched The bath may comprise 1 or more alk. earth salts, preferably the halides, or a mixt of 1 or more such salts with 1 or more alkali metal salts, preferably the halides and (or) carbonates, or 1 or more alkali cyanides and (or) 1 or more alk. earth cyanides Among org N compds that may be used are arylamines, alkylamines, pyridine, ammoniated org substances, e. g., ammoniated peat, or org CN compds, e. g., HCN or a polymer thereof, CNNH<sub>2</sub>, dicyanodiamide or melon

Case-hardening tools I G Farbenind A G (Hermann Holler, inventor) Ger 619,694, Oct 11, 1935 (Cl 18c 112) In case hardening, the tools are heated by a blow flame and chilled A pyrometer and switch arrangement ensure that the gases of the flame are stopped at a desired temp and a chilling agent such as water applied

Heat-treating steels and iron alloys Shinta Matsunawa, Fusatoshi Nozawa and Masahiro Suzuki Brit 435,223, Sept 17, 1935 The heated steel, etc., are quenched in a cooling medium comprising an emulsified mixt. of H<sub>2</sub>O and mineral and (or) fatty oil contg up to 15% of an emulsifying agent, e. g., an inorg. base, e. g., NaOH, KOH, Ca(OH)<sub>2</sub>, an org. acid, e. g., oleic, a soap or a mixt contg 2 or more such substances mixed or combined, except petroleum when a mineral oil is used alone In an example, a spring is heated to 850° and quenched in an emulsified mixt of fish oil 75, NaOH 0.1 and H<sub>2</sub>O 25%

Apparatus (with a muffle-surrounded nickel container) for heat-treating high-speed steel with fused boric oxide Chester B. Sadtler (to Barber-Colman Co.) U S 2,027,065, Jan 7 Various structural and operative details

Heat treating cobalt-tungsten-chromium alloys Werner Köster (to Veteinigte Stahlwerke A -G) U S 2,027,780, Jan 14 An alloy contg Co about 40-85, W 5-40 and Cr 10-40% and not over 0.3 C, is subjected to a pptn. hardening by heating to about 1000° or higher but below the m. p., rapidly cooling and annealing at 500-900°

Annealing metal bands Matthias Ludwig Ger 619,835, Oct. 9, 1935 (Cl 18c 610) App for even cooling or chilling is described

Annealing steel Vereinigte Stahlwerke A -G (Franz Eisenstecken, inventor) Ger. 622,078, Nov 19, 1935 (Cl 18c 880) Addn to 621,346 (C. A. 30, 10204) The process of Ger. 621,346 is modified for using Co or Mo or an alloy thereof as the catalyst for deoxidizing the protective gas

Steel-making slags Augustus B Kinzel (to Electro Metallurgical Co.) U S 2,027,868, Jan 14 For improving the working characteristics of basic open-hearth steel slags, there is added to them a substantial quantity (suitably about 5-10%) of Fe oxide so that the fluidity of the slags is increased and their cutting action on the hearth and lining of the furnace is decreased

Phospho magnesium slags Forges et aciéries du nord et de l'est. Fr 787,803, Sept 30, 1935 In making slags in a Thomas converter, 30-50% of the lime, according to the amt. of metal to be treated, is replaced by fritted dolomite in pieces the size of which is appropriate to the content of MgO to be obtained

Sintered hard metals Gustav Boecker (to "Acidia")

U. S. 2,027,763, Jan. 14 See Fr. 780,081 (C. A. 29, 58039)

Metal powders. Établissements Expert-Bezançon Fr. 788,358, Oct. 9, 1935 Metals of relatively low m. p., e. g., Zn, Sn, Sb, Cd, Al and especially Pb, are poured molten into a heated vessel in which they are subjected to the action of an agitator which pulverizes them to fine droplets A gaseous current poor in O, or inert or having a reducing action is passed through the vessel and carries the fog of metal to deposition chambers where it is collected as a powder

Aluminum powder Hartstoff-Metall A -G. (Hametag). Ger 619,624, Oct 4, 1935 (Cl 22f 13) Al powder is obtained by grinding Al in an atm of indifferent gas with a 1-3% content of O or water vapor The powder is suitable for making Al paint

Fluxes for aluminum Horace C Hall Brit 435,270, Sept 18, 1935 A flux for Al and its alloys is prep'd by mixing ZnCl<sub>2</sub> with NaF, with or without KF, in the presence of H<sub>2</sub>O so that ZnF<sub>2</sub> is formed. The ZnCl<sub>2</sub> may be replaced up to 10% by MgCl<sub>2</sub> and the NaF or KF may be replaced up to 20% by the phosphates or borates BaCl<sub>2</sub> and SrCl<sub>2</sub> may be added to reduce the m. p. and increase stability under heat, and NaMnO<sub>2</sub> or KMnO<sub>4</sub>, K<sub>2</sub>CrO<sub>4</sub> or Na-CrO<sub>4</sub>, K<sub>2</sub>SO<sub>4</sub> or Na<sub>2</sub>SO<sub>4</sub> to increase reactivity

Refining aluminum and its alloys British Non-Ferrous Metals Research Association, Daniel Hanson and Ian G Slater Brit 435,104, Sept 16, 1935 In treating molten Al, etc., prior to casting, the metal is agitated while maintained in an atm., free from H, H<sub>2</sub>O-vapor or other H gas, supplied to the space above the metal, the surface of the Al, etc., being simultaneously treated with a flux that dissolves any oxide present or formed during the process The atm may consist of dry N, CO or CO<sub>2</sub> and the flux consists of KCl 50, NaCl 40 and NaF 10 parts

Flux for cadmium and its alloys American Smelting & Refining Co. Fr 788,493, Oct 10, 1935. A flux for use in fusing Cd and its alloys is composed of CdCl<sub>2</sub> and (or) CdBr<sub>2</sub> and one or more alkali salts of the group comprising chlorides and bromides of Li, K and Na An example contains CdCl<sub>2</sub> 58, KCl 22 and NaCl 20%

Metallic lithium Miles G Hanson U S 2,023,390, Jan 21 Reduction of Li-O by Mg or Al is effected at a pressure below 6 mm. of Hg and at a temp. of 760-900° and the liberated metal is condensed on a cooled portion of the app used

Magnesium Oesterreichisch Amerikanische Magnesit A -G Brit 435,231, Sept 17, 1935 See Fr. 787,476 (C. A. 30, 10174)

Obtaining hard bodies Anton Kratky Brit 434,830, Sept 10, 1935 Hard bodies, particularly for tools, are obtained from carbides, etc., with or without addns. such as nitrides, metals, etc., by heating a shaped block or blank of the material above a matrix into which the material is then forced under pressure App is described. Cf C. A. 30, 10207

Carbides, nitrides, borides, silicides, carbonitrides and titanides Vereinigte Edelmetall A -G. Brit 435,751, Sept 23, 1935 Carbides, etc., adapted for the manuf. of tools, etc., are obtained from compds. contg the desired elements, e. g., chlorides, fluorides, iodides, oxides, carbonyls, by supplying heat to these in the gaseous or vaporous state within the free space of a chamber which is heated solely from the exterior thereof to 800-2500°, the desired compds. being ppt'd. in solid form. In a modification, at least 1, but not all, of the elements is supplied in the gaseous or vaporous elemental form. Among examples, (1) a mixt of hydrocarbon gas and H<sub>2</sub> is passed through a C-H<sub>2</sub> soln. of W carbonyl and then through a C tube heated to 1200-1400° to produce W<sub>2</sub>C, and (2) N is passed through MoO<sub>3</sub> heated to 1400° in an alumina crucible, the resulting gases being blown into a reaction chamber at 800-1400° contg a hydrocarbon to produce MoC. The carbides, etc., may be fused or sintered together or embedded in a matrix of a binding agent, e. g., a metal. Alternatively, the carbides, etc., may be

obtained from the gaseous phase simultaneously with the binding agents, both carbide-forming and metal-forming compds being introduced into the heated chamber.

Boron carbide articles such as bearings, dies or sand blast nozzles Raymond R. Ridgway and Bruce L. Bailey (to Norton Co.). U. S. 2,027,786, Jan. 14. B carbide granules are heated in a neutral environment to a temp. at which they will cohere under pressure, pressed in a C mold to the desired shape without permitting molten material to remain in contact with the carbon, thus forming a B carbide article having a conchoidal cryst. fracture which appears substantially as a single phase free from parting planes of graphite. App. is described.

Wire-drawing die or boron carbide Raymond R. Ridgway and Bruce L. Bailey (to Norton Co.). U. S. 2,027,787, Jan. 14. Boron carbide of the formula  $B_4C$  is used. U. S. 2,027,788 relates to bearings formed of  $B_4C$  and suitable for spindles of textile machinery, etc.

Photograph reproducing and recording needles with tips of titanium carbide Louis E. Turner. U. S. 2,026,760, Jan. 7.

Permanent magnets Robert Bosch A-G. Brit. 435,544, Sept. 23, 1935. See Fr. 783,309 (C. A. 29, 79303).

Forming articles from plated metal powders Charles Hardy (to Hardy Metallurgical Co.) U. S. 2,027,532, Jan. 14. A powder such as Fe powder plated with Cu is compressed into a coherent mass while wet with water and before the original soft amorphous character of the plating has been altered by heating.

Apparatus for tin plate manufacture Alexander R. Weibel and Wm. Jones (to Jones & Laughlin Steel Corp.). U. S. 2,028,306, Jan. 21. Various structural, mech. and operative details.

Compound metal articles Frederick F. Gordon. Ger. 622,484, Nov. 29, 1935 (Cl. 49f. 5). In the manu. of plates, billets, tubes, etc., of superimposed metal layers, the layers are united with a binder comprising Mn or an alloy or mixt. thereof with Ni, Fe, Co and (or) Cr, such mixt. or alloy contg. at least 10% of Mn. The binder may be taken in the form of powder or granules or in coherent form, e. g., as sheet or strip, and may be used alone or with a flux, e. g., borax. The metal layers are united by heating them with the interposed binder to about 1350°, with or without pressure. The process is particularly suitable for uniting layers of iron or steel. Numerous binding compns. are described, and various details are given.

Apparatus for making composite metal strips The Cleveland Graphite Bronze Co. Brit. 435,309, Sept. 19, 1935. A strip, e. g., of steel, is passed through a bath of molten coating metal, e. g., especially of Babbitt-metal in the manu. of bearings, and is withdrawn through a die opening, molten metal being removed from the lower side by a scraper.

Raw pig iron Max Paschke and Eugen Peetz. Fr. 788,009, Oct. 2, 1935. Raw pig iron is made from a fusion bath rich in acids or S by means of a fuel contg. more or less S by adding the substances to form the slag, e. g.,  $CaCO_3$ , only in such amts. that an easily fusible acid slag is formed and crude iron having a high S content. This crude iron is desulfurized after casting by a basic slag or other usual means. The operation may be carried out in a blast, oscillating or drum furnace.

Malleable iron Karl Roesch and Otto J. Schleimer. Brit. 434,846, Sept. 10, 1935. See Fr. 774,805 (C. A. 29, 21414). In 435,602, Sept. 24, 1935, addn. to 434,846, the castings contain C 2.2-3.2, S less than 0.15, Si less than 0.8 and Mn at least twice the Si but not greater than 2%.

Sponge iron Casimir J. Head. U. S. 2,028,105, Jan. 14. See Brit. 421,566 (C. A. 29, 36454).

Wrought-iron articles Martin J. Conway. Brit. 434,910, Sept. 11, 1935. See U. S. 2,012,599 (C. A. 29, 6874).

Cementation of iron and steel Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. Ger. 622,445, Nov. 28, 1935 (Cl. 18c. 3.15). A fused cementation bath

comprises inert salts, an alk. material and C. A typical bath comprises a fused NaCl-KCl mixt. contg. subdivided wood charcoal 3 and NaOH 10%.

Plating iron with steel. Tenner Walzwerk A-G. Ger. 622,422, Nov. 29, 1935 (Cl. 49f. 5). Addn. to 589,298 (C. A. 28, 13174). The mech. process of plating Fe with Cu or Ni or their alloys, described in Ger. 589,298, is now applied to the plating of strip iron, preheated to 500-700°, with Cr-Ni steel.

Steels Marc Burkitt. Brit. 435,576, Sept. 24, 1935. Steels for the manu. of poppet and like valves contain C, Cr and Si with 1 or more of the metals Ni, Mn, Mo, Co and W in specified proportions.

Steel Veremigte Stahlwerke A-G (Eduard Herzog, inventor). Ger. 622,077, Nov. 19, 1935 (Cl. 18b. 10). A plant is described for making highly deoxidized steel by passing the fused metal through a slag of high  $SiO_2$  content. Cf. C. A. 29, 47282.

Steel Walther Mathesius. Fr. 788,476, Oct. 10, 1935. A Ti steel is made by covering a bath of steel with a slag poor in silicic acid and composed approx. of  $TiO_2$  70 and  $CaO$  30%, the content of Ti in the bath by reduction of  $TiO_2$  of the slag being sufficiently high so that, after satn. of the N, S and O of the bath by Ti, the C enters into combination as  $TiC$ . A suitable slag is obtained by refining an ordinary bath of steel with the correct amt. of titaniferous Fe ore and lime. For tempered steels the content of C should be 0.3% and more, for constructional steels 0.1-0.2%, for steels resistant to corrosion 0.1% and for sheet steel to be subjected to deep stamping below 0.1%.

High-speed steels Herarus Vacuum-Schmelze A-G and Werner Hessebruch. Brit. 435,552, Sept. 19, 1935. Great hardness is produced in steels contg. Fe 0.1-3, C 0.2-2.5, W 12-25, Mo 0-17.5 and V 0-7.5% by quenching from 1100-1350° and then aging or annealing at 400-700°. The steels may also contain Cr about 3-5 and Co about 3-15%. In an example, a steel contg. W 18, Cr 4, C 0.8, Mn 0.2, Si 0.2 and Be 0.6 is quenched in oil from 1320° and annealed at 500° for 1 hr.

Open-hearth steel Alvin A. Claassen (one-half to James L. Hyland). U. S. 2,027,300, Jan. 7. A molten metal charge in an open-hearth furnace, covered with a thick layer of slag, is heated, worked and refined and finally tapped out of the furnace, and the C content of the molten metal is controlled by introducing beneath the slag-covered surface of the molten metal a liquid material contg. C which can be absorbed by the molten metal.

Extrusion apparatus for production of steel tubes or the like Ernest F. Jones. U. S. 2,026,979, Jan. 7. Mech. features.

Alloy steel The Earl of Dudley's Pound Oak Works Ltd. and John T. Wright. Brit. 434,828, Sept. 10, 1935. Steel for structural purposes contains C up to 0.3, Cr up to 0.69, Cu 0.2-0.45, Mn 0.4-1, Si 0.05-0.3 and Ni 0.1-0.5, the sum of the Ni and Si exceeding the Cu.

Austenitic chrome nickel steel alloy products Paul Schafmeister and Erwin A. Spenle (to Fried. Krupp A-G). U. S. 2,027,554, Jan. 14. In treating products such as metal sheets having a stable surface, a first heating within the carbide pptn. range is followed by cold working and then reheating above the recryst. temp. but below the temp. at which the carbides redissolve, to effect recryst. of the cold worked metal.

Magnet steel containing nickel and aluminum Tokushichi Mishima. U. S. 2,027,994, Jan. 14. Permanent magnets are formed of metal contg. Fe together with Ni 5-40 and Al 7-20%. U. S. 2,027,995 relates to magnets of similar alloys with the further addn. of Cr 1-5, W 0.5-8 or Mo 10% or less. U. S. 2,027,996 relates to magnets contg. Fe together with Ni 5-40, Al 7-20 and Co 0.5-30%. U. S. 2,027,997 relates to magnets formed of alloys contg. Fe together with Ni 5-40, Al 7-20 and Cu from a trace to 20%. U. S. 2,027,998 relates to magnets formed of alloys contg. Fe together with Ni 5-40, Al 7.20, Co 0.5-30 and Cr 1-5%. U. S. 2,027,999 relates to magnets formed of alloys contg. Fe together with Ni 5-40, Al 7-20 and Mn 0.5-10%. U. S. 2,028,000 relates to magnets formed of

alloys contg. Fe together with Ni 5-40, Al 7-20 and V 1 from a trace to 10%.

**Nitrogen-containing metals and alloys.** Wm. C. Read (to Electro Metallurgical Co.). U. S. 2,027,837, Jan. 14. An exothermic mixt. of a N-contg. oxidizing agent such as  $\text{NaNO}_3$  and a strong reducing agent such as Si or Al is caused to react in close assocn. with metallic materials such as *ferro-Cr*, at least one of which has a substantial affinity for N and which are substantially free from metal oxides, so that N is liberated and brought into contact with the molten constituents.

**Die-casting alloys.** John R. Freeman, Jr. (to American Brass Co.). U. S. 2,023,129, Dec. 3. Zinc alloys which as cast have a good surface finish contain Cu 0.5-7.5, Pb 0.25-3.0, Sn 0.25-3.0, Al 0.05-1.0 and Si 0.05-0.5%.

**Hard alloys.** Fried. Krupp A.-G. (Karl Schröter, Kurt Agte, Kurt Moers and Hans Wolff, inventors). Ger. 622,341, Nov. 26, 1935 (Cl. 40b 17). W carbide contg. 3-7% of C is mixed with an auxiliary metal of lower m. p., e. g., Fe, Ni, or Co, and also with Ti carbide, and the powd. mixt. is compressed and sintered. The proportions are W carbide at least 35, metal up to 25, and Ti carbide up to 40%.

**Sintered hard metallic alloys.** Karl Becker, Karl Schröter and Hans Wolff (to General Elec. Co.). U. S. 2,026,938, Jan. 7. Hard material suitable for tools, etc., contains a W carbide contg. C 3-7, an auxiliary metal such as Fe, Co or Ni not over 20, together with V carbide 0.01-30%.

**Wheel runs, etc., of special alloys.** Herman J. van Royen. U. S. 2,028,029, Jan. 14. Wheel runs, etc., of high resistance to abrasion and of lamellar-pearlitic structure are formed of steel contg. C 0.65-0.78, Si 0.4-0.8, Mn 0.4-0.85%, together with a double carbide-forming metal such as Cr 0.3-0.8, W 0.25-0.7 or Mo 0.22-0.65%, the balance being mainly Fe and the total amt. of double carbide-forming metals being such that the compn. is eutectoid and that the finished article of the steel has a lamellar pearlitic structure. Cf. C. A. 30, 1019<sup>1</sup>.

**Dental alloy.** Arthur W. Gray. Can. 353,526, Oct. 15. An alloy for dental amalgam is composed of finely divided solid particles, nearly all of which are hard, dense and free from cracks. The particles have an av. length of about 30  $\mu$  with a width and thickness well within the same order of magnitude. The alloy comprises Ag 67-70, Sn 25-9, Cu 3-5, and Zn less than 1%. When it is combined with Hg, a plastic mixt. results which will harden rapidly into a solid metallic body.

**Aluminum alloys.** Fred Keller, Jr., and Richard S. Merritt (to Aluminum Ltd.). Brit. 434,925, Sept. 11, 1935. This corresponds to U. S. 2,003,524 (C. A. 29, 4729<sup>1</sup>), the Cu being 0.05-0.45%.

**Aluminum alloys.** Aluminum Ltd. Fr. 787,996, Oct. 2, 1935. Solid  $\text{CO}_2$  is placed on the surface of molten Al alloys whereby a dry relatively cold atm. of  $\text{CO}_2$  is formed which prevents access of air to the alloy.

**Aluminum alloys.** Aluminum Ltd. Fr. 788,458, Oct. 10, 1935. Al alloys which are not submitted to a thermal treatment to improve their phys. properties contain Mn 0.1-3.5, Cu 0.1-0.45, Mn 0.1-1 and Cr 0.1-0.5%, e. g., 1.4, 0.5, 0.4 and 0.25%.

**Beryllium alloys.** Compagnie de produits chimiques et électrometallurgiques Alais, Fröges et Camargue. Brit. 433,747, Sept. 26, 1935. In making alloys by reducing Be compds by an alloy of Mg with a metal less electropos. than Be, e. g., Cu, Al, Zn, which it is desired to alloy with Be, alkali double fluorides are used contg. less alkali fluoride in proportion to the  $\text{BeF}_2$  than is indicated by the formula  $\text{BeF}_2 \cdot 2\text{NaF}$ . An alloy of Zn and Be thus obtained may be remelted in a flux of equal parts of  $\text{BaCl}_2$  and  $\text{BaF}_2$  to distill off the Zn and leave molten Be. Be alloys may be formed with any metal that alloys with Zn and is not volatile at 1300°, e. g., Fe, Ni. Thus, a Zn alloy contg. Mg 17.1 and Fe 6.13% is melted in a crucible with  $\text{BeF}_2 \cdot \text{NaF}$  to give a Zn-Fe-Be alloy contg. Be 7, Fe 7 and Mg 1-3%. This is heated to 1300° in a suitable flux and yields an alloy contg. Fe 50 and Be 50%. A Cu-Mg alloy contg. 12% Be is obtained by adding a

Cu-Mg alloy contg. 26.7% Mg to  $\text{BeF}_2 \cdot \text{NaF}$  in a crucible heated to 1000°, pouring the Cu alloy contg. Be 11 and Mg 3%, and re-melting to get rid of the Mg.

**Alloys containing boron.** Soc. d'électrochimie, d'électrometallurgie et des aciéries électriques d'Ugine and Jean L. Andrieux. Fr. 788,145, Oct. 4, 1935. A calcd. amt. of an alk. earth boride, particularly of Ca or Ba, is mixed with the metal to be alloyed with B, or with the alloy into which B is to be introduced, or an oxide or a salt of the metal to be alloyed with B, and the mixt. is then heated under a layer of flux to a temp. sufficient to melt the whole. The reduction of the oxide or salt may be effected by means of the boride itself or by Al, Mg, Ca, Na, C or Si.

**Cadmium alloys.** General Motors Corp. Fr. 788,117, Oct. 4, 1935. Alloys for journal bearings contain Cd and a small amt. of Ag, and preferably, a small amt. of Cu and (or) Ni, e. g., Cd 97-8, Ag 1.75-2.5 and Cu or Ni 0.25-0.5%.

**Columbium alloys.** Electro Metallurgical Co. Fr. 787,843, Sept. 30, 1935. See Brit. 434,400 (C. A. 30, 717<sup>1</sup>).

**Copper alloys.** Elmer L. Munson (to American Brass Co.). U. S. 2,027,750, Jan. 14. Alloys which have good hardness, strength and ductility contain Cu about 91.75-98.75, Ni 2.0-5.0, Al 0.5-2.0 and Be 0.75-1.25%.

**Ductile copper base alloys.** Henry L. Burghoff and David E. Lawson (to Chase Cos. Inc.). U. S. 2,027,807, Jan. 14. Alloys contg. Cu 75% or more, Te 0.05-1.75% and which also may contain Si, Zn, etc., have substantially better machinability than similar alloys not contg. Te.

**Iron alloys.** Hans von Kantzow. Brit. 434,849, Sept. 10, 1935. An Fe alloy for an elec. heating resistance contains Cr 2-30, Al 2-6, Si 2-20 and Co 0.1-30, with or without Mn 0.5-12 and V (or) Zr 1.8%. C may be present up to 0.5%.

**Iron alloys.** Crane Ltd. and Horace H. Shepherd. Brit. 435,656, Sept. 25, 1935. The phys. properties of malleable and gray cast Fe are improved by adding Cu 0.25-3, Ti 0.05-0.5 and, optionally, Al 0.015-0.15%.

**Cast-iron alloys.** James E. Hurst and Bradley & Foster Ltd. Brit. 435,316, Sept. 19, 1935. In the production of N-hardenable cast Fe alloy castings contg. Si, Al and Cr, with or without Ni, Mo, Cu, Ti, etc., the castings are formed in a sand mold and the alloy contains C 2.5-4, Si up to 1, Al 0.3-1.75 and Cr 0.5-2.0%. The Al varying inversely with the total of Si, Ni or other graphitizing elements and the Cr varying directly with the graphitizing elements so that the casting is devoid of graphite either in the as-cast condition or after a short annealing. The combined content of Ni, Mo, Cu or Ti does not exceed 3%. Prior to nitriding, the castings are annealed at 850-1050° and slowly cooled or are hardened by quenching from 800-875° in an oil or molten salt bath at 150-200° and then reheated to 700-750°.

**Iron-silicon alloys.** Isabellen-Hütte G. m. b. H. Brit. 434,847, Sept. 10, 1935. See Ger. 608,194 (C. A. 29, 2501<sup>1</sup>).

**Iron-silicon alloys.** Isabellen-Hütte G. m. b. H. and Friedrich Heusler. Brit. 435,093, Sept. 13, 1935. Divided on and added to 434,847 (preceding abstr.). In a modification of 434,847, the alloys are prep'd by melting down the pig Fe, adding Si or Fe-Si to the melt and removing the graphite that seps from the melt. The alloys may also contain Ni or Cu up to 3%. Cf. C. A. 29, 2501<sup>1</sup>.

**Ferromanganese.** Mannesmannröhren-Werke and "Sachtleben" A.-G. für Bergbau und chemische Industrie. Fr. 788,070, Oct. 3, 1935. Ores or slags contg. Mn and Fe but not in the correct proportions for Fe-Mn, are fused, if necessary with the addn. of  $\text{CaCO}_3$ , fluorspar, etc., to produce a slag of protoxide of Mn of low f. p., and a reduction is carried out at a temp. below that of the reduction of Mn, so as to withdraw an amt. of Fe to leave an intermediate product contg. the correct amt. of Fe and Mn. This is then reduced in the usual manner to Fe-Mn.

**Zinc alloys.** The New Jersey Zinc Co. Ger. 622,240,



Nov 23, 1935 (CI 406 13). See U S 1,716,599 (C. A. 30, 3657)

Treatment of zinc alloys. Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. Ger 622,449, Nov. 23, 1935 (CI 404 220) Zn alloys not susceptible to cementation e. g., brass, are improved in their mech. properties by treatment for short time in a fused bath comprising inert salts, e. g., NaCl and KCl, and a cyanide. The proportion of cyanide in the bath must exceed 5% and may be up to 30%. The alloys may be heated in a non-oxidizing gas before the treatment

Bronzing Henry Michault. Fr. 787,938, Oct. 1, 1935 Fe and steel are bronzed rapidly by dipping them into a bath contg. e. g., water 100, litharge 20, KCN 8, FeSO<sub>4</sub> 5, PbSO<sub>4</sub> 10 and MnO<sub>2</sub> 0.04 g

Producing polychromatic designs on aluminum and its alloys Ernest Windsor-Bowen and Charles Hugh Roberts Gower. Ger 622,186, Nov 23, 1935 (CI 484.3). See Brit 412,193 (C. A. 28, 7242\*)

Bath for galvanizing coils for evaporators and other heat exchangers by treatment with fused tin Wilhelm J. Heilenbroich. Ger 622,050, Nov 18, 1935 (CI 480.2) Structural features are described

Preventing corrosion John Graham Brit 435,420, Sept. 20, 1935 Corrosion of a metal article formed by working a metal body at an elevated temp. is prevented by applying a paint, e. g., comprising white lead and boiled linseed oil, to the new surfaces exposed by the working after the body has cooled down to 60-120°. The work piece may be decaled prior to painting

Rust preventing Victor Chemical Works. Fr. 788,036, Oct. 2, 1935 Fe is protected against rust by treating it with an aq. soln. of a hexavalent Cr compd. (chromates, dichromates, chromic acid and anhydride) and H<sub>2</sub>PO<sub>4</sub> and then drying. The ratio of, e. g., dichromate to H<sub>2</sub>PO<sub>4</sub> is about 25 to 100 and the concn. of H<sub>2</sub>PO<sub>4</sub> about 10-20%

Metals resistant to gaseous corrosion Soc. d'Electrochimie, d'Electrometallurgie et des aciers Electriques d'Ugine. Fr 788,359, Oct. 9, 1935 A metal such as Fe contg. 1 or more other metals, e. g., Al 4 or Cr 12%, is made resistant to the action of a corroding gas such as O or at a vapor at a high temp., by subjecting the metal at a

1 sufficiently high temp., to the action of the said gas or vapor at a partial pressure appreciably higher than the disocion fension, at that temp., of the noncorrodable compd. which it is desired to form at the surface of the metal, but keeping the partial pressure sufficiently low so that the layer does not become permeable by formation of too great a proportion of the noncorrodable compd.

2 Counteracting electrochemical corrosion of metal apparatus Fried. Krupp A.-G. (Andreas Gotta, inventor). Ger. 622,444, Dec. 9, 1935 (CI 127.3). Electrochem. corrosion in app. where surfaces of two different metals are in contact is counteracted by interposing between the surfaces a removable layer of the less electropos. metal. When the interposed layer has been sufficiently corroded, it is removed and replaced by another similar layer

3 Inhibiting corrosion of magnesium and its alloys Josef M. Michel (to Magnesium Development Corp.). U. S. 2,028,343, Jan. 21 Corrosion by solns. such as those of glycol or glycerol is inhibited by incorporating in the liquid about 0.1% or more of an alkali fluoride such as KF

4 Welding-rod alloys. Richard A. Wilkins (to Revere Copper and Brass Inc.). U. S. 2,027,330, Jan. 7. Hot and cold workable alloys capable, when deposited by a welding process, of forming a dense, machinable, hard weld metal of high-tensile strength, and characterized by materially reduced fuming as compared to brass of the same Zn content, contain Cu together with Si 0.05-0.3, Ni 0.25-5, Fe 0.25-3 and Zn 35-50%

5 Welding-rod alloys Homer W. Butterbaugh (to American Brass Co.). U. S. 2,028,317, Jan. 21. A filler material for welding or brazing comprises an alloy contg. Cu 55-65, Ni 0.1-3.0, Si 0.02-0.75 and Zn 31.25-44.88%

6 Welded ferrous metal pipe Geo. G. Walker (to Republic Steel Corp.). U. S. 2,028,096, Jan. 14. A ferrous metal pipe, having longitudinal elec. resistance butt weld and a tensile strength in excess of about 75,000 lb. per sq. in. of metal, contains Fe together with C about 0.1-0.45, Mn 0.7-1.7, P about 0.015-0.020, Cu about 0.5-1.05% and substantially no Si. Various details of app. and mfg. operation are described.

## 10—ORGANIC CHEMISTRY

CHAS. A. ROULLIER AND CLARENCE J. WEST

The structure of organic compounds K. K. Dubrovai "Sotsialist. Rekonstruktsiya Nauka" 1935, No. 4, 17-32, No. 5, 44-67, No. 6, 20-49 —A theory is advanced of the tetrahedral structure of org. compds. continuing fully the ideas of van't Hoff on the basis of generalizations of modern roentgenoscopic material leading to new structural conceptions. The mol. is considered as a static system with a definite direction of valences which are forces resulting from the forces of attraction and repulsion. In methane between the C and H atoms there is attraction while the H atoms are mutually being repulsed. The dimension of the central angle between 2 valences in the tetrahedron of methane is 109°28'. The C atoms are in the center of the tetrahedral system and therefore the direct rectilinear combination of 2 such atoms in the form of "C-C connection" is impossible, which also invalidates the Bayer strain theory. Connection of 2 atoms occurs always on a broken line and the breaking point where one by one or two by two or more valences of 2 such atoms are interconnected is called the nodal point of the connection. These points act as regards attraction as do the H atoms. The hydrocarbon radicals are considered as tetrahedral systems with a central C atom and with free valences where the interconnection of the radicals takes place. Having studied the distribution space of a series of compds. consisting of 2 interconnected radicals, D. gives calcs. of the distance in angstrom units between the atoms in the case of interconnected hydrocarbon radicals in paraffin, olefin and acetylene chains. These distances are for

C<sub>1</sub>-C<sub>2</sub> 1.54 (the same as for diamond), 1.45 (the same as for graphite) and 1.03; for H-H 2.52, 2.52 and 4.10; for C-H in all cases 1.54; for H<sub>1</sub>-H<sub>2</sub> 1.45, for C<sub>1</sub>-H<sub>1</sub> 1.61, 2.11 and 2.56; the rate of the chain-lengthening as one homolog passes into another one is 1.27. Rejecting the plane structure of benzene given by Kekulé-Tsje, D. gives his own structure of benzene and also a description and pictures of C<sub>6</sub>Me<sub>6</sub>, Ph<sub>6</sub>, cyclooctatetrene, C<sub>10</sub>H<sub>8</sub>, an thracene and phenanthrene structures as deduced from this theory.

B. V. Shvartberg  
The electron in organic chemistry. IV. Monohydroxy compounds M. S. Kharasch, Otto Rennuth and F. R. Mayo. J. Chem. Education 13, 7-19 (1936), cf. C. A. 28, 3381\*

Characterization of hydrocarbons Ernst Galle, Rudolf Klatt and Walter Friedl. Petroleum Z. 31, No. 36, 1-14 (1935).—See C. A. 30, 427\*

C. B. Jenni  
Mechanism of the slow oxidation of propane. Robert N. Pease. J. Am. Chem. Soc. 57, 2296-9 (1935), cf. C. A. 23, 3436 —The reaction was studied around 300° with mixts. contg. 10-30% O. The indicated stoichiometric equation is C<sub>3</sub>H<sub>8</sub> + 2O<sub>2</sub> → MeOH + HCHO + CO + H<sub>2</sub>O. The mechanism is discussed in terms of Rice's radical chain theory with MeO and C<sub>2</sub>H<sub>5</sub> as the chain carriers. The starting and stopping of the chains is considered.

H. J. Rosenbaum  
Chemistry of the acetylenes. III. Cracked gasoline as a source of α-olefins for the preparation of acetylenes. Homer J. Hall and G. Bryant Bachman, Ind. Eng.

Chem 28, 57-9(1936); cf. C. A. 30, 77-1-Pentene and 1-hexene were prep'd. in pure form by bromination and subsequent debromination of the corresponding olefins which were fractionated from Gyro process gasoline. The plays consist, were for 1-pentene,  $b_p$  39.5-41.0°,  $d_4^{20}$  0.6945,  $n_D^{20}$  1.3847, Hg deriv.,  $m$  117.0 8.83°, and for 1-hexene,  $b_p$  71-2°,  $d_4^{20}$  0.7170,  $n_D^{20}$  1.3988, Hg deriv.,  $m$  90.0-6.4°, 13 references. E. W. Tillotson

Oxidation of 3-methyl-3-pentene and 2-methyl-2-pentene by selenium dioxide André Guillemonat *Compt rend* 201, 904-6(1935) cf. C. A. 26, 5085a— $Me_3C MeCH(OH)Me$ ,  $b_p$  139-40°,  $d_4^{20}$  0.860,  $n_D^{20}$  1.4430 (acetate,  $b_p$  62-3°,  $d_4^{20}$  0.9010,  $n_D^{20}$  1.4318),  $Me_2C CHCH(OH)Me$ ,  $b_p$  61-3°,  $d_4^{20}$  0.857,  $n_D^{20}$  1.446 (acetate,  $b_p$  61-3°,  $d_4^{20}$  0.9037,  $n_D^{20}$  1.4318), were prep'd. in the manner previously described The Raman spectra for the above are given. Julius White

Raman effect and organic chemistry Study of isopropylacetylene, isopropylethylene and several of their derivatives Blanche Giedy *Bull soc chim* [5], 2, 1951-8(1935), cf. C. A. 29, 5807- Treatment of the mixt. of chlorides formed by the action of 77 g of  $Me_3CHAc$  on 250 g of  $PCl_5$  with  $NaNH_2$  contg. a trace of  $NaOH$  gave 3.5 g of isopropylacetylene (I),  $b$  28-9°, and a residual monochloride (II),  $Me_2C CClMe$ ,  $b$  94-9°,  $n_D^{20}$  1.4380,  $d_4^{20}$  0.925,  $M$  29.66 (calcd 29.69) Raman spectra detns. showed the presence of traces of the isomer,  $Me_2CHCHCl CH_3$ . The mixt. of  $Me_2CHCHCl CH_3$  and  $Me_2C CClMe$  obtained by the dehydration of iso- $AmOH$  over  $Al_2O_3$  at 450° was passed into chilled  $Br$  and the resulting mixt. of dibromides was fractionally distd. The main fraction consisted of 133% of  $Me_2CHCHBrCH_3$ ,  $Br$  which, on distn. from  $KOH$ , yielded 700 g (90%) of  $Me_2CHCHBrCH_3$  (III),  $b$  102-10°,  $n_D^{20}$  1.4574,  $d_4^{20}$  1.356,  $M$  28.84 (calcd 32.50), contg. a trace of  $Me_2CHCHClCHBr$  as indicated by the Raman frequency 1620. Treatment of 650 g of III with  $NaNH_2$  at 150° for 7 hrs. gave 75 g (25%) of I,  $b$  27.5-8.5°,  $n_D^{20}$  1.3785,  $d_4^{20}$  0.666,  $M$  28.54 (calcd 23.29) The methylation of 20 g of I with 25 g of  $NaNH_2$  and 70 g  $Me_2SO_4$  produced 9 g (36%) of  $Me_2CHC CMe$  (IV),  $b$  72.0-2.5°,  $n_D^{20}$  1.4078,  $d_4^{20}$  0.716,  $M$  28.18 (calcd 27.01), catalytically reduced to pure cis-4-methyl-2-pentene,  $Me_2CHCH CMe$  (V),  $b$  58.0-8.6°,  $n_D^{20}$  1.3858,  $d_4^{20}$  0.663,  $M$  29.72 (calcd 29.44) The reaction of 30 g of I with the  $EtMgBr$  from 15 g.  $Mg$  and 85 g. of  $EtBr$  in the cold and the addn. of 50 g. of  $AcH$  gave, after 2 hrs. heating and the usual treatment, 30 g. of  $Me_2CHC CCH(OH)Me$  (VI),  $b$  155-7°,  $n_D^{20}$  1.4418,  $d_4^{20}$  0.85,  $M$  34.25 (calcd 34.05), giving, on treatment with  $NaNH_2$ , the brick-red  $Na$  deriv. characteristic of acetylenic secondary alcs. The Raman spectra contained a line at 1680 attributed to an impurity due to the polymerization of the  $AcH$ . Methylation of 24 g. of VI with 12 g.  $NaNH_2$  and 42 g.  $Me_2SO_4$  in anhyd.  $Et_2O$  yielded 16 g. of  $Me_2CHC CCH(OH)Me$  (VII),  $b$  133.0-3.5°,  $n_D^{20}$  1.4190,  $d_4^{20}$  0.803,  $M$  39.60 (calcd 38.78), showing a slight elevation corresponding to that previously noted (C. A. 29, 6829) The lower-boiling fractions 3 and 4 (213 g.) of  $Me_2CHCHBrCH_3$  were treated with 130 g. of  $Zn$  in abs. alc. Distn. of the hydrocarbon produced gave a main fraction consisting of  $Me_2CHCH CH_3$  (VIII),  $b$  20.0-0.5°,  $n_D^{20}$  1.3725. The detn. of the Raman spectra of VIII completes the study of this series. I has the lines 2119 and 3304 characteristic of the true acetylenes but the line 345, characteristic of the radical  $-CH_2C CH$ , is missing as a matter of course. The strong line 375 generally found in the  $Me$ -substituted acetylenes does not appear in the spectra of IV or in those of  $CH_3C CMe$  and  $C_2H_5C CMe$ . Apparently the line 375 is characteristic of the group  $-CH_2C CMe$ . VI is similar to  $CH_3C CCH(OH)Me$  in possessing only 1 acetylenic line at 2241. The  $Me$  ether, VII, is similar to the other secondary ethers studied in having a unique strong line at 2241 but differs somewhat in showing no other lines at higher frequencies. VIII differs from the other pentenes but possesses the 5 lines 1286, 1417, 1641, 2998 and 3083 characteristic of a terminal double linkage. Comparison of the

Raman spectrum of V with that of the *trans*-isomer shows that the ethylenic line is displaced from 1674 to 1659 in passing from the *trans*- to the *cis*-isomer, that the lines 1351 and 1301 are weakened while the band 1257 is intensified. The influence of the  $Br$  atom is shown in the occurrence of the ethylenic line at 1630 in III and a similar influence of the  $Cl$  atom is shown by the line 1672 in II. All the compds. studied, with the exception of II, show a line whose frequency varies from 2065 to 2080. This line is not generally found in straight-chain aliphatic compds. and is attributed to the vibration of the isolated  $H$  atom of the iso- $Pr$  radical. C. R. Addinall

The synthesis of ethyl alcohol from artificially prepared gases Oil Institute in Baku *Seizhalist Rekonstruktsiya i Aukha* 1935, No. 4, 118—At a semiautomatic plant the utilization of not less than 80%  $C_2H_6$  was attained while employing up to 2.1% of  $H_2SO_4$  for a unit of alc. An exp't. plant based on the utilization of oil gases is being constructed. B. V. Shvartberg

Polymerization of methyl esters of higher unsaturated fat acids XVI. Polymerization of methyl esters of stearic acid Kichuro Kano *Sci. Papers Inst. Phys. Chem. Research (Tokyo)* 28, 140-5(1935), cf. C. A. 29, 2913- The central position of the triple bond in stearic acid (I) might confer properties other than those possessed by  $CH_3CH$  or long-chain mols. with a terminal triple linkage. Polymerization exp'ts. were carried out to examine the behavior of I in contrast to that of mols. contg. a double bond. A soln. of 1 kg. of oleic acid dibromide in 1 l. of com.  $AmOH$  was heated with 800 g. of  $KOH$  for 4 hrs. at 150°. The resulting soap was decomp'd. with dil.  $HCl$  and the crude acid after recrystn. from 80%  $EtOH$  gave I,  $m$  47-8°,  $l$  no (Wsp) 87.2 (calcd 89.93), neutralization no. 200.2 (calcd 200.15), mol. wt. 273 (calcd 280.26),  $Me$  ester (II),  $l$  no 85.3 (calcd 86.3), sapon no. 196.0 (calcd 190.64), mol. wt. 259 (calcd 294.3). II was heated in a  $H$  atm. for 20 hrs. and the product was sepd. into  $MeOH$ -sol. and -insol. portions. The latter showed the presence of a dimer (III),  $l$  no 82.0 (calcd 89.93), neutralization no. 182.4 (calcd 200.2), and mol. wt. 560 (calcd 560.5). III probably contains a double linkage. Color changes noted during the Wyp detn. indicate that, structurally, the polymerization of I takes place similarly to that of the  $Me$  esters of linolic and linolenic acids. C. R. Addinall

Compounds of bivalent carbon. XI. The carbon monoxide acetal cleavage of the esters and amides of diphenoxyacetic acid which occurs in chemical reactions Helmut Scheibler and Maximilian Depner. *Ber.* 68B, 2136-43(1935); cf. C. A. 29, 124-— $(PhO)_2CHCO_2Et$  (I) with  $NaOEt$  gives  $(PhO)_2C C(OEt)_2$  (II) and at the same time the  $C-C$  union is ruptured, with formation of  $C(O)Ph$ . In the prep'n. of I from  $Cl_2CHCO_2Et$  and  $NaOPh$  it was frequently observed that the yield was considerably lower than when it was prep'd. under the same conditions from  $Cl_2CHCO_2Na$ . This suggested that the  $NaOPh$  does not merely substitute the  $Cl$  atoms of the  $Cl_2CHCO_2Et$  but also adds to the ester group and the resulting ortho ester at once undergoes the carbon monoxide acetal cleavage. To obtain light on the cleavage process the action of  $KOPh$  on I and of  $NaOEt$  on  $(PhO)_2CHCO_2Ph$  (III) in benzene was studied. Among the volatile products which distd. over with the benzene  $HCO_2H$  was detected in both cases, while the residue, after treatment with water, contained  $PhOH$  and alkali formate. The reactions with I may be represented as follows:  $I + PhOK \rightarrow (PhO)_2CHC(OEt)(OPh)OK \rightarrow C(O)Ph + HC(OEt)(OPh)OK$  (IV)  $\rightarrow C(O)Ph + HC(OEt)(OPh)OK$ .  $C(O)Ph$  could not be isolated and only its hydrolysis products,  $PhOH$  and  $HCO_2H$ , were obtained. In the reaction of III with  $NaOEt$  there occurred, along with the above cleavage, the other reaction, resulting in the formation of II. It may be assumed that III first changes into I which with the  $NaOEt$  then forms II. The carbon monoxide acetal cleavage occurs more rapidly in alc. than in benzene, as in the prep'n. of I from  $Cl_2CHCO_2Et$  and  $PhONa$ , where considerable  $HCO_2Na$  is formed at the expense of the yield of I. The carbon mon-

oxide acetal cleavage is also observed in the action of  $\text{Et}_3\text{NMgBr}$  on I, which gives a neutral unsaponifiable product,  $b_p$  55°, contg. the  $\text{EtO}$  group. The low  $b_p$  indicates that it is a  $\text{HCO}_2\text{H}$  deriv., possibly  $\text{HC}(\text{OEt})_2\text{OPh}$ , formed by exchange of the ester groups (*Umsetzung*) in  $\text{HC}(\text{OPh})_2\text{OEt}$ , the addn. product of  $\text{EtOH}$  to  $\text{C}(\text{OPh})_2$ . Such a reaction presupposes the splitting off of  $\text{EtOH}$ , which might take place thus:  $(\text{PhO})_2\text{CHC}(\text{OEt})(\text{OMgBr})\text{NEt}_2 \rightarrow \text{C}(\text{OPh})_2 + \text{HC}(\text{OEt})(\text{OMgBr})\text{NEt}_2 \rightarrow \text{EtOH} + \text{C}(\text{OMgBr})\text{NEt}_2 \rightarrow \text{HNEt}_2 + \text{HCO}_2\text{MgBr}$ . A considerably higher yield of such a neutral,  $\text{EtO}$ -contg. compd. was obtained when the product of the reaction between  $\text{Et}_3\text{NMgBr}$  and I was heated with alc. under pressure at 150°. There was also obtained  $\text{PhOEt}$ , probably formed thus:  $\text{HC}(\text{OPh})_2\text{OEt} \rightarrow \text{PhOEt} + \text{HCO}_2\text{Ph}$ . To prevent the reaction with the  $\text{C}(\text{OPh})_2$  of the  $\text{EtOH}$  split off, the  $\text{EtO}$ -free compds. III and  $(\text{PhO})_2\text{CHCONH}_2$  (V), instead of I were treated with  $\text{Et}_3\text{NMgBr}$ . III proved to be considerably more stable than I, it reacted only when heated in benzene suspension at 150° under pressure. Fractionation of the product yielded  $\text{PhOH}$  and  $\text{HCO}_2\text{H}$ , i. e., the  $\text{PhOH}$  split off has as much a disturbing influence as does  $\text{EtOH}$  in the case of I. With V, only splitting off of  $\text{PhOH}$  was observed. *Ph diphenoxycetic acid* (III), obtained in 80% yield from  $(\text{PhO})_2\text{CHCO}_2\text{H}$  treated in ether with 2 mols. pyridine, then slowly with 1 mol.  $\text{SOCl}_2$ , and finally with 1.1 mols.  $\text{PhOH}$  and heated to 40°, in 95°. *Diphenoxycetic acid* (50.6% from I and  $\text{NH}_4\text{Et}$  at 150° in sealed tubes), in 137°. XII. The carbon monoxide acetal cleavage of diphenoxycetic acid and its derivatives produced by thermal decomposition. *Ibid* 2144-51 —  $(\text{PhO})_2\text{CHCO}_2\text{H}$  (I) cannot be distd.; on heating it breaks down into  $\text{PhOH}$  with evolution of gas. Already at 120° several cleavage reactions occur simultaneously with formation of different products, chiefly  $\text{PhOH}$  and  $\text{CO}$ , along with  $\text{CO}$ ,  $\text{H}_2\text{O}$ ,  $\text{HCO}_2\text{Ph}$ ,  $\text{Ph}_2\text{CO}$ , and a high-boiling substance (II), probably diphenoxymaleic anhydride. Because of the usually predominant  $\text{PhOH}$  formation, the carbon monoxide cleavage plays no significant role, since in addn. to  $\text{CO}$  only the small amt. of  $\text{HCO}_2\text{Ph}$  formed can be regarded as the product of such cleavage. A no. of derivs. of I have been prepd. in the hope of finding some which were thermolabile. The chloride (III), from I cautiously treated with  $\text{SOCl}_2$ , splits off  $\text{CO}$  at 170-80° and yields  $(\text{PhO})_2\text{CHCl}$  on distn.;  $\text{C}(\text{OPh})_2$  and  $\text{HCOCl}$  are probably intermediate products. The mixed acetic diphenoxycetic anhydride (IV), from III and  $\text{AgOAc}$  (or from the  $\text{Ag}$  salt of I and  $\text{AcCl}$ ), very easily loses  $\text{AcOH}$ , even during its prepn. in ether, giving  $(\text{PhO})_2\text{C}=\text{CO}$  (V). In attempts to prep. the anide from III and  $\text{NaN}_3$ , the reaction did not proceed as expected but followed the course  $\text{III} + \text{NaN}_3 \rightarrow \text{V} + \text{NaCl} + \text{NH}_3$ . From the reaction mixt. was isolated  $(\text{PhO})_2\text{CHCO}_2\text{Ph}$  (VI), formed by the action of  $\text{PhOH}$  on V. The  $\text{PhOH}$  itself was probably formed by hydrolysis of part of the di- $\text{PhO}$  groups by traces of moisture in the presence of  $\text{NaN}_3$ . VI was also formed in the reaction between III and  $\text{Ag}_2\text{O}$  in benzene. To det. whether V is an intermediate product in the prepn. of VI from III and  $\text{PhOH}$  in pyridine, III in ether was treated with pyridine and then with  $\text{PhN}^+\text{CHPh}$ , but instead of the lactam  $(\text{PhO})_2\text{C}=\text{CO}^+\text{NPh}$   $\text{CHPh}$  the product was the

anide, *m.* 124°, of I, the pyridine in this case does not split off  $\text{HCl}$ . Cleavage of  $\text{HCl}$  was effected, however, by the action of  $p\text{-HOC}_6\text{H}_4\text{N}_3\text{Ph}$  (VII) on III, the resulting unstable primary mol. compd. immediately decomposed into the known  $\text{HCl}$  addn. product, *m.* 169°, of VII. The triphenylmethyl ester (VIII) of I, from the  $\text{Ag}$  salt and  $\text{Ph}_3\text{CCl}$ , decomposed in a high vacuum only at 160°, giving  $\text{CO}$ ,  $\text{CHPh}$ , and  $\text{PhOH}$ . II *b.p.* 160-8°, *m.* 153°, is highly unsatd. toward  $\text{KMnO}_4$  in  $\text{Na}_2\text{CO}_3$ , adds  $\text{Br}$  vigorously, reacts strongly acid. III, light yellow oil. IV, light yellow oil. VIII (yield, 75%), *m.* 112°. XIII. Diphenoxychloromethane (formyl chloride diphenyl acetal) and its conversion into tetraphenoxethylene [di(carbon monoxide) tetraphenyl acetal] *Ibid* 2151-3 —  $(\text{PhO})_2\text{CHCOCl}$  on heating loses  $\text{CO}$  to form di-

phenoxychloromethane (II) (see preceding abstr.), which may be regarded as formyl chloride di- $\text{Ph}$  acetal. It is characterized by the great reactivity of its Cl atom. Even atm. moisture decomposes it, partially into  $\text{HCO}_2\text{Ph}$ ,  $\text{PhOH}$  and  $\text{HCl}$ . The  $\text{HCO}_2\text{Ph}$  readily changes further into  $\text{PhOH}$  and  $\text{HCO}_2\text{H}$ , and the  $\text{PhOH}$  reacts with  $\text{NaOH}$  to form the qu'et stable crystall. tri- $\text{Ph}$  orthoformate which seps. when I is exposed a long time to moist air. Attempts to split off  $\text{HCl}$  from I with tertiary bases (quinoline,  $\text{NEt}_3$ ) had resulted (Schneider, *Duss. Tech. Hochschule Zurich*, 1916) in the formation of addn. products which had not been investigated with any great thoroughness. Nor could  $\text{HCl}$  be split off by heating I. The desired cleavage can be effected indirectly, however.  $\text{Ph}_3\text{CN}$  with I might have been expected to yield  $(\text{PhO})_2\text{CHCPh}_3$  (II), but in spite of the mild conditions of the reaction (in ether at 15-20°) only the cleavage products of this acetal were obtained, besides  $\text{CHPh}_3$  there was formed a compd. (III) having the compo. of  $\text{C}(\text{OPh})_2$ , but with double the mol. wt. Hence III is di(carbon monoxide) tetra- $\text{Ph}$  acetal (diphenoxylene di- $\text{Ph}$  acetal, tetraphenoxethylene),  $(\text{PhO})_2\text{C}=\text{C}(\text{OPh})_2$ . This structure is confirmed by the hydrolysis of III to  $(\text{PhO})_2\text{CHCO}_2\text{Ph}$ . The primarily formed II therefore immediately breaks down into  $\text{CHPh}_3$  and  $\text{C}(\text{OPh})_2$ , which under the exptl. conditions dimerizes to III. I (64% yield from  $(\text{PhO})_2\text{CHCOCl}$  in a Clausen flask slowly heated (with an abundance of clay fragments) to 180°, the gases being removed (through a  $\text{CaCl}_2$  tube) by means of a water pump), light yellow oil, *b.p.* 130-2°. III, *m.* 168°, mol. wt. (Rast) 390, immediately decolorizes alc.  $\text{KMnO}_4$ - $\text{Na}_2\text{CO}_3$ , adds  $\text{Br}$  energetically, with evolution of heat, in  $\text{CHCl}_3$ . C. A. R.

Ester enolates and ketone acetals XIX Diphenylketene diphenyl acetal and diphenylmethyl phenyl ether Helmut Scheibler and Maximilian Depper *Ber.* 65B, 2154-7 (1933); cf. C. A. 28, 6700; preceding abstr. — Dimethylketene di-Et acetal,  $\text{Me}_2\text{C}=\text{C}(\text{OEt})_2$ , cannot be prepd. in the usual way from  $\text{Me}_2\text{CHCO}_2\text{Et}$  because the latter reacts only with great efficiency with  $\text{NaOEt}$ . Diphenylketene di-Et acetal is the primary product of the action of pyridine on  $\text{Ph}_2\text{CClCH}(\text{OEt})_2$  (I). It has not yet been obtained in pure form, however, as it splits off  $\text{C}_6\text{H}_6$  to form  $\text{PhCHCO}_2\text{Et}$  when distd. Diphenylketene di- $\text{Ph}$  acetal (II), from  $(\text{PhO})_2\text{CHCO}_2\text{Et}$  and  $\text{PhMgBr}$ , gives with  $\text{SOCl}_2$  and pyridine in ether diphenylketene di- $\text{Ph}$  acetal (III). While I with  $\text{PhMgBr}$  forms  $\text{PhCl}$  and an organomagnesium compd. which undergoes the carbon monoxide acetal cleavage ( $\text{I} + \text{PhMgBr} \rightarrow \text{PhCl} + (\text{EtO})_2\text{CHCPh}_2\text{MgBr} \rightarrow \text{C}(\text{OEt})_2 + \text{HCCPh}_2\text{MgBr}$ ), III gives the enol ether, *o,o*-diphenylmethyl  $\text{Ph}$  ether,  $\text{PhOCH}=\text{CPh}_2$  (IV), and  $\text{PhOMgBr}$ . The same reaction, with splitting off of a  $\text{PhO}$  group, is effected with  $\text{Zn}$  in  $\text{AcOH}$ -pyridine. III readily splits off  $\text{HCl}$ , yielding the *cis*-diphenylketene di- $\text{Ph}$  acetal (V), which can be distd. without decompn. in a high vacuum. The reaction is best carried out in quinoline. V is also obtained from II with  $\text{P}_2\text{O}_5$  and  $\text{NEt}_3$ . It can be hydrolyzed to  $\text{PhCHCO}_2\text{H}$  or its esters, the  $\text{Ph}$  ester is obtained with  $\text{HBr}$ - $\text{AcOH}$  at 15-20°, and the  $\text{Et}$  ester with  $\text{HCl}$ - $\text{EtOH}$ . The sapon. takes place so readily that  $\text{Br}$  in  $\text{CCl}_4$ , which is rapidly decolorized, forms no  $\text{Br}$  addn. product but only  $\text{PhCHCO}_2\text{Ph}$  and  $\text{Br}_2\text{C}_6\text{H}_5\text{OH}$ . Traces of moisture therefore first liberate  $\text{PhOH}$  which reacts with the  $\text{Br}$ , forming the  $\text{HBr}$  necessary for the further hydrolysis of V. II (yield, 95.8%), *m.* 103°. III (97.5% yield), viscous oil solidifying to a glass which gives off  $\text{HCl}$  when heated above 100°. V (obtained in 69% yield by treating III in benzene with quinoline, allowing the benzene to evaporate gradually and heating the residue 8 hrs. at 160°), *m.* 112°, *b.p.* 155°, strongly reduces  $\text{Na}_2\text{CO}_3$ ,  $\text{KMnO}_4$ , and vigorously adds  $\text{Br}$ . The splitting off of  $\text{HCl}$  can also be effected with heavy metal salts ( $\text{Ag}_2\text{CO}_3$ ,  $\text{AgCN}$ ,  $\text{Hg}(\text{CN})_2$ ) but the isolation of V is then difficult. IV (4.2 g. from 4 g. III with  $\text{PhMgBr}$ ), *b.p.* 165°, *m.* 60°, is strongly unsatd. toward  $\text{KMnO}_4$ , adds  $\text{Br}$  energetically, is not attacked by boiling dil.  $\text{H}_2\text{SO}_4$  or alc.  $\text{KOH}$ , is also obtained with  $\text{Zn}$  dust in pyridine,  $\text{AcOH}$  and a few drops of water.

C. A. R.

The introduction of chlorine into nitromethionic acid. H. J. Backer. *Rec. trav. chim.* 54, 899-904 (1935); cf. C A 25, 915.—The secondary bromine and Ba salts and the tertiary K, Ti and Ba salts of  $\text{CH}(\text{SO}_3\text{H})_2\text{NO}_2$  (I) were prep'd. The tertiary salts were explosive. When Cl was passed through a soln of the di-K salt of I at  $60^\circ$ , the di-K salt of chloronitromethionic acid (II) was formed. II was isolated as the tetrahydrate which lost  $\text{H}_2\text{O}$  over  $\text{P}_2\text{O}_5$ , giving the dihydrate, m.  $95.5^\circ$ . The strychnine, brucine, Ba, Ti, Ag and Ca salts of II were prep'd. II reacted with  $\text{K}_2\text{SO}_4$  to give I, but not with  $\text{AgNO}_3$ .

E. W. Scott

Multimembered heterocyclic compounds VIII Higher mono- and polylactone rings M. Stoll and A. Rouvé. *Helv. Chim. Acta* 18, 1087-1125 (1935). cf. C A 29, 50741.—By the method previously described (C A 29, 10637) the following acids were lactonized in boiling  $\text{C}_6\text{H}_6$  with  $\text{PhSO}_3\text{H}$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave predominantly the monomeric lactone  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  (I) gave 13%, unchanged I. 32.6% lactone, b.  $98-100^\circ$ , d.  $1.0693$ , 1.4611, 3.8% dimeric lactone, b.  $120-30^\circ$ , m.  $11-11.5^\circ$ , d.  $1.0268$ , n.  $1.4418$ , n.  $1.4379$ , and 3.8% trimeric lactone b.  $190-205^\circ$ , d.  $1.052$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave impure lactone, dimeric lactone, m.  $40-1^\circ$ , d.  $1.018$ , n.  $1.4690$ , and trimeric lactone, b.  $202-5^\circ$ , d.  $1.051$ , n.  $1.4671$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 0.95% lactone, d.  $0.9848$ , and 97% dimeric lactone, m.  $92.5-3.5^\circ$ , b.  $150-2^\circ$ , d.  $1.0739$ , n.  $1.4444$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  (II) gave 4% dimeric II, 1.2% lactone, b.  $71-8^\circ$ , m.  $24-6.5^\circ$ , d.  $1.013$ , n.  $1.4691$ , 80% dimeric lactone, m.  $56-7^\circ$ , b.  $155-8^\circ$ , d.  $0.9583$ , d.  $0.9819$ , d.  $1.0171$ , n.  $1.4563$ , and 14% trimeric lactone, b.  $240^\circ$ , m.  $20^\circ$ , d.  $1.007$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  (III) gave 11% mono- and polymeric III, 1.4% lactone, b.  $67-75^\circ$ , m.  $4-5^\circ$ , d.  $1.001$ , n.  $1.4673$ , 68% dimeric lactone, m.  $96-7^\circ$ , b.  $190-2^\circ$ , and 12% trimeric lactone, m.  $28-9^\circ$ , b.  $267-70^\circ$ , d.  $0.9942$ , n.  $1.4711$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 11% lactone, b.  $58-60^\circ$ , d.  $0.9828$ , n.  $1.4727$ , 69% dimeric lactone, m.  $71.5-2^\circ$ , and 20% trimeric lactone.  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 16% lactone, b.  $88-90^\circ$ , m.  $0^\circ$ , d.  $0.9743$ , n.  $1.4697$ , 70% dimeric lactone, m.  $100-1^\circ$ , and 12% trimeric lactone.  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 41% lactone, b.  $139-42^\circ$ , m.  $50-1^\circ$ , d.  $0.9014$ , n.  $1.4707$ , 39% dimeric lactone, m.  $80-1^\circ$ , and 15% trimeric lactone,  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 53% lactone, b.  $106-9^\circ$ , and 36% dimeric lactone, m.  $106-7^\circ$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 67% lactone, b.  $105-7^\circ$ , m.  $34-5^\circ$ , 27% dimeric lactone, m.  $108-8.5^\circ$ , and 1.7% trimeric lactone, m.  $70-2^\circ$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 58% lactone, b.  $135-8^\circ$ , and 24% dimeric lactone, m.  $96-7^\circ$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 76% lactone, b.  $136-8^\circ$ , m.  $36-7^\circ$ , d.  $0.9075$ , d.  $0.9344$ , n.  $1.4681$ , and 20% dimeric lactone, m.  $113-14^\circ$ .  $\text{HO}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 61% lactone, b.  $174-6^\circ$ , m.  $35-6^\circ$ , d.  $0.9118$ , d.  $0.8971$ , n.  $1.4678$ , and 33% dimeric lactone, m.  $104-5^\circ$ .  $\text{Me}(\text{HO})\text{CH}(\text{CH}_2)_n\text{CO}_2\text{H}$  gave 42% lactone, b.  $108-9^\circ$ , and 20% dimeric lactone, m.  $117.5-18^\circ$ . Sapon. of the lactones was accomplished by extn. with pentane, in which the dimeric lactones are insol., and distn. of the sol. mono- and trimeric lactones. To a soln. of Na (7.4 g) in  $[\text{CH}_3(\text{OH})\text{CH}_2]_2\text{O}$  (15 g) was added  $\text{Br}(\text{CH}_2)_n\text{CO}_2\text{Me}$  (90 g). After 1 day at  $150^\circ$  the soln. was poured into  $\text{H}_2\text{O}$  (3 l). Extn. with  $\text{Et}_2\text{O}$ , sapon. and recrystn. from  $\text{C}_6\text{H}_6$ -pentane gave  $\text{HO}(\text{CH}_2)_n\text{O}(\text{CH}_2)_n\text{O}(\text{CH}_2)_n\text{CO}_2\text{H}$  (IV) (36 g), m.  $32-3^\circ$ . IV on lactonization gave 73% lactone, b.  $120-5^\circ$ , d.  $1.017$ , n.  $1.4662$ , and 5.8% dimeric lactone, m.  $54.5-55^\circ$ .  $\text{HO}(\text{CH}_2)_n\text{O}(\text{CH}_2)_n\text{CO}_2\text{H}$ , m.  $50-50.5^\circ$  (Et extn), b.  $164-7^\circ$ , m.  $22-3^\circ$ , d.  $0.9352$ , n.  $1.4510$ , was prep'd. as IV. On lactonization this gave 67.4% lactone, b.  $14-5^\circ$ , d.  $0.9904$ , n.  $1.4679$ , and 21.4% dimeric lactone, m.  $50-60^\circ$ . From the data thus obtained values for the cyclization const., C, and the "degree of cyclization,"  $\gamma$ , are calcd. C is shown to be directly proportional to the intermol. collision frequency. In lactone rings contg. more than 8 members neither sapon. side-reactions nor reactions involving interchange of ester groups or resinification affect the end results under these exptl. conditions, and the percentage compn. of the

1 reaction product gives a true picture of the course of the reaction. Other theoretical considerations and their bearing on the formation of large lactone rings are discussed. R. C. Elderfield

General method for the synthesis and the chemical properties of  $\beta$ -ethylene isocyanates,  $\text{RCH}(\text{CR}'\text{NCO})\text{CO}$ . Joseph Hoch. *Compt. rend.* 201, 733-5 (1935). cf. C A 30, 4306.—The treatment of  $\text{RCH}_2\text{CR}'\text{NCO}$ -Et as previously described gave the following:  $\text{EtCH}(\text{C}_6\text{H}_5)_2\text{NCO}$ , b.  $55-6^\circ$  (86% yield), phenylurea, m.  $102^\circ$ .  $\text{C}_6\text{H}_5\text{NCO}$  (I), b.  $61-3^\circ$  (82% yield), phenylurea, m.  $107^\circ$ .  $\beta$ -Me- $\text{C}_6\text{H}_4\text{NCO}$ , b.  $73-4^\circ$  (74% yield); phenylurea, m.  $170^\circ$ .  $\text{CH}_3\text{C}_6\text{H}_4\text{NCO}$ , b.  $99^\circ$  (40% yield), phenylurea, m.  $142^\circ$ .  $\text{MeCH}(\text{C}_6\text{H}_5)_2\text{NCO}$ , b.  $110-12^\circ$  (40% yield), phenylurea, m.  $153^\circ$ . I gave the following derivs.  $\text{C}_6\text{H}_5\text{NHCONH}_2$ , m.  $130^\circ$  (impure),  $(\text{C}_6\text{H}_5)_2\text{NHCONH}_2$ , m.  $196^\circ$ ,  $\text{C}_6\text{H}_5\text{NHCONHMe}$ , m.  $88^\circ$ ;  $\text{C}_6\text{H}_5\text{NHCOEt}$ , b.  $14-9^\circ$ ,  $\text{C}_6\text{H}_5\text{NHCOCHMe}$ , b.  $147-8^\circ$ ,  $\text{C}_6\text{H}_5\text{NHCOPh}$ , m.  $106-7^\circ$ ,  $\text{C}_6\text{H}_5\text{NHCONC}_6\text{H}_5$ , m.  $119^\circ$  ( $\text{C}_6\text{H}_5 = \Delta^1$ -cyclohexenyl). Julius White

Effect of an element or a negative group on the relative mobilities of alkyl radicals in their chloroformates. Perre Carré and Henn Pascardouet. *Compt. rend.* 201, 898-900 (1935). C A 29, 5417.—The effect of an element or a neg. group on the mobility of an alkyl radical in the chloroformates is the reverse of the chlorosulfites. Hence the previous explanation offered for this is ineffective and the mechanism must be more complex.

Julius White

New uses of magnesium in organic syntheses II. The barbituric acid condensation Hakon Lund. *Apt. Danske Vidensk. Selskab. Math.-fys. Medd.* 13, No. 13, 11 pp (1935). C A 28, 2333.—Substituted barbituric acids were readily prep'd in good yields by using  $\text{Mg}(\text{OCH}_3)_2$  as a condensing agent.  $\text{Mg}$  (3 g) was dissolved in dry  $\text{MeOH}$  (75 cc), 10 g. of urea was added and then 0.1 mol of the appropriate malonic ester. The soln. was refluxed for 24-48 hrs. After deducting the recovered unchanged ester, the yields varied from 81-66%. Using thiourea, malonic ester gave only a small amt of 2-thiobarbituric acid. However 2-amino-3-isopropylbarbituric acid, m.  $178^\circ$ , was obtained in 68% yield. F. W. S.

The remarks of K. Freudenberg and E. Braun on 2,3,6-trimethylglucose anhydride. K. Hess and F. Neumann. *Ber.* 68B, 2167 (1935); cf. F. and B., C A 30, 719.

C. A. R.

The synthesis of monoalkylamino acids of the chitosamic type. E. Votoček and R. Lukeš. *Collection Czechoslov. Chem. Communications* 7, 424-9 (1935); *Chem. Listy* 29, 338-10 (1935). cf. C. A. 28, 4387.—The hitherto unknown acids of the type  $\text{CH}_2(\text{OH})_4[\text{CH}(\text{OH})_2]\text{CH}(\text{NHR})\text{CO}_2\text{H}$  are easily prep'd with satisfactory yields by the method shown in the following example: *Monomethyl-d-glucosaminic acid*, m.  $230^\circ$  (decompn.) from 50%  $\text{EtOH}$ ,  $[\alpha]_D^{20}$  (initial in  $\text{HCl}$ )  $4.0^\circ$ , (final)  $7.5^\circ$ , was obtained thus: d-Arabinose (41 g) was dissolved in 63 g 25%  $\text{MeNH}_2$  and left for 6 days, after which 40 cc of 25%  $\text{HCN}$  was added and the whole left for 2 weeks. Then the mixt. was chilled and added to 200 cc. concd.  $\text{HCl}$ . After a few days the soln. was evaporated to dryness *in vacuo* and the residue hydrolyzed by being steam-distd in  $\text{Ba}(\text{OH})_2$ ; this also removed excess  $\text{MeNH}_2$ . All the more ions were then ppt'd by a series of reactions and the soln. was evaporated almost to dryness below  $50^\circ$ . Some crystals were frozen out and more were obtained by adding  $\text{EtOH}$ . *Monomethyl-d-glucosaminic acid*, m.  $228-9^\circ$  (decompn.),  $[\alpha]_D^{20}$  (initial in  $\text{HCl}$ )  $-18.2^\circ$ , (final)  $-33.5^\circ$ , was prep'd in a similar manner from d-glucose. Its *ethyl isocyanate deriv.*, m.  $210^\circ$  from aq  $\text{EtOH}$ , was prep'd. by treating the acid in aq.  $\text{KOH}$  with  $\text{PhNCO}$ .

John E. Mulberry

Cellulose (reply to the latest communications of K. Hess and K. Drenzel and of C. Tregus and K. Hess). L. Zechmeister and G. Tóth. *Ber.* 68B, 2134-6 (1935); cf. H. and D., C. A. 29, 7069<sup>1</sup>, T. and H., C. A. 29, 6813<sup>1</sup>.

C. A. R.

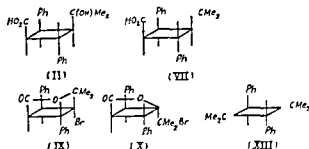
The phenylbenzylsuccinic acids R. Stoermer and H. Stroh. *Ber.* 68B, 2112-16 (1935).—Avery and Upson



8°. 3-Isopropylidene acid (VII), m. 143–4°; it is best prepd. by passing HCl gas through II in boiling concd. HCl for 15–30 min. VIII, m. 167–8°. XI, m. 161–2°. XII, m. 210–11°. Me ester of XV, m. 72°. p-Nitrophenylhydrazide of XV, m. 181°. Me ester of XVI, m. 64°. XIV, m. 183–4°. di-Me ester, m. 125°. XVII, m. 111–12°, anilide, prepd through the chloride, m. 165–6°

benzene occasionally yielded, along with VII and  $\gamma$ -truxonic acid, a very small amt. of an indifferent substance (VIII), m. 77–8°, whose compn. corresponded quite closely to that of a truxillic ketone but whose formation apparently depends on the quality of the  $\text{AlCl}_3$ , for it was not obtained with another sample of  $\text{AlCl}_3$ . Often, however, with 0.6–0.7 part  $\text{AlCl}_3$  there was obtained (about 5%) a likewise soda-insol compd. m. 195°, of the compn.  $(\text{C}_{11}\text{H}_{11}\text{O}_2)_n$ . Mono-Me ester of I, prepd from the monoanilide by treatment with  $\text{Me}_2\text{SO}$ , and subsequent decmpn with  $\text{HNO}_3$  or better by partial sapon of the di-Me ester with KOH in boiling MeOH, m. 131°. 2',4'-Diphenyl-3-( $\alpha$ -hydroxyisopropyl)cyclobutane-1'-carboxylic acid (II), m. 145°. Me ester, prepd. with  $\text{CH}_3\text{N}_3$ , does not melt.  $\alpha$ -Tetramethyltruxillic acid, m. 146°. 2',4'-Diphenyl-1',3'-dibenzoylcyclobutane (IV) (yld, up to 50%), m. 207–8°. V, m. 222°. 2',4'-Diphenyl-3-benzoyl-1'-cyclobutanecarboxylic acid (VI), from  $\gamma$ -truxillic anhydride,  $\text{AlCl}_3$ , and benzene, m. 227°. Me ester, m. 169°. Et ester, m. 162°. phenylhydrazide, m. 100° with vigorous foaming (apparently forming the lactam) and becomes insol in soda. On heating or on short treatment with concd  $\text{H}_2\text{SO}_4$ , VI is depolymerized to PhCH $_2$ CHCO $_2$ H and PhCOCH $_2$ CHPh. Chloride of VI, prepd with  $\text{SOCl}_2$  in benzene, m. 150–2°. The hemitruxonic acids of the truxillic acid series and the still missing truxone, *pers*-truxone R Stoermer and Fr. Möller *Ibid* 2124–34—Of the 5 truxillic acids (I), the  $\alpha$ -,  $\gamma$ -, *pers*- and *epi*-acids can theoretically yield hemitruxonic (or, for short, truxonic) acids (II), and of the 4 latter, in turn, only the  $\alpha$ - and *pers*-acids can form diketones or truxones (III).  $\alpha$ -III has long been known, and the *cis*- or *pers*-truxone, which was as yet lacking, has now been prepd  $\gamma$ -II, previously made by internal condensation of the truxillic anhydride with  $\text{AlCl}_3$ , was prepd. from the mono-Me ester chloride with  $\text{AlCl}_3$  in  $\text{CS}_2$  on the water bath. It is characterized by its resistance to all attempted rearrangement methods which ordinarily convert the  $\gamma$ - into the  $\alpha$ -derivs. It forms an oxime and a phenylhydrazide. *pers*-II was prepd from the anhydride, while  $\alpha$ - and *epi*-II were made from the ester chlorides. Some difficulty was encountered in the prepn of  $\alpha$ -II, for the Me ester on alk. sapon rearranged into  $\gamma$ -II, but acid hydrolysis yielded  $\alpha$ -II. *pers*- and *epi*-II offered considerably greater difficulties because, on the one hand, of the lability of the *pers*-II, and, on the other hand, of the labor involved in the prepn of *pers*-I, from which alone *epi*-I can be prepd. The prepn of *pers*-I was improved by heating the necessary  $\gamma$ -anhydride in a tube under N to only about 280° instead of in a high vacuum at 300°. Decmpn of the  $\gamma$ -acid by atm O is thereby largely avoided, the acid being recovered pure in considerable amt and the yield of *pers*-anhydride being thereby indirectly increased to about 29%. In the rearrangement there seems to be established, between the 2 anhydrides, an equilibrium which below 260° still lies completely on the  $\gamma$ -anhydride side but shifts more and more to the *pers*-anhydride side with increasing temp. Above 280° it would probably shift still further in this direction were it not that incipient decmpn limits the yield. Further progress required a careful study and a detn of the constitution of some derivs of *pers*- and *epi*-I, which resulted in the prepn of the 2 *epi*-ester acids. There had already (C. A. 18, 2172) been obtained from the *pers*-anhydride with hot concd  $\text{NH}_4\text{OH}$  an amidic acid (IV) which with  $\text{HNO}_3$  gave *epi*-I, showing that the *pers*-form is rearranged into the *epi*-form not only by strong alkalis but also by weak alkalis at high temps. The exceedingly stable monomol truxillic imide (V) formed at the same time is a deriv. of *pers*-I, which alone can yield such an imide. The true *pers*-truxillic acid (VI) is obtained by treating the *pers*-anhydride in alc. suspension with  $\text{NH}_3$  gas; when heated with aq.  $\text{NH}_4\text{OH}$  it changes over, with partial formation of V, into the *epi*-amidic acid (IV). Sapon of VI with  $\text{HNO}_3$  or mere boiling in AcOH gave very pure *pers*-anhydride. The 2 CO $_2$ H groups in *epi*-I are not equiv.; the one standing alone with H atoms on 1 side of the cyclobutane ring is designated  $\delta$ , and the other, on the

The peculiar position of  $\alpha$ -truxillic acid. Truxillic ketones R Stoermer and K Cruse *Ibid* 2117–21—Of the 3 *cis*-dicarboxylic acids ( $\gamma$ -, *pers*-,  $\alpha$ -), the wholly sym  $\alpha$ -acid (I) (with 2 *cis*-CO $_2$ H and 2 *trans*-Ph groups) alone does not form a monomeric anhydride, although it gives a monomol imide and phenylimide. Renewed attempts, in the most varied directions, to prep such an anhydride have again been without success. The methods of which most was hoped were heating the acid at 250° *in vacuo* or depolymerizing the polymeric anhydride under 0.5 mm. The latter method resulted in a surprising isomerization, accompanying the depolymerization, to the stable monomol.  $\gamma$ -anhydride and even to the *pers*-anhydride. This is the only known case of a change in the configuration of the  $\alpha$ -acid. The stability of I led to an attempt to prep the corresponding alc acid (II) with MeMgI as described for the  $\alpha$ - and  $\gamma$ -acids in the preceding abstr., dehydrate the resulting II to the unsatd acid (III) and convert the III by ozonization into a diphenylcyclobutanecarboxylic acid of definite configuration or, by ring cleavage, into I of the 2 phenylbenzylsuccinic acids. But here again the peculiar position occupied by I came disagreeably into evidence. The mono-Me ester of I gave with the Grignard reagent a very considerable amt of the ditertiary alc, this was reduced somewhat by decreasing the amt of MeMgBr but was still much greater than with the other acids and pointed to a primary disproportionation of the mono-Me ester to the di-Me ester and free I, resulting in a greatly decreased yield of II. With dehydrating agents II in no case gave III, the whole mol was completely broken down in a manner hitherto never observed in the truxillic acid series, giving PhCH $_2$ CHCO $_2$ H, together with a soda-insol, high-boiling, and completely unsaponifiable oil. With PhMgBr instead of MeMgI, none of the HIO acid at all was obtained; the products were a large amt of free I and a soda-insol, unsaponifiable, satd di-Ph diketone (IV), formed in considerable amt even when an excess of PhMgBr was used. Like the Stobbe truxillic ketones (C. A. 24, 92), IV forms no oxime or semicarbazone but with alc.  $\text{NH}_3\text{OH}$  gives an isomeric diketone (V), apparently not identical with any of Stobbe's products (the latter, however, were not available for direct comparison). The structures of IV and V were established by depolymerization; both, on cautious distn under atm. pressure, gave PhCOCH $_2$ CHPh. The  $\alpha$ -configuration may be assumed for IV; that of V is uncertain. Attempts were made to convert the different truxillic acids directly into the Ph ketones. The dichloride of the  $\alpha$ -acid with  $\text{AlCl}_3$  in benzene gave exclusively the known  $\alpha$ -truxone; with the  $\gamma$ -acid it was therefore attempted to effect the reaction in steps by first converting the acid into the Bz acid (VI), but the chloride of VI gave with  $\text{AlCl}_3$  and benzene only an exceedingly small amt. of an indifferent substance (VII), m. 92–3°, whose compn. approximated only grossly that of a truxillicone. Truxillic anhydride with 2 parts somewhat weathered  $\text{AlCl}_3$  in



same side of the ring as the  $\text{CO}_2\text{H}$  groups, is designated  $\alpha$ . In the rearrangement of VI into IV, only the  $\text{CONH}_2$  changes position, hence IV is the  $\beta$ -amino acid. Esterification of VI with  $\text{CH}_3\text{I}$ , and sapon of the ester with  $\text{HNO}_3$  gives without rearrangement the known *pers*-ester acid, m. 193°. The pure  $\alpha$ -Me ester (VII) of *epi*-I m. 204.5°, the isomeric  $\beta$ -ester (VIII) m. 141°. The Me ester, m. 228°, of IV with  $\text{HNO}_3$  gives VII, which had formerly been obtained by half sapon of the di-Me ester of *epi*-I, the  $\beta$ - $\text{CO}_2\text{Me}$  group in the di-Me ester is sapon more rapidly than the  $\alpha$ - $\text{CO}_2\text{Me}$  standing between the Ph groups. Conversely, on esterification of the free *epi*-I, the  $\beta$ - $\text{CO}_2\text{H}$  group is esterified first; with  $\text{MeOH}$ -HCl the product obtained is VIII. VII and VIII yield the same di-Me ester, m. 111-12°. In the acid chloride of VIII, the  $\alpha$ -COCl group should react with the adjacent Ph group in the Friedel-Crafts reaction, and as a matter of fact the chloride does give the Me ester, m. 106-7°, of *epi* II. *pers* II has a higher m. p. than any of its isomers but stands between  $\alpha$ - and  $\gamma$ -II in its soly. All its salts are readily sol. With alk.  $\text{NH}_4\text{OH}$  it gives the oxime of *epi*-II. No rearrangement occurs in the formation of the phenylhydrazones, which, however, does not melt sharply, this indicates that *pers* II, in spite of repeated purification, is not completely homogeneous. The  $\text{AlCl}_3$  used in its prep. gives rise to a rearrangement which had hitherto not been observed in this field, even when the purest *pers*-anhydride is used there is formed, along with *pers*-II, some  $\gamma$ -II. The chloride of *pers* II with  $\text{AlCl}_3$  in  $\text{CS}_2$  gives *pers* III, which differs from the  $\alpha$  isomer in being easily sol. in AcOH and acetone and melts almost 100° lower. It could not be rearranged into the  $\alpha$ -compd. either by heating it alone above its m. p. or with concd. HCl in a sealed tube at 180°. The mono-Me ester, m. 184°, of  $\gamma$ -II, formerly prep. from the anhydride with  $\text{MeOH}$  and  $\text{Na}_2\text{CO}_3$ , is also very conveniently obtained in 68% yield by half-sapon of the di-Me ester in cold  $\text{MeOH}$  with HCl gas. Boiled in benzene suspension with excess of  $\text{SOCl}_2$ , it readily forms the ester chloride, m. about 86°, which with 5 parts  $\text{AlCl}_3$  in  $\text{CS}_2$ , on the water bath gives 83% of the Me ester, m. 127-8°, of II, sapon. by alc. KOH to the free  $\gamma$ -II, m. 152° (oxime, m. 207°, amide, from the chloride (prep. with  $\text{PCl}_5$ ) and  $\text{NH}_3$  gas in ether, m. 236-7°, phenylhydrazones, light yellow, m. 225°). Mono Me ester of  $\alpha$ -II, m. 195°. Me ester of  $\alpha$ -II (88% yield), m. 186°, sapon. by heating in AcOH contg. a few drops of concd.  $\text{H}_2\text{SO}_4$  and a little water to the free  $\alpha$ -II, m. 216° (phenylhydrazones, m. 239°)  $\alpha$  III, m. 293°. Anhydride of *pers* I, obtained in 12% yield, together with 62% recovered *pers*-I, m. 287°. VI, m. 256.5°; Me ester, obtained with  $\text{CH}_3\text{N}$  in  $(\text{CH}_3\text{OH})$ , m. 197-8°. V, m. 237°, mol wt (Rast) 264.2-295.7. IV, m. 263°. *epi*-II, from its Me ester with KOH in  $\text{MeOH}$ , m. 100°; oxime, m. 260° (decomp.) *pers*-II, m. 221-4°. phenylhydrazones, canary-yellow, m. 189°. *pers*-III (yield, 90-3%), m. 194°. monophenylhydrazones, from III heated with  $\text{PhHNH}_2$ -AcOH, yellow, m. 217°; diphenylhydrazones, from the monophenylhydrazones or III itself heated with  $\text{PhNHNH}_2$  at 150-60° in a current of N, m. about 280° (decomp.) C. A. R.

Plane "radiary" compounds I. Hexahydroxymethylbenzene and its derivatives. H. J. Backer. *Rec. trav. chim.* 54, 833-7 (1935), cf. C. A. 27, 639.—Hexa-acetoxymethylbenzene (I), m. 163°, was prep. by heating hexabromomethylbenzene (II) with  $\text{AcOK}$  and  $\text{Ac}_2\text{O}$  for 5 hrs. at 135°. I was sapon. in EtOH with KOH to hexahydroxymethylbenzene (III), m. 310-11°. The hexapropionate of III, m. 99-100°, was prep. from II and  $\text{EtCO}_2\text{K}$ . The hexamethyl, hexaethyl and hexaphenyl ethers of III, m. 100°, 93° and 221°, resp., were prep. from II and  $\text{NaOMe}$ ,  $\text{NaOEt}$  and  $\text{NaOPh}$ . III in warm  $\text{HNO}_3$  formed the hexanitrate, m. 176.5° (decomp.) III possessed the properties of an extremely compact mol., i. e. high m. p., low soly and ease of crystn. Its derivs. showed similar properties to a lesser extent. II. Hexamercaptomethylbenzene and its thio ethers. *Ibid.* 905-15.—Hexabromomethylbenzene (I) was heated with thiourea in abs. EtOH to give the hexahydrobromide of

1 hexa-southioure-methylbenzene (II), decomp. 250°, which with cold  $\text{NaOH}$  (6 mols.) formed II, decomp. 290°, or, by heating with excess  $\text{NaOH}$  (15 mols.) and neutralizing the excess  $\text{NaOH}$ , hexamercaptomethylbenzene (III), m. 225-6°. The following hexaethers of III were prep. by heating I and a Na mercaptide in EtOH or  $\text{AmOH}$ : Me, m. 220-222°, Et, m. 131°, Pr, m. 55.5°, *iso*-Pr, m. 232.0-2.5°, Bu, m. 34.0-4.5°, *iso*-Bu, m. 94°, *sec*-Bu, m. 172°, *tert*-Bu, m. 304°, Am, m. 25.5-6.0°, *tert*-Am, m. 293°, hexyl, m. 11.5-12.0°, Ph, m. 191-2°, *p*-tolyl, m. 183.5-4.0°; *p*-chlorophenyl, m. 179-80°, *p*-*tert*-butylphenyl, m. 182.5-3.0°; benzyl, m. 143°. III and its derivs. showed the properties of filled mols. The *n*-ethers of III showed a lowering of the m. p. as the alkyl group was increased. Branching of the alkyl group caused an elevation of m. p. as the mols. thus became more compact. E. W. Scott.

2 The catalytic dehydrogenation of ethylbenzene to styrene. Yu. S. Zal kind and G. L. Bulavskii. *Plasticheskie Massy* 1935, No. 3, 9-12.—PhEt mixed with N is passed over a catalyst of 10%  $\text{ZnO}$  and 90%  $\text{Al}_2\text{O}_3$  at 10-13 mm. pressure, 600-70°, and a rate of 1 g. per min. to yield 82.74% styrene. H. M. Leicester.

3 Action of phenylmagnesium bromide on *N*-dimethyl- $\alpha$ -bromobutyramide. Sou. Phou. Ti. *Bull. soc. chim.* [5], 2, 1799-1800 (1935); cf. C. A. 25, 4525.—The action of org. Mg derivs. on  $\alpha$ -halogenated amides gives tertiary alcs. of the general formula  $\text{Et}_3\text{NCH}_2\text{C}(\text{OH})\text{R}_2$  in the case of  $\text{CH}_3\text{CONEt}_2$ . To study this curious transposition with amides of higher mol. wt.,  $\text{PhMgBr}$  was reacted in toluene with  $\text{EtCHBrCONMe}_2$  (I), b. 123-4°, prep. by the condensation of  $\text{EtCHBrCOBr}$  and  $\text{NHMe}_2$  in  $\text{Et}_2\text{O}$  at -10°. The reaction gave a small yield of  $\text{PhBr}$ , a considerable amt. of  $\text{PrCONMe}_2$  (II), b. 162-5°, some  $\text{MeCH}=\text{CHCONMe}_2$  (III), traces of  $\text{PrCO}_2\text{Ph}$  (IV) and  $\text{Me}_2\text{NCH}(\text{OH})\text{Ph}$  (V). Sapon of III, b. 200°, gave  $\text{NHMe}_2$  and  $\text{MeCH}=\text{CHCO}_2\text{H}$ , converted into  $\text{MeCHBrCHBrCO}_2\text{H}$ , m. 87°. The semicarbazone of IV was identical with that of a sample prep. by the condensation of  $\text{PrCOCl}$  and benzene in the presence of  $\text{AlCl}_3$ . V, m. 58° (picrate, m. 155°), was purified through the perchlorate, m. 182°. Treated with  $\text{AcCl}$  in  $\text{CHCl}_3$  it furnished  $\text{Me}_2\text{NCH}(\text{Et})\text{C}(\text{OAc})\text{Ph}$  (picrate, m. 138°). V was decomposed by treatment with 48%  $\text{HBr}$  into  $\text{NHMe}_2$  and  $\text{EtCOCH}_2\text{Ph}$  through the intermediate  $\text{Me}_2\text{NC}(\text{Et})\text{CPh}$ . In the presence of an excess of Mg a part of I is probably converted into  $\text{EtCH}(\text{MgBr})\text{CONMe}_2$  (VI), which is decomposed by  $\text{H}_2\text{O}$  to give II. By the action of  $\text{PhMgBr}$  on the CO group of VI an intermediate compd. is formed which is hydrolyzed to IV. In all probability III is formed by the action of  $\text{Me}_2\text{N}(\text{OH})$ , on unreacted I.

7 Purification of dimethylaniline. Use of aqueous formic acid to remove aniline and monomethylaniline. Frank O. Ritter. *Ind. Eng. Chem.* 28, 33-5 (1936).—By treatment of a mixt. of aniline, mono- and di-methylaniline with 85%  $\text{HCO}_2\text{H}$ , the primary and secondary amines are formylated.  $\text{PhNMe}_2$  of high purity then can be distd. from the higher boiling formamides. E. W. Tillitson.

8 Alkylated ethanediamine derivatives. IV. The reaction between 1,2-bis(benzylideneamino)ethane, phenyl isocyanate and water. J. van Alphen. *Rec. trav. chim.* 54, 883-7 (1935), cf. C. A. 29, 3991.—The reaction of 1,2-bis(benzylideneamino)ethane (I) with 2 mols. of  $\text{PhNCO}$  (II) in moist  $\text{Et}_2\text{O}$  gave 1-(phenyl-3'-ureido-2'-ethyl)-2-keto-3,4-diphenylisazane-1,3-cyclobutane, m. 208° (decomp.). With equimol. quantities of I and II in moist  $\text{Et}_2\text{O}$ , 1-benzylideneamino-2-(phenyl-3'-ureido)-ethane (III) was formed. III was hydrolyzed by dil. HCl and the product was benzoylated giving 1-(phenyl-3'-ureido)-2-benzoylaminoethane, m. 210°. Reduction of III with Na and  $\text{EtOH}$  and benzoylation gave 1-(benzylideneamino)-2-(phenyl-3'-ureido)ethane. E. W. S.

9 as Diphenylhydrazine (melt undercooling and molecular configuration). D. Vorlander and Carmen Butins. *Ber.* 68B, 229-9-76 (1935).—as-Diphenylhydrazine (I) on account of its mol. dissymmetry (cf. C. A. 24, 4944) should and does have a greater tendency to remain melted than

PhNHNH<sub>2</sub> (II) and hydrazobenzene (III) under the same conditions of undercooling. This behavior is not connected with lower *m*, *p* since I *m*. 44°, II *m*. 19.5°, and III *m*. 127°. *N*-Formylphenylhydrazine, *m*. 117°, and the *N,N*-di-*Ac*, *m*. 125°, and dipropionyl, *m*. 99–101°, derivs are easily undercooled but the *N*-mono-*Ac* (IV), *m*. 186°, and monopropionyl, *m*. 179°, derivs cannot be undercooled. There are various processes of crystg. and melting depending on the mol. configuration, uniformity and phys. sphere of the compd. It only remains certain that the arrangement of I is found in many derivs. The derivs of I are not cryst. liquids. *Na α,α*-diphenylhydrazine-β-acetate (V), *m*. 80° in its H<sub>2</sub>O of crystn, decomp. 180–5°, was prepd by heating 9.2 g, 1.41 g. BrCH<sub>2</sub>CO<sub>2</sub>Et and 25 cc alc. or I HCl, NaOEt soln and BrCH<sub>2</sub>CO<sub>2</sub>Et on a H<sub>2</sub>O bath 1.5 hrs and treating with 10–15% NaOH to hydrolyze the ester and ppt the salt. Pb, Hg, Mg, Ca, Sr, Zn, Cd and Al salts can be pptd from the H<sub>2</sub>O soln of V but the Ba and K salts are also sol. The Ag ppt is reduced to Ag on warming. *α,α*-Diphenylhydrazine-β-acetic acid (VI), *m*. 87° (cloudy, clear 96°), ppts from H<sub>2</sub>O solns of V on addn of AcOH, as an oil which crystallizes after a few hrs at 15–20°. It often seps as an oil even on dropping the alc soln on ice. On heating, the crystal powder changes into doubly refracting 4-sided rhombic leaf-like leaves which will not seed the undercooled melt (damorphim<sup>3</sup>). Oxidizing agents give color reactions, FeCl<sub>3</sub> red-violet, concd H<sub>2</sub>SO<sub>4</sub> moss-green, AcOH and PbO<sub>2</sub> red-violet. *N*-Acetyl-α-diphenylhydrazineacetic acid (VII), *m*. 185° (cor +2°), from heating V 0.5 hr at 95° with 5 pts Ac<sub>2</sub>O; remains only on slight undercooling. With concd HNO<sub>3</sub> in AcOH soln at 15–20° it gives a diminoacetylphenylhydrazineacetic acid, *m*. 210°. When I reacts with BrCH<sub>2</sub>CO<sub>2</sub>H or ClCH<sub>2</sub>CO<sub>2</sub>H or ester in the presence of H<sub>2</sub>O, soda or NaOAc a compd, *m*. 214°, probably the diphenylhydrazide of VI, seps its *Ac* deriv, *m*. 229° (cor +4°). *α,α*-Diphenylhydrazine-*p*,*β*-diacetic acid, an oil, resulted from the action of excess BrCH<sub>2</sub>CO<sub>2</sub>H on I in alc. or from BrCH<sub>2</sub>CO<sub>2</sub>Na and V in H<sub>2</sub>O. *β*-(*p*-nitrobenzenesulfonyl)-*p*-phenyl-*α*-acetylhydrazine, *m*. 241° (cor +5°), was formed by dropping an AcOH soln (30 cc) of Na *p*-nitrophenylnitrosamine (VIII) (1.9 g) into a soln of IV (1.1 g) in 25 cc AcOH. Similarly, *β*-(*p*-nitrobenzenesulfonyl)-*p*-phenyl-*α*-acetylhydrazine-*α*-acetic acid was formed from VII and VIII. Both compds form an amorphous lacquer on melting and cooling. Even with great excess of VIII only 1 of the 2 Ph groups reacts, possibly because the influence of the amine N is distributed unevenly. Janet C. Austin.

The hydrogenation of technical phenols in the vapor phase. M. I. Belotzerkovskii, *Prikladnaya Khimiya* 1935, No. 3, 12–16.—Ordinary Ni catalysts are rapidly poisoned by technical PhOH. A catalyst prepd by heating Ni(NO<sub>3</sub>)<sub>2</sub> to 200° and passing H<sub>2</sub> over it will hydrogenate tech. PhOH at 175–80°. Cresols can also be hydrogenated if they are first freed from thiophene. Higher temps. favor ketone formation. H. M. Leicester.

The constitution of *β*-ergosterol. F. Laucht, *Z. physiol. Chem.* 237, 239–46 (1935).—Hydrogenation of ergosterol occurs in 2 stages, (1) the double bond at C<sub>5</sub>-C<sub>6</sub> becomes satd and the C<sub>7</sub>-C<sub>8</sub> double bond in ring B shifts to C<sub>7</sub>-C<sub>8</sub> in ring C, yielding *α*-dihydroergosterol, (2) the double bond in the side chain of the latter at C<sub>22</sub>-C<sub>23</sub> becomes satd, yielding *α*-ergosterol. The same product is obtained in a single step by hydrogenation of 22-dihydroergosterol, whereby the C<sub>5</sub>-C<sub>6</sub> bond is satd and the C<sub>7</sub>-C<sub>8</sub> bond shifted, or by shifting the bond in *γ*-ergosterol. The double bond in the isomeric *β*-ergosterol is now shown to be at C<sub>5</sub>-C<sub>6</sub> in ring D. The proof consists in ozonization of *β*-ergosterol acetate to a keto alc. which represents a removal of ring D and the side chain, leaving Me at C<sub>13</sub> and O at C<sub>14</sub>, and Se dehydrogenation of the ketone to 2-methylphenanthrene. The 3 isomeric ergosterols represent, therefore, successive shifts of the double bond to an adjacent position; thus *γ*-C<sub>5</sub>-C<sub>6</sub> → *α*-C<sub>5</sub>-C<sub>6</sub> → *β*-C<sub>5</sub>-C<sub>6</sub>. Ozonization of *α*-ergosterol acetate and decompn. of the ozonide by Zn dust gave a mixt. of 2 isomeric dehydroergosterols, *m*.

132°, [α]<sub>D</sub><sup>20</sup> -56.1°, and *m*. 114–6°, [α]<sub>D</sub><sup>20</sup> -81°, resp. Ozonization of *β*-ergosterol acetate and distn. of the neutral portion gave the acetate of a keto alc. C<sub>21</sub>H<sub>34</sub>O<sub>2</sub>, and the primary ozonization product, C<sub>21</sub>H<sub>34</sub>O<sub>4</sub>, *m*. 144°, [α]<sub>D</sub><sup>20</sup> 92.5°, which on hydrolysis and CH<sub>3</sub>N<sub>3</sub> esterification gave a Me ester C<sub>21</sub>H<sub>34</sub>O<sub>4</sub>, *m*. 51°, [α]<sub>D</sub><sup>20</sup> -24.6° (acetate *m*. 124°, [α]<sub>D</sub><sup>20</sup> 14.3°). By heating the keto alc. acetate to 320° with Se the 3-ring system was stripped of H and the Me at C<sub>13</sub>, yielding 2-methylphenanthrene, *m*. 53° and identical with the synthetic substance. Ring D and the side chain had been split off as an aldehyde from which the semicarbazide C<sub>21</sub>H<sub>28</sub>N<sub>2</sub>O, *m*. 125–31°, was prepd.

A. W. Dox.  
Action of isobutylene on methyl phenol ether and the structure of pseudobutyl methyl phenol ethers. B. M. Dubinin, *Compt. rend. acad. sci. U. R. S. S. (N. S.)*, 3, 263–6 (1933).—Excellent yields of Me<sub>2</sub>C(CH<sub>3</sub>)<sub>2</sub> (I) were obtained by passing Me<sub>2</sub>C(CH<sub>3</sub>)<sub>2</sub>OH over clay heated to 340–60° and drying the product over KCl. The passage of I into 5% solns of AlCl<sub>3</sub> in phenol ethers gave 53, 61 and 71% yields of the pseudo-Bu derivs of *o*-MeOC<sub>6</sub>H<sub>4</sub>-Me (II), *b<sub>1</sub>* 112–13°, *p*-MeOC<sub>6</sub>H<sub>4</sub>-Me (III), *b<sub>1</sub>* 94–5°, and *m*-MeOC<sub>6</sub>H<sub>4</sub>-Me (IV), *b<sub>1</sub>* 120–2°. The latter reaction also gave 3,4,5-Me<sub>3</sub>O(Me<sub>2</sub>C)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>OMe (V), *m*. 75°, *b<sub>1</sub>* 123–31°, sepd. from IV by crystn. from petroleum ether. The structure of III was detd. by bromination in CHCl<sub>3</sub> to give 2,4-BrMeC<sub>6</sub>H<sub>2</sub>OMe (VI), *b<sub>1</sub>* 126–30°, which was converted into 5,2-Me(MeO)C<sub>6</sub>H<sub>2</sub>CO<sub>2</sub>H, *m*. 67–8°, through the Mg deriv 5,2-Me(MeO)C<sub>6</sub>H<sub>2</sub>MgBr (VII). The addn. of 35 g. of Me<sub>2</sub>CBr to the orig. Mg compd from 52 g. of VI, 6.27 g. of Mg and 90 cc. of Et<sub>2</sub>O gave, after heating at 100° for 1 hr and the usual decompn. procedure, 11 g. of III, *b<sub>1</sub>* 111–3°. Both this compd. and III as prepd. above were nitrated to 5,2,3-Me(MeO)(NO<sub>2</sub>)C<sub>6</sub>H<sub>2</sub>Me<sub>2</sub> (VIII), *m*. 93.5°. The further nitration of VIII by addn. to HNO<sub>3</sub> (d 1.51) at -5° to -8° formed 5,2,3,4-Me<sub>4</sub>(MeO)(NO<sub>2</sub>)C<sub>6</sub>H<sub>2</sub>NO<sub>2</sub>, *m*. 122°, by replacement of the CMe<sub>2</sub> group. In the nitration of IV, especially in AcOH solns, an intense blue color is produced. This is attributed to the formation of an oxonium quinoid salt which is only possible from phenol ethers wherein the *p*-position with respect to the OH group is free. The absence of blue color on nitrating V shows that both *p*-positions to the MeO groups are occupied by Me<sub>2</sub>C groups. A soln of 20 g. of 4,5-Me<sub>2</sub>C(MeO)C<sub>6</sub>H<sub>2</sub>OMe in 40 cc. of benzene was added dropwise to 200 cc. of cooled HNO<sub>3</sub> (d 1.4). The sepd. benzene layer was washed free from acid, dried, and, on concn., yielded 11 g. of 2,4,5-O<sub>2</sub>N-(Me<sub>2</sub>C)(MeO)C<sub>6</sub>H<sub>2</sub>OMe, *m*. 188–92°, *m*. 99–100°, which was converted by nitration with HNO<sub>3</sub> (d 1.51) in the presence of Ac<sub>2</sub>O into 2,4,5-(O<sub>2</sub>N)<sub>2</sub>(Me<sub>2</sub>C)C<sub>6</sub>H<sub>2</sub>OMe (IX), *m*. 157°. The nitration of V gave 2,4,5-O<sub>2</sub>N-(Me<sub>2</sub>C)(MeO)C<sub>6</sub>H<sub>2</sub>OMe, *m*. 99–100°, which on further nitration was converted into IX. C. R. Addinall.

Halogen derivatives of procaine II (*p*-bromo-*α*-amino-benzoyldiethylaminoethanol). J. Frejka and F. Vymetal, *Collection Czechoslov. Chem. Communications* 7, 436–43 (1935), cf. C. A. 19, 2332.—In order to study further the effect of halogens in the ring of procaine on the physiol. properties of the compds it was decided to invert the positions of the Br and NH<sub>2</sub> in bromoprocaine to obtain 4-bromo-2-amino-benzoyldiethylaminoethanol. This was found to be an oil that gave a HCl salt, *m*. 166°. Both caused an intense anesthesia of the tongue. The HCl salt was prepd. (17% yield) by heating 1 g. 4-bromo-2-amino-benzoyldiethylaminoethanol (I), *m*. 80–1°, and 0.6 g. NH<sub>4</sub>Cl in a sealed tube at 110° for 10 hrs. The contents of the tube were extd. with abs. Et<sub>2</sub>O, this soln. was extd. with the calcd. amt. of aq. HCl, the aq. soln. was treated with the calcd. amt. of K<sub>2</sub>CO<sub>3</sub> and extd. with Et<sub>2</sub>O, and finally the Et<sub>2</sub>O soln. was pptd. with HCl in EtOH. I was prepd. (23%) by heating 1 g. of 4-bromo-2-amino-benzonic acid (II), *m*. 222°, and 0.7 g. CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>OH in 3 cc. concd. H<sub>2</sub>SO<sub>4</sub> for 6 hrs. on a water bath. Considerable difficulty was experienced in finding an efficient method of prep. II. Three methods were tried: (1) NaNO<sub>2</sub> (9 g.) in 50 cc. concd. H<sub>2</sub>SO<sub>4</sub> was added to 20 g. 4-BrC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>Ac in H<sub>2</sub>SO<sub>4</sub> to give 80% 2-nitro-4-bromo-



acetanilide, m 103-4°. This was saponified with 50%  $\text{H}_2\text{SO}_4$  to give 83% 2-nitro-4-bromoaniline, m 112°. This was very difficult to diazotize and gave only 12-15% of 2-nitro-4-bromobenzenitrile, m 98-9°, which was hydrolyzed by 50%  $\text{H}_2\text{SO}_4$  to give 75% of 2-nitro-4-bromobenzoic acid (III), m 163-4°. This was reduced by Fe and HCl to give 76% of II (2). When 2  $\text{O}_2\text{N}_2\text{C}_6\text{H}_3\text{Br}$  Me was brominated, using Fe, a mixt of isomers were formed from which 15% 2-nitro-4-bromotoluene (IV), m 43°, was isolated. It was almost impossible to oxidize IV to III. The only practical method, heating it in a sealed tube at 150-70° for 10 hrs with 30%  $\text{HNO}_3$ , gave only 48% (3). However, reduction of IV with Fe and HCl and steam distn gave 80% 2-amino-4-bromotoluene, an oil, acetate, m 165°. One g of this acetate in 500 cc  $\text{H}_2\text{O}$  contg  $\text{MgSO}_4$  was easily oxidized by  $\text{KMnO}_4$  in 6 hrs to give 90% 2-acetyl-4-bromobenzoic acid, m 290° (220° ?), which was hydrolyzed by 50%  $\text{H}_2\text{SO}_4$  to give 93% II. J F M

Configurational relationships of methylphenyl- and methylhexylacetic acids and an attempt at the correlation of the configurations of 2 hydroxy acids with those of disubstituted acetic acids containing a methyl group. P A Levene and Stanton A Harris. *J Biol Chem* 112, 195-208 (1935).—The configurations of  $\text{MeCHPhCO}_2\text{H}$  and  $\text{MeEt}(\text{C}_6\text{H}_{11})\text{CH}$  are correlated. Acids contg a Ph or cyclohexyl group are correlated among themselves and to the corresponding normal acids. Successive increase in the distance of a Ph group from the asym center of a substituted AcOH causes an alternation in the shift of the mol rotations. Substitution of  $\text{C}_6\text{H}_5$  for Ph and  $\text{C}_6\text{H}_{11}$  for Ph( $\text{CH}_2$ ), enhances the rotation. With cyclohexyl no periodicity or marked difference was noted in either the normal or cyclic series. The 2 HO acids are correlated with the disubstituted AcOH series on theoretical grounds based on Boys' theory. Carbinols of the type  $\text{R}(\text{CH}_2)_n\text{CH}(\text{OH})\text{Me}$  are correlated with hydrocarbons of the type  $\text{R}(\text{CH}_2)_n\text{CH}(\text{Me})\text{Et}$ , where R is Ph or cyclohexyl. When  $n = 0$  or 1 substitution of  $\text{C}_6\text{H}_5$  by  $\text{C}_6\text{H}_{11}$  has a similar effect on the mol rotation in both alcs and hydrocarbons. The effect of Ph is in the opposite sense in the carbinols with that of the hydrocarbons. When  $n = 2$  substitution of  $\text{C}_6\text{H}_5$  for  $\text{C}_6\text{H}_{11}$  has little effect on the rotation, substitution by Ph produces an identical effect in both cases. To  $\text{BrMg}(\text{CH}_2)_2\text{MgBr}$  (from  $\text{Br}(\text{CH}_2)_2\text{Br}$  (90 g)) was added 1-EtMeCHCO<sub>2</sub>Et. Hydrolysis with  $\text{NH}_4\text{Cl}$  soln gave a mixt of 1-1 sec-butyl-1-cyclohexanol and sec-butylcyclohexene 1 carbinol,  $b_p$  161-162°,  $[M]_D^{25}$  3.96°. To this (19.5 g) in  $\text{CHCl}_3$  at 0° was added Br in  $\text{CHCl}_3$  until no further decoloration occurred. After washing, the product was fractionated, giving 1-1 sec-butyl-1-cyclohexene (I), b 172-4°,  $d_4^{25}$  0.820,  $n_D^{25}$  1.4590,  $[M]_D^{25}$  -4.06°, and a compd,  $\text{C}_{10}\text{H}_{18}\text{OBr}_2$ ,  $b_p$  161°, of undetd structure. I reduced with  $\text{PtO}_2$  gave d-2 cyclohexylbutane, b 176-8°,  $d_4^{25}$  0.815,  $n_D^{25}$  1.4460,  $[M]_D^{25}$  0.59°. Active  $\text{AmMgBr}$  and  $\text{C}_6\text{H}_5\text{CHO}$  gave 3-methyl-5-undecanol,  $b_p$  70-5°,  $b_n$  115°,  $d_4^{25}$  0.8272,  $n_D^{25}$  1.4307,  $[M]_D^{25}$  17.4°. This with anhyd HI gave 3-methyl-5-iododecane, b 88°,  $d_4^{25}$  1.1972,  $[M]_D^{25}$  45.7°, which, reduced with Raney's catalyst, gave methylcyclohexylmethane,  $b_p$  94°,  $d_4^{25}$  0.7491,  $n_D^{25}$  1.4216,  $[M]_D^{25}$  17.4°. R C E

Synthesis of alceines, derivatives of o-phenylacetic and o-phenylphenylglycolic acids. E Plazek, Z Rodewald and D Krzyżanek. *Rozprawy Chem* 15, 360-4 (in German 364) (1935).—Esters of these acids with tropine and  $\text{Me}_2\text{NCH}_2\text{CH}_2\text{OH}$  were prepd. Equimol ams of o-Ph $\text{C}_6\text{H}_4\text{OCH}_2\text{CO}_2\text{H}$  and tropine were heated to 110-20° for 4 hrs, dry HCl being simultaneously blown through the molten mass, then concd  $\text{K}_2\text{CO}_3$  added. The alcine was extd with ether and distd at 208° under 1 mm pressure. By mixing ether solns contg equimol ams of o-Ph $\text{C}_6\text{H}_4\text{OCH}_2\text{COCl}$  and  $\text{Me}_2\text{NCH}_2\text{CH}_2\text{OH}$ , oil of alcine-HCl was formed, then the free alcine was obtained by treating with  $\text{K}_2\text{CO}_3$ . Extn with ether gives a product  $b_p$  148-150°. Esterification of o-phenylacetic acid by tropine gives the product  $b_p$  149-150°. The product of esterification of o-phenylacetic acid by  $\text{Me}_2\text{NCH}_2\text{CH}_2\text{OH}$   $b_p$  110°, m 75°. M Wojciechowski

Constituents of red sandalwood. Homopteroecarpin. H. Leonhardt and E Oechler. *Arch Pharm* 273, 447-52 (1935), cf C A 29, 2543°.—Homopteroecarpin shows agreement with pterocarpin in the function of 2 O atoms. The detection of the quinoid character of dihydrohomopteroecarpin is important from the standpoint of the sandalwood dyestuffs. Dihydrophenylhydrazones of homopteroecarpin,  $\text{C}_{20}\text{H}_{20}\text{O}_4\text{N}_2$ , brownish red needles, m 292° (decompn). Dihydrohomopteroecarpone,  $\text{C}_{20}\text{H}_{20}\text{O}_4$ , by oxidation of 1-dihydrohomopteroecarpin, lemon-yellow needles, m 178.5°, dinitrophenylhydrazone,  $\text{C}_{20}\text{H}_{20}\text{O}_4\text{N}_2$ , decomp 258° without melting, p-nitrophenylhydrazone,  $\text{C}_{20}\text{H}_{20}\text{O}_4\text{N}_2$ , decomp 148° without melting, monoxime,  $\text{C}_{20}\text{H}_{20}\text{O}_4\text{N}$ , yellow needles decomp 225° (dinitrophenylhydrazone,  $\text{C}_{20}\text{H}_{20}\text{O}_4\text{N}_2$ , copper needles decomp 199°). Acetylation of dihydrohomopteroecarpin in the presence of nascent H gives an Ac deriv  $\text{C}_{20}\text{H}_{20}\text{O}_4$ , m 122-3°. W O E

Obtaining bornyl chloride. E N Rostovskii and T V Sheremeteva. *Plasticheskie Massy* 1935, No 3, 87-4.—Bornyl chloride can be prepd without decrease in yield by satg pinene with HCl at 90°. Cu and Pb surfaces cause a smaller yield at this temp, and are themselves corroded unless they are coated with lacquer. H M Leicester

Synthesis of methylcyclohexanethrenecarboxylic acid and 4-methylphenanthrene. Georges Darzens and André Lévy. *Compt rend* 201, 730 3 (1935), cf C A 29, 6232°.—Di-Et tetrahydro- $\beta$ -naphthylmethylmalonate,  $b_p$  179°, m 123°, on decompn by heat gave tetrahydro-naphthylpropionic acid, m 127°. Di-Et tetrahydro- $\beta$ -naphthylmethylallylmalonate, b 189-90°, gave on sapon the malonic acid which decomposed on heating and gave tetrahydronaphthylallylpropionic acid (I). Cyclization of I as previously described gave octahydromethylphenanthrenecarboxylic acid (II),  $b_p$  187°, m 148-9°. II heated with Se caused dehydrogenation as well as decarboxylation and gave 4-methylphenanthrene, b 160°, m 116°. Julius White

Preparation of  $\beta$ -(1-naphthyl)propionic,  $\beta$ -(2-naphthyl)propionic and  $\beta$ -(2-tetrahydronaphthyl)propionic acids. Synthesis of dihydrophenalene and tetrahydro 5,6-benzindan-1-one. Georges Darzens and André Lévy. *Compt rend* 201, 902-4 (1935), cf preceding abstr— $\text{C}_{10}\text{H}_7(\text{CH}_2)_2\text{CO}_2\text{H}$  (I),  $b_p$  179°, m 156° (amide, m 133°, Me ester, b 162°, acid chloride (II), m 179-80°, m 26°), was prepd by heating the corresponding malonic acid. These consts are different from those reported by others. Likewise  $\beta$ - $\text{C}_{10}\text{H}_7(\text{CH}_2)_2\text{CO}_2\text{H}$  (III), m 127° (amide, m 133°, acid chloride, b 162°, m 2°, Me ester,  $b_p$  183°), and  $\beta$ - $\text{C}_{10}\text{H}_7(\text{CH}_2)_2\text{CO}_2\text{H}$ , m 135°, were prepd. II, treated with  $\text{AlCl}_3$  in  $\text{PhNO}_2$ , gave dihydrophenalene (IV), m 86° (oxime, m 126°). III treated gave tetrahydro-5,6-benzindan-1-one (V),  $b_p$  167°, m 47° (oxime, m 224°). V on oxidation gave 1,2,4,5-C<sub>10</sub>H<sub>6</sub>(CO)<sub>2</sub>H, m 275°. Julius White



(IV)



(V)

Terpene furoates. J N Borghin. *Ind Eng Chem* 28, 31-2 (1936).—The prepn of fenchyl furoate,  $b_p$  275°, n 1.6019, acid no 0.7, bornyl furoate,  $b_p$  275°, acid no 1.8, and fenchyl tetrahydrofuroate, n 1.4790, are described and their properties as plasticizers discussed.

E W Tillitson

Sulfur compounds of terpenes. VIII. Actions of gold chloride on thiorborneol and thioacaphor. Akira Nakatsuchi. *J Soc Chem Ind, Japan* 38, Suppl binding 617 (1935), cf C A 30, 1042°.—An oily compd was obtained from the reaction of thiorborneol and aq  $\text{AuCl}_3$  and with alc a white ppt was formed. The Au compd

was sol in  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$  and  $\text{CHCl}_3$ , m 194–5°, decomp. at 204°, and contained 53.36% Au and 9.00% S. The reaction product from  $\text{AuCl}_3$  and thiocamphor at 80° gave a greenish black amorphous ppt when treated with  $\text{Et}_2\text{O}$ . The ppt was sparingly sol in  $\text{CHCl}_3$  and did not show any definite decomp point. It contained 81.18% Au and 10.37% S. Analogous results were obtained when the reaction took place at 29°, but different results at 0–1°, when a compd, sol in  $\text{Et}_2\text{O}$ , was obtained which m 179–80° (decomp) and contained 63.71% Au and 10.05% S. IX Actions of gold chloride on various terpene sulfur compounds. *Ibid* 617.—The S compd from  $\Delta^2$ -p menthene gave an amorphous white ppt with  $\text{AuCl}_3$ , which was sol in  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$  and  $\text{CHCl}_3$ , m 92° and decomp at 171°. It contained 53.40% Au and 9.01% S. Analogous results were obtained with the S compd from  $\Delta^{10}$ -p menthene. The S compds of  $\alpha$ -phellandrene (I), linalyl acetate (II) and linaloolene (III) were treated with aq.  $\text{AuCl}_3$  under ice cooling, followed by heating to 95°. By treating the reaction product with alc Au, decomp point 158–63°, 77.93% Au and 8.22% S, II, 192–3°, 73.00 and 9.23, III, 161°, 74.25 and 9.44. These compds were sol in  $\text{CHCl}_3$  and sparingly sol in  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$  and alc. Karl Kammermeyer

Addition compounds of benzidine with some phenols and acids. Konstanty Hrynakowski, Henryk Staszewski and Marja Szmyt. *Roczniki Chem* 15, 391.8 (in German 398–9) (1935).—Thermal analysis of binary systems of benzidine with phenol and acids proves the high reactivity of benzidine in the formation of addn compds. There were found compds with resorcinol, hydroquinone, salicylic acid,  $\text{BzOH}$ ,  $\alpha$ - and  $\beta$ -naphthol. M Wojciechowski

Benzophenones from carboxylic acids. H P Newton and P. H Groggins. *Ind. Eng. Chem* 27, 1397–9 (1935).—Condensation of  $\text{BzCO}_2\text{H}$ ,  $p$ - $\text{ClC}_6\text{H}_4\text{COCO}_2\text{H}$ ,  $p$ - $\text{MeC}_6\text{H}_4\text{COCO}_2\text{H}$  and terephthalic acid with chlorobenzene by the Friedel-Craft reaction is reported, with practically quant. yields, the formation of  $o$ - and  $p$ -isomeric benzophenones is established. In the ammonolysis of halo-benzophenones the presence of Cu is desirable for obtaining a satisfactory rate of amination. Under suitable conditions in the amination of (4- $\text{ClC}_6\text{H}_4$ ) $_2\text{CO}$  it is possible to effect the replacement of only 1 halogen. 3,3',4,4'-Tetrachloro-4'-benzoylbenzophenone, m 69–71°, is prepd by condensing terephthalic acid with  $\text{PhCl}$  in the presence of anhyd  $\text{AlCl}_3$ ; oxime, m 210°, anilide, m 288°. 4-Chloro-4'-methylbenzophenone, m 129.5–9°, 3,4-dichlorobenzophenone, m 101–1.5°, 2-chloro-4-methylbenzophenone, m 99.5°, 4-amino-4'-methylbenzophenone, m 186–7°. O W Wilcox

4,4'-Dibenzylidiphenylmethane. Edmond Connerade. *Bull. soc. chim. Belg* 44, 411–24 (1935).—To 50 g  $\text{Ph}_2\text{CH}_2$  (I) and 15 g methylal in 300 g  $\text{AcOH}$  was added slowly with vigorous shaking 50 g concd  $\text{H}_2\text{SO}_4$ . After several hrs a mixt. of 300 g of each of the 2 acids was added and finally, after several hrs of shaking, 500 g concd  $\text{H}_2\text{SO}_4$ . After diln with  $\text{H}_2\text{O}$ , extn with hot  $\text{PhMe}$  and removal of  $\text{PhMe}$  in *vacuo*, the mass was extd with boiling  $\text{EtOH}$ . I from the  $\text{EtOH}$  soln there crystd 4,4'-dibenzylidiphenylmethane (II) (2–3% yield), m 91.5°. Oxidized by  $\text{CrO}_3$  in boiling  $\text{AcOH}$  II gave 4,4'-dibenzoylbenzophenone (III), m 227.5°. The reaction of  $\text{PhLi}$  with ( $p$ - $\text{ClC}_6\text{H}_4$ ) $_2\text{C}(\text{CH}_3)_2$  (IV) gave an amorphous product and  $\text{Ph}_2\text{IV}$  with  $\text{C}_6\text{H}_5$  in the presence of  $\text{AlCl}_3$  gave I and a resinous product, but not II. The action of ( $p$ - $\text{ClC}_6\text{H}_4$ ) $_2\text{CO}$  with  $\text{C}_6\text{H}_5$  in the presence of  $\text{AlCl}_3$  gave a quant. yield of 4,4'-dibenzoylbenzophenone (V), m 227.5°, which was obtained in cryst. form only with difficulty. Oxime, m 130–1°, decomposes in light, and is easily hydrolyzed by acids. V, reduced with Na-Hg in  $\text{EtOH}$ , gave the carbinol, which could not be crystd. Oxidized with  $\text{CrO}_3$  in boiling  $\text{AcOH}$ , V gave III. The trioxime of III is unstable. The mixt. obtained by treating ( $p$ - $\text{ClC}_6\text{H}_4$ ) $_2\text{CO}$  in  $\text{C}_6\text{H}_5$  with  $\text{AlCl}_3$  was extd. with boiling  $\text{AcOH}$ . From this ext. III was obtained in 20–5% yield. The residue insol in  $\text{AcOH}$  was a mixt. of benzophenone-3,4'-dicarboxylic acid, m

360°, and 4-benzoylbenzophenone-4'-carboxylic acid, m. 268°. Reduction of III with Na-Hg in 95%  $\text{EtOH}$  at 70° gave 2 forms of [ $\text{PhCH}(\text{OH})\text{C}_6\text{H}_4$ ] $_2\text{CHOH}$ . One form, supposed to be the *syn* form (VI) because of its relative stability, m 176.5° and decomposes 180°; the *anti*-form (VII), m 105–10° and decomposes 115°. Both were oxidized by  $\text{CrO}_3$  to III. When suspended in  $\text{C}_6\text{H}_6$  and treated with dry  $\text{HCl}$ , VI gave a *tri-Cl deriv.*, yellowish, m 73–4°, and VII gave a *tri-Cl deriv.*, which could not be crystd. Louise Kelley

The hydrogenation of unsaturated compounds. N. K. Yurashevskii. *J. Gen. Chem.* (U. S. S. R.) 5, 1098–1107 (1935).—When mixts of olefin derivs. are catalytically hydrogenated, it is not always true that the component with the greatest no. of H atoms attached to the doubly linked carbons hydrogenates first, to the exclusion of the other components. A higher energy content (heat of combustion) also favors hydrogenation. *Mixts. studied.*—Benzoylbenzene(I)—stilbene (II) simultaneous absorption of H, a little more rapidly by I. I—Na cinnamate (III) at 50°, complete hydrogenation, I was 70% hydrogenated, III 30%. III pulegone (IV) IV begins to absorb H only after the reaction with III is complete. IV—fumaric acid (V). V hydrogenates first, but IV begins to take up H before the reaction with V is complete. IV—allyl alc. (VI), III–VI in both pairs VI reacts completely before the other component begins. II–VI simultaneous absorption of H. Limonene–III. III is hydrogenated first. IV–MeC<sub>6</sub>CHME (VII). IV hydrogenates first, then VII. III–Me cinnamate (VIII), cinnamic acid–VIII. VIII hydrogenates first. *Exptl.*—Hydrogenation was carried out according to the directions of Ginsberg and Ivanov (*C. A.* 25, 4173). One g Ni, 0.1 g  $\text{PdCl}_2$ , 2NaCl, 30 cc solvent (alc alone or 30 cc alc plus varying amts of  $\text{H}_2\text{O}$  or  $\text{AcMe}$ ), and electrolytic H were employed. A correction was made for H absorbed by the catalyst. The course of hydrogenation was the same with the mixt. III–VI whether the solvent was 40 cc alc plus 3 cc  $\text{H}_2\text{O}$  or 15 cc  $\text{AcMe}$  plus 15 cc alc plus 5 cc  $\text{H}_2\text{O}$ . L. W. B.

The nitration of  $\alpha$ -ethylphenylalene. Georges Lévy. *Compt. rend.* 201, 900–2 (1935), *C. A.* 27, 717.—The nitration of  $\alpha$ - $\text{C}_6\text{H}_4\text{Et}$  as previously described gave a mixt. of isomers from which 1,4- $\text{C}_6\text{H}_4\text{Et}(\text{NO}_2)$  was isolated and identified by reduction to the amine. Julius White

Addition of substituents in the anthraquinone series. Ch. Marschall. *Bull. soc. chim.* [5], 2, 1809–30 (1935), *C. A.* 22, 74.—Certain sulfonic acids of the anthraquinone series are characterized by the peculiar mobility of the conjugated  $\text{SO}_3\text{H}$  group. A study of the action of alk. cyanides on 1,4-dihydroxy-2-anthraquinonesulfonic acid (I) has shown that the final product is 2,3-dicyanoquinizarin (II) and that the reaction is not a simple substitution. To a soln of 10 g of the Na salt of I, prepd according to Ger. pat. 287,867, in 500 g  $\text{H}_2\text{O}$  and 5 g  $\text{Na}_2\text{CO}_3$  was added 10 g KCN and the mixt. was heated at 90° for 30 min. Oxidation of the leuco form with 3.5 g  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  gave a blue-violet salt which was filtered off, washed free from KCN and decompd with boiling dil  $\text{HCl}$ . The orange ppt. was recrystd. from  $\text{PhCl}$  and yielded 7 g of orange-brown crystals of II,  $\text{C}_{14}\text{H}_6\text{N}_2\text{O}_2$ , m above 300°. By heating a suspension of 5 g of quinizarin (III) in a soln of 40 g KCN in 200 cc  $\text{H}_2\text{O}$  for 5 hrs and operating as described above a yield of 1.7–1.9 g of II was obtained. The intermediary formation of a quinizarin-quinone and the interpretation of the reaction as the addn of HCN are excluded since all operations are carried out in the absence of air and in the presence of a reducing agent. Sulfonation of 0.5 g of II with 10 cc of 20% oleum at 140–50° in the presence of a trace of yellow  $\text{HgO}$  yielded a sulfonic acid, sol in  $\text{H}_2\text{O}$  and which gave a blue Al lake similar to that produced by the transformation product of 2,6-quinizarindisulfonic acid with KCN which contains a conjugated  $\text{SO}_3\text{H}$  group that renders it sol in  $\text{H}_2\text{O}$ . A soln of 30 g of 1-amino-4-hydroxyanthraquinone-2-sulfonic acid (IV) (Ger. pat. 286,308) in 2000 cc  $\text{H}_2\text{O}$  contg. 45 g KCN and 10 g  $\text{Na}_2\text{CO}_3$  was heated for 3–4 hrs at 90–5°. The cooled soln. was treated with

10 g (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and yielded 9 g of 1-amino-4-hydroxy-2,3-dicyanoanthraquinone (V). Similar treatment of the 3-sulfonic acid isomer of IV gave an analogous dinitrile. Heating 5 parts of II in 40 parts by vol. of 90% H<sub>2</sub>SO<sub>4</sub> at 150° for 1-1.5 hrs gave the insol anhydride of 1,4-dihydroxy-2,3-anthraquinonedicarboxylic acid (VI), sol in dil alk carbonates or NaOH. The acidulation of these alk salts gave dull-gold leaflets of the corresponding acid (VII), identical with the acid prep'd from 1,4-dihydroxy-2,3-dimethylantraquinone (Fr pat. 739,506). This identification establishes the correctness of the formulation of II, VI and VII. The partial decarboxylation of VII by the action of conc'd H<sub>2</sub>SO<sub>4</sub> in the presence of H<sub>2</sub>BO<sub>3</sub> at 200° gave 2-quinizarincarboxylic acid (VIII), m 244-6°, identical in its sulfoboric acid and spectra with the acid prep'd by the oxidation of 2-methylquinizarin (Ger pat. 273,341). Oxidation of VIII with MnO<sub>2</sub> transformed it into 2-purpurincarboxylic acid, m 222-4°. By the above method of hydrolysis 10 g. of II was transformed directly into 6 g. of VIII. Controlled alk hydrolysis of VI gave VIII but if the heating with 5% NaOH was continued for several hrs the entire material was converted into III. A suspension of 10 g. of VI in 1100 cc. of 1.5% Na<sub>2</sub>CO<sub>3</sub> was boiled for 10 min, dild with 1100 cc. H<sub>2</sub>O and filtered. The soln was cooled to 2° and to it was added, dropwise, 4 g. of 87% Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>. After standing for 1 hr below 4° in the absence of air 60 cc. of 30% NaOH was added and the leuco deriv. formed was oxidized by a current of air. Purification of the product gave 8 g. of VIII, m 245-7°. Operation at room temp. with an excess of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> gave dihydroquinizarin, m 158°. The constitution of II demonstrates the complexity of the fundamental reaction and it is certain that the CN group at position 3 must be the result of an addn reaction. The cyanides act slowly on I at room temp. and it was found possible by this means to isolate the intermediate in the formation of II. Air was bubbled through a cooled soln of 20 g. of the Na salt of I, 20 g. Na<sub>2</sub>CO<sub>3</sub> and 16 g. KCN in 4 l. H<sub>2</sub>O for 5-6 days. The soln was acidulated with HCl, boiled and filtered and salted out with KCl, yielding 1,4-dihydroxy-3-cyanoanthraquinone-2-sulfonic acid (IX) which is readily transformed into 1,4-dihydroxy-3-cyanoanthraquinone (X), m 228-9°, by reductive desulfonation. The constitution of X results from its hydrolysis products, 1,4-dihydroxy-2-anthraquinonecarbamide, m. above 300°, and VIII. Both IX and X are readily converted into II by the action of KCN in alk. soln. It is concluded that the conjugated SO<sub>3</sub>H group of I activates the mol. and enables it to add HCN and fix a CN group in position 3. The displaced H atom together with the acid H atom convert the quinone into the leuco form of IX which is desulfonated by the action of the alk. soln and transformed into X. Thus certain substituents such as SO<sub>3</sub>H, CN, CO<sub>2</sub>H, CO<sub>2</sub>R, CONH<sub>2</sub> in the 2-position of III accentuate the state of unsatn of the mol and render it capable of subsequent addn. III acts in an analogous fashion, though more slowly and less definitely, as is shown by the formation of some 1,4-dihydroxy-2,3-dicyanoanthraquinone in the leuco form by the action of alk. KCN. It may be supposed that the introduction of 2-substituents may inhibit secondary reactions. The formation of such bimol. substances as the 2,2'-biquinizarins may be due to the unsatd state of III and result from addn reactions. The unsatd nature of III and its derivs was exam'd by the reactions with dienes. In org. solvents, even in a sealed tube at 140°, no normal diene synthesis was observed but I, III, VIII and X reacted with cyclopentadiene in aq. alk. media at 70-80°. A similar reaction was observed with naphthazarin. It has been shown that the dinitrile formed by the action of KCN on 1,4-diamino-2-anthraquinone-sulfonic acid is 1,4-diamino-2,3-dicyanoanthraquinone which can be degraded to II by controlled oxidation. VI condenses readily with NH<sub>3</sub>, amines and amino carboxylic acids. In the presence of AlCl<sub>3</sub> and Na it reacts with hydroquinone to give a product which is probably a tetrahydroxyphenanthraquinone. C. R. Adnall.

1-Keto-1,2,3,4-tetrahydro-7-hydroxyphenanthrene. Adolf Butenandt and Gerhard Schramm. Ber.

68B, 2083-6(1935); cf. C. A. 28, 2082<sup>1</sup>-6-HO-S-1-C<sub>10</sub>H<sub>7</sub>NH<sub>2</sub>, fused with KOH at 310°, gave 6-HO-1-C<sub>10</sub>H<sub>7</sub>NH<sub>2</sub>, which on treatment with Ac<sub>2</sub>O gave 6-HO-C<sub>10</sub>H<sub>7</sub>NHAc (I). I, treated with Me<sub>2</sub>SO<sub>4</sub>, gave 6-MeOC<sub>10</sub>H<sub>7</sub>NHAc (II). II in EtOH, heated 2 hrs with conc'd HCl, gave 6-MeOC<sub>10</sub>H<sub>7</sub>NH<sub>2</sub> (III), m 205-20° (decomp.); 6-MeOC<sub>10</sub>H<sub>7</sub>NH<sub>2</sub>, m. 74°. III, treated with NaNO<sub>2</sub> and a mixt. of Cu<sub>2</sub>SO<sub>4</sub> and KCN, gave 6-MeOC<sub>10</sub>H<sub>7</sub>CH<sub>2</sub>N, m 79°. III, diazotized and treated with KI, gave 6-MeOC<sub>10</sub>H<sub>7</sub>I, b 175-8°, m. 33°. 6-MeOC<sub>10</sub>H<sub>7</sub>I (IV), caused to react with CHO(CH<sub>2</sub>)<sub>3</sub>CO<sub>2</sub>Et, gave 6-MeOC<sub>10</sub>H<sub>7</sub>CH(CH<sub>2</sub>)<sub>3</sub>CO<sub>2</sub>H (V), m 156°, Me ester, m 60°. V, reduced over Pt or Pd, gave 6-MeOC<sub>10</sub>H<sub>7</sub>(CH<sub>2</sub>)<sub>3</sub>CO<sub>2</sub>H (VI), m 149°. VI, treated with SnCl<sub>4</sub> at 105° for 1 hr, gave 7-methoxy-1-keto-1,2,3,4-tetrahydrophenanthrene (VII), m 101°. VII, treated with HOAc and HBr, gave 7-hydroxy-1,2,3,4-tetrahydrophenanthrene (VIII), m 232°. Bz ester, m 212°. VIII was physiologically inactive. IV, treated with CHO(CH<sub>2</sub>)<sub>3</sub>CO<sub>2</sub>Me, gave 6-MeOC<sub>10</sub>H<sub>7</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H which formed the lactone (IX), b<sub>10</sub> 160-70°, m 114°. IX, boiled with KOH and treated with MeN<sub>3</sub>, gave 7-MeOC<sub>10</sub>H<sub>7</sub>CH(CH<sub>2</sub>)<sub>3</sub>CO<sub>2</sub>Me (X), b<sub>10</sub> 140°. X, reduced with Pt or Pd and then saponified, gave VI.

Julius White

Dicarboxylic acid esters of tetrahydrofurfuryl alcohol. J. N. Borglin. Ind Eng Chem 28, 35-6(1936).—The tetrahydrofurfuryl esters of the following dicarboxylic acids were prep'd and their values as plasticizers discussed. Tetrahydrofurfuryl alc (I) and terpene-maleic anhydride gave an ester, acid no 66.5, which was further esterified with butanol, reducing the acid no to 45, n<sub>D</sub><sup>20</sup> 1.5023. Ester from I and maleic anhydride, acid no 69, n<sub>D</sub><sup>20</sup> 1.4897, d<sub>4</sub><sup>20</sup> 1.1931. Ester from I and phthalic anhydride, acid no 64.5, n<sub>D</sub><sup>20</sup> 1.5250, d 1.2165.

E. W. Tillotson

N-Phenethyl-3-phenyl-5-pyrazolone. E. Votokel and O. Wichterle. Collection Czechoslov Chem Communications 7, 388-91(1935); cf. C. A. 27, 3475.—N-Phenethyl-3-phenyl-5-pyrazolone (I), m 145.5°, was obtained in good yield (75% even with crude hydrazine) by the spontaneous reaction of PhCH<sub>2</sub>CH<sub>2</sub>NHNH<sub>2</sub> and PhCO-CH<sub>2</sub>CO<sub>2</sub>Et in aq. EtOH. It was sol in alkalies but not in acids. The 4-isomero (II), m 138-9°, was obtained (90%) by treating I in EtOH with HCl and NaNO<sub>2</sub> (red crystals from AcOH; yellow from EtOH). Reduction of II with Zn in AcOH gave a few colorless crystals which apparently were the 4-amino deriv. of I. These gave an oil with BzH. After they were filtered off, the addn of H<sub>2</sub>O to the mother liquor gave a dark red ppt which was probably a homolog of rubanic acid. CO N-

(CH<sub>2</sub>CH<sub>2</sub>Ph) . N CPh . CHN C . CPh N N(CH<sub>2</sub>CH<sub>2</sub>Ph).CO, m 165°. When 10 g I, 10 g MeOH and 10 g

MeI were kept at 110° for 5-8 hrs in a sealed tube, 50-55% pure hydriodide of the 2-Me deriv., PhC CH CO N

(CH<sub>2</sub>CH<sub>2</sub>Ph) NMe HI, m 174-5°, were formed. The

free base was an oil. John E. Milbery  
γ-Tetrahydropyranaldehyde. V. Prelog and E. Cerkovnikov. Collection Czechoslov Chem Communications 7, 430-5(1935).—P and C. desired this comp'd in order to prep long γ-chains in tetrahydropyran. Attempts to prep it by the dry distn of Ba γ-tetrahydropyranate and (HCO<sub>2</sub>)<sub>2</sub> Ba gave very poor results, so CH<sub>2</sub> CH<sub>2</sub> O CH<sub>2</sub>-

CH<sub>2</sub> CHCOCl was reduced with H<sub>2</sub> and Pd (C. A. 15,

2435) with better, although not always reliable, results. The following is a typical run. To 40 g. of the acid chloride in 150 g. xylene were added 4.7 g. BaSO<sub>4</sub>, contg 5% Pd and 235 (?) g. quinoline sulfate. This mixt was boiled and H<sub>2</sub> was passed in for 24 hrs. After being filtered the mixt was distilled at 15 mm Hg to give 6.8 g. impure aldehyde. A second distn gave pure γ-tetrahydropyranaldehyde (I), b<sub>10</sub> 74-7°; phenylhydrazones, m. 63-4°.

2,4-dinitrophenylhydrazones, m. 103°; semicarbazone, m. 181.5–2.5°, and 191°. The aldehyde was very unstable and rapidly polymerized to the crystalline trimer, m. 221°. When a mixt. of 3 g. I, 3.5 g. rhodanin, 3 g. AcONa, 10 cc. AcOH, and 10 cc. Ac<sub>2</sub>O was agitated for an hr. at 100–10°, 3.27 g. of a condensation product,  $\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{CO}$ —

NHCS<sub>2</sub>, m. 234°, was formed. When this product was warmed with 15% NaOH soln *γ*-tetrahydropranyl-thiopyruvic acid,  $\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{CSCo}_2\text{H}$ ,

m. 151–2°, was formed. John F. Mulbrey  
2-Aminopyridine series III. Salicyl-2-aminopyridine and its electrolytic reduction to *o*-hydroxybenzyl-2-aminopyridine. K. Feist, W. Awe and W. Völksen. *Arch. Pharm.* 273, 476–8 (1935).—The studies previously reported in this field (cf. C. A. 28, 3407°, 29, 792°) have been continued. Starting with *o*-AcOC<sub>6</sub>H<sub>4</sub>COCl, salicyl-2-aminopyridine was prepd and the latter electrolytically reduced to *o*-hydroxybenzyl-2-aminopyridine, C<sub>10</sub>H<sub>10</sub>ON, m. 105°, identical with the product obtained from *o*-hydroxybenzylidene-2-aminopyridine (picrate, m. 185–6°). Similar expts. with phthaloyl-2-aminopyridine yielded products indicative of cleavage during reduction.

W. O. E.  
3-Aminopyridine II (Methylated derivatives, acetylaminopyridine, formylaminopyridine). E. Plazek, A. Marcinkow and Ch. Stammer. *Roczniki Chem.* 15, 365–76 (in German 376–7) (1935), cf. C. A. 29, 2535°.—The authors found that all derivs. of 3-aminopyridine are chemically more inactive than those of 2-aminopyridine. The following compds. were prepd: 3-Methylaminopyridine, obtained in 4.6 g. yield by heating in a sealed tube to 125–135° for 20 hrs. 10 g. 3-bromopyridine and 25 cc. 40% aq. MeNH<sub>2</sub> with addn of 1 g. CuSO<sub>4</sub> as catalyst. The product was extd. with ether and distd. at 118–120° under 12 mm. pressure. 3-Acetylmethylaminopyridine, obtained in 9 g. yield by boiling 1 hr. 8 g. amine and 11 g. Ac<sub>2</sub>O. The product b<sub>p</sub> 145°, when crystd. from petr. ether, it m. 64°. 2,6-Dibromo-3-methylaminopyridine, obtained in 4.6 g. yield by adding to 1.06 g. amine in 5 g. AcOH 6.1 g. Br in 5 cc. AcOH, crystd. from EtOH-H<sub>2</sub>O, it m. 69°. 3-Methylnitrosamine, obtained in 4 g. yield by diazotization of 5 g. amine, b<sub>p</sub> 135°. 3-Pyridylmethylhydrazine, obtained by the reduction of the nitro compd. with Zn powder in AcOH, b<sub>p</sub> 191°. 3-Methylnitrosaminopyridine, obtained in 2.5 g. yield by adding at 0° to 4.5 g. amine in 10 cc. concd. H<sub>2</sub>SO<sub>4</sub> a mixt. of 3 cc. HNO<sub>3</sub> (d. 1.52) and 5 cc. concd. H<sub>2</sub>SO<sub>4</sub>, m. 54–5°, b<sub>p</sub> 124–5°. 2-Nitro-3-methylaminopyridine, obtained in 2 g. yield by introducing slowly 5 g. 3-methylnitrosamine into 25 cc. concd. H<sub>2</sub>SO<sub>4</sub> at 0–5°, m. 110°. 3-Methylamino-2-aminopyridine, obtained by reduction of the 2 nitro compd. with SnCl<sub>2</sub> or by amidation of 3-methylaminopyridine with NaNH<sub>2</sub>, m. 124°. 6-Amino-3-methylaminopyridine, obtained by heating at 110–115° for 20 hrs. 6 g. 3-bromo-6-aminopyridine with 15 cc. of 40% aq. MeNH<sub>2</sub> and 0.5 g. CuSO<sub>4</sub> as catalyst, m. 70°. Bromination of 3-dimethylaminopyridine gives 2,6-dibromodimethylaminopyridine, b<sub>p</sub> 113–15°, and bromo-3-dimethylaminopyridine, m. 64–6°. Formylaminopyridine obtained by boiling for 4 hrs. 5 g. 3-aminopyridine and 10 cc. HCO<sub>2</sub>H, b<sub>p</sub> 200° under 25 mm. pressure, m. 90°. There was also obtained 3-acetylaminopyridine nitrate, m. 165°. M. Wojciechowski.

The derivatives of 3,3'-bipyridine. Wanda Brydówna and Władysław Wiszniewski. *Roczniki Chem.* 15, 378–81 (in French 381–2) (1935); cf. C. A. 29, 2535°.—The di-*t* ester of 3,3'-bipyridine-2,2'-dicarboxylic acid, obtained in 60% yield by boiling for 2.5 hrs. 7 g. of the neutral salt of this acid with 14 g. of EtI and 10 g. of anhyd. benzene, m. 82–4°. The di-*m* ester, m. 153–5°, was also prepd. The diamide, obtained in 86% yield by heating in a sealed tube to 135–145° for 8 hrs. 2 g. of the di-*t* ester with 12 cc. of anhyd. EtOH, said with NH<sub>3</sub> at –10°, m. 268.5–70°. The chloride, obtained by boiling 10 g. of the acid and 100 g. of SOCl<sub>2</sub> for 3 hrs., m. 147–

150°. 2,2'-Diamino-3,3'-bipyridine, m. 181.5–2.5°, and 3,3'-bipyridine-2,2'-pyridone-2°, m. 366–8°, were also obtained. M. Wojciechowski.

4-Bromopyridine. J. P. Wibaut, J. Overhoff and H. Geldof. *Rec. trav. chim.* 54, 807–12 (1935).—The prepn of 4-bromopyridine (II) was carried out in 2 ways: 4-hydroxypyridine (γ-pyridone) was heated in PBr<sub>3</sub> and POBr<sub>3</sub> to form I which was isolated as the picrate, decomp. 223°, 4-nitroaminopyridine was mixed with concd. HBr soln, cooled to –5°, treated with NaNO<sub>2</sub> (3 mols.) and then heated. I was obtained by steam distn. and extn. of the distillate with Et<sub>2</sub>O which was evapd. at 15 mm. without heating. I b<sub>p</sub> 27–30°, m. 0–1°, n<sub>D</sub><sup>20</sup> 1.5679, decomposed on standing in a sealed tube, forming a yellow-brown solid which was possibly a pyridylpyridinium compd. I formed a HgCl<sub>2</sub> salt decomp. 270°, and chloroplatinate, and reacted with NH<sub>3</sub> at 200°, forming 4-aminopyridine. E. W. Scott.

Reactions of aromatic mixed ketones with disubstituted derivatives of thiourea containing different aromatic radicals. K. Dzwiewski, L. Gziler and J. Moszew. *Roczniki Chem.* 15, 400–6 (in French 407) (1935).—The following compds. were prepd: 2-phenyl-4-aminol-6-methylquinoline, obtained in 8 g. yield by heating 20 g. PhCOMe and 40.3 g. phenyl *p*-tolylthiourea at 180–210° for 5 hrs., then a short time at 230°, m. 161°, sol in AcOH. 2-Phenyl-4-nitrosoaminol-6-methylquinoline, yellow, m. 137–8°. 2-Phenyl-4-acetylaminol-6-methylquinoline, m. 142°. 2-Phenyl-4-aminol-6-methylquinoline-Mel, m. 235°. 2-Phenyl-4-phenylmethylaminol-6-methylquinoline, yellow, m. 167°. 2-Phenyl-4-hydroxy-6-methylquinoline, m. 201°. 2-Phenyl-4-β-naphthylaminol-5,6-benzoquinoline, obtained by treating 34.8 g. β-naphthylphenylthiourea and 15 g. PhCOMe at 180° for 4 hrs., then at 280° for 0.5 hr., m. 202–3°. The HCl salt, m. 286.7°, picrate, m. 260–1°, and methiodide, m. 293–4°, of 2-phenyl-4-β-naphthylaminol-5,6-benzoquinoline were prepd. 2-Phenyl-4-(β-naphthylacetylaminol)-5,6-benzoquinoline, m. 172–3°. 2-Phenyl-4-(β-naphthylnitrosaminol)-5,6-benzoquinoline, yellow, m. 250–1°. 2-phenyl-4-hydroxy-5,6-benzoquinoline, m. 280–1°, were also obtained. 2-(*p*-Tolyl)-4-(β-naphthylaminol)-5,6-benzoquinoline, obtained by heating at 180–260° for 5 hrs. 140 g. β-naphthylthiourea and 67 g. *p*-MeC<sub>6</sub>H<sub>4</sub>COMe, then a short time at 290°, m. 226°. 2-*p*-Tolyl-4-(β-naphthylnitrosaminol)-5,6-benzoquinoline, yellowish orange, m. 195°, and 2-*p*-tolyl-4-hydroxy-5,6-benzoquinoline, m. 314–15°, were prepd. M. Wojciechowski.

The preparation of 2,2'-biquinolyl by the catalytic dehydrogenation of quinoline. J. P. Wibaut, H. D. Tjeenk Willems, Jr. and W. E. Nieuwenhuis. *Rec. trav. chim.* 54, 804–7 (1935).—Quinoline, heated in a sealed glass tube to 325–35° in the presence of Ni (suspended on Al<sub>2</sub>O<sub>3</sub>), was dehydrogenated, forming 2,2'-biquinolyl (I) in 15% yield. I was also formed in very small amts. when I was used as a catalyst. E. W. Scott.

Malaria medicaments. Syntheses in the acridine group. I. Kh. Fed'man and E. L. Kopelovich. *Arch. Pharm.* 273, 488–90 (1935).—In all the acridine compds. described in the literature as possessing antimalarial action the halogen occupies the 6-position. Accordingly, in the present study the authors decided to prep. compds. (1) which possess in the positions 2 and 9 the same radicals as occur in acrinin (atebrin), in which, however, the halogen (Cl) occupies position 6 instead of 7, (2) compds. of the same type, but with the side chain –NHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> in position 9, (3) compds. with 2 halogen (Cl) atoms in positions 6 and 7 of the acridine nucleus. The compds. thus obtained were tested on finches and sparrows, their chemotherapeutic indices were lower than that of acrinin (atebrin), indeed for compds. (1) and (2) 7.5 times, for compds. (3) 2.5 times less. On removal of the Cl atom from position 6 in Magidson and Grigorowski's compd. the chemotherapeutic action on the malaria plasmodium is completely nullified. Furthermore, it appears that in the transfer of the Cl atom from position 6 to 7, its action is almost completely nullified, the chemotherapeutic index being only 2, instead of 15 for acri-

chim. Not less interesting is the fact that introduction of a 2nd Cl atom in the acricin mol. causes marked diminution in its activity, the index being then only 6 instead of 15. For purposes of this study the following substances were prepd: 2-Methoxyphenyl-4'-chloroanthranic acid,  $C_{14}H_{11}NO_4Cl$ , m 185-6°, 2-methoxy-7,9-dichloroacridine,  $C_{14}H_9NO_2Cl_2$ , m 203-4°, 2-methoxy-7-chloro-9-(2-diethylamino-4-methylbutylamino)acridine-2 HCl,  $C_{24}H_{31}N_3O_2Cl$ , canary yellow, m 221-2°, 2-methoxy-7-chloro-9-(2-diethylaminopropylamino)acridine-2 HCl,  $C_{24}H_{31}N_3O_2Cl$ , yellow, m 246-7°, 2,4,5-Trichlorotoluene,  $C_7H_5Cl_3$ , m 80-1° oxidized with  $HNO_3$  it yields the 2,4,5-trichlorobenzoic acid, m 160-3°, 4-methoxydiphenylamino-3',4'-dichloro-6'-carboxylic acid,  $C_{18}H_{11}NO_5Cl_2$ , green, m 233-4°, 2-methoxy-6,7,9-trichloroacridine,  $C_{14}H_7NO_2Cl_3$ , yellowish green, m 192-3°, 2-methoxy-6,7-dichloro-9-(2-diethylaminomethylbutylamino)acridine-2 HCl, orange, m 236-7°.

**Arylselenium halides VI Cleavage of ketosulfido-carboxylic acids** 2 Otto Behagel and Wilhelm Muller *Ber* 68B, 2164-6(1935), cf *C A* 29, 7960°—In the course of the study of the cleavage of ketosulfido-carboxylic acids, 3 keto 2,3-dihydrobenzo-1,4-selenazine (I) was included as a representative of heterocyclic ketones with 2 hetero atoms. From this the 2 Br deriv was to be prepd by a method analogous to that used by Zahn (*C A* 17, 2425) for the S comp, but with Br or Cl I first gave addn products,  $C_{11}H_7NHSO_2$ ,  $SeX_2$ , which on long

standing, rapidly on boiling in PhMe or AcOH, lost HX and formed the 2 halogen derivs of I by migration of the halogen from the Se to the adjacent C atoms. These halogen derivs with  $HSCH_2CO_2H$  gave the corresponding ketosulfido-carboxylic acid (II) which was broken down by alkali to I and  $HOSCH_2CO_2H$ , the latter being hydrolyzed to  $H_2S$ ,  $OHCCO_2H$  (or  $HOCH_2CO_2H$ ) and  $(CO_2H)_2$ . I, m 182°, was prepd by reduction of  $\alpha$ -O $_2$ C $_6$ H $_4$ Se-CH $_2$ CO $_2$ H with NaOH-Na $_2$ S, 1,1-Dibromide, yellow-brown, m 211°, 2-Br deriv, m 213-14°, 1,1-Dichloride, yellow brown, m 222°, 2-Cl deriv, m 227-8°. The dibalides boiled with water yield the oxide, m about 160° II, yellowish, m 209 10°.

**Alkaloids of the leaves of Jaborandi. Synthesis of plocarpidine** N A Preobrazhenskii, A M Polyakova and V A Preobrazhenskii *Compt rend acad sci U S R R [N S]*, 3, 267-9(1935) cf *C A* 29, 4740°—The syntheses of d homopilocarpic acid (I) is complicated by spatial rearrangement during the procedure. Accordingly, the diazo ketone from d plocarpic acid (II) was converted into I by the method of Arndt and Eistert (*C A* 29, 3323°). A mixt of 1 g of II, m 121-2-2°,  $[\alpha]_D^{25}$  54.60°, and an excess of  $SOCl_2$  was heated for 5 hrs at 35-40°. The resulting chloroanhydride was dissolved in Et $_2$ O and added to an Et $_2$ O soln of  $CH_3N_2$  prepd from 10 cc of  $EtO_2CN(N_2O)Me$ . The cooled mixt pptd an almost quant yield of yellowish crystals of diazomethyl d-pilocarpic ketone (III), m 106-8°. A soln of 0.97 g of III in 100 cc of  $H_2O$  at 50° was added dropwise to a mixt of 1.03 g of  $Na_2S_2O_3$  and 0.68 g of  $Ag_2O$  in 35 cc of  $H_2O$ . Extn of the reaction mixt. gave 0.78 g of I,  $C_{12}H_{15}NO_4$ , which was converted into a chloromethyl d-homopilocarpic ketone, m 88.5-9.2° identical with a similar deriv. from I obtained from natural sources.

**C R Addinall**  
**Mitraphylline** Raymond-Hamet and Louis Millat *Bull sci pharmacol* 42, 602-11(1935)—Mitraphylline (I), the alkaloid extd by Michiels from the bark of *Mitragyna stipulosa* O Kuntze, is not identical with mitramerine (II), obtained from the same source. I m 258-67°, II m 206-16°. The  $\alpha_D$  of I is -7.7°, its probable formula  $C_{24}H_{31}N_3O_4$ ,  $\alpha_D$  of II is -23.1°, its formula  $C_{24}H_{31}N_3O_4$ . Ziesel's method shows 1 OMe group in I and two in II, the latter being probably the methylated deriv of the former.

**A I Meyer**  
**The constitution of the alkaloid nymphaeae** F Bureš and I Pzák, Jr *Časopis Českoslov Lékarnictva* 15, 223-6(1933)—The alkaloid, nymphaeae, was extd from the dried roots of *Nymphaea alba* L. To 1 kg of dried roots 10 l of 5% HCl was added and the material

left to stand for 7-8 days after which the ext. was pressed out of the roots and filtered. To the filtrate a satd soln of Ba(OH) $_2$  was added until a ppt had formed. After standing the clear supernatant liquid was poured off and the remaining ppt. washed with water. This procedure was repeated several times, the ppt. then being placed on a filter, washed several times more and finally dried in air. The finely pulverized material was then extd by petr ether in a Soxhlet app. The exts obtained were combined, almost all the petr ether distd off and the thick soln extd with 5%  $H_2SO_4$ . The acid layer was sepd, decolorized with animal charcoal, made alk with dil ammonia and the nymphaeae which had sepd was filtered off, washed with water and dried. Nymphaeae is a very fluffy amorphous substance easily sol in  $CHCl_3$ , benzene, ether and AmOH. Its m p is 76-7° and formula is  $C_{14}H_{19}NO_3$ . Cf *C A* 28, 5460°.

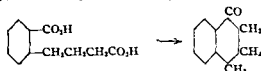
**V D Karpenko**  
**Alkaloids of the Papaver genus I Alkaloids of Papaver armeniacum and Papaver orientale** R Kononova, S Yunussov and A Orekhov *Ber* 68B, 2158-63 (1935).—As many as possible of the native species of *Papaver* in Transcaucasia have been collected for study. Those most thoroughly studied thus far are *P. armeniacum*, *P. orientale* and *P. floribundum*. The first, of which only 8.5 kg was available, was the richest in alkaloids (0.65%) of all the species thus far studied. After a quite tedious purification there was isolated an alkaloid  $C_{17}H_{21}NO_2N$ , for which the name *armepavine* (I) is suggested. It has a phenolic character and contains 1 HO group (Zerevitinov), the other 2 O atoms being present as MeO groups (Viehböck). The N is tertiary and carries a Me group, hence I may be represented by the formula  $C_{17}H_{21}(NMe)(OMe)_2O$ . A preliminary extn on I treated with  $ClCO_2Et$  gave an only product with no basic properties and no optical activity, indicating that I is a tetrahydroisoquinoline deriv.

**As the yield of pure I was only about 0.2%, the plant probably contains considerable amts of other bases, in fact, the presence of nonphenolic bases was observed, but they could not be purified for lack of material. In the case of P. orientale it was especially desired to establish the structure of Gadamir and Klee's isothebaine (C A 5, 2078), but no trace of this base was found in the plant. Instead, the plant, which contained only 0.16% total alkaloids, yielded, along with thebaine, a new phenolic base, erispavine (II),  $C_{17}H_{21}NO_2N$ , with 1 NMe, 1 OMe and 1 OH group; nothing definite can as yet be stated as to the function of the 3rd O atom. From its empirical compn II is also probably a benzyltetrahydroisoquinoline. In the isolation of thebaine and II there also sepd considerable amts of oily and resinous bases whose sepn will be attempted when more material is available. II m 200 1°,  $[\alpha]_D$  -211.8° ( $CHCl_3$ ), easily sol in 10% NaOH, when a 1% soln of the HCl salt is made alk with  $NaHCO_3$ , treated with a drop of alc I and shaken with ether, the ether becomes intensely green and the aq layer pink. HCl salt, m 244-5°, methiodide, m 207.8°, darkens in the light. I, needles with 1  $H_2O$ , m 100°, resolidsifies and again m 148-9°,  $[\alpha]_D$  -118.7°, quite sensitive to light and air, soon turns yellow-brown, is colored bright yellow by concd  $HNO_3$ , HCl salt, m 151-2°, exalate, m 211-12°, methiodide, m 199-200°.**

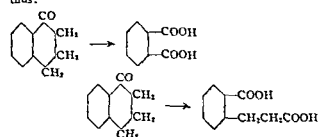
**O-Methylarmepavine-Mel, from I in MeOH refluxed with NaOMe and MeI, m 120-2°, insol in alkalies.**

**C A R**  
**Picrotoxin. XII Degradation of picrotoxin acid,  $C_{23}H_{31}O_7$ , to the dibasic acids  $C_{12}H_{15}O_4$  and  $C_{11}H_{13}O_4$ , P Hermann and Kurt Thilo *Arch Pharm* 273, 433-45 (1935)—Picrotoxin acid (I) yields with  $KMnO_4$  in alk. soln the dibasic acid  $C_{12}H_{15}O_4$  (II), this involving oxidation of the Me group to  $CO_2H$ . It is shown that the  $CO_2H$  group originally present in I is combined with the aromatic nucleus by means of the residue  $CH_2$ . This nucleus carries in addn, a Me as substituent. Dry distn of II causes the elements of  $CO_2$  and  $H_2O$  to split off with formation of the ketone  $C_{11}H_{13}O_4$  (III), which from its nature must be a cyclic ketone, otherwise oxidation with  $HNO_3$  could not yield a dibasic acid of bkt C and H content,  $C_{11}H_{13}O_4$  (IV). The simultaneous formation of the dibasic acid  $C_{11}H_{13}O_4$  (V) involves oxidation of the cyclic structure**

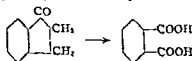
outside the nucleus. Further degradation of IV to the ketone  $C_{10}H_{16}O_2$  leads to ring formation, this compound containing a  $CH_2$  less than the ring of III. This assumption is strengthened by the formation of V through oxidation of IV. The simplest explanation of the constitution of V is that it represents a phthalic acid, since by yielding an anhydride it must be a substituted  $\alpha$ -phthalic acid. It differs from the acid II, the 1st oxidation product of I, by the lesser content of  $C_2H_4$ . It appears therefore that in the Hofmann degradation a substituent of I consists of the residue  $-C_2H_4CO_2H$ . Assuming that the  $CO_2$  group, resulting from oxidation of the Me group and leading to the formation of the dibasic acid II, stands in the  $\alpha$ -position to the residue  $-C_2H_4CO_2H$ , then the rise of a 6-ring ketone with sepn. of  $CO_2$  and  $H_2O$  becomes clear, thus



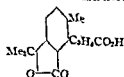
Oxidation of this ketone with  $HNO_3$  attacks the CO group. A considerable portion is oxidized up to the aromatic nucleus, whereby the dibasic acid V results. Simultaneously, there is formed by cleavage of the ring and maintenance of the C and H content the dibasic acid IV, thus.



This acid can in turn by dry distn. be converted through loss of  $CO_2$  and  $H_2O$  into the compd.,



which with  $HNO_3$  yields the dibasic acid V. The residual complex in all these acids has the constitution of a dimethylphthalide, characterized by high stability. The KOH-cleavage carried out on I also substantiates this assumption, because it proceeds with sepn. of  $AcMe$  and formation of the dibasic acid  $C_{10}H_{16}O_4$  (VI), which is to be regarded as a benzenedicarboxylic acid, a compd. still contains the same residues Me and  $C_2H_4CO_2H$  peculiar to I. The expts. have further shown that the  $CO_2H$  group in the residue  $C_2H_4CO_2H$  is in terminal union, and not, as might be concluded from the course of ozonide formation, in medial combination in the chain  $C_2H_4$ . The following constitution for this acid I is therefore suggested



By cleavage of pinolindicarboxylic acid,  $C_{10}H_{16}O_4$ , with aq.  $Ba(OH)_2$ , by the action of  $HCl$  and P in the usual way, I was obtained in a 64% yield. Phenylhydrazones,  $C_{10}H_{16}O_4 \cdot N_2H_4$ , m. 185°. The dibasic acid VI in 138° (Ag salt,  $C_{10}H_{16}O_4Ag_2$ ). The ketone III m. 147°, by 225° (semicarbazone, m. 322° (decompn.)); phenylhydrazones, m. 184°; at 187° (decompn.). The oxidation product of III or the dibasic acid IV m. 213° (decompn.) (Ag salt,  $C_{10}H_{16}O_4Ag_2$ ). The

dibasic acid V m. 293° (slow heating), heated more rapidly the substance m. 209-10°, then solidifies immediately and again m. 293° (decompn.) (Ag salt,  $C_{10}H_{16}O_4Ag_2$ , anhydride,  $C_{10}H_{16}O_2$ , m. 293° (decompn.)). The ketone,  $C_{10}H_{16}O$ , from IV by distn., m. 187° (phenylhydrazones, yellow, m. 210°, semicarbazones, m. 356° (decompn.)). The dibasic acid V m. 293° (decompn.) (Ag salt,  $C_{10}H_{16}O_4Ag_2$ ). W. O. E.

A new substitute for the androsterone group. Adolf Butenandt, Kurt Tscherning and Gunter Hamisch. Ber. 68B, 2097-2102 (1935), cf. C. A. 30, 7541.—Isoandrosterone (prepd. from the catalytic reduction of dehydroandrosterone) in  $PrOH$  and heated 90 min. with Na gave isoandrostanediol (I), m. 164°,  $[a]_D^{25}$  4.2°, diacetate (II), m. 123-4°. II, saponified and then oxidized with  $CrO_3$ , gave androstan-17 $\alpha$ -3-one (III). Testosterone, treated with H and Pt, gave the semicarbazone of III, m. 237-43°. The mother liquor contained II, m. 178°,  $[a]_D^{25}$  32.4° (in EtOH), acetate m. 157°. III oxidized with  $CrO_3$ , gave androstan-3,17-dione, m. 133°,  $[a]_D^{25}$  111° (in EtOH). Julius White.

The bromination of cholestanone and coprostanone.  $\Delta^1$ -Cholestan-3-one. Adolf Butenandt and Alexander Wolff. Ber. 68B, 2091-4 (1935), cf. C. A. 30, 4807.—Cholestanone in  $H_2O$  accont. gave a HBr-HOAc mixt. and treated with Br gave 2-bromocholestanone (I), m. 169-70°. I, suspended in 21%  $Ca(OAc)_2$ -HOAc soln. and heated 5 hrs. at 200° in a bomb, gave  $\Delta^1$ -cholestenone, m. 111-12°,  $[a]_D^{25}$  -31.1° (in EtOH),  $\lambda$  240 m $\mu$ , oxime, m. 146-7°. Coprostanone, similarly brominated, gave 4-bromocoprostanone (II), m. 110-11°. II, heated in  $C_2H_5N$  for 12 hrs., gave cholestenone, m. 79-80°,  $[a]_D^{25}$  88.6°. Julius White.

Catalytic hydrogenation of progesterone. Adolf Butenandt and Gerhard Fleischer. Ber. 68B, 2094-7 (1935), cf. preceding abstr.—Progesterone, treated with Pt and H and subsequently oxidized with  $CrO_3$ , gave alopregnanedione (I), monosemicarbazone, m. 245°. The mother liquor of I contained pregnandione. Pregnen-3-ol-20-one acetate, treated with Pt and H and then oxidized, gave I. Julius White.

Sex hormones and related substances. VI. Breakdown of acetyldehydrocholesterol and acetyldehydrocholesterol to  $\beta$ -3-hydroxynorallocholic acid and 3-hydroxynorallocholic acid. Wilhelm Dirscherl. Z. physiol. Chem. 237, 268-72 (1935), cf. C. A. 30, 4834.—Oxidation of the side chain of  $\Delta^4$  sterols by  $CrO_3$  is a means of detg. the location of the C at which the terminal branching occurs. Thus, dihydrocholesterol yields  $\beta$ -3-hydroxyallocholic acid, while ergosterol and stigmasterol yield  $\beta$ -3-hydroxynorallocholic acid, with chain lengths of 4 and 3 C, resp. The same procedure is now applied to dihydrocholesterol. Oxidation of acetyldehydrocholesterol and subsequent hydrolysis of the Ac yielded  $\beta$ -3-hydroxynorallocholic acid, m. 226°,  $[a]_D^{25}$  32° (Me ester, by  $CH_3N$ , m. 156°), while acetyldehydrocholesterol yielded 3-hydroxynorallocholic acid, 209-10° (Me ester, m. 175-6°),  $[a]_D^{25}$  21.2° (acetate of Me ester, m. 194-5°). The terminal branching in the cinchols therefore occurs on the C fourth removed from the point of attachment. A. W. Drex.

Physiologically active crystalline esters of vitamin A. Sadaaki Hamano. Sci. Papers Inst. Phys. Chem. Research (Tokyo) 28, 69-73 (1935), cf. C. A. 29, 2535.—A soln. of 9 g. of vitamin A concentrate (6000 C. I. O. units) prepd. from *Theragra chalcogramma* liver oil, in 30 cc. anhyd. pyridine was warmed for 30 min. with a soln. of  $\beta$ - $C_{10}H_{17}COCl$  in 30 cc. of benzene in a current of  $CO_2$ . The cooled reaction mixt. was taken up in 300 cc. of Et $_2$ O and freed from pyridine and excess  $\beta$ - $C_{10}H_{17}CO_2H$ . The evap. residue crystd. in cold  $Me_2CO$  at -40° to -50° and yielded spindle shaped or rhombic crystals of vitamin A  $\beta$ -naphthoate (I),  $C_{26}H_{40}O$ , m. 26°, hydrolyzed with 10% KOH to pure vitamin A (II),  $C_{20}H_{32}O$ , reddish-yellow oil, and the K salt of  $\beta$ -naphthoic acid (m. 183°). I has an activity of 7300 C. I. O. units, readily promotes the growth of albino rats and cures xerophthalmia. A 1 in 140,000 mol. soln. in MeOH shows absorption bands at 3505, 3385, 3060, 2920 and 2816, together with a max

at 3225. II also forms an *anthraquinone*  $\beta$ -carboxylate,  $C_{14}H_8O_4$ , m 124°, with an activity of 6000 C. L. O. units. Activity charts and photographs of I and II are reproduced. C. R. Addinall

**Reaction between toluquinone and cinnamaldehyde** under the action of light. A. Angeletti, *Atti accad. sci. Torino, Classe sci. fis. mat.* nat 70, 326-30 (1935).—When a mixt. of cinnamaldehyde and toluquinone in  $C_6H_6$  is exposed to sunlight there are formed cinnamic acid, toluidyhydroquinone and a crust product, m 163°, which was identified as a monocinnamate of toluidyhydroquinone. With the Me group in the 2 position this compd may be either the 1-cinnamate or the 4-cinnamate (I), with I the more probable because of the hindrance that the Me group may exert over the OH in  $\alpha$ -position. No compds corresponding to the 2,5-dihydroxybenzophenone reported by Klinger (C. A. 5, 3458) were found. A. W. Contieri

**Selenium derivatives of salicylic acid.** R. E. Nelson and G. S. Boase, *Proc. Indiana Acad. Sci.* 44, 135-7 (1934).—Bromination of 5,5'-selenodisalicylic acid in  $CHCl_3$  yields 3,5-dibromosalicylic acid, m 223°. Me salicylate (6.5 g) added to 20 g of di-Me 5,5'-selenodisalicylate *Se-dichloride* (I) in the presence of 12 g of anhyd.  $AlCl_3$  in anhyd. ether yields 45% of di-Me 5,5'-selenodisalicylate (II), m 188°. Bromination of II in  $CHCl_3$  yields the *Se-dibromide*, m 143°. Attempted methylation of II with  $Me_2SO$ , gives 5,5'-selenodisalicylic acid, m 272°. I (22.6 g) with 4.9 g of NaCN in 100 cc of  $H_2O$  in the cold for 2 hrs gives 15% of 5,5'-selenodisalicylate *Se-dithionamide*, m 137°. W. J. Peterson

**Absorption spectra of nitrophenylhydrazines** (Macbeth, Price) 3. Infrared absorption spectra of halogen derivs of nuclear hydrocarbons (Lecomte) 3. Fluorescence of alics (Canals, *et al.*) 3. Photochem. decomposition of nitrophenols (Molnar) 3. Primary photochem. reactions VII. Decomposition of isovaleraldehyde and dipropyl ketone (Bamford, Norris) 3. Temps. and heat tones of genotypical transformations of the alk. salts of long-chain fatty acids (Thessen, *et al.*) 2. Electrolytic reduction potential of org. compds (Tachi) 4. Glycerol ethers (Brit. pat. 436,143) 13.

**Aliphatic hydrocarbons.** Studien- und Verwertungsgesellschaft. Fr. 788,286, Oct. 7, 1933. The yield of aliphatic hydrocarbons contg. more than 1 C atom in the mol. by catalytic synthesis from CO and  $H_2$  is increased by transforming the CO and  $H_2$  only incompletely at first, this being effected by using relatively low temps. and (or) insufficient  $H_2$  and (or) high speed of flow. The reaction products are condensed and the remaining gases returned for further treatment.

**Purifying hydrocarbons.** I. G. Farbenind. A.-G. Fr. 785,831, Aug. 21, 1935. Dienes such as methylcyclopentadiene and cyclopentadiene are removed as addn. compds from hydrocarbons, e. g., crude benzene or cracked products, by treatment with  $\beta$ -quinones, e. g.,  $\alpha$ -naphthoquinone. Fr. 785,832. Dienes are removed as addn. compds by means of aliphatic carboxylic compds unsatd. in the  $\alpha$ - and  $\beta$ -positions, e. g., maleic anhydride. The compds are added in fractions so as to isolate the addn. products separately.

**Oxidizing hydrocarbons.** Gutehoffnungshütte Oberhausen A.-G. Fr. 788,533, Oct. 11, 1935. Hydrocarbons of the paraffin series contg. more than 1 C atom in the mol., and  $O_2$  or a gas contg.  $O_2$ , are passed, along with 1% or less of  $NO$ , at 500-800°, over catalysts compnsing elements of the 2nd and 4th group as well as Cr, V, Mo, Al, W, Mn and Co and their oxides and carbonates. Oxidation products such as  $CH_3O$ ,  $AcH$  and  $HCOOH$  are obtained.

**Catalysts for the hydration of olefins.** Geo. F. Horsley and Imperial Chemical Industries Ltd. Brit. 435,769, Sept. 27, 1935. These comprise 1 or 2 mol. proportions of  $Al(PO_4)_3$  associated with about 1 mol. proportion of a metaphosphate of Ca, Cd, Zn or bivalent Cu, Ni, Sn or

Pb. The hydration is preferably conducted in the vapor phase at 250-300° and 100 atm. or over.

**Alcohols from olefins and steam.** The Distillers Co. Ltd. Ger. 622,267, Nov. 23, 1935 (Cl. 12a 5 02). See Brit. 402,613 (C. A. 28, 2723°).

**Amino alcohols.** I. G. Farbenind. A.-G. (Max Bockmuhl, Gustav Ehrhart and Leonhard Stern, inventors) Ger. 619,332, Sept. 28, 1935 (Cl. 12g 32.21). Amino alcs. of the formula  $RCH(OH)CH_2R''$ , where R is a 3,4-ethylenedioxyphenyl residue, which may be substituted in the ethylene group, R' and R'' are H or hydrocarbon groups, and R''' is H or an alkyl group, are prep'd from ketones of the formula  $ROCH_2R'''$  (1) by halogenation followed by reaction with an amine and reduction or (2) by conversion into an isomeric compd followed by reduction.  $\alpha$ -Dihydroxyaryl alkyl ketones may also be used as starting materials, the hydroxy groups being converted into an ethylenedioxy or substituted ethylenedioxy group at a convenient stage in the process. Examples are given in which (1) 3,4-ethylenedioxypropylphenone (I) yields a bromo deriv., b. 180-2°, which by reaction with  $PhCH_2NHMe$  yields 3,4-ethylenedioxyphenyl(methylbenzylamino)propanone, m 71-3°, from which (3,4-ethylenedioxyphenyl(methylamino)propanol-HCl, m. 225-7°, is obtained by catalytic hydrogenation, the benzyl group being split off. (2) I is converted into its isomeric compd, m 142-3°, from which (3,4-ethylenedioxyphenyl)aminopropanol, m. 145° (HCl salt, m. 213°), is obtained by catalytic hydrogenation; (3) 3,4-ethylenedioxyacetophenone  $\rightarrow$  a bromo compd, m. 114-16°,  $\rightarrow$  methylbenzylamino-3,4-ethylenedioxyacetophenone  $\rightarrow$  (3,4-ethylenedioxyphenyl(methylamino)ethanol-HCl, m. 125-6°), (4) 3,4-(3'-hydroxypropylene-1',2')dioxyphe-nyl ethyl ketone (II)  $\rightarrow$  isomeric compd, m. 141-2°,  $\rightarrow$  [3,4-(3'-hydroxypropylene-1',2')dioxyphe-nyl]aminopropanol-HCl, m. 188-90°; (5) 3,4-dihydroxybutylphenone  $\rightarrow$  3,4-ethylenedioxybutylphenone, b. 145°,  $\rightarrow$  isomeric compd, m. 125-6°,  $\rightarrow$  (3,4-ethylenedioxyphenyl)aminobutanol-HCl, m. 180°. The products affect the uterus and are of therapeutic value. II, b. 210°, is prep'd from 3,4-dihydroxypropylphenone and  $\alpha$ , $\beta$ -dibromopropyl alc.

**Aromatic diamino alcohols.** Chemische Fabriken Joachim Wiernik & Co. A.-G. (Heinrich Hornstein, inventor) Ger. 619,756, Oct. 5, 1935 (Cl. 12g 32.21). Addn. to 611,501 (C. A. 29, 6064°). The method of 611,501 for producing diamino alcs. is modified by treating 1-phenyl-2-methylamino-1-propanol with halides or sulfonic acid esters of dialkylamino alcs. other than di-alkylmethanol, or their salts. Thus, ephe-drine in  $C_6H_6$  is heated with 1-diethylamino-3-chloropropane for 10 hrs. A crystn. mass of ephe-drine-HCl seps. The liquid is sep'd from this and boiled. The liquid is again sep'd from the small amt. of crystals pptd. and the  $C_6H_6$  distd. off. The product is 1-phenyl-2-(methyl)diethylaminopropylamino-1-propanol, b. 165-166°. The prep'n of 1-phenyl-2-(dimethylaminobutylmethylamino)-1-propanol is also described.

**Hydrogenation of aliphatic alcohols and esters.** Wilbur A. Lazier (to E. J. du Pont de Nemours & Co.). U. S. 2,027,182, Jan. 7. In a process such as that of satg. the unsatd. alcs., esters and other compds obtained in the high pressure catalytic dehydrogenation of EtOH to form EtOAc, or in converting crotonyl alc. into butanol, the initial material and H are brought into contact with a hydrogenating catalyst at a temp. of 50° or higher and under superatm. pressure. Several examples with details are given.

**Converting alcohols to acids.** Wm. J. Hale. U. S. 2,027,378, Jan. 14. Reaction such as the conversion of EtOH, PrOH and BuOH to produce HIOAc, propionic acid and butyric acid, resp., are effected by burning the vapors of the alc. and water into contact with a dehydrogenation catalyst such as Cu and a partially hydrolyzable salt of an amphoteric base and an org. acid such as basic acetates of Cu, Mn and Al. Various examples are given.

**Substituted aldols.** Kurt Biling (to I. G. Farbenind. A.-G.). U. S. 2,027,856, Jan. 14. A homolog of acetalde-

hyde, e.g., butyraldehyde for the production of butyraldol, is mixed with an aliphatic amine such as diethylamine so that condensation is effected in the presence of the latter. Several examples are given.

**Converting aldehydes to acids.** Wm J Hale U.S. 2,027,577, Jan. 14. In effecting reactions such as production of HOAc from vapors of acetaldehyde and water, a catalyst is used comprising a hydroxide of Cu, Mn or Cr which may be carried on pumice, and a dehydrogenative metal catalyst such as Cu is also employed. Numerous details and examples are given, including the treatment of both aliphatic and aromatic aldehydes.

**Unsaturated aldehydes and ketones.** Herbert P A Groll (to N. A. de Banafoche Petroleum Maatschappij) Brit. 435,494, Sept. 23, 1935. Unsaturated primary or secondary alks having a branched chain are treated with dehydrogenating catalysts at above 230°. Among examples (1) 2-methyl-1-buten-3-ol is converted into methyl isopropenyl ketone by passage over brass spelter in a Cu tube, and (2) 2-methyl-2-buten-4-ol is converted into  $\beta$ , $\beta$ -dimethylacrolein in similar manner. The products may be utilized as resin components alone or with aldehydes, ketones, phenols, anilines, etc., or may be used to introduce alkyl groups into org. compds by condensation or by the use of organometallic compds, or may be oxidized to the corresponding acids and have varied uses in pharmaceutical chemistry. Cf. C. A. 29, 6606.

**Amines.** I. G. Farbenindustrie A.-G. Brit. 435,563, Sept. 23, 1935. Amines are prep'd by causing halogen sulfonic acid esters of aliphatic, cycloaliphatic or mixed aromatic-aliphatic alks with at least 8 C atoms to react with basic-reacting, N-contg. inorg. or org. compds, e.g.,  $\text{NH}_3$ ,  $\text{N}_2\text{H}_4$ ,  $\text{NH}_2\text{OH}$ , aliphatic or cycloaliphatic amines, polypeptides, albuminous substances, tertiary and aromatic amines and substituted amines such as those contg. halogen atoms. Among examples, dodecyl chlorosulfonate, dissolved in  $\text{Et}_2\text{O}$ , is caused to react with liquid  $\text{NH}_3$  at -30° to produce a mixt. of the corresponding primary, secondary and tertiary amines, and the same ester is allowed to flow into a soln. of diethylaniline in spirit at 0-5° to give dodecylmethylaniline.

**Amines.** I. G. Farbenindustrie A.-G. Brit. 436,214, Sept. 30, 1935. Amines are prep'd from aldehydes or ketones by treatment with  $\text{H}_2$  and  $\text{NH}_3$  at raised temp. and pressure in the presence of a catalyst comprising an O-contg. or S-contg. compd. of a heavy metal, which is practically not reduced to metal under the working conditions. Amines are prep'd also from oximes or from compds. of the type of Schiff's bases derived from aldehydes or ketones by condensation with primary amines by treatment with  $\text{H}_2$  at a raised temp. and pressure in the presence of a catalyst as above but in the presence or absence of  $\text{NH}_3$ . Cf. C. A. 29, 69029, 30, 7029.

**Amines; organic acids.** British Celanese Ltd., Horace F. Osley, Edward B. Thomas and Henry Drevfus. Brit. 436,224, Oct. 4, 1935. Aliphatic amines or  $\text{NH}_3$  are recovered from their salts with org. acids by heating the salts to temps. at which the acidic and basic components are set free, removing 1 of the components concurrently with its liberation and separately removing the other component either simultaneously with or after the removal of the 1st component. The salts may be decomposed in aq. soln. or in suspension in other inert liquids.

**Arylamines.** Herbert A. Lubs and John F. Cole (to F. I. du Pont de Nemours & Co.). U.S. 2,023,576, Dec. 31. 1-Arylaminoarylenethianolines are hydrolyzed to simultaneously produce aryl amines having the formulas  $\text{XSRNH}_2$  (I) and  $\text{R'NH}_2$  (II), X being an alkali metal, and the compds. having the formula I are further treated with chloroacetic acid to produce aminoarylthioglycolic acids. E.g., 2,4-dichloroaniline and 2-mercapto-4-chloroaniline are prep'd by hydrolyzing 1-(2,4-dichlorophenyl)-5-chlorobenzenethianol under alk. conditions; and 2-methyl-4-chloroaniline and 2-mercapto-4-chloro-6-methylaniline are similarly obtained from 1-p-chloro-o-tolueno-3-methyl-3-chlorobenzenethianol.

**Secondary aromatic amines.** Werner M. Lauter (to

Wingfoot Corp.). U.S. 2,028,074, Jan. 14. Reaction under substantially anhyd. conditions and in the presence of an alkali metal bisulfate, pyrosulfate or acid phosphate acting as a dehydrating agent is effected between a hydroxy aromatic compd. such as a naphthol, hydroxy phenol or alkyl phenol and aniline, an alkyl phenylamine, naphthylamine, cyclohexylamine, furfurylamine, benzylamine,  $\beta$ -phenetidine and amino biphenyl, an amino phenol, a halo naphthylamine or a halo phenylamine, e.g., phenyl- $\beta$ -naphthylamine is formed from  $\beta$ -naphthol and aniline.

**Amides of carboxylic acids containing sulfur.** Henkel & Cie G. m. b. H. Ger. 619,299, Sept. 26, 1935 (Cl. 12a 16). Amides of the formula  $\text{RCONH}^*\text{R}'$ , where R is a haloacetylated hydrocarbon radical, R' is an aliphatic radical of at least 6 C atoms or a hydroaromatic radical, and R\* is H or a hydrocarbon radical, which may be the same as R', are treated with thiosulfates or with inorg. or org. derivs. of  $\text{H}_2\text{S}$ , e.g., inorg. sulfides or polysulfides, or aliphatic or aromatic mercaptans. The halogen in the group R is thus replaced by a group contg. S. Examples are given in which (1)  $\text{ClCH}_2\text{CONHC}_6\text{H}_5$  (I) and  $\text{Na}_2\text{S}_2\text{O}_3$  yield  $\text{NaSO}_3\text{SCH}_2\text{CONHC}_6\text{H}_5$  (2) I and  $\text{PhSH}$  yield  $\text{PhSCH}_2\text{CONHC}_6\text{H}_5$  in 47-8%, (3) an amide mixt. of the formula  $\text{ClCH}_2\text{CONHC}_6\text{H}_4\text{CH}_3$  (CH<sub>3</sub>)<sub>2</sub>CH, where x is 6, 8, 10 and 12, obtained from the amines derivable from cresol-fat alks., is treated with  $\text{HSCH}_2\text{COONa}$  to yield a mixt. of products of the formula  $\text{NaOOCCH}_2\text{SCH}_2\text{CONHC}_6\text{H}_4\text{CH}_3$  (CH<sub>3</sub>)<sub>2</sub>CH, (4)  $\text{HSCH}_2\text{CH}_2\text{SO}_3\text{Na}$  and the amide mixt. of (3) yield a mixt. of products of the formula  $\text{NaSO}_3\text{CH}_2\text{CH}_2\text{SCH}_2\text{CONHC}_6\text{H}_4\text{CH}_3$  (CH<sub>3</sub>)<sub>2</sub>CH, (5)  $\text{HSCH}_2\text{CH}_2\text{OH}$  (CH<sub>3</sub>)<sub>2</sub>CH and the amide mixt. of (3) yield a mixt. of products of the formula  $\text{SO}_3\text{NaCH}_2\text{CH}_2\text{OH}$  (CH<sub>3</sub>)<sub>2</sub>CH,  $\text{SCH}_2\text{CONHC}_6\text{H}_4\text{CH}_3$  (CH<sub>3</sub>)<sub>2</sub>CH. Numerous other starting materials are specified. The reactions may be effected in boiling alk. aq. or aq. alk. soln. Such of the products as are water-insol. are useful as softening agents, e.g., in the rubber industry. The water-sol. products have saponaceous properties.

**Polynuclear substituted cyclic ketones.** Soc. pour l'ind. chim. & Bille. Brit. 435,553, Sept. 24, 1935. The ketones are prep'd from sterols and bile acids, e.g., said nuclei, or from derivs. or partial degradation products thereof, by oxidizing derivs. of the parent materials in which the OH group or groups have been protected from oxidation by substitution, e.g., the neutral portion of the oxidation product, isolating the required cyclic ketones from the nonvolatile neutral portion, and, optionally, removing the protecting group to produce OH-substituted cyclic ketones. Among examples, (1) dihydrocholesterol chloride is oxidized in glacial AcOH with  $\text{CrO}_3$  and the chloro ketone formed is sep'd from the nonvolatile neutral oxidation product by use of semicarbazide as the ketone reagent, and (2) the semicarbazone of the acetate of the hydroxy ketone resulting from the oxidation of the acetate of  $\alpha$ - $\beta$ -dihydrocholesterol is submitted to a gentle hydrolysis to yield the acetate of the hydroxy ketone.

**Cyclic ketones.** I. G. Farbenindustrie A.-G. Fr. 753,540, Aug. 12, 1935. Lactones of aliphatic carboxylic acids or the corresponding hydroxy acids or unsat'd acids are treated with dehydrogenating catalysts belonging to the group of activated bleaching earths. Thus, a ketone, b, 105-110° (semicarbazone, m. 156-7°), is prep'd from  $\alpha$ -heptyl- $\gamma$ -methylbutyrolactone, one, b, 110-15° (semicarbazone, m. 155-160°) from the  $\alpha$ -heptyl- $\gamma$ -ethyl compd., one, b, 80-85° (semicarbazone, m. 148-9°) from  $\gamma$ -methylbutyrolactone, heptylphenylacetone, b, 100-5° (semicarbazone, m. 155-4°) from  $\gamma$ -heptylbutyrolactone, and 2-butyl-4-cyclopenten-1-one, b, 90-5° (semicarbazone, m. 200-1°) from butyraldehyde acid. The ketones have a jasmine perfume.

**Ketols.** Ivor A. Davies and Imperial Chemical Industries Ltd. Brit. 435,446, Sept. 23, 1935. Ketols are made by causing aldehydes and ketones to react in the presence of a very small amt. of alkali metal hydride, e.g., less than 0.5 cc. of an alk. soln. thereof per l. of reaction mixt. In an example, hydroxyacetone is produced



by adding AcH to Me<sub>2</sub>CO rendered just alk to cresol red by a few drops of a MeOH soln of KOH, the temp. being kept at about 30° and N<sub>2</sub> free from CO<sub>2</sub>, being passed through the reaction vessel

Ethers F I du Pont de Nemours & Co Brit 435,110, Sept 16, 1935 Halogen or H halide is removed from 1 mol of a halogenated dialkyl ether or from 2 mols of the same or different halogenated dialkyl ethers by means of finely divided metals. The reaction, which may be effected in the liquid or gaseous phase, yields open chain or cyclic ethers, e.g., ethylene oxide, ethylene glycol dimethyl ether, dioxane and homologs thereof. The latter are solvents or plasticizers for cellulose derivatives. In examples, monochlorodimethyl ether is heated with Na and dichlorodimethyl ether with finely divided Cu.

Halo ethers I G Farbenind A-G Fr 788,341, Oct 8, 1935 Alkoxyalkyl halides of the formula RHC(X)OR' (R is H, alkyl, aryl or aralkyl, R' is alkyl, aryl or aralkyl and X is halogen) are caused to react with compounds containing at least 1 olefinic double bond preferably in the presence of a catalyst and, if necessary, solvents. Examples are given of the prepn of 1-phenyl-1-methoxy-5-chloro-3-pentene, b<sub>10</sub> 93° (from liquefied butadiene and  $\alpha$ -methoxybenzyl chloride, with HgCl<sub>2</sub> as catalyst), 1-methoxy-5-chloro-3-pentene, b<sub>118</sub> 118°, 1-phenyl-1-methoxy-5-bromo-3-pentene, b<sub>118</sub> 92°, 1-methoxybenzyl-2-chlorocyclohexane, b<sub>118</sub> 94.5°,  $\alpha$ -methoxybenzyl- (b<sub>118</sub> 112°) and  $\alpha$ -methoxymethylchlorocyclohexane, b<sub>118</sub> 81°, 1-methoxy-2,3-dimethyl-3- (b<sub>118</sub> 45-6°) and 1-phenyl-1-methoxy-2,3-dimethyl-3-chlorobutane, b<sub>118</sub> 133-5°.

Esters of aliphatic alcohols Walter F Lawson (to Canadian Industries Ltd.) Can 3,345,08, Oct 8, 1935 A polycarboxylic acid is heated with a mixt. of alcs. b above 160° and an inert org. solvent. The alc. mixt. is obtained by the catalytic hydrogenation of carbon oxides at an elevated temp. The mixed acid esters which are formed are recovered and heated in a mixt. comprising the mixed acid esters, butyl alc. and an inert org. solvent.

Esters of methacrylic acid E Emmet Reid (to E. I. du Pont de Nemours & Co.) U.S. 2,028,012, Jan 14, 1935 Phosgene or other carbonyl halide is caused to react with C<sub>3</sub>H<sub>7</sub> (suitably in an initially formed quantity of chloroisobutyl chloride contg. AlCl<sub>3</sub>) to produce  $\beta$ -chloroisobutyl chloride and the latter is treated with an alc. such as Me, Et or Pr alc., ethylene glycol, glycerol or an aromatic alc., cyclic alc. or ether alc. to form a  $\beta$ -chloroisobutyrate which is then dehydrohalogenated to the ester of methacrylic acid. Various other condensation catalysts can be used instead of AlCl<sub>3</sub>.

Lactone esters Henck & Co. A-G Brit 435,605, Sept 24, 1935 Condensation products are obtained by causing esters of trihalo fatty acids, substituted in the  $\alpha$ -position, to react with esters of levulinic acid in the presence of metals, e.g., Zn. Among examples, (1) Et tribromovalerate is condensed with Et levulinate (I) to give the  $\gamma$ -lactone of  $\alpha$ -allyl- $\beta$ -methyl- $\beta$ -hydroxy adipic ester, and (2) the  $\gamma$ -lactone of  $\alpha$ -hexenyl- $\beta$ -methyl- $\beta$ -hydroxyadipic ester is obtained from Et tribromocaprylate and I.

Esters of etherified glycolic acids Wolfgang Lübbert and Ernst L. Müller (to Henkel & Cie G.m.b.H.) U.S. 2,027,991, Jan 14, 1935 Esters having the general formula R<sub>1</sub>XCH<sub>2</sub>COOR<sub>2</sub> in which X signifies O or S, R<sub>1</sub> represents a hydroxyethyl or phenyl or carboxymethyl radical, and R<sub>2</sub> represents an alkyl radical having at least 6 C atoms, which possess some emulsifying properties and may be used in the perfume industry or as softening agents are obtained when hydroxy or mercapto fatty acids which are alkylated, cycloalkylated or arylated at the O or S, or their derivs or substitution products, are esterified with aliphatic or hydroaromatic compounds which contain at least one hydroxyl or mercapto group and at least 6 C atoms; e.g., dodecanol, octadecanol, hexadecanol, tetradecanol or their mixts are esterified with phenoxyacetyl chloride, forming esters of wax-like character. Numerous examples with details of procedure are given.

Tetrasulfuric acid ester of leuco-1,2,2',1'-anthraquinonamine Georg Röscher, Josef Haller and Fritz

1 Helwert (to Durand & Huguenin A.-G.) U.S. 2,022,218, Nov. 20 The leuco sulfuric acid ester of anthraquinone- $\beta$ -sulfamic acid, or a salt of this ester, in a neutral or alk. aq. medium, is reacted upon with PhO<sub>2</sub> or a metal ferric cyanide, preferably an alkali metal or alk. earth metal ferric cyanide (various examples and details of procedure being given).

Polyuclear organic compounds I. G. Farbenindustrie A.-G. Brit. 435,254, Sept. 9, 1935 Unitary org. compounds containing 4 or more nuclei are recovered from the destructive hydrogenation products of bituminous, resinous or ligneous materials other than mineral coal high-temp. tar, and particularly from the high-boiling fractions of these products. The methods of recovery may be those ordinarily used in the recovery of polyuclear compounds from mineral coal high-temp. tar, e.g., cooling, pptn., crystn., selective solution and distn., each of which may be fractional, combinations of such methods may also be used, as well as chem. methods, e.g., fusion with KOH, NaNH<sub>2</sub>, or alkali metals, or sulfonation. Alternatively, use may be made of methods involving treatment of the destructive hydrogenation products by (1) dehydrogenation, (2) distn. with the addn. of metals or condensing or polymerizing agents, (3) isomerization, (4) removal of asphalts, pitch-forming constituents and paraffin, or (5) combinations of such treatments. Some of the processes may be applied also to the recovery of polyuclear compounds containing 4 or more nuclei, with or without side chains, from the destructive hydrogenation products of mineral coal high-temp. tar and also to the recovery of polyuclear compounds containing less than 4 nuclei, with or without side chains, from the destructive hydrogenation products of bituminous, resinous and ligneous materials generally. Numerous specific methods are described in detail. 26 examples are given. Fluoranthene, pyrene, chrysene, retene, piceene, phenanthrene, fluorene, etc., are recovered.

Stabilizing halogen compounds Chemische Fabrik von Heyden A.-G. Brit. 436,054, Oct. 3, 1935 See Fr 784,238 (C A 30, 106°). The org. compound is either dissolved or incorporated in the Cl substitution product, which must not be attacked by Cl at ordinary temp. in the absence of catalysts and light.

Phenylated compounds Walther Diltthey (to I. G. Farbenindustrie A.-G.) Brit. 435,708, Sept. 26, 1935 See Fr 770,790 (C A 29, 939°).

Polymerized vinyl compounds I. G. Farbenindustrie A.-G. Fr. 786,984, Sept. 14, 1935 Compds. which at the most are liable to swell in aromatic hydrocarbons but do not dissolve are made by submitting a monomeric vinyl compound containing besides a vinyl group at least one C atom not forming part of an aromatic ring, to a polymerization in the presence of a compound of the formula XRY (X and Y are radicals contg. a vinyl group, R is a hydrocarbon radical of the C<sub>6</sub>H<sub>5</sub> series). Such compounds include divinylbenzene, methyldivinylbenzene and vinylisopropenylbenzene. Examples are given of the prepn. of compounds insol in C<sub>6</sub>H<sub>6</sub> from vinyl acetate, methyl and butylacrylic ester and indene.

Thiazole compounds Earl E. Beard and Wm. L. Rintelman (to E. I. du Pont de Nemours & Co.) U.S. 2,028,118, Jan 14, 1935 Thiazole derivs of the naphthalene and the anthraquinone series may be prepd. in relatively pure form and in high yields by the reaction of benzaldehyde compounds of the naphthalene or anthraquinone series with S in the presence of at least stoichiometrical amounts of Br, which is present in the reaction in combined form, added in the form of free Br or as S bromides. The amino-naphthalene or aminoanthraquinone compound is first caused to react with benzaldehyde or other aromatic aldehyde such as those of the naphthalene or anthraquinone series to form the azomethine compound. This reaction may be carried out in trichlorobenzene or other inert solvent, and the product so formed may be caused to react without isolation with S monobromide to give the corresponding thiazole. Thus the thiazole is obtained in a very pure form, and may be readily isolated by filtering directly from the reaction mass. Several examples with details of procedure are given.

Addition compounds of biacetyl and glycerol. Vanillin-  
Fabrik G. m. b. H. Ger. 619,628, Oct. 11, 1935 (Cl.  
12.5 09). Biacetyl and glycerol are allowed to react on  
each other for a long time to form compds. of cryst. form  
and readily decomposable into the constituents. The  
solid addn. products are removed by pressing, washing or  
by centrifuge. An example is given. The product is  
used in the margarine industry.

Solid diazo compositions I. G. Farbenind. A-G  
(Karl Schützpatz, inventor) Ger. 622,306, Nov. 25,  
1935 (Cl. 8m 13). The acid diazonium sulfate obtainable  
from *m*-nitroaniline is pptd. from aq. soln. by cooling and  
(or) salting out, and is mixed directly, without inter-  
mediate drying, with a mild alk. substance, e. g., soda,  
borax, or Ca(OH)<sub>2</sub>, in an amt. sufficient to neutralize the  
compd. and any free H<sub>2</sub>SO<sub>4</sub> adhering thereto. Solid  
diluent may be added. Sp. compns. are described.

Solid inorganic acid diazonium salts from halo-*o*-  
aminodiphenyl ethers. Karl Schützpatz (to General  
Aniline Works) U. S. 2,027,066, Jan. 7. Inorg. acid  
diazonium salts of halo-*o*-aminodiphenyl ethers are caused  
to sep. from their aq. solns. in solid form by adding an  
inorg. acid to the soln. Various examples are given of the  
production of products such as the solid diazonium chloro-  
ride of 4-amino-2,2',5'-trichlorodiphenyl ether and the  
solid diazonium sulfate from 4-amino-4'-chlorodiphenyl  
ether, etc. Cf. C. A. 30, 110<sup>6</sup>.

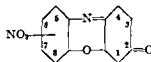
Diazo preparations. Soc. pour l'ind. chim. à Bâle.  
Brit. 434,158, Aug. 27, 1935. Diazo preps. are made by  
salting out from aq. soln. mineral acid salts of diazo  
compds. of the formula 4-acyl-NH-5-X-2-Y-C<sub>6</sub>H<sub>3</sub>N<sub>2</sub>OH  
in which X and Y are halo, alkyl or alkoxy and the acyl  
residue contains an aromatic nucleus. In examples, the  
diazo hydrochlorides of the following amines are prepd.:  
4-phenoxyacetyl- and -(1'-methylphenoxyacetyl)amino-  
and 4-(1'-, 2'- or 3'-chlorophenoxyacetyl)amino-2,5-  
dimethoxy- or -diethoxyaniline, 1-amino-2-methoxy-5-  
methyl-, 1-amino-2,5-dimethoxy- or -diethoxy-, 1-amino-  
2-methoxy-5-chloro- and 1-amino-2-chloro-5-methoxy-4-  
benzoylamino benzene, the diazo sulfate of 1-amino-2-  
methoxy-5-chloro-4-benzoylamino benzene is also prepd.  
Cf. C. A. 29, 4021<sup>1</sup>.

Halogenated products of phenyl phenylphenyl ether  
Wesley C. Stoesser (to Dow Chemical Co.). U. S. 2,028,  
081, Jan. 14. When a phenyl phenylphenyl ether, or a  
mixture of isomeric phenyl phenylphenyl ethers, is chlorinated  
or brominated, products are obtained which, according  
to their increasing halogen content, vary in phys.  
characteristics at room temp. from cryst. materials  
through the stage of viscous liquids to that of solid,  
noncryst. resins. Even though only a single phenyl  
phenylphenyl ether be halogenated, the product usually  
consists of an intimate mixture of isomeric and other closely  
related compds. Although individual compds. can in  
some instances be sep'd. from such mixts., the mixts.  
themselves are useful com. products. All of such mixts.  
are practically nonflammable, are substantially stable  
against decompn. at high temps., e. g., 300-350°, possess  
high dielec. consts. as compared with oil, have low elec.  
power factors comparable with those of commonly used  
condenser oils, and are capable of withstanding excep-  
tionally high elec. potentials without breakdown. The liquid  
products are suited to use as heat storage and transfer  
agents. The resinous products can be used in plastics,  
varnishes, etc., e. g., when the final products are to be  
employed for elec. insulation purposes, e. g., as varnish  
films over elec. wires. Numerous examples with details  
of procedure are given.

Reaction products of substituted methyleneanthrones  
Trichlar, Ger. 619,246, Sept. 26, 1935 (Cl. 12a. 10).  
Methyleneanthrone derivs. substituted in the CH<sub>2</sub> group  
are heated with compds. contg. reactive double bonds in  
the presence of a mild oxidizing agent. Benzanthrone  
derivs., useful as intermediates for dyes, are obtained.  
Examples are given in which (1) benzylideneanthrone,  
maleic anhydride and PhNO<sub>2</sub> yield a product, m. 208°,  
probably *Bz*-3-phenylbenzanthrone-*Bz*-1,2-dicarboxylic  
anhydride, (2) 2'-anthraquinonyl-*meso*-methylenean-

throne (I), maleic anhydride and PhNO<sub>2</sub> yield a product, m.  
316-17°, probably *Bz*-3-(2'-anthraquinonyl)benzanthrone-  
*Bz*-1,2-dicarboxylic anhydride, (3) benzylideneanthrone,  
fumaric acid and *p*-nitrotoluene yield *Bz*-3-phenylbenz-  
anthrone-*Bz*-2-carboxylic acid I, m. 279-80°, is prep'd.  
from *o*-dichloro-2-methylanthraquinone and anthrone in  
alc. soln. in the presence of piperidine.

Condensation products of the oxazine series. Georg Kai-  
scher and Werner Zerweck (to General Aniline Works).  
U. S. 2,020,651, Nov. 12. Oxazinone compds. of the general  
formula



in which both nuclei may be linked with a benzo nucleus,  
both nuclei may contain halogen atoms, which products  
are intensely colored, are obtained by the condensation of  
*o*-amino phenols of the benzene or naphthalene series contg.  
a nitro group, with quinones and halogenated quinones of  
the benzene or naphthalene series. 1,4-Quinones, which  
may be substituted by halogen in the 2- and 5-positions,  
may be caused to react with either one or two mol.  
proportions of nitro-*o*-aminophenols. Numerous examples  
are given and nitro oxazines thus obtainable in a very good  
yield are intensely colored compds. which may be used as  
pigment dyes, in part they are intermediates for the pro-  
duction of further conversion products, particularly of  
sulfur dyes. They can be transformed by usual methods  
into the corresponding amino compds. and their *N*-alkyl-,  
-aryl-, -aralkyl and -acyl derivs. Their corresponding  
diazo compds. are capable of the well-known reactions of  
this type, so that the fundamental unsubstituted oxazi-  
ones and otherwise substituted ethers are obtainable.

Aromatic hydroxy derivatives. I. G. Farbenind. A-G.  
Fr. 788,261, Oct. 7, 1935. Aromatic hydrocarbons and  
their derivs. free from O, e. g., C<sub>6</sub>H<sub>6</sub>, PhCl and toluene,  
are converted to OH derivs. by heating them with O or  
gases contg. O, in the absence of oxidation catalysts, to a  
temp. higher than the upper explosion limit of the mixt.  
used, e. g., 650-750°.

Mono-*N*-alkanol derivatives of aromatic diamines and  
polyamines. Erich Lehmann (to General Aniline Works).  
U. S. 2,022,245, Nov. 26. Mono-*N*-alkanol derivs. of  
aromatic di- or polyamines are made by condensation of  
equimol. proportions of the amine and a halo alkanol such  
as  $\alpha$ -monochlorohydrin or  $\beta$ -monochlorohydrin in the  
presence of a suitable solvent such as MeOH and an acid-  
binding agent such as KOH. Details are given of the  
production of *N*-( $\beta$ , $\gamma$ -dihydroxypropyl)-*p*-phenylenedi-  
amine, *N*-( $\beta$ , $\gamma$ -dihydroxypropyl)-chloro-*m*-phenyl-  
enediamine, *N*-( $\beta$ -hydroxyethyl-*m*-tolylethylenediamine and  
*N*-( $\beta$ , $\gamma$ -dihydroxypropyl)-1,2,4-triazinobenzene and, as  
being new compds., claim is made to all compds. of the  
general formula X-m-H<sub>2</sub>CN<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>C(OH)(OH)CH<sub>2</sub>OH,  
where X means H, halogen, alkyl, *o*-alkyl, NH<sub>2</sub>, NO<sub>2</sub>,  
of basic character, easily sol. in water and alcs., difficultly  
sol. in ether and insol. in aliphatic hydrocarbons, and  
forming salts with acids which are easily sol. in water.  
These compds. may be used as dye intermediates, etc.

Arsenobenzene derivatives. I. G. Farbenind. A-G.  
Fr. 787,025, Sept. 16, 1935. Monosulfoxylates of arseno-  
benzenes of the formula ZO<sub>2</sub>SH<sub>2</sub>CN(R<sup>1</sup>R<sup>2</sup>)ASR<sup>3</sup>NX<sup>4</sup> (R  
and R<sup>1</sup> are benzene rings, R<sup>2</sup> is alkyl which may contain  
at least 1 OH group, X and Y are alkyl which contain  
at least 1 OH group and Z is an alkali metal) by introduc-  
ing 2 hydroxyalkyl groups by transformation with alkylene  
oxide into 1 of the NH<sub>2</sub> groups of a diaminoarsenobenzene  
which may also contain other substituents in the C<sub>6</sub>H<sub>4</sub>  
rings, and a hydroxyalkyl or alkyl group and the formalde-  
hydesulfoxylate radical into the other NH<sub>2</sub> group. They  
may also be obtained by transforming aminobenzenes-  
arsonic acids to arsenobenzenes and introducing the other  
radicals at any stage of the process. Examples are given  
of the prep'n. of the sulfoxylates of 3-hydroxy-4-[bis-  
(dihydroxypropyl)amino]-3'-methylamino-, 3-[bis-

(dihydroxypropyl)amino]-4-hydroxy-3'-dihydroxypropylamino-, 3-hydroxy-4-[bis(hydroxyethyl)amino]-3'-methylamino- and 3-[(dihydroxypropyl)-(hydroxyethyl)amino]-4-hydroxy-3'-methylamino-4-hydroxyarsenobenzene and also the prepn of 3-hydroxy-4-[bis(hydroxyethyl)amino]benzene-, 3-hydroxy-4-[bis(hydroxyethyl)amino]benzene- and 3-[(dihydroxypropyl)-(hydroxyethyl)amino]-4-hydroxybenzene-arsonic acid. Cf. C. A. 29, 5241.

**Naphthalene derivatives.** I. G. Farbernd A-G (Karl Köberle and Kuno Maurach, inventors). Ger. 622,304, Nov. 25, 1931 (Cl. 12q 7). *o*-Amino carboxylic acids of naphthalene (I) are prep'd by heating *o*-nitromethyl derivs of I with alkali hydroxides or equivalent reagents, e. g., alkali alcoholates. Thus, 1-nitro-2-methyl-I, boiled for 12 hrs with aq. KOH, yields 1-amino-1,2-carboxylic acid, m. 198-9°. Other examples are given, the products obtained including 3-, 6- and 7-methyl-1-amino-1,2-carboxylic acids.

**4-Aminodiphenylamine derivatives.** Arthur Zutscher and Wilhelm Seidenfaden (to General Aniline Works). U. S. 2,028,373, Jan. 21. See Ger. 592,201 (C. A. 28, 3419).

**Pyrazolone derivatives.** I. G. Farbernd A-G (Herbert Kracker and Richard Schmid, inventors). Ger. 622,113, Nov. 19, 1935 (Cl. 12p 8.01). 1,3-Diaryl-5-pyrazolones, *contg.* at least one COOH or SO<sub>3</sub>H group in at least one of the aryl radicals, are prep'd by forming hydrazones from correspondingly substituted aryl acetic esters and aryl hydrazines, and subjecting the hydrazones to ring closure with elimination of an alk. The hydrazone formation is effected in an aq., preferably alk., medium. The ring closure is effected in an aq., preferably alk., medium, and may be effected without intermediate isolation of the hydrazone. In typical examples, (1) Ph-COCH<sub>2</sub>COOEt (I) and 2-sulfo-II, where II is PhNHNH<sub>2</sub>, yield 1-(2'-sulphenyl)-3-phenyl-5-pyrazolone, (2) 4-carboxy I and II yield 1-phenyl-3-(4'-carboxyphenyl)-5-pyrazolone. Pyrazolones have been prep'd also from the following components: (a) I and 3-sulfo II, 4-sulfo-II, 2-chloro-4-sulfo-II, 2-chloro-5-sulfo-II, 3,4-dichloro-6-sulfo-II, 2-hydroxy-3-carboxy-5-sulfo-II, 2-methyl-4-sulfo-II, 4-carboxy-II, 3-methyl-4-chloro-6-sulfo-II, 2,5-dichloro-4-sulfo II, 4'-hydroxy-3'-carboxy-4-sulfo-1,1'-diphenyl sulfone-2-hydrazine, 1-sulfo-2 naphthylhydrazine, 4-nitro-2-sulfo-II, 2-nitro-4-sulfo-II, 2,5-disulfo-II, and 2-methyl-4,5-disulfo II, (b) 4-carboxy-I and 2-chloro-5-sulfo II, 4-sulfo-II, 2-sulfo-II, 3-carboxy-II, 2-hydroxy-3-carboxy-5-sulfo-II, 4-nitro-II, and 1-sulfo-2-naphthylhydrazine, (c) 4-chloro I and 3-sulfo-II, (d) 3-nitro-I and 2-sulfo-II; (e) 4-methyl I and 2-chloro-5-sulfo II, (f) 4-methoxy-I and 3-carboxy-3-hydroxy-5-sulfo-II, (g) 2,4-dichloro-I and 4-sulfo-II, (h) 4-phenyl-I and 3-sulfo-II, (i) 3-chloro-I and 4-nitro-2-sulfo II, (j) 4-nitro I and 2-nitro-4-sulfo II; (k) *o*-naphthylacetic ester and 1-sulfo-2 naphthylhydrazine, (l) *o*-naphthylacetic ester and 3,4-dichloro-6-sulfo-II.

**N-Substituted amino phenols.** Miles A. Dahlen (to E. I. du Pont de Nemours & Co.). U. S. 2,027,902, Jan. 14. In producing compds such as *N*-benzyl-*p*-aminophenol, a nitrophenol such as *p*-nitrophenol is subjected to reduction, as by catalytic hydrogenation, to form the corresponding aminophenol in the presence of an inert solvent such as water, and the resulting soln. is caused to react with an aldehyde such as BzH. Numerous details and examples of similar reactions are given.

**Separating mixtures of pentanones.** N. V. de Bataafsche Petroleum Maatschappij. Ger. 622,122, Nov. 21, 1935 (Cl. 12o 10). This corresponds to Brit. 417,101 (C. A. 29, 1100?), but gives addnl. examples.

**Carbazoles.** I. G. Farberndustrie A-G. Brit. 436,110, Oct. 4, 1935. Compds contg. a carbazole radical are prep'd by heating in the absence of H a mixt. of a hydrogenated compd. contg. a carbazole radical and, as substances absorbing H, a phenol, aldehyde, C. oxide or unsat'd aliphatic compd., with the addn. of a hydrogenation catalyst. The hydrogenated carbazole derivs. are completely dehydrogenated in the heterocyclic ring system

Among examples, tetrahydrocarbazole and PhOH are heated in an autoclave at 200-230° in presence of a Ni-bleaching earth catalyst.

**Thiazoles substituted by primary alkylamino groups.** Treat B. Johnson (to Winthrop Chemical Co.) U. S. 2,020,650, Nov. 12. Thiazole compds of the general formula R<sup>1</sup>-[RC(CX)S(CR<sup>2</sup>)N], where R stands for an

aliphatic radical which may be substituted by a Ph radical, and where X represents either H or alkyl, or benzyl, and where R<sup>1</sup> stands for phenyl, benzyl, lower-alkyl-substituted phenyl, hydroxyphenyl, lower-alkoxyphenyl, alkyl, lower-hydroxy-alkyl or lower-alkoxy-alkyl, m stands for one of the nos. 1 and 2, and R<sup>2</sup> stands for an alkyl group which contains an NH<sub>2</sub> group, said compds generally being well crystallized in the form of their hydrochloric acid salts, are claimed as a new class of compds and details of procedure are given for the production of 2-phenyl-4-thiazolethylamine (HCl salt, m. 91-2°),  $\beta$ , $\beta$ '-bis(2-phenyl-4-thiazyl)isopropylamine (HCl salt, m. 235-8°), 2-*p*-hydroxyphenyl-4-thiazolethylamine, and 2-(3,4-dimethoxyphenyl)-4-thiazolethylamine (HCl salt, m. 225-7°).

**Benzisothiazolones, etc.** Norbert Steiger and Fritz Schulte (to General Aniline Works). U. S. 2,020,622, Nov. 12. A process for producing benzisothiazolones

corresponding to the general formula R.CO.NH.S, where R stands for a phenylene radical substituted by halogen, alkyl or an alkoxy group, comprises acting on substituted phenyl cyanosulfonyl chlorides of the general formula R(CN)SO-Cl, where R has the aforesaid signification, in a nonoxidizing acid medium at temps. below about 60° with an amt. of a metal reducing agent corresponding to about four reactive H atoms, care being taken that the reaction is continued only to a point at which a test just fails to show the known mercaptan reaction. Details are given of the production of 4-ethyl-6-chloro-3-benzisothiazolone, m. 220°, and 6-ethoxy-3-benzisothiazolone, m. 225°, and a general description is given of the manual of a 3-naphthylisothiazolone, m. about 205°.

**Polysulfones.** I. G. Farbernd A-G (Erich Fischer, Emil Mahler and August Modersohn, inventors). Ger. 622,494, Nov. 29, 1935 (Cl. 12o 23 (3)). Mono- or poly-nitro polysulfones are prep'd by the action of aromatic, hydroaromatic or aliphatic sulfonic acids or their salts on aromatic halonitro or halopoly-nitro sulfones or on aromatic nitro sulfones contg. a halogenated side chain. Mono- or poly-amino sulfones are obtainable by reducing the products. Thus, 4-chloro-3-nitro-I (I is PhSO<sub>2</sub>Ph), boiled in alc. with MeC<sub>4</sub>H<sub>9</sub>SO<sub>3</sub>Na, yields 3-nitro-4-*p*-toluenesulfonyl I, m. 183°, from which 3-amino-4-*p*-toluenesulfonyl-I, m. 185°, is obtainable by reduction with Fe and HCl. Examples are given also of the prep'n of 3-nitro-4-benzenesulfonyl-I, m. 162-3°, 3-nitro-4-*p*-methoxybenzenesulfonyl I, m. 199.5°, and the corresponding amine, m. 189-90°, 3-nitro-4-methanesulfonyl-I, m. 210-11°, and the corresponding amine, m. 149-50°, 3-nitro-4-cyclohexanesulfonyl-I, m. 168°, 3,3'-dinitro-4,4'-bis(*p*-toluenesulfonyl)-I, m. 310-11°, 3,3'-dinitro-4,4'-bis(benzenesulfonyl)-I, m. 313-13°, 3,3'-dinitro-4,4'-bis(methanesulfonyl)-I, m. above 327°, 3-nitro-6-benzenesulfonyl I, m. 208-9°, 3-nitro-6-methanesulfonyl-I, m. 211-2°, and the corresponding amine, m. 222°, 3-nitro-4-(*p*-hydroxy-*m*-carboxybenzenesulfonyl) I, m. 270-75°, 3-nitro-3'-sulfo-4-benzenesulfonyl I, 1-nitro-2,5-bis(methylsulfonyl)benzene, m. 209-10°, and the corresponding amine, m. 160°, and 2,4-(NO<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>SO<sub>2</sub>CH<sub>2</sub>SO<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>(-)-, m. 184-5°.

**Alkyl chlorides and bromides.** Edward H. Strange and Thomas Kane. Brit. 435,858, Oct. 1, 1935; Fr. 787,340, Sept. 20, 1935. Halogen acids are caused to react with olefins in presence of an adsorbent "hydrous metal oxide gel," i. e., a partially or nearly dehydrated mass obtained from a hydrogel or gelatinous ppt.

**Reaction of alkali metals with aromatic hydrocarbons.** Norman D. Scott (to E. I. du Pont de Nemours & Co.).

U. S. 2,027,000, Jan. 7. Reactions such as those of Na with biphenyl, phenanthrene, naphthalene and anthracene are effected in a reaction medium contg. an ether such as dimethyl ether, methyl ethyl ether, methyl propyl ether, methyl iso-Pr ether, methyllal, glycol dimethyl ether, glycol formal, dioxane, glycerol trimethyl ether, dimethyl-pentaerythritol or glycerol formal methyl ether. By adding CO<sub>2</sub> to the reaction product, carboxylic acids, such as dihydrophenanthrenedicarboxylic acid, are obtained. Cf. C. A. 30, 734<sup>1</sup>

Organic disulfides. Luther B. Turner (to Standard Oil Development Co.) U. S. 2,028,303, Jan. 21. Mercaptans, vaporizable without decompn., are subjected in the vapor phase to a limited oxidation (suitably with air and activated charcoal at 225°) and a reaction product contg. corresponding disulfides is withdrawn.

Calcium formate. Chem. Fab. Kalk G. m. b. H. and Hermann Oehrne. Ger. 622,149, Nov. 21, 1935 (Cl. 12o 11). A suspension of Ca(OH)<sub>2</sub> in a satd soln. of (HCOO)<sub>2</sub>Ca is treated with CO at a raised temp. and pressure, e. g., 160–200° and 5–25 atm.

Acid calcium citrate. Alexander H. Bennett. U. S. 2,027,204, Jan. 7, Brit. 435,586, Sept. 24, 1935. Successive portions of lime or CaCO<sub>3</sub> are added to a soln. of citric acid and pptn. of acid citrate is effected after each addn., a total addn. being made of about 2/3 the amt. of lime or CaCO<sub>3</sub> which would be required for complete neutralization of the acid in the soln.

Basic titanic oxalate. Sydney F. W. Crundall (to Peter Spence & Sons, Ltd.) U. S. 2,027,812, Jan. 14. An inorg. acid soln. of Ti contg. about 0.6 mols. of acid, expressed as SO<sub>3</sub>, for each mol. of TiO<sub>2</sub> is treated with approx. 0.2 mols. of oxalic acid for each mol. of TiO<sub>2</sub> and the soln. is dild. to ppt. basic titanic oxalate (the soln. being maintained at a temp. below 50°).

Tertiary nitriles. I. G. Farbenind. A.-G. Ger. 622,357, Nov. 26, 1935 (Cl. 12o 11). Addn. to 616,876 (C. A. 30, 1111<sup>1</sup>). The Na compds. of secondary nitriles, used as intermediates in the process of Ger. 616,876, are made by treating the nitriles with a Na-alkyl compd. which has been prepd. by treating an alkyl halide with Na in the presence of an inert solvent. The Na-alkyl compd. need not be isolated.

Hydrogenated heterocyclic acids. E. I. du Pont de Nemours & Co. Brit. 435,461, Sept. 23, 1935. Alkali salts of mononuclear heterocyclic carboxylic acids are hydrogenated by treatment with H<sub>2</sub>, e. g., at 100–250°, and preferably under pressure, in presence of a Ni catalyst in which preferably at least part of the Ni is free. The free hydrogenated acid is obtained from its salt by treating with a mineral acid. In examples, (1) aq. pyromucic acid is neutralized with NaOH and hydrogenated at 110–120° and 2000 lb. pressure with a Ni-kieselguhr catalyst, and (2) aq. Na nicotinate is hydrogenated at 190° and 120–170 atm. with the same catalyst.

Amino acids. Gesellschaft für Kohlenteknik. Brit. 435,839, Sept. 30, 1935. The sepn. of amino acids from their mixts. with (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> is effected by (1) saltg. an aq. mother lye contg. them with NH<sub>3</sub>, sepg. the pptd. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and removing the free NH<sub>3</sub>, adding a mixt. of amino acid and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> to the filtrate, sepg. the pptd. amino acid and returning the mother lye to the process, or (2) introducing into a mother lye contg. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and amino acid a soln. of the bisulfates of amino acid and NH<sub>3</sub>, such as is obtained in the hydrolysis of an amino nitrile, then saltg. with NH<sub>3</sub>, removing pptd. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, distg. off free NH<sub>3</sub>, and the added H<sub>2</sub>O, sepg. the pptd. amino acid and returning the mother lye to the process. Examples are given.

Acrylic acid. Rowland Hill (to Imperial Chemical Industries Ltd.) U. S. 2,026,894, Jan. 7. A mixt. contg. an antioxidant, such as Cu or pyrogallol, and ethylene cyanohydrin is treated with H<sub>2</sub>SO<sub>4</sub>, while stirring and cooling, and water is added to the resulting mixt. and it is heated until reaction is initiated; the input of heat is stopped until the vigorous evolution of exothermic heat has subsided, and the reaction mixt. is subsequently heated until the reaction is substantially complete.

Glycolic acid. Ernest F. Grether and Russell B. Du Vall (to Dow Chemical Co.). U. S. 2,028,064, Jan. 14. Chloroacetic acid is hydrolyzed by heating it with an aq. soln. contg. approx. twice its chem. equiv. of an alkali metal base such as Na<sub>2</sub>CO<sub>3</sub> and the hydrolysis mixt. is treated at a temp. above 70° (suitably about the b. p.) with a water-sol. Ca salt such as CaCl<sub>2</sub> in an amt. not greater than the chem. equiv. of the chloroacetic acid hydrolyzed, and the Ca glycolate formed is crystd. from the mixt. while maintaining it at a temp. above 70° so that crystals are obtained in anhyd form.

2-Keto-l-gulonic acid. F. Hoffmann-La Roche & Co. A.-G. Brit. 435,971, Oct. 2, 1935. Addn. to 427,286 (C. A. 29, 5863<sup>1</sup>). This is prepd. by a modification of the process of 427,286 by using alicyclic ketones in the same manner as the ketones in general in 427,286. In an example, l-sorbose is treated with cyclohexanone and H<sub>2</sub>SO<sub>4</sub> to produce dicyclohexanone-l-sorbose which is oxidized with KMnO<sub>4</sub> in alk. soln. to form K dicyclohexanone-2-keto-l-gulonate which is treated with H<sub>2</sub>SO<sub>4</sub> to liberate the acid and finally decomp. by boiling with H<sub>2</sub>O to yield 2-keto-l-gulonic acid. Cf. C. A. 30, 111<sup>1</sup>.

Quinonic acid. C. F. Boehringer & Soehne G. m. b. H. (Wilhelm Dürschel and Heinrich Thron, inventors). Ger. 619,824, Oct. 7, 1935 (Cl. 12p 1 10). Quinonic acid (6-methoxyquinoline-4-carboxylic acid), is prepd. by oxidizing the noncryst. residues of cinchona bark extr., i. e., the residues after extg. the quinine, cinchonidine, cinchonine and quinidine, with H<sub>2</sub>CrO<sub>4</sub> and Mn compds. in acid soln. Thus, the residue contg. quinidine is boiled with H<sub>2</sub>SO<sub>4</sub> or AcOH and K<sub>2</sub>MnO<sub>4</sub> to give the above.

Sulfonic acids. Henkel & Cie G. m. b. H. Fr. 788,606, Oct. 14, 1935. Aliphatic and cycloaliphatic sulfonic acids of high mol. wt. are prepd. by oxidizing in aq. soln. the corresponding esters of thiosulfuric acid or their salts. Thus, dodecanesulfonic acid is prepd. by oxidizing the Na salt of the ester of dodecylthiosulfuric acid.

Aliphatic acid anhydrides. Carl J. Malm and Webster E. Fisher (to Eastman Kodak Co.). U. S. 2,026,985, Jan. 7. In a process such as the production of butyric or stearic anhydride from butyric or stearic acid, the acid in admixt. with Ac<sub>2</sub>O vapor is passed in contact with Mg(ClO<sub>4</sub>)<sub>2</sub> in countercurrent through a fractionating column. App. is described.

Chlorinating phthalic anhydride. Michael N. Dvornikoff (to Monsanto Chemical Co.). U. S. 2,028,383, Jan. 21. Cl<sub>2</sub> is introduced into molten phthalic anhydride contg. an Fe salt of a nonoxidizing, strong inorg. acid such as FeCl<sub>3</sub> or Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> as a chlorinating catalyst.

Treating methane to produce more easily liquefiable hydrocarbon gases. Henry Renneke. U. S. 2,028,014, Jan. 14. CH<sub>4</sub> is passed, under sub-atm. pressure, through a reaction tube in which a silent elec. discharge is maintained, the flow of gas being such that the contact of the mols. of the gas and the elec. field is less than 0.03 sec. The product contains C<sub>2</sub>H<sub>6</sub>, etc.

Acetone. Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler (Hans Walter, inventor). Ger. 622,493, Nov. 29, 1935 (Cl. 12o 10). EtOH or AcH is led with steam at 350–500° over a contact mass contg. porous Cu and a catalyst, e. g., ZnO, CdO, MnO or Fe<sub>2</sub>O<sub>3</sub>. Compds. derivable from EtOH or AcH, e. g., AcOEt, Et<sub>2</sub>O, Me-CH(OAc), MeCOOCH<sub>3</sub> or EtOCH<sub>3</sub>, may be treated in the same way.

Dichloroethylene. Compagnie de produits chimiques et électrométallurgiques Alais, Fröges et Camargue. Brit. 436,133, Oct. 4, 1935. See Fr. 786,803 (C. A. 30, 737<sup>1</sup>).

Methylamines. I. G. Farbenind. A.-G. (Eduard Lemch, inventor). Ger. 619,754, Oct. 5, 1935 (Cl. 12g 5). In prep. methylamines by heating MeOH and NH<sub>3</sub> or NH<sub>4</sub> salts or urea in a closed vessel, the reaction is carried out in the presence of oxide, chloride or sulfate of Cu or Fe. Acids or alkali salts may also be present if desired. In an example, a suspension of CuO in MeOH is treated with NH<sub>3</sub>, till satd. at –10° and heated for 3 hrs. in a closed vessel to 370° and 168 atm. The products are 12.9% monomethylamine, 1.7% di- and tri-methylamine and 85.4% NH<sub>4</sub>. Other examples are given.

**Ethylenediamine** Frederick C Bersworth (to Frank Kottke) U S 2,028,041, Jan 14 Confined liquid  $\text{NH}_3$  is heated sufficiently to vaporize it and to generate superatm pressure, and ethylene dichloride is injected into the hot, compressed  $\text{NH}_3$  vapor, forming ethylenediamine-HCl

**2 Chloro-1,3 butadiene** Granville A Perkins (to Carbide and Carbon Chemicals Corp.) U S 2,027,550, Jan 14 Vinyl acetylene is treated with aq HCl in the presence of a  $\text{Cu}_2\text{Cl}_2$  catalyst and of a water-immiscible inert solvent such as toluene in which the product is dissolved

**Chloroethyl chloroethoxyethyl ether.** Carbide & Carbon Chemicals Corp Fr 788,281, Oct. 7, 1935. See U S 2,017,811 (C A 29, 8320\*)

**Cresol and ditolylamine** Wm J Hale (to Dow Chemical Co.) U S 2,028,065, Jan 14 Chlorotoluene is heated under superatm pressure to above  $260^\circ$  but below the temp at which the reaction products would decompose (suitably at about  $300^\circ$ ) with at least 0.3 its chem equiv of  $\text{NH}_3$ , at least 0.2 its chem equiv. of a cuprous compd such as  $\text{Cu}_2\text{O}$  and at least its chem equiv. of a base such as  $\text{Ca}(\text{OH})_2$  and with an amt of water at least twice the wt of such base Other aryl chlorides such as PhCl may be similarly decompd to form a phenol and a monoaryl amine

**Pyrocatechualdehyde** Marion S Carpenter and Eric C Kunz (to Givaudan-Delawanna, Inc.) U S 2,027,148, Jan 7 Heliotropin is treated with anhyd  $\text{AlCl}_3$  in the presence of an aliphatic chlorinated hydrocarbon solvent such as ethylene dichloride followed by treatment with an aromatic hydrocarbon such as toluene and with use of sufficient heliotropin to prevent reaction between the solvent and the aromatic hydrocarbon

**1,2 Diaminobenzene** Soc pour l'ind chim à Bâle Fr 788,348, Oct 8, 1935 This is made by treating 1,2-dichloro- or 1-amino 2-chloro benzene with an aq soln of  $\text{NH}_3$ , at about  $150^\circ$ , in the presence of Cu, so that at the end of the reaction the pressure of the  $\text{NH}_3$ -water system is at least about 80 atm at  $150^\circ$  The Cu is removed after the reaction as sulfide

**Monomethyl p aminophenol** Harold von Bramer and Albert C Ruggles Brit 435,721, Sept 26, 1935  $\text{NH}_4\text{Me}$  is caused to react at elevated temp and pressure, on hydroquinone in an unsatd aq soln The sulfate is obtained by adding  $\text{H}_2\text{SO}_4$  to the reaction mixt and sepg the product

**Cumylphenol** Edgar C Britton and Lawrence F.

Martin (to Dow Chemical Co.) U S 2,028,043, Jan 14 This compd, b, about  $182^\circ$ , m about  $73-5^\circ$ , is made by causing phenol to react with cumyl chloride in the presence of  $\text{AlCl}_3$

**Naphthalene** La Société Industrielle des carburants et solvants Brit. 435,717, Sept. 26, 1935 Crude  $\text{C}_{10}\text{H}_8$  is purified with a view to subsequent hydrogenation by vaporizing and conveying through 2 purifying chambers in series, of which the 1st contains Fe, Cu, Ni, Co or Cr, disposed so as to afford an extensive contact surface, and the 2nd contains a mixt of porous C and Ca, Fe, Cu, Ni, Co or Cr, advantageously obtained by calcining the compds resulting from the action of  $\text{CaH}_2$  on these metals, or of C and the oxides of the said metals, the purifiers being maintained at  $250-400^\circ$

**3-Aminopyridine** Chemische Fabrik von Heyden A-G (Arthur Binz and Otto v. Schickl, inventors) Ger 2,322,345, Nov. 26, 1935 (Cl 12p, 1 01). When halo 3-nitropyridines are treated with H in the presence of a hydrogenation catalyst 3-aminopyridine is formed The reaction may be effected at atm. temp and pressure

**1-Phenyl-3-methyl-5-pyrazolone and related compounds** Carbide & Carbon Chemicals Corp. Fr 788,282, Oct. 7, 1935 See U S 2,017,815 (C. A. 29, 8003\*)

**Diacetylmorphine** Hermann Fischer (Anton Baselgia, inventor) Ger 622,231, Nov 23, 1935 (Cl 12p 14)

**Morphine** is acetylated by reaction with ketene Preferably, a suspension of morphine in a solvent for diacetylmorphine, e g,  $\text{Et}_2\text{O}$ , is treated with a stream of gaseous ketene

**Polymerizing monosaccharides** such as dextrose and xylose Eduard Farber (to N. V. Internationale Suiker en Alcohol Compagnie (International Sugar and Alcohol Co "Isaco")) U S 2,027,904, Jan 14 A monose is melted with materially less than 0.1% (suitably about 0.01-0.02%) its wt of free strong inorg acid such as  $\text{H}_2\text{SO}_4$  and is heated to remove water, at temps of  $120-180^\circ$  below temps which cause material decompn

**Objects made from polystyrene.** Siemens & Halske A-G Fr. 788,345, Oct. 8, 1935 The surface of the object is rendered mat for making marks thereon or for coloring by treatment with acetone for a short time

**Unsaturated aliphatic aldehydes** Max Hoffer. Swiss 173,737, Apr 1, 1935 (Cl 36a)  $\text{H}_2\text{O}$  is split off from said aldehydes of the formula  $\text{RCH}_2\text{CHO}$ , secondary org bases or their salts being used as catalysts. Preferably acetates of secondary bases are used In an example,  $\text{EtCHO}$  is treated with piperidine acetate to give an 80% yield of methylpentenal. Other examples are given

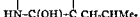
## 11—BIOLOGICAL CHEMISTRY

PAUL E HOWE

### A—GENERAL

ARTHUR W DOX

**Acid autoclaving of blood albumin at  $220^\circ$ .** V. S. Sadikov and E. V. Lindkvist-Russakova *Compt rend acad sci U R S S* [N S], 3, 271-2 (1935), cf C A 29, 5470! —On autoclaving 6 kg of blood albumin with 4%  $\text{H}_2\text{SO}_4$  for 10 min at  $220^\circ$  the whole protein passed into soln and, on cooling, 54 g of a solid amorphous mass contg cryst cyclopeptides and amorphous substances of the cyclopeptide type sepd out spontaneously This fraction was washed with  $\text{Et}_2\text{O}$  and after 20 recrystns from hot  $\text{EtOH}$  yielded 3-4 g of a cyclopeptide (I),  $\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_5$ , m  $275-6^\circ$ , it contains no  $\alpha$ - $\text{NH}_2$  groups Closer investigation showed that I is free from tyrosine, tryptophan, arginine, histidine, lysine, aspartic and glutamic acids, cystine and methionine. The hydrolyzate from the treatment of 0.5 g of I with 25% HCl for 36 hrs was freed from HCl and was pptd by  $\text{Ag}_2\text{O}$  The Ag was removed and the free amino acids were converted into Cu salts which were sepd into sol and insol fractions (leucine salt) Investigation of the sol fraction showed the presence of an isovaline and I is evidently an inactive cycloleucylisovaline with the structure



The same compd occurs in the alc autoclavyzate of blood albumin C R Addinall

**The digestive enzymes of some cephalopods** C Romijn *Arch neerland zool* 1, 373-431 (1935), cf C. A. 29, 4782! —The enzymes contained in glycerol exts of the middle digestive tract (liver and pancreas) and of the salivary glands of *Sepia officinalis* (1), *Loligo vulgaris* (2) and *Eledone cirrhosa* (3) were studied The salivary gland exts contained no digestive enzymes The reaction of the gastric juice of (1) changes from slightly acid to neutral The exts of the middle digestive tract and gastric juice contain lipase The liver lipase has a  $\text{pH}$  optimum of 6.02, the pancreas lipase, 6.35 The liver ext digests native albumin, peptone, chloroacetyl-L-tyrosine, leucylglycine and glycylglycine. The crude ext. is not activated with  $\text{H}_2\text{S}$ , glutathione or enterokinase The  $\text{pH}$  optimum is 6.1 for casein, 5.6 for gelatin, 6.1 for peptone, 5.5 for chloroacetyl-L-tyrosine, 8.3 for leucylglycine and 8.2 for glycylglycine The crude ext. of the pancreas does not split casein, peptone or chloroacetyl-L-tyrosine, but it does split leucylglycine and glycylglycine After

activation with enterokinase or an ext. from the cecum walls, the ext. can attack casein, peptone and chloroacetyl-L-tyrosine. The  $pH$  optimum is 7.7 for leucylglycine and 7.8 for glycylglycine. In gastric juice, the  $pH$  optimum is 6.8 for casein, 6.6 for gelatin and 6.2 for peptone. The  $pH$  optimum of the peptidases is on the alkali side. Liver ext. and gastric juice contain rennin, pancreas does not (1) and (2) contain amylase, (3) contains amylase, maltase and sucrose. Liver ext. has more action on starch, pancreas ext., more action on glycogen. The  $pH$  optimum is 6.7 to 7.0 for the breakdown of starch by gastric juice, liver and pancreas exts., 7.10 for glycogen breakdown in the gastric juice, 6.26 in liver ext. and 6.14 in the pancreas. E S G B

The biological properties of heavy water. C H Libérall. *Rev. chim. farm. (Brazil)* 1, 59-62 (1975). Review. E S G B

Enzymes of fermentation. IV The specificity of yeast phosphatase. Irwin Bauer, Anton Schaffner and Fritz Krumey. *Z. physiol. Chem.* 237, 191-8 (1975), cf. C A 29, 546/57. The specificity of yeast phosphatase for  $\alpha$ -in preference to  $\beta$ -glycerophosphoric acid is corroborated by the use of dry preps of Prager and Patzenhofer yeasts. The  $\alpha$  is hydrolyzed several times as rapidly as the  $\beta$ -isomer, and about 8 times as rapidly as dihydroxyacetonephosphoric acid. The difference is most striking in the early stages of hydrolysis, where the cleavage ratio may be as high as 10:1. Bottom yeast is richer in phosphatase than top yeast. A W Dux

Liver enzymes. V The aldehyde dehydrogenase of the liver. Ludwig Reichel and Kristian Løkken. *Z. physiol. Chem.* 237, 214-20 (1975), cf. C A 30, 115. The dehydrogenase obtained by dialysis of the dry prep has only slight desmutative activity toward  $F_2CHO$  because the coenzyme has been removed. It becomes active when small quantities of coenzyme are added. The method of prep. also destroys the indophenol oxidase and removes the flavin originally present. For the dehydrogenation process flavoprotein and lactoflavin can function as intermediate acceptors. Of the 4 synthetic flavins tested only 9-d-araboflavin was a  $H$ -acceptor. If the acceptor is not present in adequate concn the dehydrogenation is accompanied by dismutation. A W Dux

Activators of glucolysis. II Hans v. Euler and Gunnar Günther. *Z. physiol. Chem.* 237, 221-6 (1975), cf. C A 29, 6908. Reiteration of the previous expts., but with a highly purified and more active coenzyme prep., confirms the observation that heating destroys the activating power toward fermentation while the activation of glucolysis remains unimpaired. The activation of lactic acid formation from glycogen in the presence of hexosediphosphate by heated coenzyme is nearly the same as that by adenylypyrophosphoric acid but less than that by active coenzyme. A W Dux

The dehydrogenation of citric acid and isocitric acid by the dehydrogenase of cucumber seeds. Th. Wagner-Jauregg and H. Rauen. *Z. physiol. Chem.* 237, 227-32 (1975), cf. C A 29, 1531. Citric acid dehydrogenase has been considered a typical "anaero-dehydrogenase" since only such substances as  $CaH_2(NO_3)_2$ , methylene blue and certain dyes, but not  $O_2$ , can serve as  $H$ -acceptors. It is now shown that the dehydrogenase system after completion by flavin enzyme can take up  $O_2$ . The extent of respiration is dependent on the addn. of flavin enzyme and to a less extent on coenzyme. For each mol of citric acid consumed about 0.5 mol. of  $O_2$  is taken up. Neither  $OC(CH_3)CO_2H$ , nor  $MeAc$  is formed from citric acid by citric dehydrogenase even in the presence of yellow enzyme. In the aerobic breakdown of citric acid  $OC(CH_3)CO_2H$  cannot be an intermediate product. Other degradation products of citric acid, such as ketopie, acetoacetic, acetic, itaconic, hydroxylumaric, hydroxymaleic and acetic acids, have no substrate action toward the dehydrogenase. On the other hand, the isomeric isocitric acid,  $HO_2CCH(OH)CH_2CO_2H$  is more rapidly dehydrogenated than citric acid itself and the reaction is strongly accelerated by yellow enzyme. Isocitric acid

1 has been found only in blackberries but a much wider distribution may be assumed. A W. Dux

Enzymic dehydrogenation of glycerophosphoric acid. Th. Wagner-Jauregg and H. Rauen. *Z. physiol. Chem.* 237, 233-5 (1975).—Extn. of cucumber seeds with  $KH_2PO_4$  yields a soln. contg. a dehydrogenase which decolorizes methylene blue in the presence of Na glycerophosphate. The reaction is greatly accelerated by the addn. of flavin enzyme, the decolorization time thereby being diminished from 180 to 12 min., and to 7 min. by the further addn. of coenzyme. Heated flavin enzyme is inactive, and coenzyme alone only slightly active. Even in relatively high concn  $H_2CN$  has no influence on the reaction velocity of the system. The flavin enzyme undoubtedly serves as an intermediate acceptor which transfers  $H$  from the substrate to the final acceptor, methylene blue. A W. Dux

Orientation data on the composition of the jelly mass of *Rhizostoma cuvieri*. R. Zeyneck and A. Dimer. *Z. physiol. Chem.* 237, 217-53 (1975).—After removal of the structural portions 130 kg. of jellyfish was evapd. and the concd soln. was filtered from the sepd. salts ( $CaSO_4$ ,  $NaCl$ ) and coagulated protein. Pptn. by  $EtOH$  then gave a viscous mass of org. nitrogenous substances which dissolved in  $H_2O$  at  $45^\circ$  and gelatinized on cooling. Several fractions were obtained by increasing concns of  $EtOH$ , but none could be crystd. Redissolved in  $H_2O$  they gave ppt. with phosphotungstic and tannic acids, biuret,  $PbS$ , xanthoproteic and Millon reactions were pos.  $Ba(OH)_2$  in the cold gave  $NH_3$  and  $NMe_3$ . Hydrolysis by boiling with 3%  $HCl$  gave chiefly leucine and isoleucine. Other products identified were valine, alanine, glutamic acid, aspartic acid, phenylalanine, proline, hydroxyproline, considerable lysine, some arginine, histidine, guanine, taurine, lactic acid and probably aminobutyric acid. Glycine and tyrosine were present only in traces, while cysteine, serine and leucine acid were absent. The jelly of jellyfish differs essentially from the gelatin-yielding tissues of higher animals in its greater ease of hydrolysis, its low glycine content and its high S content. A W. Dux

Emulsin. XXV. Coffee emulsin. Burckhardt Hellerich and Fritz Voratz. *Z. physiol. Chem.* 237, 254-60 (1975), cf. C A 30, 81. An ext. of raw coffee beans contains an emulsin which hydrolyzes  $\alpha$ -d-mannosides,  $\alpha$ -d-galactosides and  $\beta$ -d-galactosides. These activities are attributed to sep. enzymes since the proportions vary with different methods of prep. Coffee emulsin is very similar to lucerne emulsin but differs therefrom in its much feebler activity toward  $\alpha$ -d-glucosides. The  $\alpha$ -d-mannosidase present is much more resistant to moderate heating than is the  $\beta$ -d-galactosidase. Phenol- $\beta$ -d-galactoside and vanillin- $\beta$ -d-galactoside are hydrolyzed with equal velocity. The  $\beta$ -d-glucosides of procatechu-aldehyde, caffeine acid and vanillin are hydrolyzed so much more rapidly than phenol- $\beta$ -d-glucoside or salicin by the  $\beta$ -d-glucosidase of coffee emulsin that their use is proposed for purposes of identification. A W. Dux

$\alpha$ - and  $\beta$ -Thymonucleic acid and the enzyme (nucleogelase) which converts the  $\alpha$ - into the  $\beta$ -form. R. Løulgen. *Z. physiol. Chem.* 237, 261-7 (1975).—Com. pancreatin contains an enzyme which depolymerizes  $\alpha$ -thymonucleic acid into a  $\beta$ -form which still has the tetranucleotide structure but is no longer capable of gelatinizing. No  $H_2O_2$  is split out in this reaction. Both  $\alpha$ - and  $\beta$ -thymonucleic acids yield the same products (guanine, adenine, cytosine and thymine) on hydrolysis, and in the same proportions. The name nucleogelase is proposed for the enzyme. A W. Dux

The chemistry of heparin. Fritz Lipmann and Albert Fischer. *Z. physiol. Chem.* 237, 273-4 (1975).—A S-contg. heparin prep. was obtained from beef lung which showed the same rate of destruction by 0.1 N  $HCl$  at  $100^\circ$  as a previous S-free prep. The power of inhibiting blood clotting was destroyed much more rapidly than the  $H_2SO_4$  was liberated by hydrolysis, 70% of the activity having vanished before sulfate could be detected. A W. Dux

The influence of carbon monoxide on the respiration of yeast cells in various substrates. Model experiments in

the physiology of fertilization Åke Öström *Protoplasma* 24, 177-85(1935) —The respiration of unfertilized sea-urchin eggs is hastened by CO<sub>2</sub> but that of the fertilized is arrested. The question is raised if similar relationships are obtained with yeast by a variation of substrates. It was found that the "Eigenatmung" (respiration of the yeast without a substrate) and the "Veratmung" (consumption of the substrate) of the formate is hastened by CO<sub>2</sub> which is similar to the reactions of unfertilized eggs. The "Veratmung" of Na pyruvate, Na lactate, AcONa, AcH and alc is retarded by CO<sub>2</sub>. With the exception of alc, all these substrates have retardation curves, which correspond more or less with those of unfertilized eggs. But the retardation curve of each substrate is typical and varies from the others. If the concns in the distribution formula of Warburg are calcd (C A 22, 4568), values are found which are high at the beginning of the curve but which drop considerably later. Only alc shows a retardation corresponding to Warburg's formula. If the concn of the substrates is varied, another retardation curve is obtained for each concn. Expts with KCN give results similar to those obtained with CO<sub>2</sub>. In the discussion, possibilities for clearing up these variations from the formula of Warburg are given. F L D.

Ion action and water permeability. A contribution to the conservation theory of the plasma limiting membranes Iz de Haan *Protoplasma* 24, 186-97(1935) —Literature on the action of salts on H<sub>2</sub>O permeability and on the viscosity of protoplasm is discussed. There is much disagreement among the statements. The influence of salts at various concns and with cations of various valences on the H<sub>2</sub>O permeability of protoplasm has been studied. Multivalent cations at low concns lower the permeability and have a condensing action on the protoplasm, while higher concns increase the permeability. One salt with a univalent cation (NaNO<sub>3</sub>) causes, at all concns studied, an increase in permeability. This salt influence is best explained by the theory of Bungenberg de Jong, according to which an auto-complex system of phosphatides participates in the building up of the protoplasm or its limiting layer. Thirty references. F L D.

The chemistry of cell division. IV. The influence of hydrogen sulfide, hydrocyanic acid, carbon dioxide and some other chemicals on mitosis in Amoeba proteus Carl Voegtlin and H W Chalkley. *Protoplasma* 24, 365-83(1935) —The influence of certain chem agents on mitosis in *Amoeba proteus* was studied by exposing cells at the onset or during the later stages of mitosis to solns contg. these chemicals in subtoxic concns. H<sub>2</sub>S and HCN inhibit mitosis. The inhibition is greatest if exposure is made in prophase and decreases rapidly if it is delayed to the later stages of mitosis. The degree of inhibition is a function of the concn of these reagents in the suspension medium. No evidence of any accelerating influence was obtained with any concn used. The inhibition is completely reversible upon withdrawal of the inhibiting agents and mitosis is completed in exactly the same time interval, as if it had not been inhibited. CO<sub>2</sub> fails to modify mitosis in the absence of light and in the presence of all but traces of mol O. The conclusion is reached that mitosis *per se* apparently is independent of cell respiration. EtO causes an inhibition, reversibly if exposure is made in early prophase. The reversibility of this action is complicated by the production of cytoplasmic vacuolization and irregularities in nuclear fission. Under the conditions used, H<sub>2</sub>O<sub>2</sub>, As<sub>2</sub>O<sub>3</sub>, methylene blue, CuCl<sub>2</sub> and HgCl<sub>2</sub> exert no inhibiting influence on mitosis. CO<sub>2</sub> inhibits in early prophase, but not appreciably in the later stages. The inhibition is incompletely reversible in the sense that irregularities in the distribution of chromatin occur. The incidental observation was made that sudden and great changes in CO<sub>2</sub> tension cause death, whereas a more gradual adjustment is well tolerated. The possible biochem mechanism responsible for the modifying influence produced by these agents on mitosis is discussed with particular reference to the function of enzymes concerned in the metabolism of the cell nucleus. F L D.

Cell elongation and the electrical properties of the cell wall James Bonner and A N J Heyn *Protoplasma* 24, 466-9(1935) —Expts have been carried out to test the hypothesis of the indirect effect of growth substance upon the charges of the micelles constituting the cell wall, by the measurement of the relative charges of cell wall suspensions from the coleoptile of *Avena sativa*. It was impossible to demonstrate any direct or indirect effect of growth substance upon the charges of such suspensions. While these neg results are not absolutely conclusive, it seems unlikely that large changes of elec properties of the cell wall play an important role in elongation. F L D.

Spectrophotometric studies. II. Preparations from washed blood cells, nitric oxide hemoglobin and sulf-hemoglobin David L Drabkin and J Harold Austin *J Biol Chem* 112, 51-65(1935); cf C A 27, 762 — Absorption spectra of oxyhemoglobin (I), hemoglobin (II), CO-hemoglobin and cyanomethemoglobin (III) yielded by solns prep'd from hemolyzed washed erythrocytes are given. Such solns are to be preferred for precise spectrophotometric analysis over corresponding preps from hemolyzed whole blood. Fairly wide variations of  $pH$  do not affect the absorption curves. The absorption spectrum of nitric oxide hemoglobin (IV) was obtained under conditions which excluded the presence of O. IV is quantitatively converted into methemoglobin (V) by K<sub>3</sub>Fe(CN)<sub>6</sub>, and V into III. Hence IV is probably a relatively stable analog of I. Data are given which indicate that the absorption spectra of pure sulfhemoglobin (VI) has not been described heretofore. The curve of VI is extrapolated from data on mixts of II and VI. VI is formed from II only in the presence of O. VI was not convertible to II or III but was readily changed into typical hemochromogens. Solns of II increase in acidity upon exposure to H<sub>2</sub>S. The estn of VI was made on the blood of a patient with clinical sulfhemoglobinemia. Sulfmethemoglobin (VII) was obtained from V by treatment with H<sub>2</sub>S, and a pigment yielding the spectrum of VII was obtained similarly from III and H<sub>2</sub>S. III. Methemoglobin. J Harold Austin and David L Drabkin *Ibid* 67-83 — Spectrophotometric constns are given for methemoglobin (I), derived from washed, hemolyzed dog erythrocytes treated with K<sub>3</sub>Fe(CN)<sub>6</sub>, (II) at various  $pH$  values and known ionic strengths. From  $pH$  6.0 to 9.4 I undergoes conversion from the acid to alk form, the conversion conforming closely to  $pH = pK' + \log I_{alk}/I_{acid}$ . The value for  $pK'$  at ionic strength 0.10 is  $8.120 \pm 0.010$  and very roughly  $\Delta pK' = -0.63\sqrt{\text{ionic strength}}$ , at least below ionic strength of 0.154. At about  $pH$  9.4, where 95% of I is in the alk form, an alteration sets in, which is increased at higher  $pH$  values and which progresses with time. Spectrophotometrically this appears to be in the direction of alk. hematin. Between  $pH$  7.0 and 9.2 the change between the acid and alk form of I is reversible. Titration of oxyhemoglobin (III) with II in contact with air shows that III is converted to I completely at  $pH$  6.0 in 20 min by a mol-to-mol ratio of reductant to oxidant. As the  $pH$  rises an excess of II is required to complete the conversion, and for a given ratio of II/III the reaction proceeds more slowly. On treating III solns. with small amts of NaNO<sub>2</sub> (IV) there is a slow reaction in which the IV appears to act only indirectly on the III, the rate of reaction being accelerated at lower  $pH$ . IV added in excess of an amt characteristic for a particular prep'n gives immediate conversion to I, 1 mol of III being converted for every 0.5 to 0.7 mols of IV effective in the reaction. With amts of IV in excess of a molar ratio of 4:1 at  $pH$  7.15 the resulting absorption curve is higher at  $\lambda$  575, 560 and 540 m $\mu$  than that obtained with II. The observed curve is not inconsistent with that which would result from a mixt. of I and nitric oxide hemoglobin. At  $pH$  9.2 IV did not convert III in contact with air to I. If I be produced with IV in unbuffered soln and then buffered to  $pH$  9.2 the spectrum is essentially identical with that of I produced with II at  $pH$  9.2. There is a slow reaction between III and small amts of quinone (V) in contact with air, but progressing with the lapse of time to at least

80% conversion at a 1:1 M ratio; a very much more rapid reaction occurs with considerable excess of V. The absorption curve of I produced by V is somewhat higher than that of I produced by II, presumably owing to by-products. Addn. of KCN (VI) produces the curve of cyano-I (VII), also somewhat elevated. III in very dil soln of hemolyzed dog erythrocytes underwent, on standing 48 hrs, partial change into I. Addn. of VI converted this I into VII and during a further 48 hrs conversion of III continued in the presence of VI. Graphs are presented for various mixts of I and III at various pH values which show the ratio of  $\frac{A_{575m\mu}/A_{540m\mu}}{A_{575m\mu}/A_{540m\mu}}$  and the summation of changes defined as  $\Sigma \frac{A_{575m\mu}}{A_{540m\mu}}$  at  $\lambda$  620, 575, 540 and 540 m $\mu$ . The use and advantages of the latter in the quant estn of I in a mixt with III are discussed. IV Hemochromogens. David L Drabkin and J Harold Austin *Ibid* 89 104.—The absorption spectra of oxidized and reduced hemochromogens are described. Data are given to illustrate the importance of the time factor in conversion of hemoglobin (I) to hemochromogen with NaOH. Solns of I treated with  $C_4H_9N$  yield characteristic hemochromogen spectra. The reduced hemochromogen so formed reacts reversibly with CO, and the oxidized form reacts with KCN, presumably yielding under these conditions cyanohemoglobin. The possible significance of the observations is discussed. Nencki and Zaleski's method for prep *chloroformin* has been improved by using washed blood cells, thereby the dilg effect of serum is avoided as well as the presence of serum proteins and lipides, and a 64% yield is obtained. V A technic for the analysis of undiluted blood and concentrated hemoglobin solutions. *Ibid* 105-15.—A new spectroscopic cell, 0.07 mm in depth, is described for the study of very concd solns. It can also be used for the study of solns without exposure to environmental gases. Optical studies have been made upon hemolyzed undil blood, nonhemolyzed blood and upon solns of crystd hemoglobin (I) in concns as high as, or in excess of, those found in red blood cells. Beer's law applies for solns of I in a range of concns from 1 to 0.0001, where 1 = 25.58 millimols per l (42.72 g per 100 cc). It also applies for oxyhemoglobin over considerable range. The absorption of light by I appears to be a function of the Fe-porphyrin; therefore aggregation involving intramolecular rearrangement within the prosthetic groups appears excluded. No conclusions could be drawn as to the possibility of mol assocn involving the large I mol. With respect to the specific absorption of light, even in very concd solns, I resembles a true soln. R C Elderfield.

The renal glucose threshold in terms of the Cushny-Rehberg theory. Delfino Barbieri *Minerva med* 1935, II, 670-82.—The renal glucose threshold was detd in normal persons and diabetics by the alimentary and the Rehberg methods. The results of the two methods were comparable. Helen Lee Gruel.

Biological cell oxidation. A Bertho *Chem Ztg* 59, 953-7 (1935).—A review. E H.

The structure of silk fibroin. II Münch. *Angew Chem* 48, 797-9 (1935).—Investigations on the splitting of natural silk fibroin in various stages of spinning were carried out by means of the enzyme papain in acid soln, which acts specifically upon sericin. The degumming effect is greatly increased by addn. of activators such as  $Na_2S_2O_5$  and  $Na_2SO_4$ . A parallelism between cleavability and fiber structure could be established. K. Kammermeyer.

Yeast amylase. IV. Enzyme-chemical properties. Optimum pH and temperature. Kazuki Ono, *J Agr Chem Soc Japan* 11, 893-7 (1935).—Amylase soln. was prepd from yeast by using  $(NH_4)_2HPO_4$ . The optimum pH was 6.2-6.6 at 22.5° and 6.0-6.2 at 30°, and the optimum temp was 25-30° at pH 6.4 for the hydrolysis of starch. Y. Kihara.

Crystalline egg albumin. II The fractionation of peptic hydrolysis products. Herbert O Calvery *J Biol Chem* 112, 171-4 (1935); cf. C. A. 27, 5315. Cryst. egg albumin was digested with pepsin for 23 days

1 and an equil. was attained in which approx. 1/3 of the peptide linkages had been hydrolyzed in accord with results previously obtained. The digestion products were sep'd. into 5 fractions quite different from each other as shown by chem. and enzymic studies. Fraction A<sub>5</sub> contained only 10% peptide N, indicating that free amino acids were present. Results of the action of a specific dipeptidase indicate also that dipeptides are formed during peptic hydrolysis of egg albumin. A. P. L.

2 Activation of enzymes. III The role of metal ions in the activation of arginase. The hydrolysis of arginine induced by certain metal ions with urease. Leslie Hellerman and Marie E Perkins *J Biol Chem* 112, 175-94 (1935), cf. C. A. 29, 1113.—The arginase prepn used (obtained from freshly excised calf liver) is neither activated nor, after treatment with various oxidizing agents, reactivated by cysteine or other reducing agents that activate papain. It is effectively activated and reactivated by certain metal ions, specifically  $Co^{++}$ ,  $Ni^{++}$  or  $Mn^{++}$  (as well as  $Fe^{++}$ ).  $Co^{++}$  brings about almost complete restoration of activity after the enzyme has been inactivated by  $H_2S$  and the excess of the latter removed.  $RHgX$  compds do not inactivate, ferricyanide ion incompletely suppresses the activity and  $Cu^{++}$ ,  $Hg^{++}$  and  $Ag^{+}$  destroy the activity, in part, by pptg the enzyme. The action of HCN is depressant. "On the basis of the results,

4 the arginase mol is visualized as contg a metallic component which may be oxidized or sep'd from the rest of the mol by the action of oxidizing agents or certain other reagents. The observed actions of  $Co^{++}$  and other effective ions are interpreted, not as reductions, but rather in terms of the characteristic property of these ions to coordinate with suitable mols or groupings to form complex mols. This suggestion may include an implication regarding the role of the metallic component in the building of an enzyme-substrate compd." Arginine without arginase is hydrolyzed in the presence of  $Co^{++}$ ,  $Mn^{++}$  or  $Ni^{++}$ , and under certain conditions  $Fe^{++}$ , when urease is also present in the mixt. This effect is suppressed by organometallic suppressors of urease activity. Cyanide diminishes the effect but does not prevent the independent hydrolysis of any urea present since it is a urease activator. The bearing of these results upon analytical operations

5 involving the use of urease in the detn. of urea where arginase and certain metal ions may also be present is obvious. A. P. Lothrop.

The effect of fluorine upon the phosphatase content of plasma, bones and teeth of albino rats. Margaret Cammack Smith and Edith M Lantz, *J Biol Chem*, 112, 303-11 (1935), cf. C. A. 29, 5159.—F does not exert its characteristic damage to the teeth of rats through its effect upon the enzyme involved in tooth and bone calcification and an increase in plasma phosphatase cannot be considered a sensitive indication of fluorosis in rats as reported by Phillips for dairy cows. The phosphatase content of the plasma of normal rats decreases with age up to about 70 days when there is a precipitous drop with little change thereafter. In rats given diets contg. 0.1% NaF there is also a decrease with age but it is slower and the values are lower than those of the controls up to 70 days of age, after which they are slightly higher. The lower values appear to be an indication of less active bone growth and delayed maturity rather than a specific F effect. Phosphatase values of the same order as the controls are shown by rats receiving 0.025% NaF, a concn sufficient to mottle the teeth but not to stunt bone development. The phosphatase content of the incisor teeth of the F-fed rats is less than that of the controls. In bone the phosphatase values in the controls and F-fed litter mates are approx the same up to 70 days after which time they are slightly higher in the latter animals. When detd approx 18 hrs. after the injection of 0.3 cc of a 2.5% soln of NaF, the plasma phosphatase values in adult rats are not significantly different from those of the controls. A. P. Lothrop.

Derivatives of keratin. David R Goddard and Leonor Michaelis, *J Biol Chem*, 112, 301-71 (1935); cf. C. A. 28, 6740.—Keratine is formed from keratin by reduction



with Na thioglycolate. It is oxidized to metakeratin which differs from native keratin by its amorphous character, soly in alkali and digestibility with pepsin or trypsin. Derived proteins, differing distinctly in their solubilities and isoelec. points, can be obtained by substituting the H of the -SH group of kerateine by treatment with iodoacetate,  $\alpha$ -bromopropionate, iodoacetamide and iodoethyl alc. forming carboxymethyl-, carboxyethyl-, carbamylmethyl- and hydroxyethylkerateine, resp. All are digested by pepsin or trypsin. No substitution of amino H or loss of S occurs during the reaction. Carboxymethylkerateine is sol enough to permit fractionation with  $(\text{NH}_4)_2\text{SO}_4$ . Two fractions were obtained, a larger at 35% satn and a smaller at 60% which contained more S and less N and amino N than the original protein. These results indicate that native keratin probably consists of 2 fractions and not that the substitution leads to 2 different products. A. P. Lothrop

The amide nitrogen of ovalbumin. Agnes Shore, Hildegard Wilson and Geo Stueck. *J Biol Chem* 112, 407-13 (1935). —Deins were made of the amt. of  $\text{NH}_2$  formed at intervals during the protracted hydrolysis of ovalbumin in 5, 1 and 0.2 M HCl at 100° and 85°. Four sep preps of cryst ovalbumin were used and all gave the same amide N within the exptl error of 1 equiv. per mol in spite of variations in the method of prepn. The probable value for the amide groups is 24 equivs per mole. The hydrolysis of the amide groups is a 1st-order process. The velocity consts are  $7.15 \times 10^{-4}$  at 100° and  $1.83 \times 10^{-4}$  at 85°, natural logs being used and the time measured in secs. A. P. Lothrop

Myoglobin. I. The solubility of myoglobin in concentrated ammonium sulfate solutions. Vincent C. Morgan. *J Biol Chem* 112, 557-63 (1935). —The soly of carboxymyoglobin from horse heart in concd solns of  $(\text{NH}_4)_2\text{SO}_4$  at  $\text{pH}$  6.6° and 25° is adequately expressed by the equation  $\log S = 8.00 - 0.94(\text{r}/2)$ , where  $\text{r}/2$  is the ionic strength per l. It is quite sol in buffer solns at  $\text{pH}$  6.6 up to a phosphate concn. of at least 3 M, in which soln the soly of carboxymyoglobin is less than 1 p p m. The great difference in soly. makes possible a method for their characterization and quant sep and emphasizes the great difference between these proteins. A. P. Lothrop

Enzymic histochemistry. I. Distribution of arginase activity in rabbit kidney. Leopold Weil and J. Owen Ely. *J Biol Chem* 112, 565-77 (1935), cf C A 28, 6731. —The microarginase method of Linderström-Lang, Wedl and Holter is suitable for histoenzymic studies. The arginase activity of the rabbit kidney is specifically connected with the cells of the proximal convoluted tubules. No correlation was found between arginase activity and cells of the other structural elements of the rabbit kidney. The medulla is entirely free of arginase and does not contain any inhibitor of this enzyme. A. P. Lothrop

The enzyme synthesis from thyroid duodotyrosine peptone of an artificial protein which relieves myxedema. Wm T. Salter and Olof H. Pearson. *J Biol Chem* 112, 579-89 (1935). —"Thyroglobulin from human thyroids was subjected to peptic digestion and, after removal of thyroxine, a soln of duodotyrosine peptone was obtained which was calorimetrically inert in standard dosage. After concg the peptone, it was subjected to a peptic synthesis which reversed the original digestion process. The artificial protein so prepd was an I-contg substance of large mol. size, with chem. properties somewhat resembling the natural thyroid protein. It was found clinically to relieve myxedema as effectively as thyroglobulin in equiv I dosage. As judged by modern analytical methods it contained an appreciable I fraction resembling thyroxine. These results suggest the following conclusions. The 'inactive' duodotyrosine-contg fraction of peptone produced by peptic digestion of thyroglobulin is a potential source of active hormone. From this peptone an artificial protein can be obtained by protease synthesis ('cse' action) which reverses the better-known proteolytic phenomenon (digestion). The artificial protein resembles

1 natural thyroglobulin in its chem and biol. properties." A. P. Lothrop

The chemistry of human epidermis. II. The isoelectric points of the stratum corneum, hair and nails as determined by electrophoresis. Vernon A. Wilkerson. *J Biol Chem* 112, 329-35 (1935), cf C A 29, 1161. —The electrophoretic mobilities of skin, hair and nails suspended in a series of buffers were deid in a modified Northrop-Kunitz microelectrophoresis app. The curves of mobility in  $\mu$  per sec. per v. per cm plotted against  $\text{pH}$  were fairly const. From this the following isoelec. points were assigned, skin 3.70, hair 3.67, nails 3.78. It is suggested that "since the isoelec. points were practically the same and the basic amino acids were present in approx the same mol. ratios, possibly the amino acids responsible for the acidic groups were also present in a definite mol ratio in these 3 chemically, physically and embryologically related structures. It is further suggested that the adherence of colloidal particles in suspension to the skin would depend upon the  $\text{pH}$  of the dispersion medium, the charge that the skin assumed in contact with the colloidal suspension and the charge on the suspended particle." A. P. Lothrop

The influence of phosphorus on fibroblast culture. Kenkichi Saito. *Folia Pharmacol Japon* 21, Opera Orig 187-91, Breviaria 49-50 (1935). —Small doses of P dissolved in gum arabic soln. increased the growth of fibroblast, but large doses decreased the growth, the cells finally drying. Histological changes are discussed. G. H. W. Lucas

Experimental studies on the cumulative action of sublimate, strychnine and arsenous acid on cultures of iris epithelium in vitro and their observed after-effects. Kenkichi Saito. *Folia Pharmacol Japon* 21, Opera Orig 192-206, Breviaria 50-1 (1935). —These poisons when applied to cultivated tissues in weak concn. through 14 passages show that at first the growth is accelerated, but later owing to an accumulative action of the drugs growth is inhibited and the tissue dies. G. H. W. Lucas

The influence of boric acid and borax on the growth of fibroblast as well as iris epithelial cultures, together with an investigation of the morphological changes following the administration of these drugs. Michitada Maeda. *Folia Pharmacol Japon* 21, Opera Orig 213-22, Breviaria 52 (1935). —Boric acid and borax, when given in low concn. first accelerate growth in fibroblast and iris epithelial cultures, but with increase in concn. growth ceases and the cells are damaged. Morphological changes are discussed. G. H. W. Lucas

The chemical mechanism of glucolysis in the brain. F. P. Mazza and C. M. Valeri. *Boll. soc. ital. biol. sper* 10, 725-7 (1935). —The purpose of the investigation was to see whether the principal chem. reactions which characterize glucolysis of muscle and alc. fermentation (Emden, Meyerhof) also occur by the action of brain enzyme exts. Rabbit brain exts were inactivated by allowing them to stand at room temp for 3 hrs and then dialyzed. When added to fructosediphosphoric ester, triosephosphoric esters were produced, thus showing the presence of zymase in the brain. By the action of brain pulp on starch solns in the presence of lactates, and on hexosediphosphates in the presence of fluorides,  $\beta$ -phosphoglyceric acid was formed (isolated as the Ba salt). Therefore, the 2nd reaction in the Emden-Meyerhof mechanism takes place in the brain. Brain exts prepd in a manner similar to that used by Meyerhof for muscle exts act on phosphoglyceric acid by splitting off  $\text{H}_2\text{PO}_4$  and producing pyruvic acid, if  $\alpha$ -glycerophosphoric acid is also present there is a marked production of lactic acid. In the presence of fluorides, the scission of phosphoglyceric acid is inhibited, but if the exts act on a mixt of  $\alpha$ -glycerophosphoric acid and pyruvic acid, the latter is transformed completely into lactic acid and the reaction is not inhibited by fluorides. Particularly noticeable is the fact that  $\text{H}_2\text{PO}_4$  is not liberated. Like the glycolytic enzymes of muscle the enzymes of brain need a coenzyme (the same as that of muscle) brain exts when dialyzed lose their activity. ICH-CO-H partially inhibits the formation of lactic acid when added

to a mixt. of brain exts., phosphoglyceric acid and glycerophosphoric acid or to brain exts., pyruvic acid and phosphoglyceric acid. In the last case, it does not completely inhibit the scission of phosphoglyceric acid with liberation of  $\text{H}_2\text{CO}_3$ , but does inhibit much more so the production of lactic acid.

Peter Masucci

**The proteins of the cowpea (*Vigna sinensis*)** William H. Adolph and Hsien-Ching Chiang *Chinese J. Physiol.* 9, 347-54 (1935).—Five protein fractions have been isolated as follows: *viscin* (globulin A) 45%, *glutinin* 25%, *albumin* 15%, *globulin B* 10% and *globulin C* 5%. N distribution studies by the Van Slyke method showed that the globulins and albumin are rich in cystine and that the *glutinin* is rich in lysine.

J. A. Maynard

**The isolation of a crystalline protein possessing the properties of aneba-mosaic virus** W. M. Stanley *J. Biol. Chem.* 31, 62-3 (1936).—The prepn was obtained by the methods used in the isolation and purification of tobacco mosaic, and the needles are similar. The N content and infectivity remained unchanged after 4 successive crystallizations. The material is between 100 and 1000 times more active than ordinary juice preps.

John T. Myers

**A quantitative study of the phytoplankton in the Bay of Fundy and the Gulf of Maine** (including observations on hydrography, chemistry and turbidity) H. H. Gran and Trygve Braarud *J. Biol. Board Can.* 1, 279-407 (1935). This investigation was undertaken to det. what effect the construction of dams across the entrances of Passamaquoddy Bay might have upon the production of phytoplankton in the Bay of Fundy. During the summer of 1932 samples were collected monthly from 27 stations, 17 in the Bay of Fundy and 10 in the Gulf of Maine. These were taken at depths of 1, 10, 25, 40 and 75 m. The P and nitrate N contents of the surface waters were higher in the Bay of Fundy than in the Gulf of Maine. In the latter region the waters were supersaturated with  $\text{O}_2$  as far down as the photosynthesis of the phytoplankton was predominant, viz., to a depth of about 40 m. offshore and to a lesser depth near the shore. During the same periods waters were subsaturated in the Bay of Fundy. The lower production of phytoplankton in the Bay of Fundy is considered to be due to the high turbidity of the waters caused by the extraordinarily high tides of the region. The construction of the dams would not be expected to affect the productivity of the Bay of Fundy outside of the confined area, whereas regions inside might be benefited. Details are given of phytoplankton population.

J. R. Noller

**The permeability of the mammalian erythrocyte to deuterium oxide (heavy water)** Arthur K. Parpart *J. Cellular Comparative Physiol.* 7, 153-61 (1935).— $\text{D}_2\text{O}$  (99.5%) penetrates bull and rat erythrocytes 14% more slowly than  $\text{H}_2\text{O}$ . The 11% lower mobility and 31% higher viscosity of  $\text{D}_2\text{O}$  as compared to  $\text{H}_2\text{O}$  may account for the difference.  $\text{D}_2\text{O}$  in this concn appears to have an inhibiting effect on erythrocyte permeability.

T. H. R. Rider

**The permeability of erythrocytes to deuterium oxide (heavy water)** S. C. Brooks *J. Cellular Comparative Physiol.* 7, 163-71 (1935).—With equal concn gradients  $\text{D}_2\text{O}$  penetrates sheep erythrocytes more slowly than  $\text{H}_2\text{O}$ . This is attributed in large part to fugacity differences, aside from which there may be little difference in intrinsic penetrability.

T. H. R. Rider

**Osmotic properties of the erythrocyte** VII. The temperature coefficients of certain hemolytic processes M. H. Jacobs, H. N. Glassman and Arthur K. Parpart *J. Cellular Comparative Physiol.* 7, 197-225 (1935).—cf. C. A. 28, 67-77. The rapid hemolysis of erythrocytes of man, rat, mouse, rabbit and guinea pig is strongly retarded by  $\text{CO}_2$  and characterized by a low-temp. coeff., while erythrocytes of ox, sheep, horse, pig, dog and cat are slowly hemolyzed without retardation and with high-temp. coeffs. by isotonic glycerol solns. Times for hemolysis by glycerol, ethylene glycol and thionin for different species at const. temp. parallel the resp. temp. coeffs.

T. H. R. Rider

**The effect of x irradiation on auxin and plant growth** I. L. Skoog *J. Cellular Comparative Physiol.* 7, 227

70 (1935).—Auxin is inactivated by moderate dosages of hard x-rays, through oxidation resultant upon such irradiation. The inactivation in water requires the presence of  $\text{O}_2$ . Similar results are effected by white light in the presence of co-in. Extid. and synthetic hetero auxin ( $\beta$ -indoleacetic acid), auxin A and auxin in the plant are similarly inactivated. Other results indicate that the effect of irradiation on auxin and its formation is the major factor in the immediate inhibition of growth.

**The inhibitory action of eserine upon choline esterase in vivo** Maxwell S. Jones and Henry Fod *Biochem. J.* 29, 2212-52 (1935).—It was shown by the detn. of choline esterase (I) in the blood of patients after response to xerine (II) injections that II inhibited the action of I on acetylcholine. Pilocarpine and adrenaline caused no lowering of the activity of I. Conclusion: II acts on the parasympathetic by virtue of its inhibition of I, leading to a prolongation of the action of the acetylcholine present.

L. W. Scott

**Action of halogenated organic compounds on living cells** I. Guryov *Bull. Assoc. diploms. microbial. faculté pharm. Nancy* No. 8, 6-13 (1934).—A review on inhibition of fermentation and toxic action in general, with 24 references.

W. C. Tobie

**Action of mass and surface in the phenomena of life** G. Bohn *Conf. lab. microbial. faculté pharm. Nancy* No. 6, 20-37 (1934).—An increased no. of organisms in a soln. may be able to resist the action of poisons to which a smaller no. would succumb, or may succumb more rapidly due to toxic compds. produced by the death of the most susceptible individuals. The effect of hydroquinone on varying concns. of sea-urchin sperm, and of colloidal Ag on varying nos. of frog tadpoles illustrates autoprotection, while the increased mortality of increased nos. of *Coronula* in dil. sea water or KCl soln. illustrates autodestruction.

W. C. Tobie

**Inhibitory effect of phlorizin on an enzymic dismutation** H. Kalkar *Nature* 136, 872-3 (1935).—cf. C. A. 27, 5425, 28, 3746. The dismutative conversion of triosephosphate into phosphoglyceric acid and glycerophosphoric acid is inhibited 70-100% by phlorizin in concns. of about 0.01 N.

I. D. Walter

**Influence of heavy water upon the activity and upon the stability of pancreatic amylase** M. L. Caldwell, S. I. Doeblich and S. H. Mamin *J. Am. Chem. Soc.* 58, 81-7 (1936).—By working with highly purified  $\text{D}_2\text{O}$  and with highly purified preps. of pancreatic amylase (I), it has been found that 100%  $\text{D}_2\text{O}$  has no marked influence upon the hydrolysis of starch by I provided the conditions of the expts. are such as to minimize the deterioration of the enzyme and that the inactivation of I is more rapid and more pronounced when the I is held at 25° in highly purified  $\text{D}_2\text{O}$  than in similarly purified ordinary  $\text{H}_2\text{O}$ .

C. J. West

**Emulsin XXIV. Glucoside synthesis with emulsin** H. Hellerich and U. Lampert *Ber. 68B*, 2050-1 (1935).—cf. C. A. 30, 61. —I've g. glucose in 500 cc. 50% MeOH at 20° with 1 g. almond emulsin which has a glucosidase value of 1.2 g. will reach equl. in 41 days. If the enzyme value is 5, equl. is reached in 13 days. Increase in temp. hastens equl. PhCH<sub>2</sub>OH in place of MeOH will allow equl. at 20° in 12 days, at 37°, 9 days. If the enzyme value is 10, equl. will be reached in 1 day. The higher the aliphatic alc. up to C<sub>4</sub> the faster equl. will be reached.

Julius White

**Phosphatide autocomplex concentrates as ionic systems and their relation to the protoplasmic membrane** (Jong, Bonner) 2. Odor of HCN (Meyer) 2. D as a research tool in the biol. sciences (Johnston) 2.

## B—METHODS AND APPARATUS

STANLEY R. BENNETT

**Dioxin solutions as colorimetric standards for the determination of carotene** Harry N. Holmes and Werner H. Bromund *J. Biol. Chem.* 112, 47-51 (1936).—cf. C. A. 29, 7322. —Dioxin in  $\text{C}_6\text{H}_6$  soln. gives a stable color which matches well that given by carotene in  $\text{C}_6\text{H}_6$  and a color-

metric method for the detn of carotene in  $C_4H_8$  and  $CHCl_3$  solns where the Willstätter-Stoll method cannot be used has been devised with bixin as the color standard.

A. P. Lothrop

The microdetermination of ferrocyanide in muscle and urine J. Graham Edwards and Wilson D. Langley *J. Biol. Chem.* 112, 469-75 (1936).—A method is described for the detn of sol ferrocyanide in pure soln and in urine and minced muscle in ams of 1 to 40 mg. About 98% recovery can be made from pure solns, urine and blood and about 90% from muscle. The ferrocyanide is converted into HCN by hydrolysis with  $H_2SO_4$ , the liberated HCN is aerated into a soln of NaOH and titrated with  $AgNO_3$  in the presence of KI as the end-point indicator. So far the method has not been applied to the detn. of insol. ferrocyanides

A. P. Lothrop

Indole VI Method for determining free indole in small quantities of blood E. Macchia *Boll. soc. ital. biol. sper.* 10, 723-5 (1935), cf. C. A. 30, 1442.—Add to 2 cc serum, 0.20 cc methanol and 12 cc. purified petr. ether. Agitate vigorously for 2 min., allow to stand until the solvent seps, draw off the solvent and wash it with 2 cc. distd. water. (If the serum is steric, first wash the solvent with a concd. alkali, then 3 or 4 times with distd. water). To 10 cc of the solvent, add 2 cc. of Macchia's reagent prep'd freshly as follows: 0.05 cc of a 1% alc. soln of *p*-dimethylaminobenzaldehyde, 0.20 cc. sulfosalicylic acid 20% soln and 1.75 cc. glacial AcOH. Mix, evaporate the solvent on the water bath at 70-80°, cool and bring the colored liquid to 2 cc. with AcOH. Det. indole photometrically or colorimetrically with a series of standard tubes prep'd as follows: agitate 1 cc. of an aq. soln. of indole (1 mg. %) 2 min. with 20 cc. petr. ether. When the liquids sep., transfer 10 cc. of the petr. ether to a test tube and add 2 cc. of Macchia's reagent. Evap. the solvent and make up the colored liquid to 5 cc. with AcOH. Transfer 0.10 cc., 0.20, 0.3, . . . 1.00 cc. to a series of test tubes. Make up each to 2 cc. with AcOH. This technic was used to det. free indole in the blood serum of 60 normal individuals having a normal indican titer. The amt. of free indole found was 0.006 to 0.008 mg. %

Peter Masucci

Hydrochloric acid as reagent for proteins and their derivatives Hugo Köhl *Pharm. Zentralhalle* 76, 625-9 (1935).—The simplest substance yielding the HCl (color) test is furfural, the closely related pyrrole yields the HCl test only with aldehydes contg. a Me group; furfural and pyrrole can be formed through a cleavage of diketone compds., and these products occur, according to protein investigators, among the degradation products of protein aldehydes and ketones in combination with HCl do not give a characteristic violet color, although they cause a change in the color, in the presence of ketones there is a red color, and in the presence of aldehydes there is a brown cast. Although carbohydrates contg. aldehyde and ketone groups yield the color tests of aldehydes and ketones, they give no violet color. The HCl reaction accordingly affords a specific test for derivs. of the proteins which contain certain groups in aldehyde or ketone combination. Six references

W. O. E.

Absorption app. for the microdetn. of certain volatile substances. III. Microdetn. of chloride with application to blood, urine and tissues (Conway) I

Desiccating serums, etc. William J. Elser (to Lawrence K. Sager). Can. 354,143, Nov. 12, 1935. Serum or other protein-contg. liquid is dried at approx. 0° and *in vacuo* by cooling a distant part of the app. to a temp. much lower than 0°. The dried product can be restored to its original condition by adding  $H_2O$ .

## C-BACTERIOLOGY

LAWRENCE H. JAMES

Antigenic structure and classification of the bacilli of the *Salmonella* group. G. Hornus. *Rev. immunol.* 1, 448-500 (1935).—A review with bibliography

Eleanor W. J. Butz

1 The somatic antigens and bacterial endotoxins. I. General considerations and technics used M. Borin and L. Mesrobian. *Rev. immunol.* 1, 553-63 (1935).—A review of the methods used by these authors for extg. partial and complete antigens from certain *Salmonella*

Eleanor W. J. Butz

Succinic acid formation by *Escherichia coli*. Succinic acid formation in connection with protein synthesis Moritz Michaelis. *Z. physiol. Chem.* 237, 181-5 (1935).—In cultures of *Es. coli* on a synthetic medium contg. sucrose,  $KH_2PO_4$ , NaCl,  $(NH_4)_2SO_4$ , and traces of  $MgSO_4$ , the yield of succinic acid in percentage of sucrose decreased from 25.5 to 1.1% with increasing concn. of  $(NH_4)_2SO_4$  from 0.02 to 2.1%. No relationship was found between  $(NH_4)_2SO_4$  concn. and the yield of EtOH, lactic acid, AcOH and  $HCO_2H$ . It appears that succinic acid is a cleavage product of the sugar and is then converted by way of fumaric acid into aspartic acid and eventually protein if sufficient  $NH_4$  is present

A. W. Dor

Infectious bovine mastitis. II. Streptococci of chronic bovine mastitis Wayne N. Plastringer, E. O. Anderson, G. D. Brigham and E. H. Spaulding. Conn. Agr. Expt. Sta. (Storrs Sta.), *Bull.* No. 195, 3-19 (1934), cf. C. A. 29, 8106<sup>1</sup>.—Of 208 strains of udder streptococci isolated from chronic cases of bovine mastitis, 204 were divided into 2 principal groups on the basis of biochem. differences

4 Eighty-seven % were identical with *S. mastitidis*, 11% differed markedly from that group both in biochem. reactions and in inability to become permanently established in the bovine udder, while the rest were different from the 1st 2 groups. A plan is given for identifying weakly hemolytic streptococci of bovine origin. III. Methods of control. W. N. Plastringer, E. O. Anderson, G. C. White and L. F. Rettger. *Bull.* No. 197, 3-40 (1934).—In expts. extending over a 4-yr. period on 5 dairy herds, the results show that periodic injections of autogenous herd bacterins fail to bring about complete recovery of affected animals, they reduce but slightly the rate of spread of infectious mastitis, and they aid somewhat in retarding the occurrence of abnormal milk from animals recently affected with the disease. Lab. tests detected mastitis several months before it was revealed by the macroscopic appearance of the milk alone

C. R. F.

6 Etiology of fowl paralysis, leucemia and allied conditions in animals. I. Introduction, history and a bacterial theory of the etiology of these diseases M. W. Emmel. Fla. Agr. Expt. Sta., *Bull.* No. 284, 5-18 (1935).—E. believes the typhoid and paratyphoid groups of bacteria are the primary etiological agents in fowl paralysis and leucemia in animals. II. Intravenous injection of suspensions of *Salmonella aertrycke* in the chicken. *Ibid.* 19-53.—Repeated intravenous injections resulted in hemocytoblastosis and fowl paralysis. C. R. Fellers [Bacterial flora of spoiled eggs] E. Lagrange. *Compt. rend. soc. biol.* 120, 846-8 (1933)

L. E. Gilson

New method for the determination of indole in bacterial cultures E. Macchia. *Diagnostica tec. lab. (Napoli)* *Riv. mensile* 6, 752-7 (1935).—The culture in liquid medium is extd. with  $Li_2O$  After sepn. the  $Et_2O$  is washed and glacial AcOH, 1% alc. soln. of *p*-dimethylaminobenzaldehyde and 20% sulfosalicylic acid are added and the  $Et_2O$  is evaporated. Red-violet color indicates indole, blue indicates skatole. The detn. can be made by comparing colorimetrically with a standard

H. L. G.

The lethal dose of the toxins of some anaerobes for sheep J. H. Mason. *Onderstepoort J. Vet. Sci.* 5, 61-4 (1935).—Cultures of *Cl. septicum*, *Cl. oedematis*, *Cl. botulinum* and *Cl. welchii* type B were grown in Robertson's meat broth at 37°. After filtration, first through pulp and then through a Berkefeld candle, the filtrates were said. with  $(NH_4)_2SO_4$ , the ppt. was blotted and finally dried in vacuum over  $H_2SO_4$ . Saline solns. contg. 100 mg. of ppt. per 5 cc. were prep'd. and injected into sheep and mice. In the order named, the min. lethal doses for sheep were 3.5, 7.0, 0.1 and 1.2, and for mice 0.2, 0.07, 0.008 and 0.02 mg./kg. of body wt.

K. D. Jacob

The antigenic components of the toxins of *Cl. botulinum*

types C and D. J. H. Mason and E. M. Robinson. *Onderstepoort J. Vet. Sci.* 5, 65-75(1935).—Toxic broth filtrates of 1 A, 1 B, 6 C and 1 D types of *Cl. botulinum* (prepd. as described in the preceding abstr.) and the antitoxins produced by injecting these filtrates into goats were tested from the standpoint of working out the antigenic "make up" of the toxins. The results indicated that (1) the A and B types are monospecific, (2) the C types contain 3 components C<sub>1</sub>, C<sub>2</sub> and D, the last being present in only very slight amt. and (3) the D type contains chiefly the D but also a small quantity of the C fraction.

Study of *B. coli* mutable from an outbreak of diarrhea in the newborn. Anna Dean Dulaney and I. D. Michelson. *Am. J. Pub. Health* 25, 1241-51(1935).—Stable lactose-fermenting and unstable nonlactose-fermenting (in that lactose-fermenting descendants continued to be given off) organisms could be derived from all cultures. Dissociation to the lactose-fermenting form occurred under simulation of contact with lactose. Lactose exerted a sp effect, since no other condition of growth produced the same result. Utilization of salicin and raffinose was shown to be similar to that of lactose and depended upon the development of variants capable of fermenting these sugars. Biologically the organisms are closely related to *Es. communior*, but no close antigenic relationship was demonstrated. All strains of *B. coli* mutable isolated from sick babies were found to form a homologous group when tested with sera of rabbits immunized with red and white forms.

J. A. Kennedy. Fermentation of glycerol by gluconic acid bacteria in fruits. The production of dihydroxyacetone, glyceric acid, acetic acid, glycolic acid, succinic acid and a substance which gives a reddish violet color reaction with ferric chloride. Teizo Takahashi and Toshinobu Asai. *J. Agr. Chem. Soc. Japan* 11, 1008-16(1935).—*Glucosacetobacter cerinus* var. *ammoniacus* f. sp. *unshu* *B.* was cultured in 4.21 of yeast ext. contg. 210 g of glycerol and 105 g of CaCO<sub>3</sub> at 20-28° for 90 days. From the ether of the fermentation product, succinic acid 1.2 g, AcOH (as Ca salt) 20 g and glycolic acid (as Ca salt) 6.0 g were obtained. Dihydroxyacetone 82.6 g, l-glyceric acid (as Ca salt) 32 g and a sirup which gave a reddish violet color with FeCl<sub>3</sub> were isolated from the ether-insol. part. Glycerol may be converted into an unknown ketonic acid through dihydroxyacetone, or succinic acid or glycolic acid through glyceric acid and AcOH.

Y. Kihara. A specific polysaccharide from the bacillus Calmette-Guérin (BCG). Erwin Chargaff and Werner Schaefer. *J. Biol. Chem.* 112, 393-405(1935).—Two polysaccharides giving pptns with anti-BCG horse sera have been prepd. from defatted B. Calmette-Guérin (BCG). One (A) is a water-sol., d-rotatory (77.4°), weak acid contg. 77.2% of reducing sugars and 2.9% of amino sugars. Its main components are mannose and d-arabinose with a small amt. of inositol. The other (S) is insol. in H<sub>2</sub>O and alkali and sol. in acids. It is a strong adsorption compd. between equal parts of a polysaccharide, contg. 94% of reducing sugars, and of Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>. A protein was also isolated which showed comparatively weak tuberculin activity.

A. P. Lohrhop. The chemistry of the lipides of tubercle bacilli. XII. Studies on phthioic acid. M. A. Spielman and R. J. Anderson. *J. Biol. Chem.* 112, 750-67(1936), cf. *C. A.* 24, 878; 29, 117.—The Me ester of phthioic acid had been purified by fractional distn. until the specific rotation reached the const. value of 12.2°. The acid prepd. by sapon of the pure ester possesses a branched chain, probably Me groups in the α-position and in the neighborhood of the 11th C atom. Other branches exist but the no and position have not yet been detd. Purified phthioic acid is biologically active and produces typical tuberculous tissue when injected into normal animals. Phthioic acid C<sub>21</sub>H<sub>38</sub>O<sub>4</sub> m. 20-21° and has [α]<sub>D</sub> 12.56°; amide m. 45°; methylamide m. 27°.

A. P. Lohrhop. Microchemical tests on the sulfur bacteria. Achille Monti. *Boll. soc. ital. biol. sper.* 10, 690-1(1935).—

By immersing colonies of *Beggiatoa* in aq. solns. of AgNO<sub>3</sub> of various concns., the endocellular droplets of S are transformed into Ag<sub>2</sub>S. The preps. are stable. They can be washed, passed through EtOH and xylene without changes. They can be stained with safranin to bring out the body of the bacteria and the gelatinous magma in which the bacteria are engulfed without previous fixation in Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. AgNO<sub>3</sub> was found to be superior to Pb acetate, HgCl<sub>2</sub>, PtCl<sub>4</sub>, osmic acid or a satd. sucrose soln.

Peter Masucci. The fermentation of mannitol by *Escherichia coli* and *Bacillus lactis aerogenes*. V. Cianci. *Boll. soc. ital. biol. sper.* 10, 730-2(1935).—The fermentation of mannitol by *Es. coli* is influenced quantitatively by the presence of a phosphate buffer. Increasing the amt. of buffer increases the growth, the rate of growth, the amt. of acid produced and the gas-producing activity. *B. lactis aerogenes* shows an analogous behavior but after incubation for 8 days at 37°, the reaction turns slightly alk. in the buffered cultures.

Peter Masucci. Reactions of R and S forms of bacteria to the treatment of Lustig and Galeotti for the extraction of nucleoprotein. A. Seppilli and R. Vendramini. *Boll. ist. sierot. milanese* 13, No 5(1934), *Rev. sud-americana endocrinol. inmunol. quimioterap.* 18, 760.—While R strains respond to the treatment, S resist lysis by KOH.

A. C. Meyer. Ultrafiltration experiments with the viruses of laryngo-tracheitis and coryza of chickens. Charles S. Gibbs. *J. Bact.* 30, 411-17(1935).—The tracheal and nasal exudates are triturated with distd. water with powder Pyrex glass and 0.01 M citric acid added to the isoelec. point, the cellular debris flocculates and settles or can be centrifuged. The supernatant fluid is neutralized with 1/2 N Na<sub>2</sub>CO<sub>3</sub> to thymol blue, and then filtered through graded collodion membranes.

John T. Myers. Dissimulation of glucose by heterofermentative lactic acid bacteria. M. E. Nelson and C. H. Werkman. *J. Bact.* 30, 547-57(1935).—Lactic, acetic and carbonic acids, alc. and glycerol were the end products of the dissimulation of glucose by 3 representative species of the heterofermentative lactic acid bacteria. The millimols of CO<sub>2</sub> produced were equiv. to the sum of the AcOH and alc.

John T. Myers. Anaerobic bacteria capable of fermenting sulfite waste liquor. A. M. Partansky and B. S. Henry. *J. Bact.* 30, 559-71(1935).—The absence of proteolytic action and the ability to attack a large no. of carbohydrates, as well as the production of butyric acid as one of the end products, indicated a relationship of all 5 of the species usually grouped under the name of *Clostridium butyricum*. However, a new species was found which differed from *Cl. butyricum* morphologically, culturally and physiologically.

John T. Myers. Growth stimulants for certain Rhizobia. W. B. Sarles and J. J. Reid. *J. Bact.* 30, 651(1935).—Potato ext. stimulated growth. Asparagine stimulated growth also but to a lesser degree.

John T. Myers. Activation of the lower fatty acids by propionic acid bacteria. R. W. Stone, H. G. Wood and C. H. Werkman. *J. Bact.* 30, 652-3(1935).

John T. Myers. The relation of oxidation-reduction potential to the growth of an aerobic organism. Wm. B. Wood, Jr., Mary Lee Wood and I. L. Baldwin. *J. Bact.* 30, 593-602(1935).—Reversible oxidation-reduction indicators positive to, and including, methylene blue inhibit the growth of *B. megatherium* when in the oxidized form. Indicators negative to methylene blue in the electromotive series fail to inhibit growth. None of the compds. which inhibit growth in the oxidized form is bacteriostatic when reduced. Potentiometric measurements indicate that the compds. studied effect growth only during the lag phase and sufficient indicator was added to the medium to have an appreciable poisoning effect. Suitable amts. of an inorg. reducing agent favor the growth of *B. megatherium*.

John T. Myers. A chemical-biological study of *Escherichia coli* and three of its rough variants. Earnest H. Rennebaum. *J. Bact.*

30, 625-38(1935).—Three rough variants of *Es. coli* were developed with marked differences between each other and between them and the parent strain. As a source of energy the parent strain more readily utilized acetic, lactic and malic acids, while the variants preferred tartaric, citric and salicylic acids. The injection of a lethal dose of the parent strain into the ear vein of a rabbit caused a sudden rise in blood sugar with death in a few hrs., while the variants had no effect. It is believed that the fatal agent is a sol. exotoxin, which produces death by making the walls of the lung capillaries suddenly permeable to the noncellular elements of the blood. J. T. M.

Growth and longevity of *Rhizobia* on agar containing various energy sources. J. J. Reid and W. B. Searles. *J. Bact.* 30, 651(1935).—Sucrose was better than mannitol. John T. Myers.

Dissimilation of glycerol by intermediate bacteria of the colon-aerogenes group. Howard Reynolds and C. H. Werkman. *J. Bact.* 30, 652(1935).—The formation of pyruvic acid may be useful in differentiation. John T. Myers.

Dissimilation of glucose by the lactic acid bacteria. M. E. Nelson and C. H. Werkman. *J. Bact.* 31, 4(1936).—Typical heterofermentative lactic acid bacteria yielded  $\text{CO}_2$  equiv. to the sum of  $\text{HOAc}$  and alc. Pyruvic acid was an intermediary in the fermentation of lactic acid. Levulose is reduced to mannitol,  $\text{AcH}$  to alc. and acetylmethylcarbinol to 2,3-butyleneglycol. Glycerol is probably formed by hydrogenation of a 3-C intermediary. John T. Myers.

Aerobic dissimilation of lactic acid by the propionic acid bacteria. H. G. Wood, Carl Lrb. and C. H. Werkman. *J. Bact.* 31, 5-6(1936).—*Propionibacterium arabinosum* in buffered phosphate media at  $30^\circ$  showed active utilization of O, lactic acid being converted to pyruvic, acetic, propionic and carbonic acids. The C of the lactic acid fermented was completely accounted for in the products. The oxidation-reduction balances were satisfactory, if it is assumed that O is hydrogenated to water. O may function as a H acceptor in the propionic dissimilation. This is added evidence of the intermediary behavior of pyruvic acid in propionic acid dissimilation. John T. Myers.

The similarities between the specificities of disinfectants against a filterable virus and Gram negative organisms. Earnest C. McCulloch. *J. Bact.* 31, 7(1936).—There was close parallelism among the relative efficiencies of phenol, liquor cresolis,  $\text{HCHO}$ ,  $\text{NaClO}$ ,  $\text{NaOH}$  and several dyes against the virus of fowl pox and against Gram-negative organisms grown in a medium containing fecal debris. John T. Myers.

The relationship of certain respiratory enzymes to the maximum growth temperature of bacteria. O. F. Edwards and L. F. Rettger. *J. Bact.* 31, 12-14(1936).—A thermostable peroxidase was found in each of 106 strains of the genus *Bacillus*. There was no evidence of  $\text{H}_2\text{O}_2$ . John T. Myers.

Oxidation-reduction potentials of certain anaerobic and facultative anaerobic bacteria. I. E.  $p_n$  relationship and a possible explanation for the phenomenon of double reversion of potential during the apparent logarithmic phase. II. Preliminary observations on the differentiation of lactobacilli of intestinal and buccal origin. R. W. H. Gillespie and L. F. Rettger. *J. Bact.* 31, 14-15(1936).—A buccal strain grown in unbuffered tomato juice was characterized by a marked activity with respect to growth and acid production and had a reversal of the drift before the final maximum reducing level was reached. Shortly thereafter the negative drift was resumed. This did not occur with two strains of *L. acidophilus*. In a buffered medium of low carbohydrate content, strains of oral and intestinal origin were differentiated by a wide margin of final reduction intensity which if substantiated should make their differentiation by means of the dye indicators practical. John T. Myers.

Metabolic activity and cell volume of *Salmonella galinarum* at various phases of the culture cycle. Evelyn Huntington. *J. Bact.* 31, 15-16(1936).—Rates of  $\text{CO}_2$

production per unit vol. of bacterial protoplasm show an increase in the lag and early logarithmic phases over the initial and terminal rates as do the rates calcd. per cell. John T. Myers.

Oxygen consumption by bacterial cultures. C. P. Hegarty. *J. Bact.* 31, 18(1936).—There were considerable differences between the amts. of  $\text{CO}_2$  produced by various streptococci and lactobacilli. John T. Myers.

Factors controlling pigment production by *Mycobacterium phlei*. Mary A. Ingraham. *J. Bact.* 31, 18-19(1936).—*M. phlei* may contain as many as 10 carotenoid pigments. A number of sugars, sugar alcs., acids, aldehydes, fats and miscellaneous compds. including phytol, beta-ionone and a vitamin A concentrate all failed to improve pigmentation. Ethylene, salts of  $\text{Sc}$ ,  $\text{Na}$ ,  $\text{Li}$ ,  $\text{Ca}$  and  $\text{Mg}$ , the oxidation-reduction potential, light intensity, and temp. of incubation were without sp. effect on pigmentation. John T. Myers.

The fermentation of acetylmethylcarbinol by the *Escherichia-Aerobacter* group and its significance in the Voges-Proskauer reaction. Ralph P. Tittler. *J. Bact.* 31, 21(1936).—The frequent failure to obtain pos. Voges-Proskauer reactions in old cultures of some *Aerobacter aerogenes* and *Aerobacter oxytumor* strains is due to fermentation of the acetylmethylcarbinol. J. T. M.

The nature of the change from slow to rapid lactose utilization by a member of the colon intermediate group. C. K. Wadsworth and E. R. Hitchner. *J. Bact.* 31, 22-3(1936).—The acquisition by the slow lactose fermenters of the ability to use lactose rapidly is abrupt rather than gradual. John T. Myers.

Hydrogen sulfide production as a differential test in the colon group. Reese Vaughn and Max Levine. *J. Bact.* 31, 24(1936).—In the presence of cysteine all strains were positive. The concn. of peptone was not significant. Many more strains were positive in the absence of agar-agar; the cause of this is unknown. John T. Myers.

Reaction of *Escherichia*, *Aerobacter* and *Citrobacter* strains in boric acid and hexamine media. M. T. Bartram and L. A. Black. *J. Bact.* 31, 24-5(1936).—Hexamine did not exert sufficient inhibitive action to be useful. The results with boric acid media agree with those of Levine. John T. Myers.

The effect of sodium selenite on the growth of bacteria and its use as a basis for a new enrichment medium for the isolation of typhoid bacilli from feces, water, milk, etc. Einar Lefson. *J. Bact.* 31, 26-7(1936).—Most organisms except typhoid, cholera, proteus and *Pseudomonas aeruginosa* are inhibited. John T. Myers.

The protein-sparing action of carbohydrates in relation to anaerobic identification. Robb S. Spray and Alfred R. Stanley. *J. Bact.* 31, 27(1936).—Fermentable carbohydrates have a "protein-sparing" action, but individual peculiarities may obscure its action in sp. instances. John T. Myers.

The preparation and properties of silicic acid gels for the pure-culture isolation of bacteria. John H. Hanks and R. W. Wentraub. *J. Bact.* 31, 29-30(1936).—Most effective control of the  $p_n$  was secured with 4 times the usual concn. of  $\text{NH}_4$  salt and by dialyzing against the salt soln. minus the  $\text{NH}_4$ . John T. Myers.

Increase in toxicity due to the action upon the broth constituents by endo-enzymes of the *Salmonella* bacteria. Frances L. Kraft and C. N. Stark. *J. Bact.* 31, 42-3(1936).—Something in the nature of a heat-stable endo-enzyme complex is released by brief subjection to  $100^\circ$ , or by natural autolytic processes, and can act on the broth to produce a gradual increase in  $p_n$  and in toxicity. It probably consists of split products from the medium. John T. Myers.

The chemistry of diphtheria toxin. Monroe D. Eaton. *J. Bact.* 31, 64-5(1936).—The toxin appears to be a protein of more complex structure than the proteases in the medium on which it was formed, suggesting that it is synthesized by the bacillus. The immunological and physiol. properties of diphtheria toxin are due to a single substance. John T. Myers.

Further observations on pneumococcal hemolysis and

its inactivation Barnett Cohen and Harry Schwachman *J. Bact.* 31, 67-8(1936).—It may be possible to define a level of oxidation-reduction potential characteristic for pneumococcus (type II) hemolysin, a reversible oxidation system with the characteristics of an enzyme. The lytic activity seems to be assoc. with a thiol grouping.

John T. Myers  
Studies on the production of toxin by *Clostridium histolyticum*. Sarah E. Stewart *J. Bact.* 31, 70-1(1936).—Glucose added to the culture broth increased the potency of the toxin by the production of a hemolysin, but the role of the glucose is unknown. The proteolytic enzyme was not increased, and is independent of the exotoxin showing no decrease in nontoxic formalinized toxins.

John T. Myers  
Conditions affecting the production of toxin and "porphyrins" by the diphtheria bacillus. Mary W. Wheeler and M. O. L. Crowe *J. Bact.* 31, 73(1936).—Porphyrins were removed from diphtheria toxin without altering its toxicity by adsorption with charcoal but with no other adsorbent tried. They could not be eluted from the charcoal.

John T. Myers  
The reducing action of diphtheria bacilli. Otfried Ehrismann *Zentr. Bakt. Parasitenk.* 1 Abt. 135, 56-60 (1935).—The reducibility of dyes depends on chem. constitution as well as soly. *C. diphtheriae* reduces telurites and nitrates. Cyanide does not interfere with these reactions.

John T. Myers  
Should the term oxychlorophyll be retained? Ph. Lasseur. *Trav. lab. microbiol. faculté pharm. Nancy* 7, 21-9(1934).—A review on chlorophyll, the green pigment of *Pseudomonas chlororaphis*. Phenazine- $\alpha$ -carbonamide is perhaps a better name for the dehydrogenated form of the pigment than the term oxychlorophyll.

W. C. Tobie  
The production of chlorophyll by *Pseudomonas chlororaphis*. Ph. Lasseur. *Trav. lab. microbiol. faculté pharm. Nancy* 7, 31-40(1934).—Washed suspensions of 40-hr. cultures of *Pseudomonas chlororaphis* produce abundant crystals of the green pigment chlorophyll after 7 days, especially at  $pH$  6.97 in a layer not over 15 mm deep.

W. C. Tobie  
Autolysis of *B. caryocaneus*. Beijerinck-Duparc 1930. Ph. Lasseur and N. Gröjean. *Trav. lab. microbiol. faculté pharm. Nancy* 7, 41-3(1934).—Washed suspensions of *Bacillus caryocaneus* (20 billion per cc.) in dist. water were sealed in Pyrex glass containers and allowed to autolyze for 51 days at 37°. The cond. of the suspensions increased, while the  $pH$  fell from above 7.05 to 6.27.

W. C. Tobie  
Media for the detn. of the coli-aerogenes group in water analysis (Ruehboft) 14

## D—BOTANY

THOMAS G. PHILLIPS

The emission of additional roots by grape vines. L. Hédin. *Compt. rend. acad. agr. France* 21, 867-81 (1935).—The H-ion concn in the vicinity of the meristems plays an important role in the evolution of addnl. roots. The mineral compn. of roots as well as various sections of the stem is given; they show that CaO and K<sub>2</sub>O remain in abundance in the roots of the vine (3 yr. old) and the lower twigs have a higher CaO and MgO content than the upper ones. Salts of Ca and Mg favor the development of roots while the salts of K, especially KCl, act especially on the lenticels. In certain combinations (carbonate, nitrate, chloride) and proportions, Ca salts can hinder the development of root hairs of certain plants in a liquid medium.

J. R. Adair  
The magnesium content of grasses and legumes and the ratios between this element and the total calcium, phosphorus and nitrogen in these plants. Harlev A. Daniel *J. Am. Soc. Agron.* 27, 922-7(1935).—The av. Mg content of 19 species of mature grass was 0.16% and that of the 45 mature legumes 0.37%. The Mg content of legumes decreased as the plants matured. The Ca/Mg ratio

varied in the mature grasses and legumes from 1.10 to 5.46, the P/Mg ratios from 0.09 to 2.42, and the N/Mg ratios from 2.09 to 22.00. The av. of these ratios was slightly higher in the legume tops collected at different stages of growth than in the roots. Very little relation existed between the chem. compn. of the tops of the plants and that of the roots.

J. R. Adams  
Characteristics of the amylase of cabbage. B. A. Rubin and V. E. Trupp. *Compt. rend. acad. sci. U. R. S. S.* [N. S.] 3, 229-32(1935).—Cabbage leaves are 50, 15 and 5 times richer in amylase (I) than beets, onions and carrots. This high activity is of special interest, since the starch content of this vegetable is inconsiderable and it is probable that this so-called amylase is an enzyme that is not characterized by a strictly sp. action or that it is a complex of sp. enzymes. The loss of cellulose in connection with an increased activity of I suggests that in cabbage I is accompanied by an enzyme, cyase (II) capable of hydrolyzing cellulose itself or some of the other carbohydrates forming the cell walls. Studies of the activity of amylase in parallel with that of II established the existence in cabbage of an enzyme capable of hydrolyzing hemicellulose to a reducing sugar. II is considerably less active than I but is comparable to sucrase. Its activity in the leaves and stalks varies and this is connected with the ability of the early variety, No. 1, to "awaken" early and with the general higher toxicity of this variety. Attempts to isolate II are in progress.

C. R. Addinall  
Physiology of tanning materials in plant cells. Wilhelm Hauser. *Protoplasma* 24, 219-24(1935).—In order to investigate the mode of action of tanning materials in plasma, the conditions of the expts. must be so controlled that their pptg. action on protein-like substances is prevented. As the weak alk. reaction of plasma in the presence of salts must be considered, a method was chosen for neutralizing the tannin soln. by which its pptg. action on gelatin was prevented. Tannin in this form prevents the aggregation of the particles of a gelatin soln.

F. L. Dunlap  
The influence of salts and hydrogen-ion concentration on plasmolysis and deplasmolysis. V. S. P'in. *Protoplasma* 24, 296-318(1935).—The leaf epidermis of various plants was plasmolyzed by means of a sucrose soln. At the end of the expts., the concn. rose to 2 g. mol., that is to an osmotic pressure of 121 atm. If the cells were deplasmolyzed in a short time, they remained for the most part alive. If they remained more than 20 hrs. in the plasmolyzing soln., deplasmolysis was much more difficult and with many plants it was impossible. The aim of these expts. was to det. the conditions under which a protoplast could be plasmolyzed strongly and for a long period and yet remain alive. The reaction of the solvent played a very important role.

For the regulation of the H-ion concn., phosphate, citrate, acetate and bicarbonate were used as buffers in the form of their K salts. The plants could be divided into various groups according to their reaction to the soln. Some were rather indifferent to the H-ion concn. and could be deplasmolyzed to the end at a  $pH$  of 4.5-5.1. Others could not stand strongly acid solns. but could live in weakly acid, neutral or alk. solns. Others deplasmolyzed only in alk. solns. of a  $pH$  of 7.0 and higher. Finally there were plants for which a strongly alk. medium was harmful; these died at a  $pH$  of 7.4-8.0 and weak acid or neutral solns. were beneficial. Of greater significance is the presence of inorg. salts and perhaps other supplements. Natural spring water maintains the cells particularly well, while so-called nutrient solns., prep. with dist. H<sub>2</sub>O, are considerably more harmful. It is better to obtain a detd.  $pH$  with KHCO<sub>3</sub> than with phosphates. Citrates and acetates give better results than phosphates. K salts in great amt. are harmful to many plants, while for others the K ion is not harmful but advantageous, and they deplasmolyze the better. For the majority of plants K ions are relatively harmless and are harmful only in large amts. Some plants react strongly to the Na ion and often a weak concn. such as 0.01 and 0.03 g. mol. will thwart deplasmolysis. Some kinds can stand 0.1-0.2 g. mol. NaCl without harm. Plasmolysis

and deplasmolysis must be carried out through a series of graduated concns. It is recommended that the strengths of the solns be calcd according to their osmotic pressures *in situ*, rather than in terms of *g. mols*, for the osmotic pressure of the stronger solns increases more rapidly than the *g. mol* concn. This difference varies with different plasmolytic agents and depends on the size of the mols. The duration of the plasmolysis or deplasmolysis plays a very important role. When the exact conditions for plasmolysis and deplasmolysis have been detd, such as the  $p_H$  of the soln, chem. compn of the soln, time, difference in concn, etc., it is possible with almost all types of plants to obtain complete deplasmolysis without endangering the tissue in the least. F. L. Dunlap

**Death of plant cells in pure and balanced salt solutions.** V. S. Il'in *Protoplasma* 24, 409-30 (1935).—Complete death of all cell portions in pure K and Na salt solns does not begin at the highest concns but at some medium or even weaker one, frequently at 0.3 *g. mol*. The cells remain alive in weaker solns, such as 0.05 or 0.1 *g. mol*, and sometimes in stronger, as 1.0, 2.0 and even 5 *g. mol*. With weak and medium-strength solns death can ensue in 5-7 hrs, in stronger ones it can follow after 20 and even after 70 hrs. The described process of the death of all cell portions in Na and K salt solns, is almost typical for all plants. In  $H_2O$  or in some weak solns, the cytoplasm appears as a homogeneous mass with hardly noticeable granulation, the nucleus is also without structure. Under the influence of stronger concns, the granulation in the nucleus and cytoplasm increases, this results in coagulation with still stronger concns. The difference in the concns which kill the nucleus and the cytoplasm, and that which kills the tonoplast may be considerable. Death of the nucleus and cytoplasm takes place with great rapidity shortly after placing in the soln when it is concd. The tonoplast lives some days in the strongest solns, such as 5 *g. mol* NaCl. The permeability of the tonoplasts for salts is greater than the permeability of the whole cell. Resistant plants are distinguished by a coagulation of the nucleus and cytoplasm at higher concns which never extends to weaker ones, as is the case with less resistant types. A great difference is noticed among the plants in relation to their reaction toward Ca ions; in some all cell portions remain alive at 0.7, others only at 0.3 *g. mol*, while others die at 0.1 *g. mol*. Sixteen references.

F. L. Dunlap

**Effect of sulfur dioxide on wheat development.** Action at low concentrations. Robert E. Swan and Arthur B. Johnson *Ind. Eng. Chem.* 28, 42-7 (1936).—Wheat plants were grown in nutrient solns in 2 cabinets under duplicate conditions of temp, light intensity, humidity and circulating air vol. They were in darkness for 8 hrs and under artificial illumination for 16 hrs daily.  $SO_2$  in concns below those at which typical foliar markings are produced (0.14-0.46 p. p. m. was used) was added to the air several hrs daily. No visible injury was produced. Growth was not retarded. In plants carried through to maturity the yield of grain was slightly greater than that of the control plants. L. E. Gilson

**Study of the bark of *Aspidosperma quirandii* Hassler.** Luis Fiorani *Rev. centro estud. farm. bioquim.* 25, 373-94, 423-47 (1935).—A botanical description and proximate analysis are given. In addn to resins, tannins and other common extractives the bark contains a saponin, provisionally named *quirandii saponin*, and the following alkaloids: aspidospermene, aspidosamine, 2 new cryst. alkaloids, *haslerine* m. 237° and *quirandine* m. 218° and 4 other alkaloids not yet sepd. in pure form. Aspidospermene is not present. The lethal dose of the mixt. of total alkaloids for rabbits is 2.3-3.4 mg/kg. when given intravenously and about 10 times as much when given subcutaneously. The mixt. produces muscular paralysis and kills by causing asphyxia. L. E. Gilson

**The vital role of chemistry in determining essential materials in plants.** W. A. Leukel *Citrus Ind.* 16, No. 11, 6-7 (1935).—A brief discussion of plants as sources of important chem. compds. K. D. Jacob

**Further investigations on the bunchy-top disease of tomato.** A. P. D. McClean *Union S. Africa Dept. Agr., Sci. Bull.* 139, 46 pp (1935).—In aq. ext. the bunchy-top virus was rapidly destroyed when exposed for 10 min to temps. above 70°. Some destruction of the virus occurred between 60° and 70°, but temps. below 60° had no appreciable effect. The virus was short lived in extd. juice and died out rapidly after 12 hrs. No appreciable loss of infective power occurred when the juice was exposed for 1 hr. to alc. in concns up to 30%. Higher concns of alc. appeared to cause some destruction of the virus. K. D. Jacob

**Enzymes of grain. III. The relation between the action of the starch liquefying enzyme of rice and  $p_H$ .** Gohri Yamaguchi *J. Agr. Chem. Soc. Japan* 11, 825-35 (1935).—The action of starch-liquefying enzyme was estd. by the viscosity. The optimum  $p_H$  varied with the conditions. The optimum  $p_H$  of the  $H_2O$  ext. of the unhulled rice was 5.2-5.4 and that of the unhulled rice, the rice bran and the embryo was 4.8. When polished rice was extd. with salt soln. such as 1 *M* NaCl, 1 *M* KCl and 0.1 *M*  $K_2SO_4$ , the optimum  $p_H$  was 6.1-6.2. When polished rice was extd. with salt soln. such as 1 *M* NaCl, 1 *M* KCl and 0.1 *M*  $K_2SO_4$ , the optimum  $p_H$  was 6.1-6.2. When the polished rice was extd. with 1% papayotin soln., the optimum  $p_H$  was 4.8. When the enzyme soln. was obtained from the aq. soln. and the salt ext. of the unhulled rice by dialysis, the optimum  $p_H$  values were 4.9 and 5.0, resp. It was not changed by the addn. of NaCl to this soln. Y. Kihara

**Chemical constituents of kaohang.** Sugao Hirao *J. Agr. Chem. Soc. Japan* 11, 921-4 (1935).—The kaohang was extd. with  $H_2O$  at 80-90°. Pb acetate was added to the ext. The ppt. obtained stood overnight. The supernatant liquor was sepd. and 10% NaOH was added. A yellow ppt. was obtained. The ppt. was suspended in  $H_2O$  and decomposed by  $H_2S$ . The filtrate was extd. with ether. Succinic acid and 2 kinds of white crystals, resp. m. 240-2° and 98-100°, were isolated from the ether ext. The ether-insol. part was further extd. with acetic ether. A yellow glucoside, m. 249-250°, was obtained. It was identified with quercitrin, quercetin monoglucoside. Y. Kihara

**Unsataponifiable matter of algae fats. I.** Sterol Kiyoshi Shirahama *J. Agr. Chem. Soc. Japan* 11, 980-4 (1935).—*Peltsea wrightii* Yendo was extd. with ether. The ether-sol. portion was again extd. with petroleum ether b. 40-50°. The ext. was saponified with 10% alc. KOH soln. Colorless needles were obtained from the unsaponifiable matter. Crystals m. 125° were obtained after several recrystns.  $[\alpha]_D^{20} = -39.0^\circ$  (0.2055 *g.* in 5 cc. of  $CHCl_3$ ). It was designated as *peresterol*. The reaction for ergosterol was negative. It gave no color reaction with  $SnCl_4$  in  $CHCl_3$ . The acetate, colorless hexagonal plates m. 118°, with  $[\alpha]_D^{20} = -44.1^\circ$  (0.2497 *g.* in 5 cc. of  $CHCl_3$ ); propionate, colorless plates m. 104°, benzoate, colorless rectangular plates m. 114°, and bromide, a white powder decomposing at 132°, were prepd. *Peresterol* was also isolated from *Fucus vesiculosus* Ag. and *Laminaria longissima* Miyabe. *Codium fragile* (Sutner) Hariot contains a sterol having sitosterol-like properties, m. 130°. Y. Kihara

**Leaf starch: its isolation and some of its properties.** H. A. Specht and Harold W. Milner. *J. Biol. Chem.* 111, 679-87 (1935).—Leaves collected after they had been exposed to bright light on a warm day were exposed to  $CHCl_3$  vapors to prevent depletion of the starch. After drying in a stream of air at 50° for 2 to 24 hrs, the leaves were ground and passed through a 60 mesh sieve to sep. the stem and fibers and the material was then ground in a pebble mill for 3 hrs. Repeated extn. with  $EtOH$  at room temp. for 5 to 15 hrs. and alternate extn. with  $EtOH$  and petr. ether in the last 3 or 4 extns. removed chlorophyll and other sol. substances. Gums, pectic substances and salts were removed by repeated extn. with  $H_2O$  at room temp. until the wash  $H_2O$  was almost colorless. The starch was then extd. with hot  $H_2O$  by heating in a boiling

water bath for 1 hr and heating at 120° for an hr. The soln remaining after removal of the insol. residue was kept at -8° for 3-4 days and the starch pptd. out on thawing and was removed by centrifuging and filtration on silk. When thoroughly dried from 3 to 18% of the sepd. starch is insol. even after heating to 120° with H<sub>2</sub>O but is hydrolyzable with 1% HCl. There is also always present a small and variable amt of insol. material other than starch (in some cases largely SiO<sub>2</sub> in part at least from the pebble mill). Detns. of specific rotation and prepn of osazones indicate that glucose is the chief product of the hydrolysis of the leaf starch. Max. reducing power is obtained only after 4-5 hrs. of hydrolysis as retrograded starch (pptd. by freezing and then thawing) is more slowly hydrolyzed than "standard" potato starch. A. P. L.

The plastid pigments of the marsh dodder, *G. MacLunney* *J. Biol. Chem.* 112, 421-4 (1935).—Two species of dodder, *Cuscuta subinclusa* and *salina*, were examd. Chlorophyll is present in low concn., except possibly in sharply localized regions.  $\alpha$ -,  $\beta$ - and  $\gamma$ -Carotenes, lycopene and rubixanthine have also been isolated. Rigorous purification of the various components is difficult on account of the unusually high concn. of unsaponifiable colorless material. The marsh dodder (*salina*) is the richest source of  $\gamma$ -carotene known to M., readily available in Cal. A detailed study of *Cuscuta subinclusa* was impossible but there is apparently no major difference in the pigment complex of the 2 species. A. P. Lothrop

The chemistry of the lipides of yeast. III. Lecithin and cephalin. L. F. Salsbury and R. J. Anderson *J. Biol. Chem.* 112, 541-50 (1936), cf. *C. A.* 27, 5779.—Lecithin and cephalin from yeast phosphatides have been purified so that the lecithin was free from amino N and all of the N of the cephalin was in the NH<sub>2</sub> form. Both yield about 64% of fatty acids on hydrolysis and the component acids of both are very similar, consisting of 84-6% of liquid acids. These on catalytic reduction give a mixt. of palmitic and stearic acids. Palmitic and stearic acids are the sole components of the solid acids. Optically active glycerophosphoric acid and choline comprise the water-sol. portion of the hydrolysis products of the lecithin and optically inactive glycerophosphoric acid and aminoethyl alc. from the cephalin. A. P. Lothrop

The action of growth substance on parallelotopic plant organs. A. Th. Czaja *Ber. deut. botan. Ges.* 53, 478-90 (1935); cf. *C. A.* 30, 507<sup>1</sup>.—Growth substance produces only an increase in cell size, any increase in cell no. resulting is secondary. Lawrence P. Miller

Nutrient-solution purification for removal of heavy metals in deficiency investigations with *Aspergillus niger*. Robert A. Steinberg. *J. Agr. Research* 51, 413-24 (1935), cf. *C. A.* 29, 2197<sup>1</sup>.—Purification of nutrient soln. for the removal of heavy metals can be accomplished by treating the soln. with CaCO<sub>3</sub> and filtering while hot. The method gives good results with Fe, Zn, Cu and Mn under a wide variety of conditions and procedures. The removal of heavy metals appears to be dependent on their copptn. with an alk. earth as phosphate, carbonate or hydroxide by a decrease in acidity. The use of absorbent charcoal to supplement the action of an alk. earth purificant is unnecessary and causes a decrease in extn. precision. Extn. of the spores of *Aspergillus niger* with alk. solns. effects partial removal of Fe, Cu and Mn stored in the spores for subsequent growth, and so leads to an accentuation of deficiency effects with these metals. W. H. Ross

The synthesis of reserve carbohydrate by yeast. II. The effect of fluoride. Rachel A. McNally and Ida Smedley-MacLean. *Biochem. J.* 29, 2236-41 (1935); cf. *C. A.* 29, 8059<sup>1</sup>.—The addn. of fluoride (0.0024-0.0071 M) to glucose or maltose solns. in which yeast was incubated caused a marked inhibition of the glycogen storage. The addn. of fluoride in 0.0024 M concn. caused an increase in glycogen storage when glucose-phosphate was used. Higher concns. caused a fall. When maltose-phosphate was used, the inhibiting action of fluoride was marked only at the higher concns. In 48-hr. fermentations, the addn. of phosphate increased the decompn. of

glucose, but had no effect on the maltose solns. The effect of fluoride on the sugar solns. could be counteracted by the addn. of phosphate. There appeared to be a definite correlation between the wet weight of the yeast and its total carbohydrate content. E. W. Scott

The influence of the anions of the nutrient salts on decomposition and diseases of the potato. Karl Böning *Angew. Botan.* 17, 323-35 (1935).—Potato plants which received K as chloride developed more severe symptoms of leaf roll and streak disease than did those which received K as nitrate or sulfate. J. T. Sullivan

Transformation of sugars in plants. M. Nurmia. *Ann. Acad. Sci. Fennicae* 44A, No. 8, 105 pp (1935); cf. *C. A.* 29, 2998<sup>1</sup>, 3371<sup>1</sup>.—*Triticum sativum*, *Avena sativa*, *Trifolium pratense* and *Vicia faba* were placed in the dark to reduce their sugar content, then cut and immersed for 24 hours in a sugar soln. in the dark. Leaves and stems

were then analyzed separately. When glucose (I) or fructose (II) was fed they were readily converted into one another in the plant, and the interconversion was accompanied by a synthesis of sucrose (III). III was synthesized from I, II, galactose and maltose but not from dihydroxyacetone, xylose or glycerol. Interconversion of I and II and synthesis of III took place more readily in the stem than in the leaves. When III was fed, part was hydrolyzed in the stems, and the remainder accumulated in high concn. in the leaves. Changes were not affected by toluene. Part of I disappeared, probably by respiration, especially when fresh plants were placed in darkness, but also in feeding expts. Addn. of KCN retarded this only slightly. In *V. faba* lactic acid content was low, changes were not proportional to changes in pH of cell sap, and increase was not affected by feeding with I or II, ascorbic acid was not increased by feeding with III. Invertase activity of exts. from *V. faba* and *T. pratense* was 15-20 times greater in case of stems than leaves. When midribs were removed from leaves, invertase activity of leaves was almost nil. Since the leaf is capable of synthesizing III, it is assumed that the chem. nature of leaf invertase is different from that of stems and petioles. Attempts to accomplish interconversion of I and II and synthesis of III *in vitro* by plant exts. were unsuccessful. The method of detg. II was to oxidize I with I<sub>2</sub> and NaOH, clear with Pb(OAc)<sub>2</sub> and det. the reducing power of the residual soln.

J. T. Sullivan

Chemistry of mold tissue. X. The phospholipides of *Aspergillus sydowii*. D. M. Woolley, F. M. Strong, W. H. Peterson and E. A. Prill *J. Am. Chem. Soc.* 57, 2589-91 (1935); cf. *C. A.* 28, 3105<sup>1</sup>.—The Et<sub>2</sub>O-sol. phospholipides of *Aspergillus sydowii* consist of a mixt. of lecithin and cephalin. From the mycelium 0.4-0.7% of the lipides was isolated. Glycerophosphoric acid, choline, choline and oleic acid were identified as the chief hydrolytic products. Stearic, palmitic and a more unsatd. acid were also probably present in small amts. C. J. West

Photochemical responses of the wheat plant to spectral region E. J. Lease and W. E. Tottingham *J. Am. Chem. Soc.* 57, 2613-16 (1935).—Elimination of wave lengths shorter than about 3900 or 5200 Å. from radiation resembling sunlight decreased the assimilation of nitrate and conserved carbohydrates in young wheat plants. The primary factors to which increased reducing power under shorter radiation could be attributed were increased tissue contents of chlorophylls and sulphydryl compds. Paths are indicated by way of which these factors could function as trigger mechanisms in the reduction of nitrate. Depression of pentosan formation was assoc. with increased assimilation of nitrate. Most of the compositional effects in the tissue could be attributed to variations in the proportion of blue-violet light but the reduction of nitrate to nitrite was more distinctly assoc. with long ultraviolet radiation. Apparently the high proportion in sunlight of radiation from 3900 to 4920 Å. and the low proportion above 8000 Å. in comparison with the light sources here tested favor the assimilation of nitrates by plants. Analyses are given of wheat tops from water and soil cultures and of press sap from plants 3 weeks old. Pigment contents are also given. C. J. West



## E—NUTRITION

PHILIP B. HAWK

The effect of ingested fat on the sterol metabolism of the white rat H. C. Eckstein and Carleton R. Treadwell. *J. Biol. Chem.* 112, 373-8 (1935).—The amts of sterol in the livers of growing white rats are greater when the percentage of corn or soybean oil in the diet is increased but these larger amts cannot be ascribed to sterol mobilization because the sterol contents of the remaining tissues of the rats on the high fat diets were as great if not greater than the amts found in the remaining tissues of their litter mates on low-fat rations. It is impossible to state whether the larger amts of sterols in the livers were due to ingested sterols, fats or both.

A. P. Lothrop

The extraction and stabilities of vitamin B (B<sub>1</sub>) and of lactoflavin Bertha Bisbey and H. C. Sherman. *J. Biol. Chem.* 112, 415-20 (1935).—Practically all of the vitamin B<sub>1</sub> in the form found in spray-dried skim milk is extd. by 80% alc. and practically none of it by abs. alc. The solvent effect of the alc. is not enhanced by acidulation with 1% glacial AcOH. Additive results are obtained with mixts of ext. and residue, indicating no vitiation of the feeding method by lack of any "new" or unknown factor. The extns. and dryings were done under an atm. of N<sub>2</sub>. The vitamin G or flavin value was somewhat less conserved than that of vitamin B<sub>1</sub>, probably because of the combined effects of the visible light which penetrated the glass containers in which the extns. were performed, the small amt. of residual O<sub>2</sub> and possibly a slight influence of the alc. present. The flavin value of milk powder is not measurably extd. by abs. alc. at room temp.; by 80% alc. (neutral or acidulated) it is less completely extd. than is the vitamin B<sub>1</sub> value, about equal amts. of the flavin appearing in the ext. and residue. Quant. feeding expts. with mixts of ext. and residue give no indication of any measurable sepn. of essential factors and indicate that measurements of vitamin G hitherto made by the Bourquim (C. A. 25, 5996) method are essentially measures of flavin values, whether of the natural food or of prepd. exts. and residues.

A. P. Lothrop

The utilization of *d*-mannoheptulose (*d*-mannoketose) by adult rabbits Joseph H. Roe and C. S. Hudson. *J. Biol. Chem.* 112, 443-9 (1935).—Mannoheptulose in rabbits gives rise to a yeast-fermentable, C-reducing substance in the blood, the removal of which is stimulated by insulin. Since these properties correspond with those of glucose and fructose, the metabolic transformation of a 7-C sugar into one of lower C content seems a possibility. Rabbits have a high tolerance for this sugar. The identity of the metabolite is being investigated.

A. P. Lothrop

The production of a deficiency involving cystine and methionine by the administration of cholic acid Abraham White. *J. Biol. Chem.* 112, 503-9 (1935).—Male rats were fed a diet contg. a limited amt. of the S-contg. amino acids (6% casein) to accentuate the effects of the administered cholic acid and a nutritional deficiency was produced by the addn. of cholic acid to this diet. The deficiency was relieved by the addn. of extra cystine or methionine to the diet, taurine was without effect. The results may be due to the demand of the organism for taurine for the synthesis of taurocholic acid but the neg. results with taurine and the complete inability of glycine to alleviate the deficiency through a synthesis of glycocholic acid suggest that there is a direct detoxication of cholic acid by a mechanism involving cystine or methionine or both with the formation of a product other than the bile acid. Cholic acid is somewhat toxic but the stimulation or cessation of growth was exactly coincident with the addn. or withdrawal of the extra cystine or methionine so that its effect on growth can only in part be related to the toxicity of cholic acid.

A. P. Lothrop

Phosphatase content of blood serum and tissues in the rat following administration of vitamins D and A Paul D. Crenn and J. W. Strayer. *J. Biol. Chem.* 112, 511-15 (1935).—Approx. 20 times the amt. of phosphatase usually found in human serum is present in the serum of

rats of various ages (40-110 units per 100 cc.). A marked reduction in blood and kidney and a great increase in the small intestine occur in rats made toxic with viosterol. Findings on the effect of viosterol on bone phosphatase content were variable but there was no demonstrable effect on the small amts. present in the liver and spleen. Vitamin A-deficient rats and those fed high doses of vitamin A showed reductions of phosphatase in the serum, these were due apparently to the greatly impaired nutrition of the animals since the presence or absence of vitamin A had little or no effect on phosphatase. A. P. Lothrop

Vitamin A content of sour-cream butter, sweet-cream butter and margarine (Hathaway, Davis) 12 Physiologically active cryst. esters of vitamin A (Haman) 10.

## F—PHYSIOLOGY

HOMER W. SMITH

Influence of quinine on the realization of the morphogenetic effect of the thyroid hormone. R. I. Belkin and A. A. Votkevich. *Compt. rend. acad. sci. U. R. S. S. [N. S.]*, 3, 233-8 (1935).—To test the restricting effect of quinine (I) on the activity of the thyroid hormone (II) a group of young pigeons in the 1st molting phase and a 2nd group in an advanced stage of molt were treated with the di-HCl salt of I in daily oral doses of 50, 100 and 200 g., with 300 mg. of II and with combinations. The main feathers were counted daily for 13 days and at the end of the runs the thyroid glands were dissected out to permit an examn. of their histological picture and functional condition. The biol. activity of the glands was estd. by implantation of 1-mg. portions in tadpoles. It was demonstrated that I and II decrease the functional activity of the thyroid gland and that their effect is synergistic. The increased protein metabolism resulting from molting is reduced by the administration of I and consequently the need of the organism for thyroid is lessened. Similarly the administration of II reduces the necessity for glandular action and a typical hypofunctional picture results. I reduces the oxidative effect of II and this has its influence as is shown by the restriction of the morphogenetic effect in molting as demonstrated by the results when I and II are administered together in contrast to those obtained by treatment with II alone.

C. R. Addall

Distribution of chlorine and urea in the blood and bile Oscar Miseta. *Rev. med. quiv. patol. feminae* 5, 69-124 (1935).—The concn. of urea in the blood was found to be between 0.24 and 0.49 g. per l.; in the bile, between 0.19 and 0.44. Chlorides in the blood oscillated between 4.0 and 4.8 g. per l., in the bile, between 4.8 and 5.6.

E. S. G. Barron

Glukagon (the hyperglucemia-raising substance of the pancreas). M. Burger and W. Brandt. *Z. ges. exp. Med.* 95, 375-97 (1935).—Glukagon is in its chem. and phys. properties similar to insulin. It is a protein, contains C, H, N and S, and is isoelec. about where insulin is. It is sol. in H<sub>2</sub>O, alc., and org. solvents. Its salts are sol. in H<sub>2</sub>O, dil. alc. and dil. MeCO. It is nondialyzable. It is not inactivated by dil. NaOH or cysteine as insulin is. The most effective prepn. obtained raises the blood sugar of rabbits (about 2 kg.) by injection of 20 γ per kg. about 50% for a period of 40 to 60 min. Eleanor W. J. Butz

The influence of fat metabolism hormones of the anterior hypophysis on the acetone body secretions of rats under different experimental conditions. Chr. Sievert. *Z. ges. exp. Med.* 96, 429-41 (1935).—The injection of fat metabolism hormone in female rats held on a butter diet did not influence the daily AcMe excretion. The injection of anterior hypophysis ext. into hungry rats causes an increase in AcMe in the urine. Test animals (starved rats) were fed daily with a detd. amt. of NaCl to increase the urine output. The influence of the blood ketogenic substances after fat feeding on the AcMe excretion was then studied. The substance producing fat metabolism is the same as the hormone isolated from anterior hypophysis described by Anselmino and Hoffman (C. A. 26, 2229).

Eleanor W. J. Butz

Liver and nitrogen metabolism. F. Goebel. *Z. ges.*

*exptl. Med.* 96, 468-77 (1935).—N metabolism was studied in dogs with Eck fistula. The disturbance of the normal relation of the urea N and  $\text{NH}_3$  to total N is a result of the disturbance of the urea synthesis. The acid base equil. was not disturbed. The amt. of  $\text{Et}_2\text{O}$ -sol. substance increased in the urine.

**Eleanore W. J. Butz**  
*Fat metabolism in depancreatized dogs.* A. I. Lewin *Z. ges. exptl. Med.* 96, 548-60 (1935).—Pancreatectomy in dogs produced a considerable increase in blood lipoids, a decrease in peripherally stored fat and infiltration of fat into some organs. Both blood cholesterol and lipoid P increased. Administration of pancreatic juice did not alter the picture so the changes are to be attributed to insulin lack. Administration of insulin restored the fat metabolism to normal. Feeding of cholesterol in oil with simultaneous administration of pancreatic juice prolonged the life of the operated dogs to 110 days, decreasing the hyperglucemia and glucosuria, and preventing acetonuria.

**Eleanore W. J. Butz**  
**The influence of cations of fluorescein salts on the absorption by the intestine.** (Study with living frog and mouse intestine in ultraviolet light) Josef Pick and Fritz Zuckerkandl *Z. ges. exptl. Med.* 96, 747-51 (1935).—K, Mg and Na fluorescein were absorbed by the epithelium, first by the cytoplasm and later by the nucleus. The rates in decreasing order were K, Mg and Na. The Na salt remained concd in the epithelium, the Mg was detected in the blood and the K concd in the lymph channels.

**Eleanore W. J. Butz**  
**The oxalic acid content of blood.** Axel Thomsen *Z. physiol. Chem.* 237, 199-213 (1935).—The method of Merz and Maugeri (C. A. 26, 488) for detg. oxalic acid in blood and serum is unreliable because  $\text{Ca}(\text{OH})_2$  ppt. other reducing substances which react with  $\text{KMnO}_4$  and gives values far too high. The same objection applies to Izumi's  $\text{CeCl}_3$  method (C. A. 28, 1372<sup>a</sup>). The method recommended consists in satg. the serum with  $\text{Na}_2\text{SO}_4$  or  $(\text{NH}_4)_2\text{SO}_4$ , acidify with  $\text{H}_2\text{SO}_4$ , extg. with  $\text{Et}_2\text{O}$  and converting the  $\text{Et}_2\text{O}$  ext. into  $\text{CaC}_2\text{O}_4$ . The oxalic acid content of beef serum is thus shown to be 1 mg. % at the most. After feeding a rabbit with spinach, which contains oxalic acid, the serum contained only 1.1 mg. %. Small quantities were found also in the liver and kidneys. Samples of beef blood which gave zero value by the  $\text{Et}_2\text{O}$  extn. method gave as high as 8 mg. % by the Merz and Maugeri method.

**A. W. Dux**  
**The  $p_H$  of muscle.** W. O. Fenn and F. W. Maurer. *Protoplasma* 24, 337-45 (1935).—Frog muscle in the body is in equil. with plasma which contains 2.6 times as much bicarbonate. After the bicarbonate contained in the tissue spaces is allowed for, a  $p_H$  of 6.9 for the interior of the fibers is calcd. by the Henderson-Hasselbalch equation, while the outside of the fibers is bathed in a soln. of  $p_H$  7.34. A micro-method is described for extg. from muscles minute quantities of extracellular fluid which is shown to be alk. in reaction ( $p_H$  7.4). Fluid obtained from a site of injury is acid ( $p_H$  6.27) and this acidity persists to a lesser degree ( $p_H$  7.07) even after lactic acid production has been stopped by iodoacetic acid, which indicates intracellular acidity. When muscles are brought into equil. with Ringer soln. this wide difference in  $p_H$  between the inside and the outside of the fibers tends to disappear, but some small excess outside remains even after 5 hrs. except in the most acid solns. In alk. solns. the muscle tends to gain bicarbonate and this takes place to some extent even when the muscle is immersed after dissection in blood of the same frog. Histological examn. of muscles from which extracellular fluid had been drawn in capillary tubes presents evidence that the fluid is purely extracellular and not a mixt. of extra- and intracellular material. This evidence was obtained by studying the path of the capillary through the muscle by means of a hair which had been inserted in the tube before puncturing the muscle and which was allowed to remain in the muscle after withdrawal of the tube. Seventeen sections taken at random from a gastrocnemius muscle so treated showed no broken muscle cells. Thirty-five references.

**F. L. Dunlap**  
**Differential reduction of Janus green during develop-**

**ment of the chick.** Olin Rulon. *Protoplasma* 24, 346-64 (1935).—Chick embryos, from the primitive streak stage to the 8th day of incubation, were stained with oxidized Janus green and placed under conditions of low O. The rate, intensity and regions of reduction of the dye were noted. The differentials in reduction of the dye correspond closely to the disintegration gradients of Hyman (*Biol. Bull.* 52, 1-38, 39-50) and Hinrichs (C. A. 22, 994). Nineteen references.

**F. L. Dunlap**  
**The validity of fractional gastric analysis.** Frances A. Hellebrandt and Elizabeth Brogdon *Am. J. Digestive Diseases Nutrition* 2, 402-8 (1935).—Gastric fractional analyses were repeated from 8 to 21 times on 3 normal subjects after stimulation by oatmeal gruel, 7% alc. and by histamine. From the results of 105 expts it is concluded that the secretory response of the stomach is highly variable in the normal subject, irrespective of the quant. reproducibility of the stimulant used. Although fractional gastric analysis is of questionable value for quant. studies of gastric function, it remains the method of choice for detections of gastric acidity and for approximations of secretory capacity.

**Edward Eagle**  
**The pancreas and general metabolism.** W. N. Boldyreff *Am. J. Digestive Diseases Nutrition* 2, 413-15 (1935).—Carbohydrate, fat and protein metabolism in the whole body as well as in each separate cell are effected with the aid of certain pancreatic enzymes which are being secreted in the pancreatic juice and are absorbed into the blood (and hence into the cells) during digestion and in fasting. There is evidence to support the view that the pancreas is the main agent of all general chem. processes in the organism.

**Edward Eagle**  
**The autoregulation of the gastric secretion.** J. J. Day and D. R. Webster *Am. J. Digestive Diseases Nutrition* 2, 527-31 (1935).—Introduction into the duodenum of 0.25% HCl or of gastric juice dil. 2 to 4 times inhibits the gastric secretion stimulated (a) through the parasympathetic nervous system or (b) by the presence in the intestine of food substances or the products of their digestion. The theory is advanced that the passage of the acid chyme from the stomach into the duodenum causes the gastric secretion to diminish, this being an important factor in regulating the activity of the gastric glands.

**E. Eagle**  
**Influence on carbohydrate metabolism of experimentally induced hepatic changes.** IV. Block of the reticulo-endothelial system with special reference to the Kupffer cell. T. L. Althausen, B. E. Blomquist and E. F. Whedon. *Am. J. Digestive Diseases Nutrition* 2, 532-40 (1935), cf. C. A. 27, 2209.—Partial block of intravascular reticulo-endothelial cells causes lowering of the blood sugar, increased tolerance to dextrose, decreased mobilization of blood sugar by adrenaline and reduction in the glycogen content of the liver and muscles. The authors believe that one of the functions of the Kupffer cells is the transmission of precursors of glycogen from the blood stream to the liver, this transfer is interfered with by blocking, which results in decreased gluconeogenesis and consequent internal deficiency of carbohydrates.

**E. E.**  
**The lysozyme content of tears.** Wm. M. James. *Am. J. Ophthalmol.* 18, 1109-13 (1935).—Tears were collected with capillary pipets from 100 clinically normal persons and the bacteriolytic titer was detd. under standard conditions, with *Jf. lysodenthus* as the test organism. No relationship could be established between the titer variations and the age, sex or race of the subject.

**E. Eagle**  
**Acid-base balance of the blood.** IV. Characterization and interpretation of displacement of the acid-base balance. Nathan W. Shock and A. Baur Hastings. *J. Biol. Chem.* 112, 239-62 (1935), cf. C. A. 28, 3464<sup>a</sup>.—By use of methods and notation previously described (C. A. 28, 3434<sup>a</sup>) the details of variations in the acid-base balance of the blood of normal individuals and the rate of elimination of fixed alkali and acid have been studied. Ingestion of  $\text{NH}_4\text{Cl}$  (I) caused displacement of the balance toward decreased  $p_H$  and  $(\text{BHCO}_3)$ , without much change in  $p\text{CO}_2$ . Max. displacement occurred 2-5 hrs. after giving 1 (10 g.). After this a period of decreasing  $p\text{CO}_2$  and increasing  $p_H$  followed. The return to normal, somewhat

slower than the time of max. displacement, showed increasing  $p_{H_2}$ , (BHC $O_2$ ) and  $pCO_2$ . Ingestion of NaHCO $_3$  (20 g) caused an increase in  $pCO_2$  after the max.  $p_{H_2}$  and (BHC $O_2$ ), were attained, which occurred in 1-1.5 hrs. Return to normal showed decreasing (BHC $O_2$ ),  $pCO_2$  and  $p_{H_2}$  in 2 to 3 hrs. The line of displacement was linear and tended to remain parallel to the const.  $pCO_2$  lines. Excess  $CO_2$  as attained by re-breathing gave results similar to those obtained by  $CO_2$  titration of normal blood.  $CO_2$  deficiency attained by over-breathing followed the  $CO_2$  titration curve during the early stages, but later a rather persistent increase in fixed acid of the blood occurred. Four major paths of displacement and recovery of the acid-base balance have been found experimentally, corresponding to those derived on theoretical grounds, which are sufficiently distinct to permit characterization of deviation from the normal condition. This permits pathol. conditions affecting the acid-base balance to be classified under metabolic acidosis, metabolic alkalosis, respiratory acidosis and respiratory alkalosis. R. C. E.

Growth and development with respect to domestic animals XXXVII Interrelations between protein intake, endogenous nitrogen excretion and biological value of protein. Ural S. Ashworth. Mo. Agr. Expt. Sta., Research Bull. No. 228, 3-14 (1935); cf. C. A. 29, 54971. By use of the paired rat technique on 20 pairs of rats, A. detd. that the nature of the protein fed affected but slightly the endogenous N excretion when short exptl. periods were used. However, when the reserve protein supply of the body was reduced to a low level by long periods on N-free diets an effect of the nature of the protein fed on endogenous N excretion did appear. The results suggest the use of a short exptl. period to avoid the effect of the nature of the protein fed on the endogenous N excretion, but short exptl. periods increase the variability of (1) the amt. of reserve protein present in the body, (2) the endogenous N excretion and (3) the biol. values of the protein. Thus, short exptl. periods cannot be used to det. small differences in biol. values of proteins. Before these small differences can be detd. with reliability, a method of securing more consistent values for the endogenous N excretion must be devised. C. R. Fellers

Further examinations on the chemical structure of the salts of the bones. Jozsef Marek, Oszkar Wellmann and László Urbányi. Math. naturw. Ans. ungar. Akad. Wiss. 52, 734-8 (1935).—See C. A. 29, 80823. S. S. de F.

Action of male hormone on the genital organs of young dogs. Maso Itho and Tsungiro Kon. Compt. rend. soc. biol. 120, 678-81 (1935).—Intramuscular injections of 50 units per day for 3-6 weeks caused a marked increase in the size and wt. of the prostate, seminal canal, epididymus, penis and seminal vesicles but not the testicles.

L. E. Gilson  
Phosphatase of human serum during pregnancy. J. Cayla and F. Fabre. Compt. rend. soc. biol. 120, 748-50 (1935).—In 13 cases, 6-9 months pregnant, serum phosphatase was increased to 1.5-3 times the normal value.

L. E. Gilson  
Absence of bacteria-inhibiting substances in human saliva. A. Roney and J. Fricker. Compt. rend. soc. biol. 120, 887-90 (1935).

L. E. Gilson  
Adrenal cortex extract. Edward C. Kendall. J. Am. Med. Assoc. 105, 1486-9 (1935).—A review with 23 references.

F. P. Griffiths  
Blood-sugar concentration and the external secretion of the pancreatic gland. B. P. Babkin. J. Am. Med. Assoc. 105, 1659-62 (1935).—The concn. of dextrose in the blood affects directly the concn. of enzymes in the pancreatic juice. Twenty-seven references. F. P. Griffiths

Preliminary blood survey of Masai cattle in drought periods. M. H. French. Tanganyika Territory, Ann. Rept. Dept. Vet. Sci. 1934, 65-8.—Long intervals without water had no significant effect on the contents of Ca, K, Na and inorg. P in the blood of Masai stock. A large intake of water after a 3 days' thirst caused a slight decrease in the hemoglobin content of the blood but had no effect on the inorg. P. K. D. Jacob

A new phosphorus fraction in blood and tissues. Tauno

1 Kalaja. Suomen Kemistilehti 8B, 41-2 (1935) (in English).—The blood of man, cow, rabbit and cat contains some P compd. which spontaneously undergoes a rapid hydrolysis in protein-free filtrates. In the blood the labile compd. is present in the red blood cells, since no liberation of inorg. P was observed in serum filtrates. The liver and muscles of rat and rabbit contain a similar P compd. At 37° the rate of hydrolysis is much higher than at room temp. The compd. undergoes hydrolysis both in acid and alk. soln. while it is fairly stable in neutral soln. Detn. of blood-inorg. P by the usual colorimetric method must be made immediately after filtering. E. E. J.

The influence of the ingestion of raw pancreas upon the blood lipides of completely depancreatized dogs maintained with insulin. I. L. Chalkoff and A. Kaplan. J. Biol. Chem. 112, 155-65 (1935); cf. C. A. 28, 68237; 29, 11479.—A fall in the lipide concn. of the blood occurs in completely depancreatized dogs maintained with insulin as early as 12 days after pancreasectomy. In 8 dogs cholesterol esters were reduced to between 0 and 3 mg % at intervals of 10 to 122 days following pancreatectomy while in 3 others 7 to 14 mg % were still found in whole blood as late as 96 to 202 days. When raw pancreas is added to the diet there is a rise instead of a fall in whole blood lipides and the rise is particularly striking in the cholesterol esters (in 1 animal from 5 to 129 mg % in 23 days). The total fatty acids rose from preoperative or normal levels of 324 to 352 to 412 to 633 mg % and cholesterol esters from 28 to 43 to 73 to 152 mg %. The removal of the raw pancreas from the diet after the high lipide levels had been produced resulted in a rapid and pronounced drop in blood lipides, all participating in the fall as they had in the rise. The high values are found only so long as raw pancreas is being ingested. A. P. Lothrop

Some reactions of ammonolyzed parathyroid hormone. Richard G. Roberts, Wilbur R. Tweedy and Geo. H. Smullen. J. Biol. Chem. 112, 209-14 (1935).—Parathyroid hormone has no prosthetic group with or without Fe that has a catalytic effect, such as shown by hematin, for the action of Na on liquid NH $_3$ . It reacts as a typical protein with Na in liquid NH $_3$ . The activity is lessened by the action of liquid NH $_3$  alone and is not dependent on disulfide linkages or any other linkages that are strongly reduced by Na in liquid NH $_3$ . Its activity appears to be a function of a part of the acidic H liberated and varies inversely with the liberation of this H which most probably comes from imide groups as indicated by its slow evolution. A. P. Lothrop

Chemical studies on the pituitary gonadotropic hormone. L. C. Maxwell and Fritz Bischoff. J. Biol. Chem. 112, 215-21 (1935).—Amino, imino or OH groups or their S analogs may be concerned with the physiol. activity of pituitary gonadotropic preps since reagents known to react with these groups cause a partial to a complete inactivation of the hormone. Its activity is completely destroyed by strong oxidizing agents but is unaffected by mild oxidizing agents or by reducing agents. Complete inactivation occurs in 0.1 N NaOH and partial inactivation in N/30 NaOH in 3 hrs at 37°. Partial inactivation occurs in 0.1 N HCl in 3 hrs. Denaturation of the protein aggregate may produce the same physiol. effect as slowed tissue resorption of the original product, evidence against the dual hormone theory of gonadotropic preps. A. P. Lothrop

The phosphorus metabolism of invertebrate nerve. Geo. L. Engel and R. W. Gerard. J. Biol. Chem. 112, 379-92 (1935).—Expts. were conducted on lobster claw nerves and ganglia and crab nerves. "During rest in O $_2$  the phosphoarginine decomposed during dissection (and attendant stimulation and injury) is rebuilt, apparently by direct reaction with adenylypyrophosphate which decomposes. This, in turn, is subsequently resynthesized, the lactic acid intermediates probably serving in part as phosphate donors. During anoxia there is an extensive hydrolysis of both phosphoarginine (up to 100%) and adenylypyrophosphate with an attendant rise in the inorg. fraction. Subsequent exposure to O $_2$  results in full resynthesis of arginine phosphate in 5 hrs; and of 1/2 of the adenyly-

pyrophosphate within an hr. with no further change. Both substances are probably decreased by stimulation. The stable fraction (hexosephosphate, etc.) regularly shows a loss of bound P after several hrs., greater during or following the absence of  $O_2$ . There is no evidence that the P of any acid-insol compounds (phosphoproteins, phospholipids, nucleoproteins, etc.) is involved in the aerobic metabolism of these nerves." A. P. Lothrop

The question of the utilization of tryptophan administered subcutaneously. Vincent du Vigneaud, Robert R. Sealock and Cecil Van Eppen. *J. Biol. Chem.* 112, 451-6 (1936); cf. C. A. 27, 530.—Tryptophan injected subcutaneously is definitely utilized. From these findings there is apparently no explicit evidence to support the theories of protein metabolism promulgated by Acock (C. A. 29, 1859<sup>4</sup>), based on the nonutilization of injected tryptophan. A. P. Lothrop

Study of the serum lipides by a microgravimetric technique. Wm. R. Wilson and Arild L. Hansen. *J. Biol. Chem.* 112, 457-68 (1936).—A method is described for the sepn. of the saponifiable and unsaponifiable fractions of the blood lipides which is an adaptation of an early method of Bloor (C. A. 10, 1664). The quant. microgravimetric technique is similar to that used by Wilson and Hanner (C. A. 28, 6798<sup>7</sup>) for the total lipides. Analyses of 17 normal serum gave the following av. values per 100 cc. total lipides: 657 mg.; unsaponifiable fraction 260 mg. with an I no. of 63.4; saponifiable fraction 362 mg. with an av. mol. wt. of 291 and an I no. of 108 equis. to 1.23 double bonds per mol., figures suggesting that while 18 C unsat. acids predominate in human blood, there are appreciable quantities of longer-chain unsat. acids. The lipid P of the saponifiable fraction averaged about 81% of the total lipid P, indicating 2 types of phospholipide in the blood, one of them saponifiable and the other not. A. P. L.

The effect of changes in the concentration of plasma electrolytes on the concentration of electrolytes in the red blood cells of dogs, monkeys and rabbits. Herman Yarnet, Daniel C. Darrow and M. Katherine Cary. *J. Biol. Chem.* 112, 477-88 (1936).—In a series of 30 expts (15 on dogs, 8 on monkeys and 7 on rabbits) changes in the concn. of plasma electrolytes were produced by intraperitoneal injection of 8% glucose soln. or 1.8% NaCl soln. There is a high degree of correlation between the univalent cations of the red cells and plasma and a given change in concn. of these cations in serum is accompanied by an equiv. change in the cations of the red cells, results fitting the generally accepted concepts of osmotic equil. in body fluids. Shift of  $H_2O$  across the red cell membrane constitutes the chief, if not the only, mechanism of adjusting osmotic equil. in monkeys and rabbits but in the dog there is in addn. passage of NaCl across the membrane. Erythrocytes contg. Na as the preponderant base are permeable to Na but a similar permeability of red cells contg. K has not been demonstrated. A. P. Lothrop

The adenine nucleotide content of human blood. Mary V. Buell. *J. Biol. Chem.* 112, 523-30 (1936); cf. C. A. 29, 1473<sup>4</sup>.—There is in human blood a significant correlation between the adenine nucleotide and hemoglobin contents of the erythrocytes. If this correlation indicates a primary assoc. between nucleotide and hemoglobin, the observed correlation between nucleotide and both hematocrit and erythrocyte count follows logically. It cannot be concluded from the observations, however, whether both compounds are regulated by the same unknown factors or whether the occurrence of one fundamentally affects the other." A. P. Lothrop

The blood cholesterol in the carotid artery, venae cavae and portal vein. Frederick H. Shillito, Emul H. Bidwell and Kenneth B. Turner. *J. Biol. Chem.* 112, 351-6 (1936).—The cholesterol content of blood in the carotid artery, superior and inferior venae cavae and portal vein is the same for a given animal (cats and dogs) irrespective of whether whole blood or serum is used and whether or not cholesterol was administered before operation. No variation in the distribution of cholesterol is produced by administration of large doses of KI (1 g. daily) for 2-14 days. Passage through the lungs has no demonstrable

effect upon the blood cholesterol although the claim has been made that such is the case. Twenty-four references. A. P. Lothrop

Lipide composition and physiological activity in the ovaries of pregnant guinea pigs. Ildon M. Boyd. *J. Biol. Chem.* 112, 591-5 (1936), cf. C. A. 29, 4064<sup>3</sup>.—The phospholipide content of the ovaries of nonpregnant control guinea pigs at the proestrus stage averaged 1250 mg. % and there was no significant variation during pregnancy. At proestrus the ovary contained a mean of approx. 300 mg. % of free cholesterol which again did not significantly vary during pregnancy. These results were correlated with the fact that guinea pigs may be castrated in the litter half or 1/4 of pregnancy without abortion ensuing which indicates that no increase in physical activity occurs in the ovaries of gravid guinea pigs. A. P. L.

Respiratory metabolism in infancy and childhood. XVI. Effect of intravenous infusions of fat on the energy exchange of infants. Harry Gordon and S. Z. Levine. *Am. J. Diseases Children* 50, 894-912 (1935), cf. C. A. 29, 8071<sup>4</sup>.—Parenterally administered fats can apparently be oxidized by normal infants when the control level of the R. Q. is 0.9-1.0. Intravenous infusions of emulsified human fat, olive oil or a com. prepn. of fat, Yanol, depressed the R. Q. by 0.03-0.07. An increase in heat production due entirely to increased oxidation of fat was observed. When the control level of the R. Q. was 0.8, the combustion of fat was not increased even if the emulsified fat was given by mouth. Parenterally administered fat had no effect on the R. Q. in the myxomatous infant. An increase in heat production was observed, but could be only partially accounted for by the oxidation of fat. F. R. Main

The influence of dextrose ingestion on amino acid nitrogen, urea nitrogen and hemoglobin concentration of the blood. E. G. Schmidt and J. S. Eastland. *J. Lab. Clin. Med.* 21, 1-12 (1935).—During the course of dextrose tolerance tests, the av. max. decrease in amino acid N was 12.91% in normal persons, 10.22% in arthritis and rheumatoid conditions, 14.68% in infections and 11.51% in diabetes mellitus. The av. max. decrease in urea N was 13.55% in normal cases, 14.78% in arthritis, 15.02% in infections and 11.00% in diabetes. The degree of hyperglycemia attained during the test did not appear to be correlated with the decrease in amino acid and urea N or with the changes in blood vol. as detd. by the relative concns. of hemoglobin. E. R. Main

The morphologic sugar metabolism in the human leucocyte culture. Günter Wallbach. *J. Lab. Clin. Med.* 21, 163-8 (1935).—Normal human leucocytes cultivated in human plasma showed glycogen deposits in the first 2 days of growth but none was found after the 3rd day. After the addn. of glycogen or glycerol to the culture medium a marked increase in glycogen deposition was observed. Little or no glycogen was synthesized after the addn. of glucose, fructose, galactose, dextrin, maltose, starch or 1.0H. After the addn. of insulin or tophenin, a transient storage was observed on the 2nd day. The addn. of insulin and glucose or Hefpene (a lecithin emulsion) and glucose caused a pronounced increase in glycogen storage. Thyroxine and adrenaline did not increase glycogen storage. E. R. Main

The influence of sucrose ingestion on amino acid nitrogen and urea nitrogen concentration of the blood. E. G. Schmidt and J. S. Eastland. *J. Lab. Clin. Med.* 21, 233-5 (1935).—The ingestion of sucrose caused a somewhat smaller reduction in amino acid and urea N than the ingestion of glucose. The av. max. decrease in amino acid N was 8.07% and that of urea N, 8.74%. E. R. Main

Preliminary attempts and results obtained with cultures of the female genital apparatus in vitro with folliculin. Francesco Guerriero and Romano Arnone. *Boll. Soc. Ital. Biol. Sper.* 10, 622-4 (1935).—The prolonged treatment of female rabbits with folliculin (1000 U per day) produced in certain animals changes in the hormones which influenced the genital app. in such a manner that pieces of this tissue, especially epithelial tissue, when cultivated in vitro developed more vigorously. Peter Masucci

The isolated ameboid cells in cultures of adult rabbit uterine tissue in vitro treated with gonadotropic hormone and with folliculin Francesco Guercio *Boll. soc. ital. biol. sper.* 10, 624-6(1935).—After prolonged treatment with prolactin numerous, large, actively ameboid, isolated cells were observed on cultivating adult rabbit uterine mucosa. The cells seemed to be not epithelial but histogenic.

**Suprarenal capsules and gaseous metabolism.** I. Michele Gagliardi *Boll. soc. ital. biol. sper.* 10, 628-31 (1935).—The suprarenal capsules of albino rats were extirpated. The gaseous metabolism of those animals which died soon after the operation decreased, that of the animals which survived 36 hrs or longer increased. II. The influence of pilocarpine on the gaseous metabolism of rats whose suprarenal capsules had been extirpated. *Ibid.* 631-33.—The injection of pilocarpine produced no increase in the O consumption of albino rats whose suprarenal capsules had been extirpated. Peter Masucci.

**Preliminary observations on the variations of the ascorbic acid titer in the puerperal state.** I. Technical considerations and plan of attack. Francesco Guercio, Giuseppe Piccinelli and H. Hamburger *Boll. soc. ital. biol. sper.* 10, 640-3(1935).—Data were made on pregnant women in various periods of gestation and in labor, healthy subjects kept on the same diet were used as controls. A gradual increase in the ascorbic acid titer of the serum was noted, reaching figures which were markedly higher than the initial and the normal controls at the end of gestation. There were no significant variations in the titer during labor. Peter Masucci.

**The calcium content of human milk as influenced by administration of calcium, irradiated ergosterol and parathyroid hormone.** L. Rossi *Clinica pediatrica*, 16, No 10 (1934), *Rev. sudamericana endocrinol. inmunol. ginecoterap.* 18, 812.—Ca feeding has no influence on the Ca content of the milk. Irradiated ergosterol causes a slight, parathormone a considerable, increase. A. E. Meyer.

**Further studies on the thymus and pineal glands.** L. G. Rowntree, J. H. Clark, A. Stenberg, A. M. Hanson, N. H. Emborn and W. A. Shannon *Ann. Internal Med.*, 9, 359-75(1935), cf. C. A. 29, 7427.—Thymus ext. accelerated the rate of growth and development and hastened the onset of adolescence in the offspring of treated rats. Thymusectomy of parent rats retarded the growth of young. Pineal ext. retarded the rate of growth and accelerated the onset of adolescence. John T. Myers.

**The assay of fat-soluble androsteroneol Vladimir Korenchevsky and Marjorie Dennison.** *Biochem. J.* 29, 2122-30(1935), cf. C. A. 29, 7437.—Pure androsteroneol (I) prep. by Ruzicka was assayed by K. and D's method on rats. I in amts of about 21  $\gamma$  and 19  $\gamma$  gave one rat unit of "comb-growth activity" and of "whole male sexual activity," resp. Thus, I measured on capons had 3 times the activity of androsterone (II), on rats 8 times the activity of the II. Compared to II, I stimulated the growth of the seminal vesicles more than that of the prostate and had a greater effect on the penis and the preputial glands. E. W. Scott.

**The effects of water-soluble preparations of androsterone and androsteroneol on castrated rats.** Vladimir Korenchevsky, Marjorie Dennison and Samuel Levy Simpson. *Biochem. J.* 29, 2131-42(1935).—The Li salts of androsterone- and androsteroneol-mono succinic acid esters (I and II, resp.) were assayed on rats and the results compared to those obtained with androsterone (III) and androsteroneol (IV) in olive oil. The chief effects of I and III, and of II and IV were the same. The rat unit of "comb-growth activity" was contained in about 1600  $\gamma$  of I and 67  $\gamma$  of II. The ratio of the rat unit of III to that of I was 1.9, and of IV to III was 1.3. While on capons II showed twice the activity of I, on rats II was 25 times as active as I. E. W. Scott.

**Fat metabolism.** I. The oxidation of butyric, crotonic and  $\beta$ -hydroxybutyric acids in the presence of guinea pig liver slices. Maurice Jowett and Juda Hirsch Quastel. *Biochem. J.* 29, 2143-58(1935).—The rates of oxidation of butyric (I), crotonic (II) and  $\beta$ -hydroxybutyric (III)

acids to acetoacetic acid (IV) were studied. The rate of formation of IV ( $Q_{0.5}$ ) was a function of substrate concn with I and II but passed through a max. value with III. The  $Q_{0.5}$  from I and II varied with the K- and Ca-ion concns of the medium passing through a max. The optimum  $pH$  value was close to the physiol. value for I and III, but was higher (7.7-9) for II. The  $Q_{0.5}$  decreased in the order I, II and III. There was definite correlation between the increase in respiration and the increase in  $Q_{0.5}$  caused by these acids. Mixts. of I and II showed competition for oxidation, but mixts. of I or II with III gave partial additivity.  $BzOH$ ,  $PhCH$ ,  $CHCl_3$  and  $PhCH_2CH_2CO_2H$  strongly inhibited the oxidation of I and II to IV, but the oxidation of III was inhibited to a much smaller extent. The evidence produced supported the theory that I and II were directly oxidized to IV by the same enzyme and that III was the reversibly reduced form of IV and not an intermediary in the oxidation of I and II. II. The oxidation of normal saturated fatty acids in the presence of liver slices. *Ibid.* 2159-80.—The formation of III and IV by the oxidation of normal said acids of 2-10 C atoms in the presence of rat and guinea-pig liver slices was investigated. IV was the only  $\beta$ -keto acid produced in significant amts. Acids of 4, 6 and 8 C atoms produced IV most rapidly, decanoic acid slightly less rapidly and  $AcOH$  only slowly. Acids of 5, 7 and 9 C atoms produced

IV at small but significant rates with guinea-pig liver. Propionic acid produced no IV. The greater increase in the liver respiration caused by the odd-numbered acids suggested that they were more completely burned to  $CO_2$  and  $H_2O$  than the even-numbered acids. With guinea-pig liver, hexanoic and octanoic acids produced about 1 mol of ketone bodies, decanoic acid less than 1 mol, and butyric acid much less. With all fatty acids higher than valeric, oxidation resulted in the formation of fixed acid, more with the even-numbered than with the odd numbered acids, which was not III or IV. The oxidation of propionic and valeric acids caused a decrease of this fixed acid.  $BzONa$  inhibited the ketone body formation from I, but its effect lessened as the no. of C atoms in the substrate was increased. J. and Q. point out that the theory of successive  $\beta$ -oxidation of fatty acids does not adequately explain the facts. A theory of "multiple alternate oxidation" was suggested in which the fatty acids are supposed to be oxidized at alternate C atoms along the whole chain before the breakdown to acetoacetic and other acids takes place. III. The formation and breakdown of acetoacetic acid in animal tissues. *Ibid.* 2181-91.—Kidney, spleen and testis produced IV in small amts from fatty acids, but the brain produced none. All these tissues produced IV from III. In the absence of K and Ca ions, the kidney produced IV most rapidly. These tissues and liver destroyed IV in the presence of O. IV was also removed anaerobically by kidney. Na malonate inhibited the oxidation by the liver of  $AcOH$ , propionic and I and the breakdown of IV. Atorol, in esterase inhibiting concn, inhibited the spontaneous formation of IV by the liver. This fact led to the tentative conclusion that the formation of IV is due to the oxidation of fatty acids liberated by hydrolysis from fats in the tissue. E. W. Scott.

**Uric acid synthesis in pigeons.** I. Reginald B. Fisher. *Biochem. J.* 29, 2192-7(1935).—About 24 hrs elapsed between the ingestion of food N by blue-bar pigeons and the corresponding uric acid excretion. The amt of N ingested showed a linear relation to the uric acid excretion. Observations on birds fed with mixed corn and on whole wheat led to the conclusion that 42-50% of the ingested N was excreted as uric acid and in addn. a constant amt of 25-40 mg of uric acid was excreted per diem. II. *Ibid.* 2198-2207.—Daily doses of 50-100 mg of Na d-lactate and l-lactate were given homer pigeons. By a statistical analysis of the data obtained on the uric acid excretion, I showed that Na l-(+)-lactate caused a significant increase in uric acid excretion while Na d-(-)-lactate had no effect. Similar treatment of Wiener's data [*Bertr. chem. Physiol. Path.* 2, 42(1902)] showed that the increased uric acid excretions observed by W. when hens were fed lactate, malonate or tartaric acid were not significant. E. W. S.

The oxytocic hormone of the posterior lobe of the pituitary gland VII. A.—Ultraviolet absorption spectra, John M. Gulland and Nathaniel S. Lucas, *Biochem. J.* 29, 2209-11(1935), cf. C. A. 29, 4814.—The ultraviolet absorption spectra of posterior lobe exts. and of purified hormones solns corresponded to those of proteoses and peptones and were not characteristic of the hormone itself B and C—Adsorption and electroanalysis Mavis Freeman, John M. Gulland and Sydney B. Randall *Ibid.* 2211-20.—The purer hormone solns available could not be purified further by adsorption on silica gel, Al hydroxide C, fuller's earth, acid-treated talc, ignited talc, asbestos or purified norite. A new technique was described for purifying whole alc. exts. of the posterior lobe by adsorption on fuller's earth and norite. The hormone was inactivated by  $\text{Ac}_2\text{O}$ . At  $\text{pH}$  greater than 8, the hormone remained in the center portion of the electroanalysis cell, at  $\text{pH}$  6 or less it migrated to the cathode. Removal of the proteose material decreased the stability of the hormone to alkalis. The oxytocic and pressor hormones were not sepd at  $\text{pH}$  10-11 by electroanalysis E. W. Scott

Gluclidic metabolism in pregnancy Bernat J. Moragues *Dia med.* 6, 497(1934), *Anal. asoc. quim. Argentina* 23, 23B(1935)—In pregnancy, there are factors capable of altering normal gluco-regulation, distinct from those observed out of gestation. Marked hypoglycemia is due to latent or apparent hepatic insufficiency

E. M. Symmes  
Phosphorus content of the blood G. C. Heringa and J. Hoekstra *Acta Brevis Neerland. Physiol., Pharmacol., Microbiol.* 5, 117(1935)—Lipoid P increases slightly after compression of the blood vessels, while the total P remains the same. The fluctuations in the percentage of lipid P are greater and occur more quickly in ovalated blood collected from compressed blood vessels than from those not compressed E. D. Walter

Xylose clearance as a test of renal function in children K. de Leeuw, *Acta Brevis Neerland. Physiol., Pharmacol., Microbiol.* 5, 129-31(1935), cf. C. A. 26, 5141; 27, 2467. E. D. Walter

The specific nature of the inhibition of the coagulating effect exerted by tissue extract on plasma resulting from incubation of tissue extract with blood serum C. Moore, V. Sunitzoff and Leo Loeb, *Am. J. Physiol.* 114, 1-18(1935), cf. C. A. 16, 1800.—The effects of tissue exts. and blood serum of dogs and birds on the coagulation of dog heparin plasma and of bird plasma were tested. A sp. adaptation exists between the tissue exts. and the plasma. The effects exerted on coagulation depend on whether the 2 substances when combined are derived from homologous or heterologous species or classes of animals. With blood serums a corresponding sp. adaptation cannot be demonstrated E. D. Walter

The inhibiting action of cattle and sheep serum on kidney extracts of cattle and sheep Eric W. Thurston, J. E. Smadel and Leo Loeb, *Am. J. Physiol.* 114, 19-24(1935), cf. preceding abstr.—In mixts. of sheep or cattle serum and ext., which under ordinary conditions produce a rapid coagulation of heparin dog plasma, even after long-continued incubation, the typical curve of inhibition can be obtained through addn. of either heparin or a Ca-inactivating substance. The curve indicating increased inhibition of coagulation of plasma, with increased incubation of the mixt. of serum and ext., is presumably due to the inactivation of tissue coagulin by a substance in the serum, and to the destruction of thrombin, which progresses with increasing time of incubation. E. D. W.

The secretory metabolism of the salivary glands. David Northrup, *Am. J. Physiol.* 114, 46-52(1935); cf. Bergonzi, C. A. 26, 1023.—Simultaneous stimulation of sympathetic and parasympathetic nerves to the submaxillary gland of the dog causes an av. decrease in glycogen of 41.4%, an av. increase in lactic acid of 128%, and an av. decrease in creatine phosphate P of 63.4%. Exhaustive stimulation of only the parasympathetic nerves to the gland causes an av. decrease in glycogen of 16.1%, and an av. increase in lactic acid of 16.1%. Exhaustive stimulation of the sympathetic nerves to the gland causes an av.

1 decrease in glycogen of 26.3%, and an av. increase in lactic acid of 65%. The lesser changes during parasympathetic than sympathetic stimulation may be due to vasodilatation during the former, and a consequent increase in the rate at which recovery processes involving oxidation can occur. It is suggested that the creatine phosphate-glycogen mechanism may be a rather general one for the release of energy in various tissues where work is done. E. D. W.

2 The chemical transmission of vagal effects to the small intestine Henry Bunting, Walter J. Meek and C. A. Maaske *Am. J. Physiol.* 114, 100-6(1935), cf. C. A. 28, 4471.—By the use of an intestinal loop in an intact animal as an indicator, the production of an acetylcholine-like substance was shown to arise in the splanchnic area during vagal stimulation. Both the frog heart and the eviscerated leech responded to tests on the perfusate from the small intestine in a manner typical for acetylcholine. The action of the material was almost entirely abolished by atropine and it disappeared on standing. E. D. W.

3 "Hunger diabetes" and the utilization of glucose in the fasting dog Samuel Soskin and I. A. Mirsky, *Am. J. Physiol.* 114, 106-9(1935), cf. C. A. 29, 5171.—As judged by the utilization of sugar after evisceration, there is no suppression of carbohydrate oxidation in the normal fasting as compared to the normal fed animal. The low R. Q. of fasting as well as the other manifestations of "hunger diabetes" is not due to a lack of carbohydrate utilization, but probably result from an increased gluconeogenesis. The need for a reinterpretation of the R. Q. is emphasized E. D. Walter

Influence of hypophysectomy on gluconeogenesis in the normal and depancreatized dog Samuel Soskin, I. A. Mirsky, Leo M. Zimmerman and Nathan Crohn, *Am. J. Physiol.* 114, 110-19(1935), cf. preceding abstr.—The hypophysectomized animal differs from the normal in that the latter derives sugar from both protein and fat, while the former is unable to convert the fat and, when its carbohydrate stores are depleted and exogenous carbohydrate is not available, derives its blood sugar from protein alone E. D. Walter

Synthesis in the sex-hormone group A. Cohen *Nature* 136, 869-70(1935), cf. C. A. 29, 4371, 4372.—A description of the synthesis and structure of compds. of maleic anhydride and vinyl naphthalenes. These compds. are being extended and will be described in detail elsewhere. These compds. may be of use in the synthesis of sex hormones of the equilenin and estrin type. E. D. Walter

Isolation and identification of a paraffin hydrocarbon from urine of pregnancy. Wm. Foris Hart and M. Allen Northrup, *J. Am. Chem. Soc.* 57, 2726-7(1935)—Details are given of the isolation of 1.4 g. of hydrocarbons from 12 l. acidified urine (of pregnancy) by adsorption on fuller's earth, sepd by  $\text{MeOH}$  into heptacosane and probably pentacosane. If these hydrocarbons occur in non-pregnant female urine and male urine they must do so in an amt. far less than in urine of pregnancy. C. J. W.

The fluorine content of bone and teeth Robert Klement, *Ber.* 68B, 2012-19(1935), cf. C. A. 27, 2090.—Land-living sucking animals and human beings have a F content of bone and teeth of 0.03%. There is no difference between F content of dentine and enamel. Sea-living sucking animals have a F content of the bone of 0.55%. Bones of land birds have 0.11% F, sea birds, 0.32%, fresh-water fish, 0.03%, ocean fish, 0.43%. F replaces some of the  $\text{H}_2\text{O}$  groups in hydroxyapatite to give a mixt. of crystals Julius White

Formation of xanthurenic acid Experiments on man Francesco M. Chiancone *Boll. soc. ital. biol. sper.* 10, 578-80(1935)—Specimens of urine taken every 3 hrs. for 24 hrs. after the administration per os of 3 g. tryptophan gave no evidence of its transformation into xanthurenic acid Helen Lee Gruehl

Several chemical growth substances which cause initiation of roots and other responses in plants. P. W. Zimmerman and Frank Wilcox *Contrib. Boyce Thompson Inst.* 7, 209-29(1935); cf. C. A. 27, 5780; 26, 172.—The growth substances tested for response upon 10 varieties of plants are  $\alpha$ - and  $\beta$ -naphthaleneacetic, 5-acenaph-

theneacetic, indolebutyric, phenylacetic, fluoreneacetic and anthraceneacetic acids, and  $\alpha$ -naphthaleneacetonitrile. Substances were injected into plants through capillary tubes, admitted through an overhanging slit stem or applied in a lanolin paste. Plant responses include local initiation of adventitious roots on stem and leaves, proliferations, swelling and bending of stems, acceleration of growth and epinasty of leaves.  $\alpha$ -Naphthaleneacetic acid and indolebutyric acid were the most effective root-producing substances and indoleacetic acid was most effective for epinasty of leaves. Both responses indicate that the chemical moved upward and downward in plants for several inches. Results indicated that plants can use  $\alpha$ -naphthaleneacetonitrile to make growth substance. Production of emanations ( $\text{C}_2\text{H}_4$ ) from growing plants is increased by growth substances. N M Naylor

Sex hormones and related substances (Dirschel) 10

## G—PATHOLOGY

H GIDEON WELLS

The importance of sugar, protein and water metabolisms in the study of obstructive jaundice. C F Cárrega Cassañón and Luis M. Dottu. *Rev. asoc. med. argentina* 49, 1261-4 (1935).—Clinical. E S G. Barron. Phenolsulfonophthalein in hepatic cirrhosis. Gabriel Peco and Francisco I. Ferreira. *Rev. asoc. med. argentina* 49, 1265-8 (1935).—In some cases of disturbances of the liver the renal elimination of phenolsulfonophthalein is diminished. E S G. Barron.

Kidney and bladder stone formation and dietetic treatment. Ikuro Mizuno. *Arch. klin. Chir.* 182, 375-91 (1935).—Stone soly urates occurred in 25% of cases on a Ca-free diet and 68.6% on P-free diets. Addn. of  $\text{MgSO}_4$  to basic Ca-free, P-free and salt free diets increased the percentage of stone soly. Eleanor W. J. Butz

Gastrointestinal manifestations of hyperinsulinism. Seale Harris. *Am. J. Digestive Diseases Nutrition* 2, 557-67 (1935).—Case reports of hyperinsulinism indicate that gastrointestinal manifestations of this new disease entity are important. Dietary management or resection of the pancreas and removal of insulinomas have produced clinical cures. Studies of fasting blood sugar, dextrose-tolerance tests and glucemia detns during attacks in gastrointestinal patients may reveal hypoglycemia as the underlying factor in many cases. Edward Eagle

Gout and water supplies in Holland. J. F. Reith. *Water* 17, 1-13 (1935). *U S Pub Health Eng. Abstracts* 15, W, 104 (Oct 5, 1935), cf. C A. 29, 7832.—Investigations carried on since 1924 show that I deficiency is the cause of endemic gout. There was a reciprocal relationship between gout frequency and the I content of foods, water supplies and soils. By using common salt to which 10 mg of KI per kg has been added, about 80 parts per billion of I per day, or 30 mg per year, can be consumed without danger of I poisoning. The possible addn. of I to Dutch water supplies is discussed. C. R. Fellers

Flocculation and dispersion of colloidal hydrosols by blood serums. P. Legrand. *Bull. biol. pharm.* 1935, 407-10, et seq.—Discussion. L. E. Gilson

Lipase content of the liver in normal persons and in fatty degeneration of the liver. Noel Fiesinger and Alfred Gaydos. *Compt. rend. soc. biol.* 120, 766-8 (1935), cf. C A. 28, 6478.—In fatty degeneration the lipase of the liver and blood serum is greatly decreased. L. E. G.

Glutathione of the erythrocytes in hyper- and hypothyroidism. Alberto R. Beaux. *Rev. soc. argentina biol.* 11, 489-99 (1935). *Compt. rend. soc. biol.* 120, 822-3 (1935).—In dogs the normal glutathionemia undergoes wide fluctuations due to unknown factors. After thyroidectomy the glutathione increases and in exptl. hyperthyroidism it decreases. Results of expts with rats and guinea pigs were too irregular to permit definite conclusions. In human hyperthyroidism corpuscle glutathione decreases. After thyroidectomy it slowly increases to a little above normal. L. E. Gilson

Mechanism of the rapid production of an intense and lasting allergy in guinea pigs by the ingestion of dead

tubercle bacilli mixed with liquid petrolatum. A. Saenz. *Compt. rend. soc. biol.* 120, 870-3 (1935). L. E. G.

The reactions to the nonspecific protein treatment of infectious diseases. Ludwig Hektoen. *J. Am. Med. Assoc.* 105, 1765-7 (1935).—The effects of nonspecific proteins in infectious diseases appear due to the activation of nonspecific as well as specific antimicrobial body processes. F. P. Griffiths

Nonspecific protein therapy. Russell L. Cecil. *J. Am. Med. Assoc.* 105, 1846-54 (1935); cf. preceding abstr.—A review of the mechanism, reactions and clinical applications of therapy by injection of protein material in the treatment of disease. One hundred references. F. P. Griffiths

Behavior of serum proteins in pathological conditions of pregnancy and puerperium. R. Olivetti and G. Valle. *Minerva med.* 1935, 1, 777-83.—In normal physiological pregnancies and puerperium the total proteins were generally within low normal limits with a slight displacement of the albumin/globulin ratio. There were no particular changes in the serum proteins during various pathological conditions and in cases with edema the calcn of the colloid-osmotic pressure by Govaert's formula showed a const. ratio. Helen Lee Gruel

Carbohydrate exchange in hypophyseal conditions and diagnostic value of the glucemic curve. G. C. Dogliotti. *Minerva med.* 1935, 1, 783-5.—The glucose-tolerance test was made in 5 patients with normal glucemia after fasting. In 2 hypopituitary patients the glucemic curve rose above 2 mg/100 soon after ingestion of glucose, then fell gradually to slightly above normal after the third hr. In 3 hypopituitary patients there was a slight hyperglucemia in the first hr., followed by a slight hypoglycemia with return to normal after the third hr. Too few cases were studied to enable definite conclusions. H. L. Gruel

Experimental studies on some biological effects of ascorbic acid. II. Antigenic power of mixed diphtheria toxin and ascorbic acid. Eugenio Schwarz and Ferdinando Cislighi. *Minerva med.* 1935, II, 521-2; cf. C A. 29, 7482.—Guinea pigs injected with a mixt. of diphtheria toxin and ascorbic acid developed no immunity toward the toxin, although expts *in vitro* showed that such a mixt. lost its toxicity and pptg. power. III. The effect of ascorbic acid on anaphylactic shock. *Ibid.* 522-4.—Ascorbic acid injected simultaneously with, or 5 min. before, the shock injection of horse serum into sensitized guinea pigs apparently manifested no influence on the development of shock. Helen Lee Gruel

Nitrogen and mineral metabolism in Trypanosoma congolense disease. M. H. French. Tanganyika Territory, *Ann. Rept. Dept. Vet. Sci.* 1934, 59-64.—Infection of cattle with *T. congolense* results in an increased rate of excretion of N, Ca, K and P. Mg balances do not appear to be disturbed. The effect on Na and Cl metabolism appears to be dependent upon the level of intake, retention occurs on an adequate consumption but a neg. balance results on a low intake. Any pica which develops during the course of the disease is the result of the animal attempting to correct the excessive loss of minerals from the body and to neutralize the acidosis which develops. K. D. J.

Immunological application of placental extracts. Elhott S. Robinson and Charles F. McKhann. *Am. J. Pub. Health* 25, 1353-8 (1935). J. A. Kennedy

Studies on the minimal threshold of the dental sign of chronic endemic fluorosis (mottled enamel). H. Trendley Dean and Elias Elvove. *U S Pub Health Repts.* 50, 1719-29 (1935).—The "mottled enamel index" of Colorado Springs, Colo. is "slight." A milder type is endemic in Monmouth and Galesburg, Ill. In Pueblo, Colo., the index is "neg." The mean annual F content, based on monthly exams of the municipal water of Colorado Springs, Colo., between Nov., 1933 and Oct., 1934, was close to 2.5 p.p.m. The corresponding mean annual F contents of the municipal waters of Monmouth, Ill., Galesburg, Ill., and Pueblo, Colo., were close to 1.7, 1.8 and 0.6 p.p.m., resp. Eight references. J. A. K.

The icterus index in the newborn infant. Barnett E. Bonar. *Am. J. Diseases Children* 50, 1143-5 (1935).—

Hyperbilirubinemia in infants exists at birth and continues through the first 12 days regardless of the presence of clinical jaundice, which may not be detected until the icterus index reaches 30. The av. icterus index of the blood of the umbilical cord is 12 and that of the infants blood shortly after birth is 13. During the first 5 days the index rises to 53 and gradually decreases to 25 by the 12th day. In infants with clinical jaundice the curves showed the same general trend with a slightly higher index at birth and a higher max value reached 2 days later than the max in nonicteric infants

E. R. Main  
Phosphorus of blood IV. Phosphorus partition in the blood of children with disease Genevieve Stearns and Edna Warweg *Am. J. Diseases Children* 50, 1164-72 (1935); cf. C. A. 29, 2581\*—In infants with tetany, the changes in the blood P were similar to those observed in rickets, viz., a decrease in the ester P of the corpuscles and an increase in plasma phosphatase In malnutrition and in osteoporosis, the ester P of the corpuscles was lowered although not to the degree observed in rickets The ester P of the serum was markedly increased and that of the corpuscles was often decreased in osteomyelitis In acute renal disturbance, alterations in all forms of P were observed No characteristic changes in blood P were observed in children with hypercalcemia, hyperthyroidism, mild diabetes, dwarfism or osteopetrosis E. R. M.

The quinine test for hyperthyroidism Israel Bram *J. Lab. Clin. Med.* 21, 123-7 (1935)—Patients with hyperthyroidism can apparently tolerate large quantities of quinine. The tolerance varies in direct proportion with the height of the basal metabolic rate Dosages of 30-90 grains of quinine sulfate per day can be tolerated without evidences of cinchonism, while normal persons can tolerate only 3-12 grains per day. The diagnostic test consists in the administration of 30 grains per day and is considered pos. if after 4 days there are no evidences of cinchonism. The test does not differentiate between toxic adenoma and exophthalmic goiter.

E. R. Main  
The incidence of nondiabetic glucosuria B. V. Glassberg, *J. Lab. Clin. Med.* 21, 152-6 (1935)—There appears to be no relation between the level of sugar in the blood and its appearance in the urine. In nondiabetic glucosuria the blood-sugar content is less than 100 mg. % and sugar is found in the urine 3 hrs. after the ingestion of 100 g. of glucose, while in diabetes the blood sugar content is usually greater than 150 mg. % after 3 hrs. Of 238 cases with a tentative diagnosis of diabetes or glucosuria, 13% were nondiabetic.

E. R. Main  
A chemical study of the alum-diphtheria toxoid precipitate Edward Buxbaum and Charles K. Greenwald *J. Lab. Clin. Med.* 21, 157-63 (1935)—Toxoids prepd. from lacto-veal broth require alum in a concn of 2% for the max. pptn. Those prepd. from fresh veal broth require a concn. of 1.5%. The toxoid should be heated to 40° before the pptn. The concd. toxoid prepd. from fresh veal broth contains a greater no. of L<sub>1</sub> units per mg. of N and appears to have more desirable phys. characteristics

E. R. Main  
Gonadotropic hormone (prolan) in relation to carcinoma of the cervix. James A. Halsted *New Engl. J. Med.* 213, 503-5 (1935).—An increased content of gonadotropic hormone in the urine was observed in 4 of 15 cases of carcinoma of the cervix uncomplicated by ovarian deficiency. This incidence does not appear to be large enough to be of diagnostic value.

E. R. Main  
Reticulocyte responses in the pigeon produced by maternal effective and noneffective in pernicious anemia with description of histologically different reactions of bone marrow Gullt Lindh Muller, *New Engl. J. Med.* 213, 1221-6 (1935)—The injection of active, purified liver exts. into pigeons maintained under standard lab. conditions causes an increase in reticulocytes. Changes in the megaloblastic bone marrow similar to those in pernicious anemia patients treated with liver ext. are observed. The intravenous injection of lysine or leucine causes a similar reticulocyte response and a histologic change in the bone marrow consisting of growth and extension of erythroblastic tissue

E. R. Main

1 Investigation of a method for testing the therapeutic effectiveness of substances in carcinoma. IV. Proteins, albumoses and amino acids B. Lustig and H. Wachtel *Z. Krebsforsch.* 42, 397-409 (1935); cf. C. A. 29, 3029\*—Casein, protoalbumoses and deuterolalbumoses do not protect cancer cells against soln. by normal serum. In vivo, these substances tend to prolong the life of tumor-bearing animals. Almost all amino acids have some anti-carcinogenic action either in vitro or in vivo. V. Amines and amino alcohols *Ibid* 409-16—Many aliphatic and aromatic amines tend to inhibit the growth of tumors and to prolong the lives of tumor-bearing animals. Indole, skatole, 1-methylindole, 2-methylindole, 7-methylindole and 3-indolepropionic acid tend to inhibit the growth of mouse tumors. Indole and the isomeric methylindoles exert a protective action on cancer cells in vitro

E. R. Main  
2 Preliminary observations on the indole and skatole contents of blood in nephropathies Mario Zappacosta *Boll. soc. ital. biol. sper.* 10, 708-10 (1935)—In acute glomerulonephritis and in nephritis, indole and skatole do not increase, in renal sclerosis and in uremia, the increase is very moderate from 0.030 mg. % to 0.10 mg. % The increase is neither const. nor proportional to the degree of renal injury. The conclusion is that these bodies contribute little if any to uremic poisoning P. Masucci

3 Gastric acidity in chronic arthritis Edward F. Hartung and Otto Steinbrocker *Ann. Internal Med.* 9, 252-7 (1935)—In 70 patients with chronic arthritis, achlorhydria occurred in 26% of the cases with rheumatoid arthritis, and in 25.6% of the cases with osteoarthritis. Hypochlorhydria was detected in 17% of the former and 3% of the latter. Subacidity is an important factor in the chem. picture of chronic arthritis. J. T. Myers

4 Von Gierke's glycogen disease Lionel M. Lindsay, Alan Ross and F. W. Wigglesworth, *Ann. Internal Med.* 9, 274-81 (1935)—Although sufficient amylase is present in the liver, blood and urine, it cannot liberate glucose from glycogen. Either the glycogen in this condition is resistant to the enzyme or there is some barrier between the glycogen and the enzyme J. T. Myers

Immunological applications of placental extracts; effectiveness by oral administration C. F. McKhann, A. A. Green, L. E. Eckles and J. A. B. Davies *Ann. Internal Med.* 9, 388-97 (1935).—Protein exts. composed of globulins from human placenta contain diphtheria and scarlet fever antitoxins, and antibodies which neutralize polio-myelitis virus, and protect susceptible children against measles. There have been some moderately severe reactions after injection. Oral administration seemed to protect against measles and there was no reaction

John T. Myers  
5 Blood cholesterol in disturbances of the basal metabolic rate Lemuel C. McGee, *Ann. Internal Med.* 9, 728-38 (1935)—The figures for fasting plasma cholesterol in 52 normal adults ranged from 150 to 228 mg. %, the av. being 193. In 145 patients there was no correlation between blood cholesterol and basal metabolic rate, or the condition of the patient. Thyroid disease is accompanied by blood cholesterol changes and plasma detns. may be of value in following the progress of treatment in an individual.

John T. Myers  
The mechanism of healing in collapse therapy. Max Pinner, *Ann. Internal Med.* 9, 501-15 (1935).—A reduction of O tension and increase in CO<sub>2</sub> tension may produce conditions less favorable to the tubercle bacillus.

John T. Myers  
Attempts at vitamin C therapy in experimental poliomyelitis Claus W. Jungeblut, *J. Biol. Sci.* 31, 34-5 (1936), cf. C. A. 29, 8127\*—There is a probability that vitamin C injected subcutaneously in the proper dose has therapeutic value in exptl. poliomyelitis John T. Myers

The action of formalized virus as a preventive of experimental equine encephalomyelitis in guinea pigs Herald R. Cox and Peter K. Ohitsky, *J. Biol. Sci.* 31, 38 (1936).—The virus in contact with 0.4% formalin for 2 to 4 days immunized guinea pigs. John T. Myers



The effect of combination with diazo compounds on the immunological reactivity of antibodies. Harry Eagle, Dorothy J. J. glesston Smith and Percy Vickers. *J. Bact.* 31, 65-6 (1936).—Sufficient coupling with diazo compds. eventually destroyed the reactivity of all antiserums studied. This was due to the progressive and simultaneous decrease in the reactivity of all antibody mols., rather than to the inactivation of an increasing number of mols. The groups in protein which participate in its reaction with diazo compds probably include aliphatic amines, the NH groups of histidine, tryptophan, proline and hydroxylamine and the tyrosine OH. John T. Myers

The immunological properties of an artificial carbohydrate protein antigen containing glucuronic acid. Walter F. Goebel. *J. Bact.* 31, 60 (1936).—An artificial glucuronic acid-protein antigen, prep'd by combining the diazonium salt of the *p*-aminobenzyl glucoside of glucuronic acid with foreign protein, will react in dilns. 1-1,000,000 with antipneumococcus horse serums, types III and VIII. A corresponding antigen contg. glucose is serologically inert. It is suggested that the chem. basis for the immunological cross reactions of these pneumococci resides in the configuration of the uronic acid constituents of the sp. sol. substances. The immunological activity of the artificial antigen may be due to the interaction of uronic acid antibodies, elicited by the highly polar uronic acid constituents of the bacterial polysaccharides, with the uronic acid radical of the artificial antigen. John T. Myers

Hydrogen ion concentration in the preparation of tuberculin protein antigen. I. Ernest B. Hanan and Sophia Zurett. *J. Bact.* 31, 64-7 (1936).—The optimum  $pH$  for the extn. of the tuberculin proteins from the organism without serious deterioration of antigenic power was 7. The optimum  $pH$  for pptn. of the protein was 2.8 with a range between 2.6 and 4.6. John T. Myers

A preliminary study of the absorption spectra of antigens. M. O. L. Crowe. *J. Bact.* 31, 67 (1936).—Differences occur between the curves of 3 types of beef heart antigen and between 2 samples of one of the types. John T. Myers

Chemical and immunological properties of bacterial proteins. Michael Hechtelberger, Arthur E. O. Menzel and Forrest E. Kendall. *J. Bact.* 31, 68 (1936). J. T. M.

Is immunological aggregation (flocculation) specific? Sanford B. Hooker. *J. Bact.* 31, 69-70 (1936).—In a mixt. of 2 pptg. antigen-antibody systems, the antibody-coated mols. of each antigen seem to enter indifferently into the formation of the mixed ppt. John T. Myers

A new method of preparing antigen for serological tests in syphilis. Augustus Wadsworth and Rachael Brown. *J. Bact.* 31, 72-3 (1936).—Fresh ground beef heart was infused at 55° with 20% NaCl soln., treated with acetone, filtered, pressed and dried in a current of air. The NaCl reduced bacterial decompn. and so altered the tissue that it could be extd. directly with alc. without preliminary ether extn. John T. Myers

The influence of snake venom on coagulability of blood. Th. Lunk. *Zentr. Bakt. Parasitenk.* 1 Abt. 135, 160-1 (1935).—Some venoms contain a substance which hinders coagulation, some increase it, some have both factors and others have neither. John T. Myers

Staphylocoxin. W. Seifert. *Zentr. Bakt. Parasitenk.* 1 Abt. 135, 100-8 (1935).—The toxin should be considered, not in terms of receptors, toxophore groups, etc., but in chem. and physiol. terms. Staphylococcus toxin is considered to be a lipoid sp. enzyme. Cytolysis naturally follows lipoid binding and a disturbed regulatory mechanism of the ions in cells and serum. Susceptibility to staphylococcus depends not on salt but on fat metabolism. The frequency of staphylococcus infections in bone marrow may be due to its high fat content. John T. Myers

Lipase of the blood serum, and diastase of the urine in diseases of the pancreas and of the liver. W. Hülcher. *Acta Brevia Neerland. Physiol., Pharmacol., Microbiol.* 5, 129 (219.5).—A discussion. F. D. Walter

## II—PHARMACOLOGY

A. N. RICHARDS

A review of burn therapy. L. Stambovsky. *Drug and Cosmetic Ind.* 37, 743-7 (1935); cf. C. A. 29, 747P.

H. M. Burlage

Poisons and first aid in the cases of poisoning. Max Grünwald. *Pharm. Post* 68, 550-2 (1935). H. M. B.

Chronic carbon tetrachloride poisoning. Julius Löwy. *Arch. gewerbepath. gewerbephys.* 6, 157-9 (1935).—Symptoms in 20 cases are described. Liver function is impaired. Eleanor W. J. Butz

The effect of sulfur in experimental carbon monoxide poisoning. K. Volt and H. H. Schmidt. *Munch. Med. Wochschr.* 82, 1393-5 (1935).—The effect of S injection into CO-poisoned guinea pigs was studied. Hyperglucemia did not develop. Eleanor W. J. Butz

Substituted naphthalenesulfonic acids with regard to blood coagulation, staining and tumor affinity. K. Kottman. *Schweiz. med. Wochschr.* 65, 533-5 (1935).—The compds. tested were 1,8-aminonaphthol-3,6-disulfonic acid (I), 1,8-iodonaphthol-3,6-disulfonic acid (II), 1-cholalylamino-8-naphthol-3,6-disulfonic acid (III), diazotized I coupled to I, diazotized I coupled to cholalyl and diazotized I coupled to iodonaphthol-3,6-disulfonic acid I and II inhibit *in vitro* coagulation of blood completely in 1% soln. III inhibits in 0.5% concn. The azo compds are up to 10 times as effective. Injected into rabbits, these compds proved inhibitory. These preps inhibit pepsin, rennet, oxidase and reductase. They exert an anticomplementary effect with Wassermann-positive sera. The naphthalenesulfonic acids cause rouleau formation and blood sedimentation. Tumor-tissue affinity was demonstrated with mouse carcinoma. E. W. J. B.

The influence of insulin on experimental hyperemia. A. I. Lewin. *Z. ges. exp. Med.* 96, 532-47 (1935).—Feeding cholesterol in oil to rabbits raised the blood cholesterol (I) considerably, and the sep. fat fractions and lipid P of the blood to a less extent. This hypercholesterolemia disappeared when feeding of I was stopped. The lipocyte coeff. increased, probably because of storage of I by the tissues. Insulin accelerated the assimilation and storage of I, the hypercholesterolemia appearing more slowly and the development of atherosclerotic changes being suppressed. Blood sugar was unchanged during the hypercholesterolemia. No conversion of fat to carbohydrate could be detected. Eleanor W. J. Butz

The action of narcotics on the condition of living substance. The infrared effect in the narcosis of the transversely striated muscles. P. J. Jundé. *Protoplasma* 24, 268-80 (1935).—The so-called muscle Raman spectra can serve as an indicator of muscle swelling, for these spectra can be extinguished in a characteristic fashion in Ringer as well as in Ringer plus narcotic. Swelling in both these media is looked on as an equiv. phenomenon. The normal sartorius muscle of the frog was investigated by swelling in Ringer and in Ringer plus narcotic. It has been shown that even in narcosis, there is no increase in the infrared effect. Model expts. on colloidal media showed that each lessening of dispersion or any coagulation is accompanied by an increase in the infrared effect. From this it may be concluded that the congelation of living substance in narcosis is not conditioned by a coagulation. J. is of the opinion that one is dealing here with a structural change in the living substance, which is to be explained by the thixotropic nature of protoplasm. Twenty eight references. F. L. Dunlap

Action of vegetative adjuvants on emulsions. Gustav Bayer and Theodor Wense. *Protoplasma* 24, 281-5 (1935); cf. C. A. 28, 7356P. F. L. Dunlap

Histidine in the treatment of peptic ulcer. John T. Eads. *Am. J. Digestive Diseases Nutrition* 2, 426-30 (1935).—A preliminary report on the histidine injection treatment of peptic ulcers. Gastric ulcers appear to show more rapid improvement than do duodenal. F. B.

The treatment of amebiasis with iodohydroxyquinoline-sulfonic acid. I. W. O'Connor and C. R. Hulse. *Am. J. Digestive Diseases Nutrition* 2, 448-9 (1935).—From studies

on 152 cases of *Endostrongylus histolytica* infection, treatment with iodoxyquinolinesulfonic acid (anayodin) seems to be effective in stopping severe symptoms rapidly and curing acute and carrier cases. I. Edward Fagle

Action of  $\beta$ -aminophenylsulfamide in experimental streptococcus infections of mice and rabbits. J. Tréhou, Mme J. Tréhou, F. Nitti and D. Boyet. *Compt rend soc. biol.* 120, 756-8 (1935).—Death of the infected animals was delayed several days by ingestion or injection of the compound. L. F. Gilson

Effects of alkaline extract of the anterior hypophysis on the mineral constituents of the blood plasma. R. Gerschlager and A. D. Marenzi. *Rev soc. argentina biol.* 11, 580-8 (1935). *Compt rend soc. biol.* 120, 817-20 (1935), cf. C. A. 29, 33-5. In normal and thyroidectomized dogs injection of large doses of the ext. causes an increase in combined CO<sub>2</sub>, glucose, Ca, Mg and inorg. P and a decrease in Cl and Na. The hypercalcemia produced by the ext. occurs after hypophysectomy or thyroidectomy but not after thyroparathyroidectomy, indicating that the ext. stimulates the parathyroids. L. I. G.

Diuretic action of potassium salts. Norman M. Kenh and Melvin W. Binger. *J. Am. Med. Assoc.* 105, 1584-91 (1935).—The diuretic effects of KNO<sub>3</sub>, KCl, K<sub>2</sub>HPO<sub>4</sub>, KOAc and K<sub>2</sub>CO<sub>3</sub> are reported. Of these of edema an increased urinary output was observed in 49. Dosage was usually 6-12 g. of KNO<sub>3</sub> or its equiv. daily. Thirty-five references. I. P. Griffiths

The use of unsaturated fat acids in the treatment of eczema. S. J. Taub and S. J. Zakon. *J. Am. Med. Assoc.* 105, 1675-8 (1935). Daily ingestion of 15-30 cc. of purified linseed oil did not benefit patients suffering from eczema. Allergy was noted in one instance. Use of the oil is discouraged. I. P. Griffiths

The treatment of acute alcoholism with ten percent carbon dioxide and ninety percent oxygen inhalation. L. J. Robinson and Sydney Selesnick. *J. Am. Med. Assoc.* 105, 1734-8 (1935).—Administration of the gases for a period of time (30 min. or more) sufficient to re-establish and maintain normal respiration may prevent death in cases of acute alk. coma and cyanosis. Twenty-three references. I. P. Griffiths

Diuretic action of intravenous (administration of) sodium dehydrocholate. Franklin A. Wiegand. *J. Am. Med. Assoc.* 105, 2031-40 (1935).—Sodium dehydrocholate, formed by the oxidation of cholic acid, is relatively nonhemolytic and nontoxic. Administration of 10 ml. of a 20% soln. in combination with salyrgan, 0.25-10 ml., resulted in increased diuresis in 8 patients. Twenty references. I. P. Griffiths

Ether oil rectal analgesia in obstetrics. Modified technique. J. T. Gwathmey and C. O. McCormick. *J. Am. Med. Assoc.* 105, 2044-7 (1935).—The rectal mixt. now used consists of quinine alkaloid 1.3 g., EtOH 3 ml., paraldehyde 8 ml., 1 i, 10 75 ml., and enough liquid paraffin or olive oil to make 120 ml. Details of technique are given. Twenty-two references. I. P. G.

The use of benzedrine for the treatment of narcolepsy. M. Prinzmetal and W. Bloomberg. *J. Am. Med. Assoc.* 105, 2051-4 (1935).—Benzedrine, a phenylisopropylamine, was found 2-5 times more effective than ephedrine in the prevention and cure of sleep attacks and cataplexy. I. P. G.

Effect of posterior hypophysis extract on inorganic phosphorus of blood and urine. Giorgio Dodero. *Arch. farmacol. sper.* 60, 422-9 (1935).—Tonephin injected subcutaneously or intravenously into rabbits produced a hyperphosphatemia but no increase in phosphaturia. Helen Lee Gruehl

Action of amino acids (tryptophan) in experimental anemia. Angelina Levi. *Arch. farmacol. sper.* 60, 437-48 (1935).—Doses of 0.1001 g. per kg. injected on alternate days for several months markedly improved exptl. anemia in rabbits and caused a return of red cells and hemoglobin to near normal values. H. I. G.

Tri tetrathylammonium phosphate in therapy. Angelo Spanio. *Minerva med.* 1935, II, 582-4. In 8 patients with various malades tri tetrathylammonium phosphate was

well tolerated and manifested a beneficial effect, stimulating heart and circulation and producing a marked tonic effect on the nervous system. Helen Lee Gruehl

Parallel between adrenaline and hypophyseal vasopressin administered intravenously in man. S. Deleamari, I. S. Arica and C. D'Arrigo. *Minerva med.* 1935, II, 584-602. Adrenaline and tonephin produced parallel effects of splenocontraction, hyperleucemia and oliguria but contrasting effects on heart and blood vol. Adrenaline caused erythrocytosis, leucocytosis, hyperplasmia and hyperglobulia whereas tonephin produced a slight diminution in erythrocytes and hemoglobin and a leucocytosis. Helen Lee Gruehl

Action of arsenobenzene on transplanted tumors. Leopoldo Bertellotti. *Minerva med.* 1935, II, 602-5. Daily subcutaneous injections of 0.2 mg. arsenobenzene had no effect on the development of adenocarcinoma of the Ehrlich type in mice. In rats daily subcutaneous injections of 2 mg. favored the development of the Galliera type of tumor. Arsenic could be detected chemically in both types of tumors. Helen Lee Gruehl

The nature of the resistance to treatment shown by some cases of bovine trypanosomiasis. H. I. Hornby. *Tanganyika Territory, Ann Rep. Dept. Vet. Sci.* 1934, 37-9, cf. C. A. 28, 11031. In the treatment of *T. congolense* infection of bovines with Sb compds. it is unlikely that the drug succeeds in reaching and killing all the trypanosomes within the body. Complete sterilization is, therefore, dependent on subsequent antibody action. Unusual refractoriness to treatment is more often due to the failure of the host to produce the antibody than special resistance to the drug on the part of the parasite. K. D. Jacob

The action of  $\beta$ -phenylenediamine on the chemical processes in striated muscle. V. Parshin. *Compt. rend. acad. sci. (I. R. S. S.) [N. S.]*, 3, 113-16 (1935).—The only effect of injection of  $\beta$ -phenylenediamine (I) into frogs on the chem. processes in striated muscle is a derangement of the lactic acid-formation process. So much lactic acid is formed due to the poisoning effect of I that there is little detectable difference in the amt. found in working and resting muscle. C. E. P. Jeffreys

The effect of hydrazine in the production of acetone bodies in the phlorizin-intoxicated animal. Max Greenberg. *J. Biol. Chem.* 112, 431-6 (1936).—Phlorizinized fasting rats excrete less acetone bodies after hydrazine intoxication than before and this decrease is independent of any decrease in deamination of ketogenic amino acids or any increase in the metabolism of glucose or protein. A. P. Lothrop

Multiple sclerosis. Effect of typhoid vaccine and adrenaline on coagulation of the blood. Benjamin Simon and Philip Solomon. *Arch. Neurol. and Psychiat.* 34, 1296 (1935).—In patients with multiple sclerosis and in normal subjects, the drop in clotting time was about the same when typhoid vaccine was given, but the duration of the drop was about 2 1/4 times as long in cases of multiple sclerosis as in the normal. When adrenaline was given the drop was 5 times as long and the duration 3.7 times as long for cases of multiple sclerosis as for normal people. G. H. W. Lucas

The colloidal metal absorption by tissue cells. I. The influence of serum, serum albumin, as well as serum globulin, on the metal absorption by the surviving rabbit liver. Kap-Soo Lee. *Folia Pharmacol. Japon.* 21, Opera Orig. 1-9 (1935).—The surviving rabbit liver was perfused with 0.02% collagol in 0.85% NaCl soln. to which was added rabbit serum, serum albumin or serum globulin. The presence of these lyophilic colloids decreased to about one half the quantity of Ag remaining in the liver tissues, as compared with that found when the soln. of collagol alone was used. II. The influence of various lyophilic colloids on the metal absorption of tissue cells in the surviving rabbit liver. *Ibid.* Opera Orig. 11-20 (1935).—The surviving rabbit liver was perfused with 0.02% collagol soln. ontg. 1% Witte's pepton, 1% egg white protein, 1% egg albumin, 0.5-1% gelatin, starch or gum arabic. The presence of these colloids de-

creased the Ag remaining in the liver practically the same as previously shown, except that the addn. of starch was almost without effect. The up-take of metal was less with gum arabic than the other colloids. III The influence of various lyophilic colloids on the metal absorption of the tissue cells of the surviving spleen and kidney. *Ibid* Opera Orig 33-40 (1935).—Spleen or kidney perfused with a simple collagol or with collagol in the presence of serum, egg white or gelatin shows that the presence of the lyophilic colloid decreases the amt. of metal absorbed by the tissue. The serum afforded a greater protection against the up-take of the metal than did the other colloids. Histological changes are discussed. G H W Lucas

The influence of the thymus hormone on the poisonous action of opium alkaloid. Kaoru Arima, *Folia Pharmacol Japon* 21, Opera Orig 41-7 (1935).—Morphine, heroine and codeine were given subcutaneously in suitable doses to young suckling rabbits, some of which were given thymus ext. and some from which the thymus gland was extirpated. The removal of the thymus rendered the animals a little less sensitive to these alkaloids, but the injection of thymus ext. greatly increased their sensitiveness to the drugs. G H W Lucas

The action of  $\beta$ - $\gamma$  hexenol, a constituent of the raw leaves of *Tea sinensis* Japonica, together with a comparison of this substance with hexyl alcohol. II The action on the vessels, skeletal muscles and motor nerve endings. Seisaku Murakami, *Folia Pharmacol Japon* 21, Opera Orig 165-74, Breviaria 48 (1935), cf C A 30, 768.—By use of a prepn. of  $\beta$ - $\gamma$ -hexenol 97-98% pure, it was found that because of a depression of muscle activity the visceral vessels in the toad were greatly dilated. In a nerve muscle prepn. of the frog gastrocnemius both the muscle and motor nerve were depressed. The action resembles that of hexyl alc., but is stronger. G H W Lucas

Pharmacological studies on the automatic movement of the rabbit testicle. II The influence of the thyroid and pancreas on the sensitivity of the testicle against acetylcholine and adrenaline. Reiji Uchihashi, *Folia Pharmacol Japon* 21, Opera Orig 175-89, Breviaria 49 (1935).—The day following thyroidectomy the sensitivity of the musculature of the rabbit testicle is increased to adrenaline and acetylcholine and then gradually approaches the normal. In animals repeatedly injected with thyroxine, the sensitivity to acetylcholine is very marked, while to adrenaline it is generally weaker. A decrease in sensitivity follows when very large doses of either poison are given. After repeated insulin injections, the sensitivity to acetylcholine is increased, but that to adrenaline may be unchanged or weaker. The thyroid and pancreas may play a role for maintaining the tonus of the motor nerves for the testicle. G H W Lucas

Changes in the blood or serum viscosity of the rabbit under the influence of alcohol. Hitoshi Wakai, *Folia Pharmacol Japon* 21, Opera Orig 207-12, Breviaria 51 (1935), cf C A 30, 767.—When dil. alc. (100 cc. in 3% EtOH per kg.) was given rabbits orally, a diuresis followed, accompanied by a decrease in serum and blood viscosity. No change in specific viscosity was apparent. If the same dose of 50% EtOH were given, which set up a deep narcosis, the protein concn. of the serum diminished while the blood viscosity and specific viscosity of the serum increased slightly. The albumin-globulin quotient increased at the same time. G H W Lucas

The diuretic action of tobacco smoke. Adolph Venusch and Rudolf Schöller, *Med. Klin* 31, 1336 (1935).—Tobacco smoking raises the blood pressure and causes a diuresis. G H W Lucas

Dilaudid as a pain depressant in obstetrics. G Steigelmann, *Med. Klin* 31, 1337 (1935).—Dilaudid may be used in obstetrics as a pain depressant intramuscularly or per os in combination with such drugs as scopolamine or picroton, without danger to mother or child. G H W Lucas

Two cases of thallium poisoning. Hans Heinz Brennecke, *Med. Klin* 31, 1494-5 (1935).—A report on 2

cases of poisoning by Tl, one medicinal and the other suicide, by use of a rat poison contg. Tl sulfate.

G H W Lucas  
Damage to heart muscle through insulin. E. Schonbrunner, *Med. Klin* 31, 1571-2 (1935).—Insulin appears to be contraindicated in diabetics who have angina.

G H W Lucas  
Health risks through carbon monoxide. Spitta, *Med. Klin* 31, 1595-9 (1935).—A review. G H W Lucas  
Hypnotics and sedatives in circulatory conditions. Robert Weiss, *Med. Klin* 31, 1665-7 (1935).—A review. G H W Lucas

Chemotherapy in leucosis of fowls. J. Engelbreth-Holm, A. Rothe Meyer and E. Uhl, *Acta Path. Microbiol. Scand* 12, 491-510 (1935).—Neither the Pb compd., R 237b, nor plasmochin, administered intravenously, had any effect on the development, duration or degree of erythroblastosis in fowls. Rhodogone caused a reduction in the no. of takes in animals inoculated with a pure erythroleucosis strain but had no effect on those inoculated with a combined erythroblastosis-sarcoma strain. The active principle is inactivated *in vitro* by Rhodogone but is unaffected by R 237b or plasmochin. E. R. Main

Cataracts and dinitrophenol. David G. Cogan and Frances C. Cogan, *New Engl. J. Med.* 213, 854-6 (1935).—A review of 20 reported cases of cataract following the use of dinitrophenol in therapeutic dosage. The formation of the cataracts appears to be caused by tissue anoxemia with consequent damage to the lens epithelium. E. R. Main

The action of certain chemical stimuli on cultures *in vitro*. I Benzene. Francesco Guerico and Rosario Arnone, *Boll. soc. ital. biol. sper.* 10, 621-2 (1935).—Benzene was emulsified in plasma, dild. with NaCl soln., and added to cultures of rabbit bone marrow *in vitro*. The results were neg.; there was no difference between the cultures with benzene and those without. The results also were neg. with cultures of the rabbit genital app. *in vitro* previously stimulated with prolactin. P. M.

Pharmacologic studies on nupercaine. I The action on the isolated heart of the frog. Comparison with cocaine. Antagonism with adrenaline. R. Santi and B. Zweifel, *Boll. soc. ital. biol. sper.* 10, 648-52 (1935).—The min. active concn. of nupercaine is 1.4-5 million on the isolated heart of *R. esculenta*. If the period of contact is long, the drug is active even in greater dilns. The drug is fixed by the cardiac muscle and accumulates until an active dose is reached. The action is reversible only after repeated washings with fresh perfusion liquid. The concn. of the drug is not as important as the time it has acted in bringing about reversibility. The min. active concn. of cocaine is about 40 times and the min. paralyzing concn. about 25 times greater than those of nupercaine. The action of cocaine is much more readily reversible than that of nupercaine. Atropine sulfate, atropine and cocaine do not influence noticeably the action of nupercaine. Adrenaline has a marked effect. When the adrenaline is allowed to act before or simultaneously with nupercaine, the functional changes caused by nupercaine appear more slowly and are very weak, when adrenaline acts after the nupercaine, the pulsations become more regular with a simultaneous increase in amplitude and frequency. Fresh perfusion liquid washes away the adrenaline more readily than the nupercaine. II The action on the isolated heart of the rabbit. Comparison with cocaine. Antagonism with adrenaline. *Ibid* 652-5.—Even in a diln. of 1.5 million after 20 min. contact nupercaine has a definite action. On substitution of fresh, pure perfusion liquid, reversibility is very slow. For cocaine, the phenomena are the same but not so intense and reversibility is more rapid. The min. active dose is about 1,200,000, the min. paralyzing dose is between 10,000 and 140,000. Atropine has no marked influence on the action of either nupercaine or cocaine, although it does retard the appearance of irregularity in pulsations and incoordination. The antagonism between adrenaline and nupercaine and adrenaline and cocaine is very evident. Peter Masucci

Radioactivity, iodine and thyroid stroma. Giacomo

Pighini. *Boll. soc. ital. biol. sper.* 10, 661-3(1935).—Radioactive water with and without the addn. of  $I_2$  was administered to rats which were later (2 to 120 days after) sacrificed, and the thyroid was removed and exam'd histologically. The animals treated with the radioactive water alone showed from the beginning more or less marked thyroid alterations. The animals which received the radioactive water plus  $I_2$  showed a thyroid with a normal structure.

**The action of physical and pharmacological stimuli on blood vessels without nerves (chick embryonic vessels).** Mario Volterra. *Boll. soc. ital. biol. sper.* 10, 667-9 (1935).—Adrenaline, acetylcholine, atropine and ephephrine are pharmacologically active when brought into direct contact with the walls of the minute vessels forming the chick embryonic vascular system, even though the vessels are without nerves or junctions. P. M.

**Insulin and weight I. Variations in the body weight, glycogen content and iodine number of adipose tissue.** F. Boeri, G. Scorz and P. Baer. *Boll. soc. ital. biol. sper.* 10, 680-2(1935).—Data obtained on rats and dogs induce the authors to formulate the hypothesis that insulin injected into animals produces an increase in body wt. by stimulating in the adipose tissue the transformation of carbohydrates into fats. II. Variations in the composition of the adipose tissue in animals treated with insulin. P. Baer, G. Scorz and F. Boeri. *Ibid.* 682-5.—Rats received 0.5 unit and dogs 2-4 units of insulin daily; 2 rats were sacrificed daily so that the first 2 animals received 1 injection, the last 2 animals, 16 injections of insulin. Samples of adipose tissue were removed from the dogs before and during treatment. During the first 5-10 days of treatment, the adipose tissue showed a gradual increase in the percentage of water, a corresponding decrease in the percentage of fats and an increase in total solids. Later, the picture changed. The water in the adipose tissue decreased while the fats increased in body wt.; a marked increase in the  $I_2$  no. of the adipose fat also took place. This process continued for 10 days when the increase in body wt. ceased and the amt. of glycogen and the  $I_2$  no. of the adipose tissue returned to normal.

**The action of thyroxine on the body weight and on the weight and nitrogen content of certain internal organs of the rat.** G. Scorz and P. L. Micheli. *Boll. soc. ital. biol. sper.* 10, 687-9(1935).—Whatever the action of thyroxine on the body wt. of rats, the wet wt. and the dry wt. of the liver, kidneys and spleen are greater than the control animals. The increase in the dry wt. of the liver and kidneys is greater than the increase in the wet wt. The percentage N of the organs of the treated animals is equal to that of the controls.

**Death by hemorrhage II. The quantity of blood contained in the viscera on death by hemorrhage.** F. Domencini. *Boll. soc. ital. biol. sper.* 10, 691-3(1935). III. The influence of adrenaline and of morphine on the return circulation. *Ibid.* 693-4.—Animals which died from hemorrhage from the femoral vein but which were previously treated with morphine gave a larger percentage loss of blood than those treated with adrenaline. Nevertheless, the blood content of the 4 organs exam'd. was as a whole greater than that of the organs of the adrenaline-treated animals.

**The influence of ascorbic acid on the coagulation of the blood (time of coagulation) in normal and pathological conditions (hemorrhagic diathesis).** L. Cott. *Boll. soc. ital. biol. sper.* 10, 697-700(1935).—Ascorbic acid injected intravenously into hemophilic subjects produces a noticeable action on the coagulation of the blood manifested by a decrease in the coagulation time and a general improvement in the clinical picture.

**The behavior of ascorbic acid and of glutathione in the organs of guinea pigs treated with bacterial poisons. I. Diphtheria toxin.** P. Nuzzi. *Boll. soc. ital. biol. sper.* 10, 710-14(1935).—Guinea pigs were injected with (a) 1 m.l. d. and (b) 0.50 m.l. d. of diphtheria toxin. The animals were killed (a) after 24 hrs.; (b) after 3 or 5 days. The organs were removed and ext'd. with 2%

$CCl_3COOH$ . Ascorbic acid and glutathione were det'd. The results show no noticeable variations in either the ascorbic acid or glutathione content of the organs of the treated animals compared with the values found in the organs of the control animals. II. Tetanus toxin. *Ibid.* 714-15.—There was no noticeable variation in the ascorbic acid content of the organs of guinea pigs treated with tetanus toxin, there was a marked decrease in glutathione in the lungs and suprarenals. III. Old tuberculin. *Ibid.* 715-17.—Guinea pigs were injected daily for 1 month with 1 cc 1 10 old tuberculin. The animals were then killed. The lungs and suprarenals showed a slight decrease in ascorbic acid and in glutathione. The spleen showed a marked increase in glutathione.

**Indole IV. The indican-indole index of the blood in various experimental conditions.** E. Macchia. *Boll. soc. ital. biol. sper.* 10, 717-20(1935), cf. C. A. 29, 81414.—After the intravenous injection of 0.50 mg. indole per kg. body wt. into normal dogs, indole is found in the blood in appreciable quantities, 0.013 mg. % after 30 min., 0.006 mg. % after 60 min. The time-concn. curve is not materially changed after the removal of a portion of the intestine, changes slightly (0.031 mg. % after 60 min.) after ligating the renal peduncles, but undergoes very marked changes after ligating the hepatic peduncle. In the last case, the indican-indole index is always less than unity. V. The indican-indole index of the blood in individuals with a normal liver and in individuals with hepatic lesions. *Ibid.* 720-2.—The indican-indole index in individuals with a normal liver does not descend below 10 (10 min. after the intravenous injection of 0.50 mg. indole per kg. body wt.). In individuals with hepatic lesions, the index is lowered in some serious cases to figures less than unity. In individuals with renal lesions or with renal and intestinal lesions, the index rises above 10.

**Hematophorphyria in the treatment of melancholia.** Alfredo Santamaria, Julio N. Quaranta, Oscar F. Gabisso and Rodolfo Nelli. *Semana med.* (Buenos Aires) 1935, II, 1747-50. A. E. Meyer

**The use of helium in the treatment of asthma and obstructive lesions in the larynx and trachea.** Alvin L. Barach. *Ann. Internal Med.* 9, 739-65(1935).—A mixt. of 80% He and 20% O has  $\frac{1}{2}$  the wt. of a comparable vol. of air. The inhalation of such a mixt. caused a decrease in pulmonary ventilation and pressure, a relative and absolute diminution in the length of expiration, and an increased rest period between respiratory cycles. It may thus relieve dyspnea.

**Protective action of certain chemicals against infection with poliomyelitis virus by the nasal route.** A. B. Sabin, P. K. Olitsky and H. R. Cox. *J. Bact.* 31, 35-6(1936).—Treatment with 4% alum or 4% tannic acid increased the resistance of monkeys.

**The action of mustard oil on cells in tissue cultures.** H. Wollmar. *Zentr. Bakt. Parasitenk.* 1 Abt., 135, 161-4(1935).—Films of mustard oil not in contact with growing cells may hinder growth or change the type of mitosis.

**Effect of various anesthetics on salivary secretion.** Benjamin H. Robbins. *J. Pharmacol.* 54, 426-32(1935).— $Ft_2O$  stimulates salivary secretion during induction and recovery if the vapors pass over the upper respiratory mucosa.  $Et_2O$ ,  $CHCl_3$ , cyclopropane,  $C_2H_6$ ,  $NO_2$ , barbital and epival all cause a cessation of salivary secretion during anesthesia by depressing the secretory center. In so far as change in rate of salivary secretion is an index of irritant action, the order of irritation is  $Et_2O$  and  $CHCl_3$ , cyclopropane,  $C_2H_6$  and  $NO_2$ , the latter being nonirritating.

**The reduction of pitressin and pitocin with cysteine.** Robt. R. Sealock and Vincent du Vigneaud. *J. Pharmacol.* 54, 433-47(1935).—Neither reduction with cysteine nor subsequent reoxidation affected the pressor and oxytocic principles of the posterior pituitary gland. Benzoylation or methylation destroyed the activity of the reduced material but did not affect the activity of the nonreduced compds. Conclusion: Pitocin and pitressin contain S

in the disulfide form, and the sulfhydryl or potential sulfhydryl group is essential to their activity T H R

The toxicity of methyl mercaptan for fresh-water fish Arch E Cole *J Pharmacol* 54, 448-53 (1935)—Methyl mercaptan affects fish in the same manner as mammals, acting on the central nervous system and producing death by a paralysis of the respiratory muscles T H R

Metabolic response of white rats to continued administration of dinitrophenol B Terada and M L Tainter *J Pharmacol* 54, 454-62 (1935)—The metabolism of young rats was unaffected by dinitrophenol (I) even in quantities of several fatal doses Continued administration of I did not produce tolerance I was without effect when fed to rats with the diet, apparently because of relative tissue-insensitivity in the young and slow gastrointestinal absorption and rapid renal secretion in both young and adult rats When excretion was interfered with by removal of the kidneys, 1 mol I caused the use of over 4000 mols O<sub>2</sub> T H R

The pharmacology of nicotine Harry Gold and Frederick Brown *J Pharmacol* 54, 463-76 (1935)—Barbital (I) antagonizes the action of nicotine (II), although it does not prevent death from 2 fatal doses Such death is without convulsions The emetic action of II is not abolished by I The disappearance of the convulsive, emetic and respiratory stimulating effects of II after repeated doses may be due to partial peripheral motor paralysis Respiratory stimulation by II appears to be central, while respiratory depression and paralysis are peripheral T H R

The relative efficiencies of a series of analeptics as antidotes to sublethal and lethal dosages of pentobarbital, chloral hydrate and tribromoethanol (avertin) O W Barlow *J Pharmacol* 55, 1-23 (1935)—Picrotoxin, metrazole, ephedrine, artificial respiration, coramine, icoral, strychnine and caffeine sodio-benzoate were found to be of more or less symptomatic value (in the order mentioned) in antidoting temporarily the depressant effect of the hypnotics T H R

Ether dosage after preanesthetic medication with narcotics (barbiturates, magnesium sulfate and morphine) Frank A Calderone *J Pharmacol* 55, 24-39 (1935)—After morphine and subanesthetic doses of Na amyltal there was no difference from normal in blood Et<sub>2</sub>O concn causing surgical anesthesia or respiratory stoppage MgSO<sub>4</sub> had little effect on blood Et<sub>2</sub>O concn for anesthesia, but did lower it for respiratory stoppage The margin of safety of Et<sub>2</sub>O anesthesia was unaltered by preliminary medication with sedative doses of morphine or several different barbiturates, and was lessened with MgSO<sub>4</sub> T H R

The pharmacological action of alkaloids of fumaraceous plants II Corydine R A Wand *J Pharmacol* 55, 40-5 (1935)—Corydine (C<sub>20</sub>H<sub>20</sub>O<sub>4</sub>N<sub>2</sub>), prisms, m 148.5°, produces in intact animals an initial stage of drowsiness later mixed with fibrillary twitches in isolated muscles, and in large doses produces strychnine like convulsions with death due to asphyxia Corydine given intravenously in rabbits produces an initial fall in blood pressure followed by a rise to normal or above and increased tone and slows the perfused frog heart The tone and height of contraction of the excised uterus were increased T H R

Barbiturates XII Factors governing the distribution of barbiturates James M Dille, Charles R Linegar and Theodore Koppanyi *J Pharmacol* 55, 40-61 (1935), cf C A 30, 128—Barbiturates can be detected in the blood as long as they are present in the tissues, and no organ tissue has any specific affinity for them, although barbital is less concd in the brain and pentobarbital sodium more concd in the brain than in other organs XIII The duration of action of barbiturates Theodore Koppanyi, Charles R Linegar and James M Dille *Ibid* 62-71.—Long-acting barbiturates are destroyed slowly, hence they are present in the brain for long periods Pentobarbital is quickly destroyed and rapidly disappears from

the central nervous system; hence it is short-acting

Narcoses and the chronaxie P K Knoefel *J Pharmacol* 55, 72-81 (1935)—Cocaine, chloral hydrate and urethan influence excitability rather than cond of nerve The chronaxie is not an index of the excitability of nerve during narcosis T H R

Alcohol injected intravenously Effect of habituation on rate of metabolism Henry W Newman and Windsor C Cutting *J Pharmacol* 55, 82-9 (1935), cf C A 30, 163—Tolerance to alc, if it exists in the dog, is due to tissue tolerance, not to an increased rate of alc metabolism T H R

Sex variation in the ketonuria of ether anesthesia in rats Geo A Emerson *J Pharmacol* 55, 90-6 (1935)—Ketonuria is greater in females Ketosis was not produced by adrenaline in doses of 0.2 mg per kg at 4-hr intervals T H R

Choline and certain of its analogs I The pharmacological activity of acetylphosphocholine and acetyl arsenocholine relative to acetylcholine Arnold DeM Welch and Martin H Roepke *J Pharmacol* 55, 118-26 (1935)—The qual actions are identical, but the P analog has only 5 to 15% and the As analog only 1 to 2.7% the activity of acetylcholine by various tests T H R

Homogluceic or hypogluceic curves for injection of glucose José A Pángaro *Dia méd* 6, 731 (1934), *Anales asoc quim Argentina* 23, 24B—Homogluceic or hypogluceic curves, in which "hyperglucosemic waves" are lacking, observed in cases with or without preprior to the test, are found to be related to latent hyperinsulinism E M Symmes

Emetine in the treatment of enterocolitis and nonamebic diarrhea Jaime Damianovich *Dia méd* 6, 969 (1934), *Anales asoc quim Argentina* 23, 24B—Emetine HCl, injected subcutaneously in initial doses of 0.01-0.015, is a valuable remedy and has no contra-effects E M S

Coagulation Bleeding and calcemia Its modification by ingestion of a mixture of calcium lactate and ammonium chloride Roque A Poletti *Dia méd* 6, 1091 (1934), *Anales asoc quim Argentina* 23, 24B—Oral administration of Ca lactate with NH<sub>4</sub>Cl causes a 5.04 times diminution of coagulation time and a 2.86 times diminution of bleeding time, as compared with the use of Ca lactate alone Calcemia is not modified E M Symmes

Utilization and tolerance of the monogluceids J Garcia-Blanco *Dia méd* 6, 1090 (1934); *Anales asoc quim Argentina* 23, 24B—Fructose, galactose and mannose appear to be converted into glucose before utilization It appears in the urine soon after its circulation in the blood is started. Xylose is absorbed slowly, and its hepatic retention is also slow, its utilization small, and elimination by the liver easy E M Symmes

The effect of adrenaline on arterial and venous plasma sugar and blood flow in dogs and cats C F Cori, R E Fisher and G T Cori *Am J Physiol* 114, 53-68 (1935), cf C A 28, 6460—Adrenaline solns were protected against destruction *in vitro* by the addn of antioxidants Intravenous injection of 0.015 and 0.003 mg per kg per hr produced hyperglucemia in amygdalized dogs and cats, resp By the use of a blood-flow recorder, rates of injection of adrenaline were found which increased plasma sugar and lactic acid without changing significantly the blood flow through muscle or through the whole leg The significance of the arterio-venous difference as a measure of sugar utilization in the tissues and the errors involved in such detns are discussed E D Walter

Influence of certain polycyclic hydrocarbons on the growth of the Jensen rat sarcoma Alexander Haddow *Nature* 136, 868-9 (1935), cf C A 28, 4129, 29, 5187—Daily intraperitoneal injections of ac colloidal suspensions of 1,2,5,6-dibenzanthracene, 1,2-benzopyrene, 5,6-cyclopenteno-1,2-benzanthracene and 1,2-benzanthracene in 0.5% gelatin caused a marked inhibition of the growth of the Jensen sarcoma On the other hand, anthracene and phenanthrene proved completely devoid of inhibitory power under the same conditions L D W

Effect on tumors of intravenous injections of new soluble complex salts of ferriacorbene. Fernand Arlong, Albert Morel and André Jossierand. *Compt. rend.* 201, 745-7(1935); cf. C. A. 29, 7471<sup>1</sup>—The Ca, Mg and Pb salts of ferriacorbene were prepd. and tested as previously described. Julius White

Concomitant variations of the chronaxie and the nervous excitability under a pharmacodynamic effect. Action of cocaine and its substitutes on the motor nerve of *Rana esculenta*. Jean Régner and André Quevauviller. *Compt. rend.* 201, 912-14(1935)—The sciatic nerve of *Rana esculenta* was isolated and chronaxie measurements were made. After the administration of cocaine or procaine,

there was a marked decrease in excitability but no change in chronaxie. Julius White  
Antagonistic effect of potassium iodide in baldness due to thallium acetate. O. V. Hyleš and F. A. Diakov. *Nature* 136, 685(1935)—Rats given 3, 4 and 6 mg. of TI-OAc (I), resp. per os per kg body wt. caused a loss of hair after 12 days. At the end of 35 days most of the rats were bald. Rats on a similar diet but supplemented with 0.75 cc 2% KI subcutaneously completely retained their hair coating. The mortality caused by the toxicity of I was also markedly reduced. J. W.

Malaria medicaments (Fel'dman, Kopplovich) 10.  
Halogen derivs of procaine (Frejka, Vymetal) 10

## 12—FOODS

F. C. BLANCE AND H. A. LEPPER

Protein and moisture content of wheat grown in New Mexico. C. W. Botkin. *New Mex. Agr. Expt. Sta. Bull.* No. 230, 16 pp (1935)—New Mexico wheats contain approx. 4.6% less moisture than the 13.5% av. for moister climates and, therefore, contain more solid per bu. than other wheats. The av. protein content of wheats grown under dry farming conditions over a 7-yr. period is 13.7%, the max. yearly av. being 15.6, calcd. on a 13.5% moisture content. The protein content increased when the yield was low and decreased when the yield was high. The hard red spring wheats were high in protein and reached a max. of 17.1%. In the soft spring-wheat varieties, the protein content was low. C. R. Fellers

Biochemical changes in wheat grain under the action of high temperatures. V. L. Kretovich and F. N. Ruzantzeva. *Compt. rend. acad. sci. U. R. S. S. (N. S.)*, 3, 409-12(1935), cf. C. A. 29, 490<sup>4</sup>—Samples of various grains were heated in a chamber and the soly of gliadin in FIOH, hydration of gluten and the activities of catalase and diastase measured. Denaturation of gliadin was increased on heating to 90-130°. The hydration of gluten was markedly decreased. Catalase activity is sharply cut on heating to 90° even with quite dry grain. The diastase activity increased markedly at 60-90°, and is relatively high temp., 105°, caused a decrease in H<sub>2</sub>O, but in phosphate-citrate buffer it is resistant to even these temps. Control of grain drying and hot-conditioning could be based upon detn. of catalase activity. C. E. P. J.

Carotenoid pigments in wheat with special reference to varieties and strains. W. W. Worzella and G. H. Cutler. *Cereal Chem.* 12, 708-13(1935)—The amt. of carotenoid pigments was studied on line-ground whole-wheat meal of 29 wheat varieties and 72 hybrid strains. The finer the wheat meal the greater the extn. of the carotenoid pigments. Significant pos., interannual correlation coeffs. for carotenoid pigments were obtained between the results of different seasons when wheats of diverse nature were grown under similar environmental conditions, this indicates that carotenoid pigmentation is an inherited varietal characteristic. Hybrid families which appear to be homozygous and others heterozygous for this character are presented. The range of the wheats studied in carotenoid pigmentation, expressed as carotene in parts per million, varied from 1.66 to 3.80 for 1932-33 to 1.80 to 3.80 for 1933-34. L. H. Bailey

Wheat embryo the inorganic and phosphoric constituents. Ennio Zunini. *Chim. ind. agr. biol.* 11, 379-42(1935)—The embryos, which are mechanically cut to produce flours of good keeping qualities, contain Fe 0.25, 8.20% (on dry basis), nuclein P 0.26%, rhytin P 0.34, lecithin P 0.10, ash 4.25, K 1.09, S 0.24, Mg 0.16, Ca 0.043, Na 0.01 and Si 0.004%. Zn, Mn, Fe, Ni, Al, Cu, Co and B were detected. It is suggested to add these powd. residues to ordinary flours every time bread is prepd., to improve its quality. G. A. Bravo

Soft winter-wheat studies. IV. Some factors producing variations in wholemeal "time" data. E. G. Bayfield. *Cereal Chem.* 12, 559-68(1935); cf. C. A. 29, 3407<sup>2</sup>—The Pelshenke and Cutler-Worzella procedures give dif-

ferent results. With a 150-cc beaker B believes a 4-g doughball will have definite advantages over larger-sized doughs. The time test is empirical in nature, and relative results are the best which may be hoped for. For truly comparable data great care must be taken to see that all tests are made under comparable conditions. Of the factors studied, variation in vessel diam. proved very important as a cause of variability in the results. The doughball should be of such a size that it receives no support from the vessel used in carrying out the test. Provided a proper size of vessel is used, the time increases with decreasing size of doughball and increasing moisture in the meal (within the limits studied). Uniformity in grinding is essential. Samples which have been heated to eliminate insects give time data which are erratic and higher than those from the same samples which have not been heated. Increases in time due to aging of the meal were found to be within the exptl. error of the detn., provided the tests were made within a few days after grinding. L. H. B.

An experimental flour mill for 100-grm wheat samples. W. F. Geddes and B. Fessell. *Cereal Chem.* 12, 691-5(1935). L. H. Bailey

An experimental milling and baking technic requiring 100 grams wheat. W. F. Geddes and T. R. Aitken. *Cereal Chem.* 12, 696-707(1935)—No major changes in the flow sheet used for the regular 3-stand Allis-Chalmers exptl. mill were necessary when the 100-g. sample mill was used. Fifty g. of flour was taken to make a dough which was divided into 2 equal parts immediately after mixing. Special baking tins were obtained to accommodate the doughs from 25 g. of flour. These tins were similar to the standard "low form" tins scaled down to suitable proportions. The only baking data recorded were loaf vol. and external appearance. L. H. Bailey

Report of the 1934-35 committee on standardization of laboratory baking. W. F. Geddes, et al. *Cereal Chem.* 12, 652-3(1935)—The comm. recommends that (1) a comprehensive study be made of baking tins, (2) a further study be made of the method of reporting the results of the Standard Baking Test. L. H. Bailey

Correlation of experimental and commercial baking tests. J. Frelich, S. McHugh and C. N. Frey. *Cereal Chem.* 12, 668-91(1935)—It was found, in comparative tests using bakery and lab. methods with the same straight dough formula and ingredients, that lab. conditions in mixing or fermentation could be so adjusted as to produce results equal to those obtained in the bakery, as indicated by the characteristics of the finished bread. L. H. B.

What the baker wants in flour. Mary M. Brooke. *Northwestern Miller* 184, 493, 516(1935)—Chemical factors, such as protein and ash, are only indicators of strength, grade and uniformity of milling. We must rely on the baking test for baking characteristics. L. H. B.

The baking strength of flour. M. J. Blush. *Northwestern Miller* 184, 817, 831-5(1935)—In this country "strength" is usually regarded as an expression of a flour's capacity to make a large well-risen loaf. For all practical considerations a flour is fundamentally as "strong" as its protein suggests. "Weakness" is a term that should be

reserved for flours of low gluten content, and possibly also for those flours whose inferiority may be traced to unsoundness or to ingredients or treatments that have been excessively superimposed upon the flour. L. H. B.

Definition and measurement of "flour strength" as an inherent property of wheat. M. J. Blish and R. M. Sandstedt. *Cereal Chem.* 12, 653-64 (1935).—Wheats of equal protein content have essentially the same baking potentialities, in terms of loaf vol., even though they do not all respond equally to a single baking formula and procedure. Therefore, for all practical purposes, protein content and inherent "flour strength" are one and the same thing. "Strength" as an inherent property is to be determined by the baking test but by the protein test. The essential purpose of the baking test is to measure the response of a flour to single and combined treatments and environments that are likely to be accorded to it under modern industrial conditions. All possible combinations should be tried if necessary. L. H. Bailey

A simple method for determining the "yellowness" and "grade" of wheat flours. A. G. Simpson. *Cereal Chem.* 12, 569-74 (1935).—No more elaborate apparatus than a standard type of colorimeter is required, the rest of the equipment being ordinary lab glassware. The method depends on the extn. of flour with acetone, by which the yellow color due to carotenes can be determined, and again with 80% acetone, from which, after making alk., measurements of the "flavone yellow" color, gives a measure of the "grade" of flour. As regards the determination of the "grade" of flour, the flavone-color determination gives a more sensitive method than ash-content determination and is applicable where the latter cannot be used. L. H. Bailey

Contingent glucidic and alcoholic fermentation of wheat flours. R. Geoffroy. *Compt. rend. acad. agr. France* 21, 847-9 (1935), cf. *C. A.* 27, 1679.—The formation of maltose during bread making is optimum at 63° and is influenced by the sanitary condition and nature of the wheat, the amt. of extn. and the method of grinding. The vols. of fermented doughs before baking are related to the ranking of the flours as classified by "diastatic powers." Factors governing the speed of fermentation are: temp. of the medium, amt. of yeast, origin of the yeast, quantity of salt and consistency of the dough. The effect of the pH on the flours is felt at the beginning of the fermentation.  $K_2BrO_4$ ,  $(NH_4)_2S_2O_8$  in traces and toluene in large amounts slightly retard the speed of fermentation. Cu sulfate and  $NH_4$  phosphate activated the alc. fermentation. Dough reaches its max. vol. at the end of 4 hrs. of normal fermentation. The speed of fermentation was studied by measuring the  $CO_2$  formed. J. R. Adams

Paprika flours from Sieged. István Horváth. *Készletügyi Közlemények* 38, 161-7 (1935).—The flours are mixtures of the pericarps and washed kernels of paprika fruits. Those which contain much kernels are dark-reddish, contain much oil and less ash and sugar. They contain 0.085-0.116% etheral oil according to Griebel, 5.32-6.06% extractable ash, 2.37-2.45% N and 14.84-12.51% sugar as invert sugar. S. S. de Finitis

Test dough mixer calibration. Quick Landis and D. Freulich. *Cereal Chem.* 12, 665-7 (1935).—Two methods of calibration are described: (1) use of the Farnograph as a direct calibration instrument, (2) indirect comparison with a mixer of known calibration or a previously calibrated flour of known development energy value. L. H. B.

Sugars of flour and dough. H. Colin and H. Belval. *Bull. soc. chim.* [5], 2, 1907-12 (1935).—White flour contains, at the most, 1% of sol. saccharides, of which at least 50% is levulose (I) (cf. Tanret, *Bull. soc. chim.* 5, 724 (1891)). Maltose occurs in the dough as a result of the amylolysis of the more fragile starch grains. In the presence of yeast, sucrose and reducing sugars are quickly eliminated from the dough by fermentation. The consequent rising of the dough is maintained by the maltose present. It persists partially in the raised dough. Flour which has been deprived of its amylolytic power by sterilization with boiling alc. gives only 0.15% of alc. on fermentation, a yield which demonstrates the unimportance of preformed fermentable sugars. C. R. Addinall

The yield and the composition of the milk of the ewe. Wm. Godden and Claude A. Puddy. *J. Dairy Research* 6, 307-12 (1935).—Averages of the compn. of all the samples taken from 8 ewes during a two-yr. period expressed in g. per 100 cc. are: total solids 19.30, total protein (N  $\times$  6.38) 6.09, casein 4.61, albumin and globulin 0.88, sugar 4.81, fat 7.43, total ash 0.97,  $CaO$  0.292,  $P_2O_5$  0.380 and  $ClO$  0.78, and sp. gr. 1.0317. The compn. of milk taken from grazing ewes in various parts of Scotland is also given. The milk did not show any marked differences in compn. as a result of wide differences in the Ca content of the rations. A. H. Johnson

The influence of high environmental temperature on the secretion and composition of milk. S. Barlett. *J. Dairy Research* 6, 283-9 (1935).—When cows were subjected to high temp. produced artificially, only slight reduction occurred in milk yield and in the butter fat and solids not-fat contents of the milk. Since summer conditions are known to effect considerably larger changes in these characteristics it is suggested that high temp. is not the only factor responsible for the low milk yield and the lower fat and solids not-fat contents of summer milk. A. H. Johnson

Application of the Schiff-Sørensen reaction in the determination of proteins in milk. Carlo Nuti. *Ann. chim. applicata* 25, 482-8 (1935).—The Schiff-Sørensen test was applied to the determination of total N and casein in a no. of milks. The N determinations are in very good agreement, but casein determinations are practically valueless. A. W. C.

Nonprotein nitrogen of cow milk. A. E. Perkins. *Ohio Agr. Expt. Sta., Bull. No. 548 (53rd Ann. Rept.)*, 62 (1935); cf. *C. A.* 28, 1110<sup>a</sup>.—The nonprotein N of milk comprises about 5% of the total N and is readily obtained in the filtrate after the pptn. of the casein and albumin. Methods are being developed for the determination of urea,  $NH_4$ , amino acids, creatine and creatinine in milk. Urea makes up about 50% of the total nonprotein N of milk and is the most variable constituent. Uric acid also shows much variation in amt. The latter is higher in milk from cows fed low-protein rations. C. R. Fellers

The milk of a typical herd of Shorthorn cows. III. Nitrogen distribution, chloride, lactose, copper and iron content over a period of two years. Wm. Lewis Davies. *J. Dairy Research* 6, 363-8 (1935).—Analyses are reported over a 2-yr. period on weekly samples of milk from Shorthorn cows covering fractional distn. of N, together with lactose, chlorides, Cu and Fe. The data are tabulated and discussed and serve as a comprehensive standard of typical Shorthorn milk on which criteria of the normality of individual samples may be based. A. H. J.

Methylene blue reduction test and the number of bacteria in milk. J. D. Filappo. *Chem. Weekblad* 32, 601-2 (1935).—Four hundred and twenty-six samples of com. milk were examined for bacteria by the time required for reduction of methylene blue and by bacterial count. The time required for milk to decolorize the dye is only roughly related to the no. of bacteria in the milk. Milk showing a reduction period shorter than 2 hrs. is not suitable for human consumption. W. Gordon Rose

Chemical analysis of milk and its application to cheese factories. Juan Minut. *Ind. lechera* 15, 595 (1933). *Anales asoc. quim. Argentina* 23, 26B.—Methods, standards and proof of watering of milk. E. M. Symmes

Comparison of thermal processes (for milk). Juan Minut. *Ind. lechera* 15, 762-3 (1933); *Anales asoc. quim. Argentina* 23, 26B.—A review of methods of investigation of heating milk. E. M. Symmes

Various methods of grading milk. M. M. Miller. *Proc. 8th Ann. State Coll. Wash. Inst. Dairying* 1935, 32-5.

Grading milk at the factory. The value of the methylene blue reductase test and the fermentation test. S. B. Thomas and D. E. Ralph. *Welsh J. Agr.* 11, 209-24 (1935).—There appeared to be some general correlation between the results obtained by the reductase and color-form tests. A high percentage of the samples placed in reductase grades III and IV contained large nos. of coli-

form organisms. Coliform organisms were not detected (in 0.1-ml samples) in 67% of 1088 samples of milk that were placed in grade I by the reductase test. There was no marked correlation between the presence of coliform organisms and the results obtained by the fermentation test.

K. D. Jacob  
Vitamin D developments F. B. McKenzie *Proc 8th Ann. State Coll Wash Inst Dairyng* 1935, 22-5. The several methods of producing vitamin D milks are discussed, viz., by feeding irradiated yeast to the cows, by adding vitamin D concentrate to the milk and by irradiating the milk.

A. H. Johnson  
The daily fluctuation of the fat content of milk Zoltán Csulák *Mezőgazdasági Kutatóisk* 8, 270-8 (1935).—Milk of cows in the 3rd 5th month of lactation averaged 2.5-5.5% fat. Evening milk contained most, morning milk the least fat. In case of 3 milkings daily the 12 night hours supplied 49.02% of the whole milk and 41.62% of the whole amt. of fat. In case of only 2 milkings daily the night 12 hrs gave 56.4% of the amount of milk and 49.83% of the amt. of fat.

S. S. de Finály  
An unusual variation in the butter-fat content of milk K. Hartley and D. W. H. Baker *J Dairy Research* 6, 353-63 (1935).—The butter-fat content of milk is consistently higher in the morning than in the evening during the wet season and the reverse is true during the dry season. The relative quantities of milk produced in the evening during the dry season are slightly less than those produced during the wet season but this difference is inadequate in explaining the change in percentage of butter fat. Some evidence is offered that the change in butter-fat content of the milk is directly due to the marked climatic differences between the 2 seasons and that it cannot be controlled by altering the diet of the cattle.

A. H. Johnson  
Comparative study of the vitamin A content of butter fat from four breeds of dairy cattle T. S. Sutton and W. E. Krauss. *Ohio Agr. Expt. Sta., Bull* No. 548 (53rd Ann. Rept.) 59 (1935).—Composite samples of butter fat obtained over a 2-yr. period showed that definite breed differences in the vitamin A activity occur. Holstein and Guernsey fats gave similar biol. responses. Ayrshire and Jersey were likewise similar but lower in vitamin A content. The biol. values of these fats were not correlated with their carotene content. This suggests that breeds differ with respect to the relative amts. of pro-vitamin A secreted in their milk. Ayrshire, Holstein, Jersey and Guernsey in winter yielded, resp., 1.45, 1.95, 2.03 and 3.45 mg. of carotene per kg.; summer values were 4.70, 8.00, 12.10 and 20.50 mg. per kg.

C. R. Fellers  
A monohydroxy-palmitic acid in butter fat A. W. Bosworth and G. E. Helz *J Biol Chem* 112, 459-92 (1936).—Butter fat contains an optically active monohydroxy-palmitic acid which differs from any of these acids previously described. The Pb soap is sol. in Et<sub>2</sub>O and the Ba soap in CH<sub>2</sub>. The m. p. of different preps varied from 16.5° to 17.5° and [α] from 2.40° to 2.47°. All samples contained traces of unsatd. acids. On reduction with H<sub>2</sub>, palmitic acid is obtained.

A. P. Lohrhop  
Are flavor and coloring sources of bacteria in ice cream? P. H. Tracy and M. J. Prucha. *Ice Cream Trade J.* 32, 17-18 (1936).

A. H. Johnson  
High-solids ice cream Paul Young *Proc 8th Ann. State Coll Wash Inst Dairyng* 1935, 35-45.—The fact that lactose crystallizes when the solids-not-fat content of ice cream is increased operates to prevent the production of ice cream of as high milk-solids content as desired. Most methods of increasing the solids-not-fat in ice cream involve a reduction of the lactose content. The following methods of reducing the lactose content in ice cream are discussed: treatment of the milk with lactase, use of edible caseinates and centrifugation of lactose crystals from the condensed product contg. added sucrose. High solids ice creams are said to have characteristics of usual ice creams of higher fat contents.

A. H. J.  
Cultural identification of peptonizing bacteria. Juan Minut. *Ind. Lechera* 15, 36-8, 57 (1933); *Arales asoc. quim. Argentina* 23, 26B.—Technic of identification in milk, by the Gassner methods, using neutral red. E. M. S.

Application of the catalase test to butter. Geo. Cruess-Callaghan. *Sci. Proc. Roy. Dublin Soc.* 21, 253-5 (1935).—The value of the catalase test for the detn. of the quality and keeping property of butter was investigated. Reasons are given for the conflicting coeff. of correlation in results of other workers varying from +0.191 to -0.06. A coeff. of correlation between the catalase figure and the quality of butter of about -0.4, and one between the catalase figure and the keeping quality of butter of the same order have been put forward as the most reliable index of the value of the catalase test. These coeffs. have been worked out from figures, probably for ripened cream butter, previously published.

J. C. J.  
Biacyetyl in cold-stored butters C. R. Barnicoat. *J. Dairy Research* 6, 397-406 (1935), cf. C. A. 30, 174.—Acetylmethylcarbinol and biacyetyl are present in New Zealand butters made from slightly ripened cream. Most of the acetylmethylcarbinol plus biacyetyl found in such butters appears to have been added to the cream with the starter rather than to have developed during the customary mild ripening process. Little change in the acetylmethylcarbinol plus biacyetyl content of butter occurred during cold storage at 14° to 17° F. for more than 6 months. Larger quantities of biacyetyl (4 p. p. m.) added to butters made from cream with and without the use of starter showed considerable losses after cold storage for several months. The losses were of the same order for both types of butter but in the case of the butters made with starter, part of the biacyetyl has been reduced to its precursor (acetylmethylcarbinol).

A. H. Johnson  
Identification of acetic acid esters added to butter to mask adulterants Silvio Bezzi and Silvio Sacconi. *Ann. chim. applicata* 25, 407-17 (1935).—In the detn. of volatile acids in butter, their proportion is constant if the butter is genuine. Therefore, if fractions are collected, each will have a certain percentage of the total volatile acids. It has been found that if the distn. is carried out as in the Reichert-Meissl test, i. e., collecting 4 fractions of 55 cc. each, and detg. the acid in each, the proportions will be for I 0.373, II 0.285, III 0.205 and IV 0.138. These are called the coeffs. of fractionation. If tracetin has been added, the values of I and II will be low, and III and IV high. The values for acidity are in cc. of 0.1 N NaOH.

A. W. Contier  
Carotene for coloring butter T. S. Sutton and R. B. Stoltz. *Ohio Agr. Expt. Sta., Bull.* No. 548 (53rd Ann. Rept.), 59-60 (1935).—An attempt was made to use a soln. of carotene to enhance the color and increase the nutritive value of winter butter. Approx. 60 mg. of the uncolored, 45 mg. of the colored, and 30 mg. of the June butter were about equal in vitamin A content as detd. by rat assays. Fifteen drops of "caritol" were used per lb. in 1 sample and 2 cc. of "Prumatene" were used in the second sample. Carotene increases the color and vitamin A content of the butter, and its use is less misleading to the consumer than other coloring compds. which have little or no nutritive value.

C. R. Fellers  
Vitamin A content of sour-cream butter, sweet-cream butter and margarine I. L. Hathaway and H. P. Davis. *Neb. Agr. Expt. Sta., Research Bull.* No. 79, 3-8 (1935).—Nineteen samples of margarine obtained from Ill., Ohio and Neb. were analyzed chemically and the vitamin A content of each one was compared with that of either sour-cream or sweet-cream butter. The fat content of the butter samples varied from 80.2 to 81.5% while the fat content of the margarine samples varied from 78.3 to 80.2%. The samples of margarine were very poor sources of vitamin A when compared with butter. One of the margarine samples caused an av. gain of 10 g. per rat and another caused an av. gain of 25 g. per rat when fed at the rate of 1 cc. daily for 8 weeks. In every other case the rats fed margarine showed a final loss of wt. and most of them did not survive the expt. Butter was fed at a daily rate equal to 1/10-1/20 of the quantity of margarine fed in all cases but 1, and the rats survived and gained, the smallest gain averaging 45 g. and the largest 111 g. during the 8-week period.

C. R. Fellers  
The biological processes in the manufacture of margarine.



rine O Palladina *Mastobolno-Zhironoe Delo* 1934, No 4, 35-7—A discussion E Bieleuss  
Biology in the cheese factory Colorimetric acidimetry. Juan Mmut *Ind lechera* 15, 351-2, 366(1933), *Anales asoc quim Argentina* 23, 26B—The Dormic method is recommended E M Symmes

A review of recent Cheddar-cheese making experiments N S Golding *Proc 8th Ann State Coll. Wash Inst Dairying* 1935, 3-10—A review of exptl work done on Cheddar cheese within the last 5 years is given The phases of the subject considered are milk quality, effects of pasteurization, conditions of use of rennet, types of starter, loss of vitality of starters, acidity, salting, ripening and cheese defects Forty-two references

A H Johnson  
The electrical resistance of pork and bacon I Method of measurement F H Banfield *J Soc. Chem Ind.* 54, 411 13T(1935)—In order to det the concn of NaCl in various parts of cured meat, the elec resistance was measured by means of a 2 pronged probe, insulated to the tips A thermionic valve oscillator, generating current at 1000 cycles, was used This high frequency was obtained by close coupling of 3 coils of No 40 enameled wire, 2 of 5000 and 1 of 3000 turns Following this oscillating circuit was a second amplifying valve with potentiometer-controlled output This allowed output control without affecting the frequency. With this high frequency generator and thermionic-valve rectifying circuit of Callan and Horrobin (*C A* 23, 789), it was necessary to use storage batteries to supply high-tension current to the rectifying valve The Kohlrausch modification of the Wheatstone bridge was used to det. the resistance Platinization of the probe tips facilitated the manipulation Readings were standardized with small cubes of meat left in NaCl solns of various concns. until equil was reached The resistance was detd. in various parts of the piece and NaCl content checked by chem analysis A curve was constructed for a certain temp The resistance falls rapidly up to 3% NaCl, then slowly. Saltpeper had the same effect as salt Since most pickle brines contain  $\frac{1}{10}$  NaCl, the NaCl content would be 90% of the amt. indicated As a test, samples of cured meat were analyzed and the probe method was checked to 0.5% of the NaCl content A Salinity Tester is now manu. by Evershed Vignoles, calibrated for 5°, 10°, and 15° and having a scale for depth penetration II The electrical resistance of salt in solutions, gels, minced pork and bacon F. H Banfield and E H Callow *Ibid.* 413-17T—The elec probe described above was used in the detns of elec resistance (E R.) In the measurement of E R. of various concns of NaCl in  $H_2O$ , agar-agar gels, gelatin gels and minced pork, an increase of NaCl caused a decrease in the E R. The addn of  $H_2O$  to a definite mixt of minced pork and bacon decreased the E R. of the mixt, probably since there was less poorly conducting material in the same vol fat (3.3% fat) minced pork had a greater E R. than lean (0.9% fat) pork, at the same NaCl concn. Although the addn of NaCl decreased the E R. of minced pork, it also led to an increase in the internal E R. of the tissue, since, as the amt of NaCl was increased, it required a larger proportionate amt. to decrease the E R. The addn of  $KNO_3$  to minced pork decreased the E R. to exactly the same extent as a chem. equiv of NaCl The E R. of various mixts of minced pork and NaCl decreased as the temp. increased. Minced pork had less E R. than an intact piece of salted pork, at the same NaCl concn, probably because of less connective tissue and less fat in the minced product Intact pieces of unsalted pork and bacon showed greater E R. across the fibers than with the fibers, when measured between parallel plates, but with the elec probe there is no difference from which direction the E R. is measured III. The penetration of salt into muscular tissue during the curing of bacon F H Banfield and E H Callow *Ibid.* 418-21T—By the elec probe method (I), the elec resistance (E R.) of muscular tissue, before and after curing, was measured The E R. of fresh pork was considerably greater in a very fat side than in a very lean one. The E R. of muscular

tissue in all 8 sides measured was the greatest near the shoulder and least in the hind leg, therefore the muscular tissue of the hind leg must contain less fat than near the shoulder The E R. was greater near the cut surface of the side than in the deep-seated parts, probably because of loss of  $H_2O$  in the former. The effect of extra fat or loss of  $H_2O$  in increasing the E R. of muscular tissue was still evident to some extent, even after curing, yet the authors were able to observe by changes in E R. the progressive penetration of salt During both dry salting and tank curing, there was a decided fall in the E R. during the first 24 hrs, indicating a penetration of salt into the innermost parts of the sides Tank curing gave more rapid penetration of salt than dry salting, even when the sides had been previously injected with pump-pickle Chem. analysis showed there was practically no difference between the concn of salt in a very fat and a very lean side, both having been treated the same Chem analysis also showed there was a definite gradient in the concn of both salt and  $H_2O$  in the sides The outer layer, in contact with the air, had the most salt and least  $H_2O$ , even if the sides had been previously injected with pump-pickle.

R L Horst  
The effect of the feeding of kitchen-refuse upon the quality of bacon S Schmidt-Nielsen and C F. Pettersen *Norg Tek Høskole, Aethandl.* 25, 787-805(1935)—Bacon produced in certain localities has been found to be of a very soft consistency, yellow in color and becomes rancid when salted It was the purpose of the authors to ascertain if highly unsatd acids in the food can be detected in the bacon and if  $C_{18}$  and  $C_{20}$  unsatd acids are present in the fat Samples of bacon were obtained from 9 swine, which are divided into 2 general groups Group I consisted of 3 animals fed a standardized mixt. of kitchen-refuse, corn and barley, and Group II, of 6, which had been fed the usual corn-milk diet The bacon fats were rendered at the temp. of boiling  $H_2O$  in a  $CO_2$  atm The analytical constns of the fats and their acids and the properties of the benzene-sol and -insol polybromides and Me esters of these fat acids were detd., as well as the constns of the fat of the kitchen-refuse food mixt No detns were made on the fat of the food given the animals of Group II The m. p. of the fat from the softer bacons of Group I was 1.4° m. lower than that of Group II Groups I and II showed an av. Hübl I no (cor according to Schmidt-Nielsen and Owe, *C A* 18, 2818) of 69.8 and 61.2, resp., a difference of 8.6 The av. thiocyanate I nos (according to Kaufmann, *C A* 20, 2256), indicative of the amt. of fat acids with 1 or 2 double bonds, were 54.4 and 52.3, resp., a difference of 2.1 The difference between the av. I no and the av. thiocyanate I no of Group I was 15.3 and of Group II, 8.9, the amt of unsatd acids in Group I being about double that in Group II The solid and liquid acids were sepd according to the soly of the Pb salts in  $EtOH$  The av. I nos of the acids of the 2 groups were 72.1 and 64.0, the av. solid fat acids, 32.3 and 38.9%, the av. I no of the solid acids, 4.16 and 2.54 and of the liquid acids, 105.9 and 99.1, and most characteristic, the av.  $EtOAc$ -insol bromides, 2.36 and 0.29%, resp. In the 2 groups, 72.8 and 57.8% of the fat acid bromides were insol in benzene and the Br content of the total bromides was 68.45 and 67.40%, resp The av. Köttstorfer no of the benzene-insol bromides in Group I was 174.7 and av. Br content 69.79% From this it is concluded that the greater part of the fat acids in Group I is  $C_{18}H_{33}O_2$  The Me ester of the fat acids subjected to fractional distn gave, in the residue for Group I, an av. I no of 109.8, compared to 73.1 for the total acids, and for Group II, 78.8 compared to 64.2, indicating more highly mol and more unsatd acids than in the distillate, confirmed by 1 of the Group I yielding 1.04% insol bromides in the distillate and 14.15% in the residue The Köttstorfer no of the first fraction in Group I was 213.7 and in Group II, 212.0, indicating that Group I was also richer in lower fat acids The authors conclude that the unsatd acids in Group I were of marine origin, that these acids were more highly unsatd, that 90% of the unsatd acids of the food were recovered in the

including seeds and juice. The latter makes up about 29% of the wt. of the fruit and has the following percentage compn.: water 79.8, protein 0.6, fat and crude fiber 0, ash 0.48, carbohydrates (by difference) 19.1, acidity as citric acid 2.3, Ca 0.005, P 0.018 and Fe 0.018. The seeds contain 12.7% protein and 8.3% fat with an ash content of 1.92%. The juice is very palatable and possesses com. possibilities. The juice may be readily utilized in cocktails, candy, syrups, jelly and cordials.

C. R. Fellers  
Vitamin C content of paprika fruit. Istvan Szanyi. *Természettudományi Közlemények* 67, 527 (1935).—Veins, stem and seeds of Hungarian paprika contain only traces of vitamin C. Green paprika fruit contained 400, brown 800–2000, fully ripe, red paprika 1048 to 2100 mg. vitamin C per kg. Flesh or juice of paprika is much higher (3160 mg./kg.). S. S. de Fina

Influence of pectin on the velocity of inversion of sucrose. Sergio Berlingozzi and Mario Testoni. *Ann. chim. applicata* 25, 483–96 (1935).—Pectin was found by B. and Liquori (C. A. 25, 1235) to retard the velocity of inversion of sucrose. The action has now been measured quantitatively. The rate of inversion of sugar soln. by citric acid in the presence of 1% pectin was detd. at 10°, 35°, 45°, 65° and 80°. The retarding action of pectin increases with the concn. Thus, in a 13% sugar soln. the value of K at the above temps was reduced 20.4, 30.5, 22.0, 22.3 and 9.7%, resp. due to the presence of pectin. In a 5% sugar soln. the reduction in K was 20.3, 32.8, 24.7, 24.8 and 17.1%, resp. It is seen that the retarding action decreases with increasing temp. and practically disappears at 107°.

A. W. Conners  
Chemical composition of certain fodder plants from Cameron Highlands. C. D. V. George. *Malayan Agr. J.* 23, 483–5 (1935).—When cut at weekly intervals at Serdang the crude protein, fat and fiber contents of guinea grass (*Panicum maximum*) were about the same as those of grass cut every 3 weeks at Cameron Highlands. Buffalo grass (*Paspalum conjugatum*) cut in flower at Cameron Highlands was of about the same compn. as that cut every 3 weeks at Serdang. Analyses are given for Dallis grass (*Paspalum dilatatum*) and for kikuyu grass (*Pennisetum clandestinum*). The clovers were of about the same compn. as like varieties grown in Europe. J. R. N.

Digestibility of artificially dried roughages. J. A. Newlander. Vt. Agr. Expt. Sta., *Bull.* No. 400, 3–12 (1935); cf. C. A. 29, 55361.—The digestibility for cows of each of the following artificially dried roughages was detd.: corn silage, both wet and dried, oat hay, sudan grass, soybean hay and Japanese millet. The amts. of digestible crude protein and total digestible nutrients were: corn silage, dried, 3.73 and 67.71; corn silage, wet, 4.57 and 68.24; oat hay, 10.50 and 70.57; sudan grass, 12.44 and 65.25; soybean hay, 9.00 and 71.43; and millet, 8.05 and 67.06%. C. R. Fellers

New methods for preserving legume forage. A. E. Perkins. Ohio Agr. Expt. Sta., *Bimonthly Bull.* No. 177, 200–5 (1935); cf. Vitanen, C. A. 29, 24171.—New methods of ensiling leguminous crops by stack, acid-treated (A. I. V.) and artificial drying are discussed. Results of the 1st year's expts with the A. I. V. acid-treated silage show that fermentation is prevented in the ensiled hay and losses of drainage water are reduced. The product is palatable to dairy cows and, in spite of the mineral acids present, appears to be harmless physiologically. A mixt. of CaO and NaHCO<sub>3</sub> is normally fed in moderate amts. along with the silage to neutralize the acids. The method is not recommended for general adoption at the present time. C. R. Fellers

Effect of season and maturity on the composition of blue grass. C. H. Hunt and W. L. Robison. Ohio Agr. Expt. Sta., *Bull.* No. 548 (53rd Ann. Rept.), 68–9 (1935); cf. C. A. 28, 13955.—Frequent clipping decrease the total yield of dry matter but increase the protein content of the dry matter. In general, the 1st cutting in May was highest in protein and fiber content and lowest in fat content. The N-free ext. increased as the season advanced. The ash content remained fairly const. C. R. Fellers

1 Composition of hays from Somogy county. László Urbányi. *Mesgazdasági Kutatók* 8, 320–7 (1935).—Natural meadow hays, calcd. to 85% dry matter content, contained crude protein 87%, CaO 9.88, MgO 4.17 and P<sub>2</sub>O<sub>5</sub> 4.91%. Alfalfa hay, calcd. to 84% dry matter, contained crude protein 19.05, CaO 24.14, MgO 4.84 and P<sub>2</sub>O<sub>5</sub> 5.92%. Red clover hay, calcd. to 84% dry matter, contained crude protein 15.38, CaO 19.25, MgO 5.23 and P<sub>2</sub>O<sub>5</sub> 4.96%. The detailed analyses show that the compn. of various meadow hays is much influenced both by the botanical types of component plants and the different factors of vegetation. S. S. de Fina

Experiments on grass silage. C. Boyle and J. J. Ryan. *Journal (Saorstát Éireann Dept. Agr.)* 33, 143–39 (1935).—Grass silage made by the A. I. V. method contained H<sub>2</sub>O 79.90, protein 3.08, oil 0.64, fiber 5.45, carbohydrate 9.29 and ash 1.64%; the compn. of the silage closely approximated that of the original grass. The effluent that escaped from the silo contained total dry matter 5.2 and protein 1.6%. The total loss in wt. was 7.3%, as compared with 10.6–11.9% when the grass was ensiled by the ordinary method. The pH of the silage was 4.0–4.2 in the top portion and 3.7–3.8 in the bottom portion. Both types of silage were excellent feeds for dairy cows and calves. K. D. Jacob

Vitamin A content of corn silage. W. E. Krass. Ohio Agr. Expt. Sta., *Bull.* No. 548 (53rd Ann. Rept.), 62 (1935).—Corn silage contained 2.5 Sherman units of vitamin A per g. Assay of the silage showed approx. twice as much vitamin A activity (rat bioassay) as the green corn from which the silage was made. C. R. Fellers

The food value of oat hulls. E. J. Sheehy. *Journal (Saorstát Éireann Dept. Agr.)* 33, 167–72 (1935).—Hulls from white oats contained H<sub>2</sub>O 10.5, crude protein 3.94, EtO ext. 1.48, sol. carbohydrate 53.17, fiber 28.26 and ash 3.63%. In expts. with cattle, the hulls gave satisfactory results when they were substituted for good quality meadow hay in the ration at the rate of 11 lbs. to 8 lbs. K. D. Jacob

Detn. of true dry substance in solid and liquid products [flours] (Monzot) 28. Analysis for fluoride—application to detn. of spray residue on food products (Hoskins, Ferris) 7. Automatic recording balance [for use in drying macaroni products] (Bunnington, Geddes) 1. Effects of arsenical sprays on grapefruit, oranges, tangerines, Temple oranges, limes and lemons (Longfield-Smith) 15. Proteins of the cowpea (Adolph, Chiang) 11A. Jars for preservation of food (Shtern) 19. Coconut fat treatment in the confection industry (Hölscher) 27. Cellulose "bulking agent" for use in foods (U. S. pat. 2,026,865) 7. 23. Addn. compds. of butacetyl and glycerol [used in the margarine industry] (Ger. pat. 619,628) 10.

Vitaminizing food. F. Hoffmann-La Roche & Co. (Soc. anon.). Fr. 788,014, Oct. 2, 1935. The vitamin C content of food is increased by adding esters, e. g., Me or Et, of 2 keto-L-gulonic acid.

Preserving food. International Sugar & Alcohol Co. "Isaco" Fr. 788,098, Oct. 3, 1935. Pure dextrose or xylose is used for preserving foods contg. much water. This avoids excessive sweetness and sepn. of "crusts" of sugar.

Preserving milk for human consumption. Georg A. Kranse (to Katadyn, Inc.) U. S. 2,028,072, Jan. 14. Milk is partially sterilized by treatment with oligodynamically active bodies such as silvered clay pellets while in a previously heated condition (suitably at a temp. of about 63°), the heating being for a time shorter than the conventional pasteurization time and insufficient to cause complete sterilization or to impart a boiled taste to the milk, and the oligodynamic treatment is interrupted within 4 min. to avoid color or taste changes in the milk, substantially the full vitamin value of the milk is retained.

Header and tube heat-exchange apparatus suitable for treating milk. Fritz G. Cornell, Jr. U. S. 2,027,809 Jan. 14. Structural details.

Filters suitable for milk, etc. Maurice A. Goldman (to Commercial Filters Corp.). U. S. 2,028,061-2, Jan. 14. Various structural details.

Canning crab meat. Carl R. Tellers. U. S. 2,027,270, Jan. 7. Meat from the "blue crab," "rock crab" or "sand crab," before initial discoloration, is treated with an aq. soln. of an Al, Zn or Sn salt such as sulfate, nitrate or chloride contg. about 50-500 p. p. m. of Al, Zn or Sn, and the treated meat is sealed in containers and sterilized.

Confection compositions such as chocolate coatings Benjamin R. Harris. U. S. 2,027,167, Jan. 7. A fat together with dispersed comminuted material such as cocoa and sugar is used with an ester of sulfuric acid having a terminal lipophile group with at least 8 C atoms and a hydrophilic sulfate group, such as sulfonated cacao butter or the like.

Apparatus for concentrating fruit juices Marcel Mallet. Fr. 787,016, Oct. 1, 1935.

Food flavorings. Arthur Alt (to Monsanto Chemical Co.) U. S. 2,027,261, Jan. 14. A flavoring for various foods such as ice cream, confectionery, bakery products, preserves, etc., comprises an alkyl ether of protocatechuic aldehyde, such as the *m* benzyl ether, dissolved in alc. Other ingredients such as coumarin, vanillin, sugar and water may be added.

Food flavorings. Lucas P. Kyrides and Henry H. Retalliau (to Monsanto Chemical Co.) U. S. 2,027,287, Jan. 14. A vanilla-like taste and odor are given to foods such as confectionery, bakery goods, preserves, syrups, etc., by use of alkyl ethers of protocatechuic aldehyde such as the Pr, Bu, Am, hexyl, heptyl or octyl ethers,

which may be used in admixt. with various other flavoring ingredients if desired.

Caffeine-free tea. Theodor Grethe. Ger. 621,557, Nov. 9, 1935 (Cl. 53k. 3). Addn. to 608,215 (C. A. 29, 2620P). The process of Ger. 608,215 is modified by treating the tea with steam for a short time, e. g., 5-8 mins., after the extn. with an org. solvent. The conditions must be such that the temp. of the tea does not exceed 65°. The steam treatment may precede or follow the treatment with air or an inert gas.

Use of solid carbon dioxide for supplying inert gas in packages of coffee, etc. Carl H. Hansen (to John Hansen) U. S. 2,027,422, Jan. 14. Various operative details are described. U. S. 2,027,430 relates to a container adapted for use in packaging operations of this character.

Preventing oxidation and rancidity of coffee, shelled nuts, cheese, powdered-milk products, milk chocolate, etc. Sidney Musher (to Musher Foundation Inc.). U. S. 2,026,697, Jan. 7. Various solid food materials have their particles mixed or coated with finely divided antioxidative vegetable material such as oat flour or finely divided barley, hominy, soybean flour, crushed sesame seed, crushed peanuts, crushed linseed cake, soybean press cake, castor-bean pomace, corn-germ cake or corn gluten, or, in the case of roasted coffee, a small proportion of finely ground unroasted coffee. Alc. exts. or the like from similar materials also may be employed as inhibitors of oxidation or rancidity. Rubber, greases, soaps, resins and various drugs, chemicals and pharmaceutical products, etc., may be similarly preserved from rancidity, oxidation or aging changes. Numerous details and examples are given.

## 13—CHEMICAL INDUSTRY AND MISCELLANEOUS INDUSTRIAL PRODUCTS

(PLASTICS, RESINOIDS, INSULATORS, ADHESIVES, ETC.)

HARLAN S. MINER

Chemical engineering in retrospect and prospect Hugh Griffiths. *Chem. Age* 33, 591-3 (1935). E. H. Danger to the human organism from industrial solvents Otto Schulz. *Die Gasmaske* 7, 126-8 (1935).—A general discussion.

2,3-Dichlorobutane as a solvent M. V. Likhoshestov, S. V. Alekseev and T. V. Shalueva. *Nitrocellulose* 6, 226-7 (1935); cf. C. A. 29, 8174. E. M. Symmes.

The year's progress in solvents and plasticizers Thos. H. Durran. *Chem. Age* 33, 606-7 (1935). E. H.

Patented improvements in the manufacture of synthetics from nitrocellulose. Krausch. *Nitrocellulose* 6, 219-20 (1935).—A review. E. M. Symmes.

Collodion cotton as a film former in artificial leather manufacture Walter M. Münzinger. *Nitrocellulose* 6, 99-100 (1935). E. M. S.

The surface tension of fat acid condensation products and fatty alcohol sulfuric acid esters. W. Weltzien and H. Ottensmeyer. *Monatsh. Seide Kunststoffe* 40, 504-7 (1935).—Expts. were conducted (1) to purify commercially available powders so as to remove admixed electrolytes without any sepn. of the various homologs, as a mixt. of the latter is at times necessary for technical purposes, (2) to det. how surface tension depends on concn. and temp. and (3) to find out whether the addn. of electrolytes produces further changes in the surface tension. Igepon T powder (A) and Gardinol WA coned. powder (B) were purified by double recrystn. from AcOEt; Igepon A powder (C) was purified by double recrystn. from 96% LiOH. Dynamic surface-tension measurements were made with Traube's stalagmometer. A shows the greater dependence of surface-tension on the concn. at 20° and 80°. Nearly identical values were obtained with B and C at 20°. At 80° the concn. factor was less pronounced in the lower concns. than it was in the cold. At a concn. of 1 g./l. the surface tensions of all 3 products are practically identical at 20° and 80°. The difference of about 10 dynes/cm. between 20° and 80° corresponds to

the change in the surface tension of pure water. The greatest decrease of the surface tension is brought about at relatively low concns. and further addns. produce no great changes. Equally coned. solns. of the com. products contained considerably smaller amts. of the surface-tension-active ingredients than did the purified products. The addn. of electrolytes to the surface-tension-active substances tested yields, particularly at higher temp., an extraordinary, unexpected addnl. decrease of the surface tension. Leopold Scheffan.

Rock wool in relation to health Lawrence T. Fairhall, Stewart H. Webster and Granville A. Bennett. *J. Ind. Hyg.* 17, 263-75 (1935).—Cats were subjected to cold vapor, hot vapor and dust expts. with varying concns. of rock wool. The chem. data indicate no hygienic hazard. Histological examn. of the lungs, the liver and kidneys reveal no const. or significant pathol. changes.

Accidents during the removal of acids (in cleaning solutions) from well pipes Kremer. *Geundh.-Ing.* 59, 26-7 (1936).—The "Flerin" used for cleaning consists essentially of 20% HCl and a protective collod. Its action may produce sufficient CO<sub>2</sub> to replace most or all of the O present. Poisonous S-contg. gases may also be produced. Either of these factors could account for the deaths of workers reported. M. G. Moore.

Red squill investigations—effectiveness of red squill extracts as raticides. Robert E. Buck and C. R. Fellers. *Ind. Eng. Chem.* 27, 1497-9 (1935); cf. O'Connor, et al., C. A. 30, 1881.—Toxic exts. prep'd from red squill powder are efficient raticides. The best solvents are MeOH and EtOH and exts. made with a Soxhlet app. were more toxic than those prep'd by shaking or stirring. Wheat bran is a suitable carrier for the dried ext. Exts. can be prep'd on a large scale by percolation. Extd. baits are more palatable and more readily standardized than red squill powder. Field tests were made. C. W. Whittaker.

Determination of phosgene in gases from experimental

fires extinguished with carbon tetrachloride fire-extinguisher liquid W. P. Yant, J. C. Olsen, H. H. Storch, J. B. Littlefield and Leopold Scheffan *Ind Eng Chem, Anal Ed* 8, 20-5 (1936).—The results previously reported by the Bur. of Mines (cf. *C. A.* 15, 294) on  $\text{COCl}_2$  in the gases from excelsior fires extinguished by  $\text{CCl}_4$ -type fire-extinguishing liquids have been questioned (cf. Olsen, *et al.*, *C. A.* 25, 2075) on the basis that the analytical method gave erroneously high results. The Bureau, with the cooperation of the manufacturers of the  $\text{CCl}_4$ -type fire extinguisher, repeated some of the earlier work under a reproduction of the former conditions except that a different analytical method was used. In the first report the  $\text{COCl}_2$  was absorbed in alc. NaOH and the NaCl formed was detd. by titration, while in this new investigation the same constituent was detd. by passing the gas sample first through a suitable purifying tower to remove interfering substances and then through a soln. of aniline water which had been said with sym. diphenylurea. Any  $\text{COCl}_2$  in the gas sample reacts with this reagent, pptg. diphenylurea which can be filtered off and weighed, this serves as a measure of the  $\text{COCl}_2$  present. The purity of the diphenylurea formed was checked by its m. p. and also microscopically by detns. of the cryst. structure and  $n$ . The report gives a detailed description of the exptl. procedure and methods and a tabulation of results obtained. The  $\text{COCl}_2$  found in 16 expts. in which excelsior fires were extinguished in 2 sealed chambers (capacity 28 and 32 cu. m., resp.) ranged from 4 p. p. m. by vol. to 92 p. p. m., with an av. of 23.6 p. p. m. When the  $\text{CCl}_4$ -type fire-extinguishing liquid was dropped on an I-beam previously heated to red heat and without fire present the amt. of  $\text{COCl}_2$  found was 119 p. p. m. The exptl. results are in substantial agreement with those formerly reported by the Bureau. The article calls attention to the importance of using materials for test chambers which absorb minute amts. of  $\text{COCl}_2$  as this is a highly reactive gas. It is suggested that neglect of this precaution may have given rise to conflicts in results reported by previous investigators. The authors state that it is not the intention of the Bureau to discourage the use of  $\text{CCl}_4$ -type fire extinguishers, which are excellent for stopping incipient fires, but rather to det. the decompn. products which may result and which should be recognized and properly evaluated. L. S.

Gas mask disinfection with a formaldehyde water vapor mixture Bau Kein-Hun, E. Heide and Wang Kan. *Die Gasmasken* 7, 115-23 (1935).—Expts. were carried out in a gastight chamber with a capacity of about 1/4 cu. m. The  $\text{HCHO}$  water vapor mixt. was generated in place by adding 8.3 g. cryst.  $\text{KMnO}_4$  to 3.3 g. paraformosoda in 10-33 cc.  $\text{H}_2\text{O}$ . The organisms used were *Es. coli*, *Staphylococcus aureus* and anthrax spores. These were applied in known nos. on cover glasses which were exposed for different times at different temps. and humidities. It was concluded that the important factors in this method of disinfection are the  $\text{HCHO}$  content of the condensed water and the duration of the exposure.  $\text{HCHO}$  in vapor form was ineffective. The following 2 formulas for disinfecting gas masks are recommended. For general use for each cu. m. of storage space use 30 g. paraformosoda (1.8), 75 g.  $\text{KMnO}_4$ , and 9 cc.  $\text{H}_2\text{O}$ . The  $\text{HCHO}$  content of condensed water is about 2%. For known contamination with contagious diseases, for each cu. m. of storage space use 45 g. paraformosoda (1.8), 112.5 g.  $\text{KMnO}_4$ , 135 cc.  $\text{H}_2\text{O}$ . The  $\text{HCHO}$  content of condensed water is about 4%. Exposures of 1 hr. are sufficient for general use but when known contamination with dangerous diseases has occurred it is advisable to expose for 8-12 hrs. Gas masks should be hung up in the disinfecting chamber dry since water on the gas masks will dil. the concn. of  $\text{HCHO}$ . A. L. Kibler

Use of higher sulfonated fatty acids in the laundry (Brewer) 25. Sturtevant for the manuf. of glues and adhesives (Wollenberg) 1. Dicarboxylic acid esters of tetrahydrofurans) alc. (plasticizers) (Borglum) 10. Halogenated products of phenyl phenylphenyl ether [used in plastics] (U. S. pat. 2,028,081) 10. Guanyl and biguanyl compds

[having wetting, frothing and dispersing properties] (Fr. pat. 788,423) 17. Dissolving rubber to form a product used in insulation (U. S. 2,028,068) 26. Esters of etherified glycolic acids [used as softening agents] (U. S. pat. 2,027,901) 10. Unsaid. aldehydes and ketones [products used as resin components] (Brit. pat. 435,494) 10.

Extracting products by solvents Unes de Melle (Soc. anon.) Brit. 435,725, Sept. 26, 1935. See Fr. 778,689 (C. A. 29, 4833). For "extd." read "extd."

Plastic compositions. Ernest Belmans. Fr. 787,980, Oct. 2, 1935. Fillers such as sawdust, cork, asbestos or vulcanized rubber are incorporated homogeneously in plastic material by passing the latter between rollers to form a layer about 0.5 mm. thick and spreading the filler evenly on to this layer.

Plastic compositions. Jean de Granville and Léopold Davion. Fr. 788,407, Oct. 10, 1935. The use of catalysts and heat in making resins from  $\text{CHO}$  and carbamide, phenols, cresols, etc., with or without casein, is replaced by the use of high frequency currents. High-frequency currents are also used for insolubilizing casein, alone or mixed with other substances.

Plastic materials. I. G. Farberman A-G. Fr. 788,645, Oct. 14, 1935. Products of high mol. wt. contg. S and resembling rubber are made by causing alkali, alk. earth or  $\text{NH}_4$  polysulfides to react with 2-chloroethyl-2'-chloroethyl ether in the presence of a diluent such as water and substances having surface activity such as  $\text{BaSO}_4$ , preferably freshly pptd. Cf. C. A. 30, 536.

Plastic compositions containing cellulose acetate Joseph R. Mares (to Monsanto Chemical Co.) U. S. 2,028,403, Jan. 21. A neutral ester of a carboxylic acid and an acetal of glycerol and  $\text{AlCl}_3$ , such as formal glycerol benzoate, is used for plasticizing cellulose acetate.

Plastic compositions suitable for coating, molding, etc. Carleton Ellis and Wm. F. ter Horst (to Ellis-Foster Co.) U. S. 2,028,575, Jan. 7. A reaction product sol. in a restricted no. of org. solvents such as hexahydrophenol, phenols and diethylene glycol, diethylene dioxide, etc., is formed from glycerol dichlorohydrin and an alkali polysulfide by heating together. Numerous examples are given.

Cellulose nitrate plastic compositions Harold J. Titterton and Imperial Chemical Industries Ltd. Brit. 436,161, Oct. 7, 1935. Plastic compns. are obtained by dissolving cellulose nitrate in monomeric Me methacrylate contg. methyl- $\alpha,\beta$ -dichloroisobutyrate and subjecting the mixt. to a treatment that effects polymerization of the Me methacrylate. Polymerization may be in presence of catalysts, e. g.,  $\text{Br}_2\text{O}$ , succinyl peroxide.

Decoratively colored plastic magnesium articles Albert E. Cleghorn (to Travatex Products Corp.). U. S. 2,027,021, Jan. 7. In the manuf. of cast articles, there is added to a plastic mixt. of magnesium,  $\text{MgCl}_2$ , etc., a metal salt (such as  $\text{Fe}(\text{SO}_4)_3$  or a Cu salt) which reacts to give a hydroxide or oxide of the desired color.

Plasticizers for nitrocellulose compositions. Compagnie française pour l'exploitation des procédés Thomson-Houston. Fr. 788,674, Oct. 14, 1935. Mixed phthalates of glycerol or glycol with an aliphatic monohydric alc. such as  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{PrOH}$ ,  $\text{BuOH}$ ,  $\text{AmOH}$  and hexyl alc. are used.

Apparatus for extruding plastic material such as in making molded rubber articles Geo. F. Brouseau and Harvey D. Ferns (to Hood Rubber Co.) U. S. 2,024,044, Jan. 14. Structural, mech. and operative details.

Synthetic resins N. V. Industriële Maatschappij Vootheen Noury & van der Lande. Fr. 788,584, Oct. 12, 1935. Drying or semi-drying oils or their esters are polymerized and the fats of the polymerization products are split, or the fats of the oils are split and the mixed fat acids are polymerized. The unpolymerized fat acids are eliminated by distn. and the polymerized products are transformed to resins by reaction with compds. used for the production of resins, e. g., glycerol, phthalic anhydride and vinyl acetate or chloride.

**Synthetic resins, varnishes, etc.** E. I. du Pont de Nemours & Co. Brit. 435,762, Sept. 23, 1935. Resinous materials sol. in varnish oils are prep'd by causing  $\text{CH}_3\text{O}$  or a deriv. thereof to react in an alk. medium with a binuclear phenol contg. at least 1 but not more than 2 reactive positions and contg. at least 4 nonaromatic C atoms, 1 of which is a secondary or tertiary C atom that is directly joined to a  $\text{C}_6\text{H}_5$  ring in 1 of the *o*- or *p*-positions to the phenolic OH. Among examples, (1) di-(4-hydroxy-3-methylphenyl)dimethylmethane is condensed with  $\text{CH}_3\text{O}$  in presence of NaOH and the product, sol. in  $\text{Me}_2\text{CO}$ , alc. and ethoxyethanol, is blended with raw China wood oil or laseed oil, or mixts thereof, and thinned with a mineral thinner and an aromatic hydrocarbon, with or without a Co drier to yield a varnish, and (2) the resin prep'd in example (1) is blended with a polyhydric alc.-polybasic alc. condensation product contg. a drying oil or a fatty acid derived therefrom and thinned, with or without the addn. of an oil, to yield a varnish. The products may be blended with cellulose derivs., natural resins and ester gums, synthetic resins and synthetic resin-forming material, bitumens, natural or synthetic waxes, pigments, plasticizers, fillers, lakes, etc., for use in the manuf. of coating compns., molding plastics, impregnating agents for paper, wood, etc., adhesives for safety glass, insulom, cements, sealing waxes, insulating materials, etc. In 435,796, Sept. 23, 1935, divided on 435,762, oil-sol. resinous materials are prep'd by condensing  $\text{CH}_3\text{O}$  with a chlorinated di- or tri-nuclear phenol having at least 1 but not more than 2 substitutable reactive positions and at least 3 non-aromatic C atoms, 1 of which is directly joined to at least 3 C atoms and is directly joined to a  $\text{C}_6\text{H}_5$  ring as described in 435,762. In an example, di-(4-hydroxy-3-chlorophenyl)diphenylmethane is condensed with  $\text{CH}_3\text{O}$  in presence of NaOH and the product is blended with China wood oil and thinned with mineral thinner and aromatic hydrocarbons to yield a varnish. In 435,797, Sept. 23, 1935, divided on 435,762,  $\text{CH}_3\text{O}$  or a polymer thereof is condensed with a mononuclear phenol having at least 1 but not more than 2 substitutable reactive positions and at least 7 non-benzenoid C atoms, 1 of which is directly joined to at least 3 C atoms and is directly joined to the  $\text{C}_6\text{H}_5$  ring as described in 435,762. In an example, *p*-terphenyl phenol is condensed with  $\text{CH}_3\text{O}$  in presence of NaOH to yield a resin.

**Synthetic resinous condensation products.** Walther Schrauth (to Deutsche Hydrerwerke A-G). U. S. 2,027,351, Jan. 7. By heating together initial materials such as phthalic anhydride, succinic acid, ricinoleyl alc. and glycerol, resins of especially good elasticity are obtained, and generally similar products may be derived from phthalic acid, phthalic anhydride, hexahydrophthalic acid, succinic acid, adipic acid, fumaric acid, maleic acid, citric acid, tartaric acid, oxalic acid, etc. The said acids may be used alone or conjointly as a mixt.; it is advantageous to co-employ besides polybasic aromatic dicarboxylic acids simultaneously aliphatic, polybasic carbon acids, such as succinic acid, adipic acid or tartaric acid. Several examples with details of procedure are given.

**Synthetic resinous products free from phenolic odor.** Gustave E. Landt (to Continental-Diamond Fibre Co.). U. S. 2,027,988, Jan. 14. An initial phenolic-aldehyde condensation product is treated with a small proportion of a halogen such as Br, an inorg. halogen oxy-acid or a salt of such an acid such as a hypochlorite or chlorate to render the product free from phenolic odor in the final, infusible state, and the reaction product is subsequently heated to convert it into the final state.

**Resinous composition.** Daniel E. Strain (to Canadian Industries Ltd.). Can. 353,925, Nov. 5, 1935. Ten parts by wt. of methyl methacrylate dissolved in 90 parts by wt. acetone is partially polymerized and then treated with a mixt. of 20%  $\text{H}_2\text{O}$  and 80%  $\text{MeOH}$ , which ppt. the polymer but not the unpolymerized substance. The new compn. softens above  $100^\circ$ .

**Moldable resinous product.** Gerald H. Mains (to Canadian Westinghouse Co. Ltd.). Can. 354,092, Nov. 12, 1935. A first fabric is impregnated with a phenolic

resin. A second fabric is treated with a phenolic resin on one surface and a urea resin on the other surface. The second fabric is placed over the first fabric with the urea-resin surface outward. A third fabric is impregnated with urea resin and placed over the second fabric. The fabrics are treated with heat and pressure to unite them.

**Mixed resins.** Adrien A. Champetier and Albert Laporte. Fr. 787,967, Oct. 2, 1935. Mixed resins sol. in oils are obtained by esterifying resinic acids of natural resins by the phenolic functions of synthetic resins, the heating necessary being carried out in an autoclave under pressure in an atm. constituted by a phenolic product, and the heating is followed by a treatment under vacuum. The reaction mass is heated to above  $260^\circ$  as rapidly as possible.

**Polyvinyl resins.** Shawinigan Chemicals Ltd. Brit. 436,072, Sept. 30, 1935. Resins are made by causing a polyvinyl ester, other than a formate, a substance, other than  $\text{AcH}$ , contg. an active carbonyl group that combines with free OH groups of a hydrolysis product of the polyvinyl ester and  $\text{H}_2\text{O}$  to react in the presence of a hydrolyzing and acetalizing catalyst and an org. solvent, other than an alc., and, if desired, in the presence of an alc., the amt. of  $\text{H}_2\text{O}$  or  $\text{H}_2\text{O}$  and alc. being sufficiently small to give homogeneous reaction conditions such that the acetal reaction may attain or exceed 87% of completion. In a modification, a partial or complete hydrolysis product of a polyvinyl ester, other than formate, formed in absence of a carbonyl compd., is caused to react with a carbonyl compd., other than  $\text{AcH}$ , in the presence of an acetalizing catalyst and an org. solvent, other than an alc., and, if desired, in the presence of  $\text{H}_2\text{O}$  or an alc., or both, in amt. sufficiently small to permit of obtaining homogeneous reaction conditions such that the acetal reaction may attain or exceed 87% of completion. Films or threads are manuf'd by extruding the reaction mixt. into air or into a pptn. bath, with or without previous neutralization of the catalyst. The products are also suitable for the production of sheets, rods or tubes, safety glass, coating compns. and moldings. Among examples, (1) polyvinyl acetate (I) having a viscosity of 15 centipoises is heated to  $70^\circ$  with  $\text{H}_2\text{O}$  and paraformaldehyde in the presence of  $\text{H}_2\text{SO}_4$ ,  $\text{AcOEt}$  and  $\text{BuOH}$ , and (2) a hydrolysis product of I (prep'd by 50% hydrolysis of I of 2.5 centipoises viscosity in  $\text{EtOH}$  contg.  $\text{H}_2\text{SO}_4$ ) is isolated and heated to  $70^\circ$  with aq.  $\text{CH}_3\text{O}$  and  $\text{H}_2\text{SO}_4$  in the presence of  $\text{AcOEt}$  and  $\text{EtOH}$ .

**Polyvinyl resin.** Howard W. Matheson and George O. Morrison (to Shawinigan Chemicals Ltd.). Can. 353,601, Oct. 15. A polyvinyl ester is hydrolyzed with an inorg. acid and water, in any extent up to 100% hydrolysis. The product is condensed with an aliphatic aldehyde and with an aromatic aldehyde.

**Phenol-aldehyde resins.** Adolf Heck (to Cook Paint & Varnish Co.). U. S. 2,027,337, Jan. 7. A phenol, about 2 mols., is condensed with 1 mol. of an anhydride of an aromatic dicarboxylic acid such as phthalic anhydride in the presence of a chloride of Al, Fe or Zn, and the resulting condensation product is caused to react with about 5-25% its wt. of an aldehyde such as "paraform" and the material is partially resimified by heating to about  $170^\circ$  until a resinous product sol. in alcs., esters and ketones is obtained.

**Use of vinyl resin compositions for tubes, toys, electrical parts, etc.** Lauchlin M. Currie (to National Carbon Co.). U. S. 2,027,961, Jan. 14. Various operative details and final heat treatment are described. U. S. 2,027,962 relates to operative details such as covering pipes, hand grips, etc., with preformed vinyl resin compn. and then heating the material to convert it into final stable form.

**Synthetic horn.** Internationale Galalith-Gesellschaft Hoff & Co. (Henri Dumont and Werner Lück, inventors). Ger. 619,532, Oct. 4, 1935 (Cl. 39b. 18). Glass-clear horn stable to light is made by adding a mixt. of alkylaryl-alkylated aniline and dichlorohydrin to moistened casein and working up the casein by usual methods. Thus, ethylbenzylamine and dichlorohydrin are added to moistened casein. Coloring matter and filling materials are added.

The casein is then molded, pressed, etc., hardened by  $\text{CH}_3\text{O}$  and, if desired, polished

Insulating material Electric & Musical Industries Ltd. Fr. 788,531, Oct. 11, 1935. Mica, for use as insulator in elec. discharge app., is heated to about 1000° for 1-5 min., whereby its thickness is increased to about 5 times. The mica may then be subjected to pressure

Thermal insulator Edward A. Toohy and Earle R. Williams (to Johns-Manville Corp.) Can. 353,583, Oct. 15. A sheet of felted asbestos fibers with a starch binder contains water-repellant material of the type of wax distributed throughout the sheet in amt. not in excess of 10% of the wt. of the sheet.

Thermal insulator John D. Cochran, Jr. (to Formica Insulation Co.) Can. 354,110, Nov. 12, 1935. A foundation body is impregnated with a heat-resistant resin. Al sheet 0.005 in. thick is placed over the body and provided with rough surfaces. Over the Al sheet is placed a fibrous sheet impregnated with a heat-reactive resin, and a thin pigmented adhesive sheet is interposed between the Al sheet and the surface sheet. The whole is heated and pressed

Nonconducting coverings for heat Friedrich Buchwald (to N. V. Internationale Alfol Maatschappij) Brit. 434,682, Sept. 6, 1935. Vessels for transporting solid  $\text{CO}_2$  have insulation comprising 2 or more layers of corrugated milboard and bright metal foil sep. by spaces through which the gaseous  $\text{CO}_2$  evolved passes outwardly to the atm.

Dielectrics for Kerr cells Sueddeutsche Telefon-Apparate-, Kabel- und Draht-Werke A.-G. Ger. 622,368, Nov. 26, 1935 (Cl. 21a<sup>1</sup> 32 50). A benzene deriv. which contains 2 or 3 nuclear substituents, is solid at atm. temp., and has elec. insulating properties and a high dipole moment, is dissolved in  $\text{PhNO}_2$  or another liquid having about the same Kerr const. Specified benzene derivs. include *o*-dimethylbenzene, *p*-nitroaniline, and 1,2,3-dichlorotoluene

Rubber-asbestos products Dewey & Almy Chem. Co. Ger. 622,320, Nov. 25, 1935 (Cl. 39b<sup>5</sup>). See Brit. 399,870 (C. A. 28, 1824<sup>1</sup>)

Rubber-asbestos products Dewey & Almy Chem. Co. Ger. 622,416, Nov. 28, 1935 (Cl. 39b<sup>5</sup>). See U. S. 1,907,017 (C. A. 27, 3571<sup>1</sup>)

Adhesives I. G. Farben und A.-G. Ger. 621,138, Nov. 2, 1935 (Cl. 22<sup>1</sup> 2). For uniting surfaces of artificial horn prep. from casein, use is made of adhesives comprising casein 100, urea, thiourea or a deriv. thereof 50-100, and water 100-200 parts, with or without appropriate addns., e. g., a pigment, a filler, a softening or a wetting agent, or a small proportion of a hardening agent

Adhesives Fritz Masold Ger. 622,212, Nov. 22, 1935 (Cl. 22<sup>1</sup> 2) Addn. to 615,594 (C. A. 29, 8179<sup>1</sup>) The compds. described in Ger. 615,509 are improved by addn. of a small proportion of wood meal or rye bran

Adhesive polymerized vinyl-ester-resin coatings. Ernest L. Kallander and Gardner R. Alden (to Dennison Mfg. Co.) U. S. 2,027,435, Jan. 14. For giving a vinyl ester-resin coating on material such as adhesive tape, a desired degree of tackiness, it is treated with a volatile moistening liquid contg. a true solvent for the resin such as alc. and an aliphatic hydrocarbon such as naphtha serving to restrain the solvent action. U. S. 2,027,436 relates to adhesive tape coated on one face with a vinyl ester resin and on the other face with another material such as cellulose acetate capable of adhesive activation by an org. liquid, both coatings being capable of adhesive activation by a common org. liquid and capable of adhering firmly to each other when so activated but without tackiness prior to such activation. Various examples are given

Dispersing rubber in alkali silicate solutions. Max H. Kheloth (to C. F. Burgess Laboratories, Inc.) U. S. 2,028,397, Jan. 21. An alkali silicate soln. such as a soln. of Na silicate has thoroughly mixed with it rubber in the form of a rubber soln. or suspension, and a fluosilicate such as that of Na which is slightly water-sol. The resulting soln. is suitable for use as an adhesive.

Cement for abrasive articles. Albert Lloyd Ball (to Carborundum Co.) Can. 353,933, Nov. 8, 1935. A cement is composed of 16-18 parts of a liquid condensation resin in the A stage sold under the trade mark of Liquid Redmanol BR-1373, 8-10 parts  $\text{H}_2\text{O}$ , about 73 parts of particles of fused  $\text{SiO}_2$ . The  $\text{SiO}_2$  consists of approx. equal parts of particles which pass a 40-mesh screen and are retained on a 200 mesh screen, and particles which pass a 200-mesh screen

Apparatus for preparing adhesive sheets such as those used in belt manufacture Charles A. Ball (to Chicago Belting Co.) U. S. 2,026,726, Jan. 7. Various structural, mech. and operative details

Adhesive tape Donald R. Anderson. U. S. 2,027,461, Jan. 14. Various details of app. and operation

Wetting agents, etc. I. G. Farbenindustrie A.-G. Brit. 435,481, Sept. 23, 1935. Products suitable as wetting, washing, leveling, dispersing and emulsifying agents are obtained by condensing fatty acids contg. at least 8 C atoms, or reactive derivs. thereof, with polypeptides contg. 1-5 peptide groups. In examples, (1) glycylglycine is condensed with lauric acid chloride in the presence of NaOH, and (2) the mixt. of polypeptides obtained by hydrolyzing leather waste with  $\text{Ca}(\text{OH})_2$ , NaOH or  $\text{H}_2\text{O}$  is condensed with oleic or lauric acid chloride or the chlorides of coconut acids or the acids obtained by oxidizing paraffin with air. The products obtained with the last-named acids may be used in mercerizing baths. The products are useful for making dye pastes, making dyed textiles fast to rubbing and stabilizing peroxide solns.

Wetting agents, etc. N. V. Chemische Fabriek Servo and Meindert D. Rozenbroek. Brit. 436,075, Sept. 30, 1935. As assistants in processes for cleansing, wetting, bleaching, dyeing, finishing or oiling fibrous materials, leather included, use is made of compds. with a straight chain of at least 8 C atoms contg. (a) a terminal  $\text{COOH}$  esterified or amidated with an alc. or amine contg. a group derived from an oxygenated acid of S, or (b) a terminal  $\text{SO}_3\text{H}$ , or (c) a terminal persulfonic group, the straight chain carrying also a side chain that contains at least 2 C atoms and is free from acidic salt forming groups. The side chain is linked to the main chain directly or through an atom of O or N, and the C atom in the main chain to which the side chain is attached must not be in the  $\alpha$ - or  $\beta$ -position. The side chain may be built up by (1) introducing an alkyl group into the straight chain, (2) esterifying with a mono- or poly-hydric alc. a  $\text{COOH}$  linked to the main chain, or converting the  $\text{COOH}$  into a substituted amide group, the  $\text{COOH}$  may be introduced into the straight-chain compd. by way of the CN group, (3) reaction of a straight-chain compd. contg. halogen with a compd. such as a malonic, tricarballic or acetoacetic ester, i. e., a compd. in which a H linked to C can be substituted by an alkali metal, (4) esterifying or etherifying a OH linked to the straight chain, e. g., by etherifying the OH group or groups of hydroxy fatty acids, e. g., ricinoleic acid, (5) substituting a carbonaceous group, e. g., by acylation, into an amino group linked to the straight chain, or (6) causing a straight-chain compd. contg. halogen to react with an amine, diamine or hydroxy- or oxo-amine; the side chain may contain substituents, e. g., OH, oxo or nitrogenous groups, or esterified or etherified OH groups. Among 13 examples, (1) the dichlorinated acid of coconut oil is converted into a dibutyl ether by reaction with  $\text{BuONa}$  and then amidated by condensation with taurine, (2) dichlorooctylsulfonic acid is heated with NaOH soln. to yield dihydroxyoctylsulfonic acid which is treated with butyric acid chloride, (3) the product of (2) is converted into a persulfonic compd. by treatment with  $\text{Na}_2\text{O}_2$ , (4) Na dichlorooctylsulfonate (1) is heated with  $\text{Bu}_3\text{NH}$ , whereby Cl is replaced by the dibutylamino group, and (5) 1 is heated with the Na compd. of di-Et malonate

Wetting and dispersing agents Henkel & Cie G. m. b. H. Fr. 788,663, Oct. 14, 1935. Unsat. organic compds. which, besides 2 free OH groups, contain at least 1 aliphatic or cycloaliphatic radical of high mol. wt. joined to the radical contg. the OH groups by a chain of

O or S, are used. Examples are mono-octyl ethers of glycerol and diglycerol, monododecyl ethers of polyglycerols or glycerol and also the tetradecyl-, hexadecyl- and octodecyl-compds.

**Bleaching, washing and other compositions.** Henkel & Cie. G in b. II Brit 435,861, Sept. 23, 1935. Comps. consisting of alkyl-reacting mixts. conig. a prep. compd. and a salt of pyro- or meta-phosphoric acid are stabilized by an addn. of Mg silicate added in the ready-formed state Cf C A 29, 8183<sup>1</sup>.

**Detergent, wetting, antispattering, emulsifying and frothing agents.** Benjamin R. Harris U S 2,023,347, Dec. 3. Numerous examples are given of the prepn of ester derivs. of at least relatively high mol. fatty acids or relatively high-mol. alcs. with at least one semi-esterified sulfuric acid group, such as a substance of the general formula  $\text{ROX}[\text{OS}(\text{O})_2\text{O}]_n\text{V}$ , where R is an acyl radical with at least 4 C atoms, X represents the residue of an aliphatic polyhydroxy substance of a class consisting of glycerol, polyglycerols, glycyls, polyglycyls, hydroxy carboxylic acids, sugars, sugar alcs., and acid derivs. of sugars which link together the acyl radical with the sulfate group, Y is a cation, and w and v are small whole numbers, at least one. Among the compds. formed are monododecyl bisulfate, monododecyl diethylene glycol sulfate, and sulfonated mixed coconut fatty acid mono esters of diethylene glycol, sulfonated monoethyl glucose (many other compds. also being mentioned). In prep. these compds., there can be used as sources of the lipophile group such materials as melissic acid, stearic acid, oleic acid, ricinoleic acid, lauric acid, palmitic acid, cetyl alc., lauryl alc., cholesterol, mixed coconut fatty acids, mixed tallow fatty acids and other materials with marked affinity for oils and fats. For linking the lipophile groups with the sulfate groups, there can be used glycerol, diglycerol, polyglycerols, glycyls, polyglycyls, hydroxy-carboxylic acids, sugars, alcohol derivs. of sugars, acid derivs. of sugars, and other di- and poly-hydroxy org. substances. For introducing the sulfate groups there may be used sulfuric acid, concentrated or fuming, chlorosulfonic acid, sulfuric chloride,  $\text{SO}_2$  and other sulfonating agents. The treatment with these agents may take place in the presence or absence of solvents and condensing agents such as pyridine and the like.

Alkyl sulfates of high molecular weight (wetting, dispersing, foaming and smoothing agents for the textile and leather industries, etc.) Heinrich Bertsch (to H. Th. Böhme A.-G.) U S 2,027,906, Jan. 14. Unsated hydrocarbons contg. not less than 10 C atoms, such as centene, etc., are converted into sulfuric esters of the corresponding hydroxy compds. by treating them with a mildly acting sulfating reagent such as  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{O}$  at a temp. not exceeding  $0^\circ$  (suitably  $-10$  to  $0^\circ$ ).

**Phosphoric acid esters of fatty acid monoglycerides (frothing, wetting, detergent and emulsifying agents).** Benjamin R. Harris U S 2,026,785, Jan. 7. Products suitable for use in various textile treatments, etc., have the general formula  $[\text{ROXOP}(\text{O})(\text{O})_2\text{O}]_n\text{Z}$ , where "R" is a lipophile group, "X" is the residue of a polyhydroxy substance, "Y" and "Z" are cations, and "w" is a small whole no. and are nonnitrogenous esters of a phosphoric acid and a polyhydroxy substance where at least one hydroxy group of the polyhydroxy substance has its H substituted by a lipophile group. The term "non-nitrogenous" indicates substances devoid of N linked directly to C. The lipophile group may include any fatty acid group such as the fatty acid radicals of, caproic acid, capric, caprylic, valeric, butyric, abietic, hydroxystearic, benzoic, benzoylbenzoic, naphthoic, toluic, palmitic acid, stearic, lauric, melissic, oleic, myristic, ricinoleic, linoleic acid or mixed fatty acids derived from animal or vegetable fats and fish oils such as lard, coconut oil, corn oil, cottonseed oil, partially or completely hydrogenated vegetable oils such as cottonseed oil, corn oil, sesame oil and fatty acids of various waxes such as beeswax and carnauba wax; or the lipophile group may be an alkyl radical derived from an alc. corresponding to any of the preceding acids,

such as cetyl alc. or lauryl alc. Examples of polyhydroxy substances, the residues of which may serve as linkages between the lipophile groups and the hydrophilic phosphate groups, are as follows: mucic acid, tartaric acid, saccharic acid, gluconic acid, glucuronic acid, gulonic acid, mannonic acid, trihydroxyglutaric acid, glyceric acid, and the like, as well as carboxylic oxidation products of polyglycerols and sugars such as: xylitol, galactose, fructose, maltose, sorbitol, glucose, dulcitol, arabitol and other sugar alcohols such as hexahydric alcohols derived from sugars, and other substances having free hydroxy groups. Various examples with details are given.

**Nitrogen-containing esters (frothing, wetting, detergent, emulsifying and penetrating agents for treating textiles, leather, ores, etc.)** Benjamin R. Harris U S 2,023,075, Dec. 3. Numerous examples are given of the production of compds. of the general formula  $\text{L-O-COCH}_2\text{N}(\text{X})_2$ , where "L" is a lipophile group of the class consisting of alkyls, ethers and esters, and having at least 4 C atoms, "X" is an amine, and "N" represents a quinequivalent N with the three indicated valence bonds satisfied by the radical of the class consisting of alkyls, aryls, and the radical of a heterocyclic ring of which the N is a member. In this general formula, "X" may represent Cl, Br, I, NO, OH,  $\text{SO}_3$ , or an acetate or other org. or inorg. amine, and N is a quinequivalent N three valence bonds of which are satisfied by alkyl, aryl or cyclic radicals. Among the compds. production of which is involved are the cholesteryl ester of betaine chloride (I), the melissyl ester of betaine bromide (II), and of I, the cetyl ester of I, the octyl ester of II, the dodecyl ester of I, the cholesteryl ester of II. Among the acids which may be employed as a source of the fatty acid lipophile radical forming a part of the lipophile ester group are: caproic, capric, caprylic, valeric, butyric, abietic, hydroxystearic, benzoic, benzoylbenzoic, naphthoic, toluic, palmitic, stearic, lauric, melissic, oleic, myristic, ricinoleic, linoleic acid or any fatty acid with at least 4 C atoms, mixed fatty acids derived from natural fats, oils, hydrogenated oils, waxes. Tertiary amines which may be used as a source of and for the purpose of forming the quaternary nitrogen in the betaine radical are: pyridine, various picolines, quinoline, isoquinoline, quinaldine,  $\text{PhNMe}_2$ ,  $\text{PhNMe}_3$ ,  $\text{Et}_3\text{N}$ ,  $\text{I}(\text{NMe})$ ,  $\text{MeN}$  and other tertiary amines which are sufficiently reactive to form quaternary ammonium compds. In general, the preferred method of making these substances is to cause a tertiary amine, either aliphatic, cyclic or mixed aliphatic-aromatic to react with a lipophile ester of monohydroxy- or monochloro-acetic acid. The "L" of the formula may also be satisfied by an ester or ether group, such as a fatty acid ester or alkyl ether of a polyhydroxy substance. The polyhydroxy substances employed may be hydroxy carboxylic acids, sugars, alcs., sugar alcs., glycyls, polyglycyls, glycerols, polyglycerols, in fact, substantially any substance having at least two esterifiable hydroxy groups. Examples of other polyhydroxy substances are: mucic acid, tartaric acid, saccharic acid, gluconic acid, glucuronic acid, gulonic acid, mannonic acid, trihydroxyglutaric acid and glyceric acid, as well as carboxylic oxidation products of polyglycerols such as represented by the formula  $\text{HOOCCH}(\text{OH})\text{CH}_2\text{OCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OCH}_2\text{COOH}$ , xylitol, sucrose, glucose, lactose, galactose, fructose, maltose, shamosse, sorbitol, dulcitol, arabitol and other substances having free hydroxy groups.

**Esters of sulfodicarboxylic acids.** Alphons O. Jaeger (to American Cyanamid & Chemical Corp.) U S 2,028,091, Jan. 14. Numerous details and examples are given of esters having wetting, detergent and emulsifying properties such as the di-Me, di-Am, dioctyl, didodecyl, didodecyl, difenyl, dibenzyl, di-Ph, bi(tetradecylsulfonyl), Me iso-Bu, di-Bu, di-Ph, di-Ph, I, t. steryl or similar esters of  $\text{H}_2\text{O}$  and unsatd. aliphatic dicarboxylic acids such as mono and disulfosuccinic, sulfodichlorosuccinic, sulfodipic, sulfopropylsuccinic, sulfoglutaric, sulfomaleic, sulfocaproic, sulfomaleic, sulfomaleic, sulfomethylsuccinic, sulfomethylglutaric, sulfomaleic, sulfopropyl-

succinic, sulfoacetylglutaric and other sulfonated dicarboxylic acids of the aliphatic series

**Sulfonic and phosphonic esters** Oranienburger chem. Fab A -G (Kurt Lindner and Johannes Zickermann, inventors). Ger 622,268, Nov 23, 1935 (Cl 12a. 23 02). Unsaid monohydric aliphatic acids contg at least 10 C atoms are partly esterified with  $H_2SO_4$  or other sulfonating agent, and esterification is then completed with  $H_3PO_4$  or a deriv thereof, e. g.,  $P_2O_5$ . The products are finally neutralized. Modified products are obtainable by adding other substances, e. g., hydrocarbons, other alics, ketones or carboxylic acids or their anhydrides or chlorides, to the reaction mixt. The products are useful as *washing and emulsifying agents*.

**Aliphatic sulfonation products** I. G. Farbenind A -G (Karl Keller and Eduard Coffeyé, inventors). Ger. 622,296, Nov 25, 1935 (Cl 12a 23 01). The known products obtained by treating polychlorinated higher paraffin hydrocarbons with alkalis are treated with sulfonating agents. Products contg both  $SO_3H$  and  $OSO_3H$  groups are obtained. Examples are given. The products are useful as *wetting and emulsifying agents*.

**Glycerol ethers** Imperial Chemical Industries Ltd., Alfred W Baldwin, Isidor M. Heilbron and Wm E. Jones. Brit 436,143, Oct 7, 1935. For the production of  $\beta$ -ethers of glycerol (I), the alkali metal compd. of an alkylidene or aralkylidene ether of I is caused to react with an inorg ester of an aliphatic alc having at least 8 C atoms and the compd. thus produced is hydrolyzed. The products are oily or waxy and, when emulsified, as by the salts of cetyl sulfuric ester, yield useful softening agents. They may be converted into detergents as described in Brit. 436,209 (following abstr.). In examples, benzylidene glycerol is treated with Na or K and condensed with cetyl iodide, cetyl Na sulfate or octadecyl iodide, the products being hydrolyzed with HCl.

**Wetting agents, etc** Imperial Chemical Industries Ltd., Alfred William Baldwin, Hugh Mills Bunbury and Isidor Morris Heilbron. Brit 436,209, Oct. 7, 1935. Addn to 398,818 (C. A. 28, 1487). Wetting, cleansing and dispersing agents are obtained by sulfonating  $\beta$ -glyceryl alkyl ethers in which the alkyl group contains 13-20 C atoms. In examples, (1) cetyl- $\beta$ -glyceryl ether is treated with Na pyrosulfate and pyridine, the product foams strongly in soln and cleanses greasy woolsens, and (2) cetyl- or octadecyl- $\beta$ -glycerylether is treated with  $CH_3SO_3Na$  in  $CCl_4$ .

**Molded product** Royal F Strickland (to Canadian General Electric Co Ltd.). Can 353,565, Oct. 15. In manuf. an article having a fragile portion, there is placed in the mold surrounding that portion a material composed of a synthetic resin, such as Durite, a hardening agent, such as hexamethylenetetramine, and a noncompressible filler, such as marble flour. This produces a base for elec. lamps composed chiefly of synthetic resin.

**Molded articles of thermoplastic material** such as battery cases of bituminous composition. Charles L. Keller (to Richardson Co.). U. S. 2,027,483, Jan 14. Various mfg. details are described.

**Molding hollow bodies** Brevets & Procédés Industriels (B. F. P. I.) and Eugen Katz. Brit 432,321, July 24, 1935. A seamless hollow body, e. g., a container, box, article of clothing, hat, toy, etc., consists of 2 shells connected by surface adhesion, 1 of the shells is molded as a seamless uncalendered hollow body with a porous surface from paper pulp in molds with pervious walls and the other is formed on the inside and (or) outside of the 1st shell and consists of latex or a latex compn. The pulp is deposited on the pervious molds by compressed air or suction and saponin, "Igepon," etc., may be added to assist the penetration of the latex into the porous surface. The pulp is of vegetable or animal origin and strong or foaming substances other than latex may be added. Vulcanizing mixts may be added to the latex and also dyes or perfumes. The latex shell may be provided with a fat, oil or volatile hydrocarbon-proof coating of Ac cellulose, gelatin or chlorinated rubber.

**Apparatus for molding blanks from powdered ma-**

terials such as bakelite. Leo C. Shippy (to General Motors Corp.). U. S. 2,025,021, Jan. 14. Various structural, mech. and operative details.

**Coloring benzylcellulose molding-compositions** Imperial Chemical Industries Ltd. and Archibald A. Harrison. Brit. 435,539, Sept. 16, 1935. The blue dyes contg Cu obtainable according to Brit 322,169 (C. A. 24, 2890), 389,842 (C. A. 27, 4941) or 410,814 (C. A. 28, 6374) are incorporated in benzylcellulose extrusion molding compns. The free dyes or lakes formed therefrom may be used.

**Coloring nitrocellulose coating-compositions** Imperial Chemical Industries Ltd. and Archibald A. Harrison. Brit 435,614, Sept. 16, 1935. Divided on 435,539 (preceding abstr.). The dyes and lakes mentioned in 435,539 are incorporated in nitrocellulose coating-compns. The compns so obtained give blue weather-resistant coatings, fast to light, on metal, fabric or leather.

**Condensation products from acetylene and phenols, etc** Walter Keppe and Ernst Keyssner (to I. G. Farbenind A.-G.). U. S. 2,027,199, Jan. 7. Products which are of resinous character are obtained by reaction of  $C_2H_2$  at temps of 100-300° on cyclic org. compds. hydroxylated in the nucleus such as PhOH or p-phenols in the presence of a catalytic org. base such as pyridine or cyclohexylamine which is resistant to the reaction temp and inert, at such temp, to the hydroxylated reacting compd.

**Vinyl polymerization products** Otto Röhm. Brit 436,084, Sept. 30, 1935. Materials useful as substitutes for glass, wood or metal are made by supporting or suspending 1 or more pieces of absorbent material, e. g., fabric, paper, fine wire mesh or plywood, in a chamber, introducing 1 or more unpolymerized or partially polymerized unsatd org. compds capable of forming hard polymerization products, if necessary with the addn of hardening agents, and polymerizing or completing the polymerization of said compds. The products may be used in the manuf. of motor bodies, parts of aeroplanes and railway carriages, film spools, toys, dishes, casings for wireless sets, floor coverings, etc.

**Polymerization of methyl methacrylate**. Emil D. Ries (to Canadian Industries Ltd.). Can 354,096, Nov 12, 1935. A mixt of 37 parts by vol of methanol, 53 parts  $H_2O$ , and 10 parts Me methacrylate is heated for 96 hrs at 65°. The polymer is obtained in the form of a soft sponge, which is readily broken up. The whole mass is removed from the polymerizing vessel, filtered, washed with methanol, and dried for 3 hrs at 20° and 3 hrs at 120-140°.

**Cleansing agents, etc** Henkel & Cie G. m. b. H. Brit. 435,465, Sept. 23, 1935. Cleansing, bleaching, dyeing, penetrating and like compns contain  $H_2O$ -sol hypophosphates, e. g.,  $Na_2P_2O_5$ , or hypophosphates of triethanolamine, cyclohexylamine or aminopropandiol. For example,  $NaBO_2$ , 12,  $Na_2P_2O_5$ , 15, soda 10 and Mg silicate 1, with or without soap 50 lb, are mixed. In 435,475, Sept. 23, 1935,  $H_2O$ -sol trimeric alkali metaphosphates are added to cleansing, bleaching and wetting agents and to liquids used in the prepn and dyeing of textiles, leather, hair, etc. For example, 35 lb. of the metaphosphate mixed with 65 of the Na salts of the sulfuric esters of higher fatty alics is used for washing delicate fabrics, e. g., wool or silk. In 435,562, Sept. 24, 1935, phosphites, pyrophosphites, e. g.,  $Na_2H_2P_2O_5$ , or hypophosphites, e. g.,  $NH_4H_2P_2O_5$ , are added to cleansing and washing agents, the mixts. may be used to bleach or to assist wetting and dispersion in dyeing or other treatment of textiles, leather, etc. For example, a mixt. of  $Na_2H_2P_2O_5$  10 lb with the Na salts of the  $H_2SO_4$  esters of higher fatty alics 60 lb is used for washing silk or wool. In 435,710, Sept. 26, 1935,  $H_2O$ -sol. polyphosphates are used as addns to known cleansing, wetting, bleaching and superfatting agents. For example, a mixt. of  $Na_2P_2O_5$  15, soda 10, perborate 10, stabilized by Mg silicate 1, and soap 50 lb is used for washing clothes.

**Detergents** Henkel & Cie G. m. b. H. Brit 435,217, Sept. 19, 1935. In cleansing-agents that contain alkali carbonate and neutral alkali pyrophosphate, the phosphate is at least half the carbonate. Soap, per salts,



waterglass, metasilicate and borax may be present. Thus, a mixt of soap 45,  $\text{NaBO}_2$  10,  $\text{Na}_2\text{P}_2\text{O}_7$  30 and  $\text{Na}_2\text{CO}_3$  15 lb is used for washing textiles

**Stenizing liquids** Katadyn A-G Brit 422,101, July 15, 1915  $\text{H}_2\text{O}$  and other liquids are sterilized by adding liquid  $\text{H}_2\text{O}_2$  or other nongaseous O-yielding substances other than halo compds together with 1 or more oligodynamically active metals or 1 or more compds of such metals in oligodynamically minute quantities, in such manner that the actions of the 2 different agents can be separately controlled Cf C A 29, 392<sup>2</sup>

**Wax emulsions** I G Farbenindustrie A-G Brit 475,618, Sept 19, 1935 See Fr 784,614 (C A 30, 1935)

**Polishes** Gayet, Beriot & Fils Fr 787,910, Oct 1, 1935 Colloidal suspensions of fats, waxes, paraffins, etc., in water contg glue, e.g., bone glue, are used. The fat is dissolved in kerosene or like solvent and mixed with a soln of glue and a soln of starch and soap

**Absorption material, apparatus for removing moisture from the air** Edmund Altmkirch Brit 434,666, Sept 6, 1935 See Fr 772,706 (C A 29, 1542<sup>2</sup>) and Ger 610,939 (C A 29, 5703<sup>3</sup>)

**Porous bodies** Maxwell L. Whitacre and Peter DeLeeuw (to Carbosundum Co.) Can 353,932, Nov 5, 1935 One of several examples of making porous articles is: Mix 4000 g of fused Al, 2000 g of a clay bond, 1600 cc of  $\text{H}_2\text{O}$  contg 4 g of acetanilide and 20 cc of EtOH for 15 min, add 300 cc of a 2.5% glue soln and mix 10 min, add 300 cc of a 1.5% soln of  $\text{H}_2\text{O}_2$  and mix for 2 min, place in a mold and dry at room temp for 48 hrs

**Dispersion of carbon black** Charles W. Tucker (to Dewey and Almy Chemical Co.) Can 354,103, Nov 12, 1935 Carbon black is mixed with  $\text{H}_2\text{O}$  in the presence of a sol salt of a compd having the structure of the product obtained by condensing substantially 1 mol of formaldehyde with substantially 2 mols of 8-naphthalenesulfonic acid. The aq dispersing agent comprises 2-6% of the quantity of carbon black

**Wood-filling composition** Earl D. Flood and John A. Hannum (to Flood Co.) U. S. 2,027,095, Jan 7, 1935 An aq. vehicle is used with a predominant proportion of finely divided mineral filler and with small proportions of cellulose acetate, rubber latex, a drying oil and Na salicylate

**Compositions for coating metal, stone, wood, paper, textile materials, etc** Paul Friedrich (to Trustringkantor Amstelland N.-V.) U. S. 2,027,686, Jan 14 Bituminous hydrocarbon materials such as tar, pitch and asphalt are mixed with smaller amts. of aliphatic chlorohydrocarbons such as  $\text{C}_2\text{HCl}$  and fluid homologs and derivs of benzene and the mixt. is heated to above 130° under superatm. pressure with rapid stirring to form a reaction product

**Gum-lac coatings** Rheinische Schellackbleiche Ernst Kalkhof A-G. Fr. 788,703, Oct 15, 1935 A coating which hardens without heating comprises a soln of gum-lac of known type to which  $\text{AlCl}_3$  or urea is added as hardening agent. Resins, cellulose derivs, fillers and dyes may also be added

**Modified castor oil, coating composition** Harold J. Barrett (to Canadian Industries Ltd.) Can 354,089, Nov. 12, 1935 This relates to improved softening agents and more particularly to nitrocellulose compns contg. these agents. E.g., a mixt. of 81.3% of castor oil and 18.7% of phthalic anhydride is heated without stirring with  $\text{CO}_2$  at 220° for about 12 hrs. until the acid no. of the mixt. reaches approx 15. The product obtained is a light-brown oil of a viscosity of 471 poises

**Tar varnish** Soc. du gaz de Paris Fr. 788,249, Oct. 7, 1935 A tar varnish contains coal-tar pitch, crude phenol and benzene, to which a pigment or insulating filler is added

**Removing halogens from fluids** Kodak Ltd Brit. 435,735, Sept 26, 1935. Halogens, except F, are removed from fluids by contact with polyvinyl aldehyde condensation products, which may be used in any suitable phys. form, e.g., powder, grains, porous masses, fibers,

threads, films, etc. The absorbed halogen is readily removed by treatment by heat, vacuum, solvents or suitable reagents. Iodine may be extd from the mother liquors obtained from the refining of seaweed ashes or  $\text{NaNO}_3$ . Halogens may be similarly removed from air or other gases. The products comprising the condensation products with halogens, particularly I, may be used as bandages, etc., for medical purposes. I solns may be prepd. by immersion thereof in a solvent, e.g., alc. The products may also be used for inhalation purposes or to remove impurities that react with halogens, e.g.,  $\text{H}_2\text{S}$ ,  $\text{SO}_2$ , from gases. A suitable filtering mass is prepd. by causing polymerized vinyl alc, dissolved in  $\text{H}_2\text{O}$  100, to react with  $\text{HCl}$  (sp gr 1.19) 24 and 40%  $\text{CH}_2\text{O}$  9 parts, stirring, molding when coagulation starts and allowing to stand

**Removing water from vaporous mixtures with anhydrides such as acetic anhydride** Henry Dreyfus U. S. 2,027,420, Jan 14 Water vapor such as that in admixt with  $\text{Ac}_2\text{O}$  is caused to react with a gaseous hydrocarbon such as  $\text{CH}_4$  (suitably at a temp of about 600-850° in the presence of Ni, Co or Cu)

**Fluid for hydraulic brakes, etc** Wagner Electric Corp. Fr 783,534, Oct 11, 1935 A preferred compn is glyceryl diricinolate 50 and diacetone alc 50 parts, but the monoester of glycerol and glycol and polyglycol esters may also be used, and other ales such as EtOH, PrOH and tetrahydrofurfuryl alc may be used. If an alc. other than diacetone alc is used it is desirable to add a small amt of a neutralizing agent such as K arsenate to avoid all acidity

**Friction material suitable for brakes and clutches** John D. Alley (to American Brakeblok Corp.) U. S. 2,026,767, Jan 7 A flexible friction material is prepd by mixing 18 parts of a drying vegetable oil such as linseed oil with S 2.7, finely divided pyrobituminous material such as bituminous coal 25, and short-fibered asbestos 65 parts, shaping as desired, and curing without pressure in a baking oven at a temp of about 50° for 4-5 hrs and then at about 150° for 5-6 hrs Cf C A 29, 2631<sup>1</sup>

**Bushings for use in steering columns, etc.** Newton Skillman (to O & S Bearing Co.) U. S. 2,027,559, Jan 14 An inner liner of lubricant-impregnated fibrous material is backed and held in place by a hardened asphaltic material of such consistency as readily to shear when forced into an undersize housing unit

**Material for seals such as pump packings resistant to chemicals.** Wilbur A. Lazier (to E. I. du Pont de Nemours & Co.) U. S. 2,027,389, Jan 14 A packing for seals such as those of pumps for acids comprises a fibrous base such as asbestos impregnated with a mist comprising a hydrogenated castor oil (hydrogenated to produce a waxy material m 70° or higher) and a solid lubricant such as graphite

**Paste of comminuted metal** Henry H. Mandie Can 354,050, Nov 12, 1935. Metal, e.g., Al, in flake form is mixed with a purified hydrocarbon of suitable volatility and boiling range. Dissolved in the mixt in a quantity less than 5% of the Al present is an alkali metal salt of a volatile aliphatic acid and an Al salt of a nonvolatile aliphatic acid. The latter is present in greater amt than the former

**Strip for sealing cartons** Old Colony Trust Co (executor of the estate of William W. McLaurin) Can 354,144, Nov 12, 1935 A strip is coated on one side with a water-resistant adhesive, such as asphalt. Over this coating is placed a coating of water-sol adhesive, such as fresh glue. The intermediate coating prevents the strip from loosening in warm climates

**Casting transparent films** Edouard M. Kratz (to Marbo Products Corp.) U. S. 2,027,829, Jan 14 In the casting of films such as those of casein or gelatin the surface of a carrier web used is protected by a coating of petroleum jelly deposited from a soln in a volatile solvent

**Lignin derivative.** Guy C. Howard. Can 353,886, Nov 5, 1935. Lignin substance is sepd from assoc nonlignaceous org matter to obtain an aq dispersion of free ligninsulfonic acid contg. some organically com-

bined base in the form of a salt of ligninsulfonic acid. This is heated and evapd under conditions of sufficiently low partial pressure of  $\text{SO}_2$  in the gas phase to induce the escape of nearly all the  $\text{SO}_2$  gas. The evapd product is dried with  $\text{H}_2\text{O}$  to produce a lignin deriv. in gel form, which is then sep'd from the mother liquor. It is useful as a bonding agent.

Apparatus for use in making synthetic carbonado diamonds. Jean H. L. DeBats. U. S. 2,027,963, Jan 14. Various structural, mech. and operative details.

Rock wool. John Buss. Brit. 435,064, Sept. 13, 1935. The wool is made from siliceous material, e. g., rock, metal or ore slag, by pouring the molten material in a thin stream onto the marginal surface of a thin metal disk rotating at high speed and utilizing the rotation of the disk to discharge air or other fluid, e. g., paraffin oil, from beneath the disk onto the mineral fibers. App. is described.

Magnetic powders. Heraeus-Vacuumschmelze A.-G. and Wilhelm Rohn. Ger. 619,749, Oct. 5, 1935 (Cl. 21g, 31 03). Metal powders with predetermined magnetic properties are obtained by reduction of crystals of mixed salts formed to give the desired end-product mixt. Mixed salts of Fe or Ni with Co, Cr, Mo, Cu, Mn, Si or Ag are used. The products are used for making magnetic cores, etc.

Proofing agents. Wingfoot Corp. Ir. 788,057, Oct. 3, 1935. A waterproofing compn. for paper, etc., comprises a mixt. of a waxy substance and a condensation deriv. of rubber. The latter may be made by boiling a soln. of rubber in  $\text{C}_2\text{H}_4$  for 1 or 2 hrs., adding 10% of a transformation reagent such as  $\text{SnCl}_4$ , and continuing the heating for several hrs.

Containers of treated paper or the like suitable for holding lubricating oils, etc. Wilbur A. Lazier and James H. Wertz. (to E. I. du Pont de Nemours & Co.). U. S. 2,027,390, Jan. 14. Material for containers such as paper bottles is coated with a compn. comprising a substantially H-sat'd ester of a fatty acid having a m. p. of  $50^\circ$  or higher such as substantially H-sat'd castor oil, hydrogenated blown drying oils or waxy esters of hydroxy stearic acids with high-mol.-wt. alcs.

Panels of wood veneer with a core such as wood. Charles B. Norris. (to Haskelite Mfg. Corp.). U. S. 2,028,076, Jan. 14. Various mfg. details are described.

Buffing wheels. Elisha W. Hall. U. S. 2,027,425, Jan. 14. Layers of fibrous buffing material such as cloth are united at spaced intervals by a tenacious adhesive such as a latex compn.

Playing courts, etc. Caleb Archidge. Brit. 435,324, Sept. 19, 1935. A tennis-court, path, etc., are provided with a layer comprising an aggregate of ground wood, ground cork, ground rubber and sand, held together by a binder of oil, e. g., linseed, with either resin or a resinous substance. The ground wood may be pretreated with linseed oil.

Dry preparations dispersible in water. Soc. pour l'ind. chim. à Bâle. Brit. 435,412, Sept. 20, 1935. See Ir. 709,930 (C. A. 29, 530\*).

Imitation straw hats. Caradine Hat Co. Brit. 435,207, Sept. 17, 1935. An imitation straw hat is made from fabricated or woven paper by applying a waterproof stiffening of transparent cellulose gum lacquer, consisting preferably of pyroxilin (I), dammar (II) and AcOEt (III), to the partly shaped hat body and, after giving the body its final shape, coating the surface with an opaque cellulose gum lacquer consisting of I, II and III, to which a filler comprising  $\text{TiO}_2$ ,  $\text{ZnO}$  and lithopone together with a stabilizing agent, e. g., picric acid.

Shoe polish. Hugh P. Griffin and Robert P. Richardson. U. S. 2,028,324, Jan. 21. A dry compn. for white shoe polish contains lithopone,  $\text{BaSO}_4$  or  $\text{ZnS}$  in mixt. with talc and  $\text{Na}_2\text{PO}_4$ .

Softening bleached and dyed ruscus. John M. Dux. U. S. 2,028,873, Jan. 7. The material is submerged in an emulsified soln. of a water-sol. sulfonated vegetable oil, glycerol and a vegetable gum, and Na benzoate also may be added, as may also  $\text{MgSO}_4$ .

Printer's blanket. Harold D. Rice. (to U. S. Rubber Co.). U. S. 2,027,322, Jan. 7. A base such as a sheet of felt carries a coating of the solids deposit of an aq. dispersion of rubber vulcanized to the base.

Attaching linings to furs. Josephus A. Kleine. Brit. 433,587, Aug. 10, 1935. Reinforcing linings are attached by adhesive rubber dispersions sprayed on as small droplets.

Catalytic material suitable for igniting cigarettes. Richard E. Berthold. (to Cardinal Products, Inc.). U. S. 2,021,475, Nov. 19. A calcined residue of cotton is sat'd with a soln. of Th nitrate, Zn nitrate, Al nitrate, Pt chloride and Ba Pt cyanide, with the total amounts of nitrates, chloride and cyanide in the ratio of about 14:2:1, and the sat'd calcined material is formed into igniting pellets which may be used as described in U. S. 1,899,008.

Floor covering. Milton O. Schur and Walter L. Hearn. (to Brown Co.). Can. 353,556, Oct. 15. A felt base is composed of compacted, waterlaid, substantially unhydrated but refined wood pulp and up to about 25% by wt. of added cementitious material such as well-hydrated cellulose and starch substantially uniformly distributed therethrough. The felt is sufficiently absorbent to be readily penetrable by molten bitumen.

Laminated phonograph records. Richard F. Warren. (to Carbide and Carbon Chemicals Corp.). U. S. 2,028,120, Jan. 14. A core of fibrous material such as paper is used, at least the surface of which is impregnated with a hardened artificial resin to render the core moisture-resistant, a playing groove receiving surface being provided contg. as the sole resinous constituent a polymerization product from a vinyl lower aliphatic acid ester such as vinyl acetate, a vinyl halide or styrene or mixts. of such products. Durable records with a "minimum" of surface noise are thus produced.

Stiffening fabric suitable for use in shoe manufacture. Joseph Fausse. (to Celastic Corp.). U. S. 2,027,968, Jan. 14. A fabric has a cellulose deriv. such as cellulose nitrate or acetate or ethyl cellulose pptd. in the interstices on one side, with the cellulose deriv. in a colloidal solvent resistant form on the other side, and is adapted for use by treating with a solvent followed by shaping and drying in the desired final form.

Stiffening fabric suitable for use in shoe manufacture. Russell Hamilton. (to Celastic Corp.). U. S. 2,027,973, Jan. 14. A fabric is impregnated throughout with a cellulose-deriv. soln. such as one of cellulose nitrate or acetate or ethyl cellulose and without drying is passed into a pptg. bath, the solvent-liquid content of the soln. and pptg. bath being at least "partially immiscible" and the fabric being subjected to the pptg. bath for a time only sufficient to ppt. part of the cellulose deriv.; the impregnated fabric is dried, treated with a cellulose-deriv. solvent to soften it, shaped and dried in the final desired form.

Impregnated fabric suitable for stiffening shoe materials. Earle H. Cameron. (to Celastic Corp.). U. S. 2,027,957, Jan. 14. A material adapted to be softened by treatment with a solvent comprises a fabric carrying a pptd. cellulose deriv. such as cellulose acetate or ethyl cellulose in its interstices and also carrying, distributed throughout the pptd. cellulose deriv., a cellulose deriv. in a relatively harder, firmer, colloidal and more solvent-resistant form.

Fumigation such as that with moist air in contact with calcium cyanide. Wm. W. Hinds. (to Calcyanide Products Corp.). U. S. 2,027,042, Jan. 7. App. and various operative details are described.

Repellent or fumigant. Evan Clifford Williams. (to Shell Development Co.). Can. 353,986, Nov. 5, 1935. As a fly repellent a compn. contains an unsat'd org. sulfide or polysulfide.

Fire extinguishers. Pierre J. Marchant. Brit. 433,757, Sept. 23, 1935.

Fire extinguishers. Soc. pour l'achat, la vente et l'exploitation de brevets, S. A. V. E. B., S. A. Ir. 788,401, Oct. 10, 1935. An alk. soln., an acid soln. and a stabilizer are used, the stabilizer being incorporated in the acid soln. and being stable in acid. Thus, a soln. of  $\text{NaHCO}_3$  and a soln. of  $\text{Al}_2(\text{SO}_4)_3$ , with or without acids,

contg. Na laurosulfonate or "Igepon" are used. A thin layer of oil may be poured on to each of the solns  
Extinguishing fires Otto Wille. Ger. 622,219, Nov

22, 1935 (Cl 61b 2). For extinguishing fire from incendiary bombs having a thermite charge, use is made of mixts. of dry sand with anhyd.  $\text{KHSO}_4$ .

## 14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

Industrial utility of public water supplies in the United States, 1932. W D Collins, W L Lamar and E W Lohr. U. S. Geol Survey, *Water Supply Paper No 658*, 140 pp (1934). U S Pub Health Eng Abstracts 15, W, 101 (Oct 5, 1935), cf C A 27, 791—Brief descriptions and chem analyses are given of public water supplies of 670 cities of the U S Treatment of different waters for boiler-feed purposes is also considered for different classes of waters C R Fellers

Water works of Madisonville, Ky, celebrates its 21st birthday Hall Arnold *Water Works Eng* 88, 1480-1 (1935), cf C A 30, 550<sup>5</sup> W A Moore

Lewiston's water system W P Hughes. *J Am Water Works Assoc* 27, 1708-11 (1935)—The supply is Clear Water River  $\text{Ca}(\text{OH})_2$  and  $\text{Al}_2(\text{SO}_4)_3$  are used, followed by sedimentation and rapid sand filtration After filtration and before the water goes to the reservoir 2 lb. Cl per million gal is added D K French

Water supply in England A E L Chorlton *Munic Eng. Sanit Record* 94, 684 (1934). U S Pub Health Eng Abstracts 15, W, 123 (Nov 9, 1935) C R F

London's water supply Sir Wm Prescott *J Roy Sanit Inst.* 55, 643-58 (1935). U. S. Pub Health Eng Abstracts 15, W, 113 (Nov 2, 1935)—An area of 573 sq miles is furnished 300,000,000 gal of water a year Great economy in operation is being effected through the adoption of a system of double filtration and the installation of Diesel engines There are 139 acres of slow sand filters and  $\frac{1}{2}$  acre of rapid sand filters C R Fellers

The Swinford waterworks of the Oxford corporation H H. Crawley. *Water and Water Eng* 37, 651-8 (1935)—Structural and operating features are given W A Moore

The municipal water supply of Paris. John B Hawley *J. Am. Water Works Assoc.* 27, 1699-1703 (1935).—Most spring waters from which the Paris supply is taken are soft, agreeable to the taste, and nearly sterile as to pathogenic bacteria Two slow sand filtration plants existed which have been improved, and chlorination app is part of the plant equipment. There is also a non-potable supply carried through sep pipe lines for other uses D K French

New water supply of Athens, Piraeus and district T. A. Liefmann. *Water* 18, 21 (1934). U S Pub Health Eng. Abstracts 15, W, 146 (Dec. 14, 1935)—The supply comes from an impounding reservoir near Marathon and is brought to Athens and Piraeus 25 km by a pipe line Until 1920, the Aqueduct of Hadrian, dating from the 2nd century, formed the only supply of water for the cities Sea water is used for street cleaning. C R Fellers

The nature of the waters at Oulmès, Morocco R. Frey. *Ann. franç. Avanc. Sci., Congrès de Rabat* 1934, 73-7; *Rer. Geol.* 15, 245—Two warm springs at Ain Karouba and Lala Afa show, resp., 2.9 and 32.1 g per l of alkali chlorides, 4.1 and 14 alkali sulfates, 6.1 and 14.5 alkali carbonates and 87 and 52 alk. earth carbonates J F Schauer

Additions to the water supply of Melfort, Sask J E Underwood *Can. Eng.* 68, No 8, 3-4 (1935). U S Pub Health Eng Abstracts 15, W, 52 (May 11, 1935)—The normal treatment of the water taken from Melfort Dam consists of Cl and alum treatment and pressure filtration. Odor and taste troubles, especially serious after freezing, were controlled by aeration, sedimentation and treatment with activated C at the rate of 2.5 oz per 66,000 gal. The alum and activated C are fed into the supply main leading to the aeration basin. Thorough mixing occurs in passage through the nozzles. The activated C is suspended in the feed water with the aid of an

ordinary elec washing machine The water is chlorinated before delivery to the pressure filters. C R Fellers

Irrigation and water supply in Victoria, Australia. L R East *J Inst Engrs Australia* 6, 389 (1934). U. S. Pub Health Eng Abstracts 15, W, 153 (Dec 28, 1935).—Descriptive C R Fellers

Water supplies from underground sources J D Restler *Surveyor* 87, 283 (1935); U S Pub Health Eng Abstracts 15, W, 147 (Dec 14, 1935)—The advantages of ground over surface sources are stressed C R Fellers

Presence of lithium in several waters in the Turn Hills Marcella Jarach *Ann chim applicata* 25, 385-8 (1935)—Amts of Li-O up to 0.01 g/l have been found in waters from the Turn Hills This is probably derived by seepage of water through the granite formations, which often contain large amts of alkalies A W C

The sparkling spring of Oberschützen in the Burgenland E Dittler and R Dworak *Chem Erde* 9, 209-85 (1935)—The mineral spring at Oberschützen near Tatzmendorf (40 m S of Vienna) is marked by its large content of  $\text{Na}^+$  (0.8789),  $\text{Mg}^{++}$  (0.2312),  $\text{SO}_4^{--}$  (0.8465) and  $\text{HCO}_3^-$  (0.00665 g/l) Total solids are 5.3717, with 2.051 g/l  $\text{CO}_2$  The Mg is probably derived from an underground extension of serpentine from the N E A boring in the neighborhood shows streaks of coal at about 70 and 140 m C A Silberrad

A selenium spring John T Miller and Horace G Ryers *Ind Eng Chem, News Ed* 13, 456 (1935).—Samples of water from, and soil and vegetation in the neighborhood of, a "poison spring" in Custer Co., S Dak were analyzed for Se and As The water was found to contain only 0.4 p. p m Se and 0.5 p. p m As, quantities insufficient to account for the death of cattle Soil contained 1.5-4.0 p. p m Se, while the vegetation contained as high as 1600 p. p m Se This latter is soil and was regarded as the primary cause of death of cattle. M G Moore

Water Charles R Hazen *Can Eng* 67, No 11, 14-17 (1934). U. S. Pub Health Eng Abstracts 15, W, 20 (Mar 23, 1935)—The properties and impurities of water in its many ind applications are discussed C R Fellers

New methods for the examination of ground-water regions L Munder. *Verh intern theort argew. Limnol* 6, 238 (1934). U S Pub Health Eng Abstracts 15, W, 116 (Nov. 2, 1935)—Carbonate hardness differs widely in different types of water, and is comparatively const for each source of water This characteristic was used in prepg a carbonate-hardness chart for a large ground-water region, in which points of equal carbonate hardness were joined by lines From the lines of hardness can be detd the direction and comparative amt of river-water infiltration and the direction of ground-water flow. The method should prove valuable in preliminary investigations of ground-water utilization and for hydrological calns. and investigations C R Fellers

Removal of lead and copper from drinking water K. Holl *Arch Hyg Bakt* 113, 296-304 (1935); U S. Pub Health Eng Abstracts 15, W, 99 (Oct 5, 1935)—Filtration of water through cotton wool may be efficacious, but the adsorbent action of the cotton is inadequate for waters contg more than 1 mg per l of Pb If present in smaller amts, a cotton wool about the size of a man's fist will remove Pb almost completely from 10-15 l of water Filter paper shreds when made into a suitable filter bed on a funnel will remove Pb up to a concn of 2.5 mg per l. Activated C gives best results in Pb removal Small-scale expts. showed that the addn. of 10 g. of the

C to 5 l of water, followed by filtration through filter paper contg. another 5-g layer of C, removed completely the Pb in water contg. 8 mg per l. Filtration through a Berkefeld candle filter will also remove Pb up to a concn. of 2.5 mg per l from many waters, but the presence of chlorides lessens the efficiency of removal. Filtration of water through a bed consisting of  $MgO$  in granular form will also remove both Pb and Cu from water. The adsorbent materials must be changed regularly for best results.

**Iron and hard water problems solved at Waupun, Wis.** H T Rudgal *Water Works Eng* 88, 1404-7(1935).—The lime treatment followed by recarbonation to reduce the alkali is used. The Fe is deposited in the clarifier basin.

**A modification of the Sanchis method for determination of fluorine in water.** C Herrero Ducloux *Analges aquim Argentina* 23 (3-6(1935)), cf. Sanchis, *C A* 28, 28134.—Prep. a 0.17% soln. of Na alizarinsulfonate (I) and an 0.87% soln. of  $Zr(NO_3)_4$  (II), to 10 cc of II add 10 cc of I, let stand several hrs., shake, dil with 80 cc of distd.  $H_2O$ , protecting from the light, this forms the indicator soln. (III). Prep. a soln. 1.5 N as to HCl and 1.5 N as to  $H_2SO_4$  (IV) and a 0.1105% NaF soln., from which take 5 cc and add to 250 cc soln., so that 1 cc = 0.01 mg F (V). Instead of using 100 cc., and heating in flasks, use 50 cc. in test tubes. From a buret, drop into 8 test tubes 1, 2, 3, 4, 5, 6, 7, 8 cc of V, make up to 50 cc with distd.  $H_2O$ , add 2 cc of IV, then 1 cc of III, shake, place the tubes in a support in a NaCl brine heating bath and heat 3 min., withdraw the tubes, let stand 5 hrs. or until the next day, and compare colors with tubes in which the sample to be analyzed has been treated similarly. E M S.

**Alterations in a lime soda water treatment plant.** F L Hana *Warne* 57, 105-7(1934), *U S Pub Health Eng Abstracts* 15, W, 156(Dec 29, 1935).—After 10 yrs' operation of a lime soda treatment plant, the appearance of turbidity in the raw water caused difficulties. These were removed by altering the method of adding reagents and the insertion of perforated bottom plates in the reaction tanks, and colloidal turbidity was removed by the addn. of 150 g. of solid  $Al_2(SO_4)_3$  per hr. at a flow of 20 cu m an hr. After the alterations the filter runs were 3 times as long as previously. C R Fellers.

**Use of powdered activated carbon in Florida.** F E Stuart *Proc 9th Ann Meeting, Fla Sec Am Water Works Assoc* 60-2, *U S Pub Health Eng Abstracts* 15, W, 46(Apr 20, 1935), cf. *C A* 28, 17677, 29, 55541.—For elimination of taste and odor the filters should be first seeded with 2 lb. of C per million gal of filter capacity. The sludge should be blanketed with 25 lb. of C per million gals of water treated per day. This step is followed by adding 8 lb. of C along with the alum. After these initial steps the dosage may be varied according to needs.

**Apparatus with a metal casing for sampling deep water and determining the corresponding water temperature.** L Schioppa *Zentr. Bakt. Parasitenk., Abt. I* 130, 301(1933), *U S Pub Health Eng Abstracts* 15, W, 24(Mar 23, 1935).—Three l. samples can be readily obtained at depths of over 400 m in 2-3 min. The samples are suitable for bacteriol. examn. C R Fellers.

**A filtration plant in southern Michigan.** D E Bicknell *Am City* 49, 67-8(1934), *U S Pub Health Eng Abstracts* 15, W, 54(May 11, 1935).—The supply of St. Joseph is from Lake Michigan. The completely electrified plant has a capacity of 6 million gal per day. The 2 mixing tanks and coagulation basins are so arranged that they can be operated in series or parallel. Filters can be washed by scp pump or from high service discharge. Cl is added to raw water suction. Alum,  $(NH_4)_2SO_4$ , and powd. activated C are added at the inlet to the mixing tank. The final dose of Cl is added in the filter effluent flume ahead of the clear well. C R Fellers.

**Design, construction and operation of 40,000,000 gallon filtration plant for the city of Hamilton, Ontario.** W L McFaul *Can Engr* 68, No 14, 62-5(1935), *U S Pub Health Eng Abstracts* 15, W, 151(Dec 21,

1935), cf. *C A* 27, 2508; 29, 55541.—The water from Lake Ontario is filtered by 12 filters of a capacity of 3.3 million gal daily. The filter underdrainage system which is of the perforated-pipe type, is covered with 20 in. of graded gravel with an effective size of 0.5 mm and uniformity coeff. of 50%. Filter runs are limited to 48 hrs. or a loss of head of 8 ft. During the summer the runs may be reduced to 20-30 hrs. by microorganisms. The coagulation basins provide 3 hrs. detention. Pretreatment by  $NH_3$  and Cl is used and alum is applied only intermittently for turbidity removal. Residual Cl is maintained throughout the plant and post-chlorination is not required. The cost of the plant was \$1,220,000. C R F.

**Problems in water filtration plant operation.** Brantford, Ont. I P Adams *J. Am. Water Works Assoc.* 27, 1725-7(1935). Hamilton, Ont. D H Matheson *Ibid.* 1727-8. Peterborough, Ont. W. G. Hunt *Ibid.* 1728-31.—The various experiences involve largely taste, odor or color in the water. Novel methods of filtration and application of chemicals were of great assistance.

**Effect of filtration through rapid filters on the operation of slow filters according to observations at the filtration plant of Warsaw.** I Piotrowski *Gas & Woda* 14, 183(1934); *U S Pub Health Eng Abstracts* 15, W, 148(Dec 14, 1935).—Rapid sand filtration without coagulation prior to slow sand filtration at Warsaw removes suspended matter and 90-95% of the microorganisms. This increases the operation period and efficiency of the slow sand filters and renders filtration more regular.

**Controlled coagulation maintains filters in good condition.** Frank W. Bouison *J. Penna. Water Works Operators Assoc.* 6, 15-16(1934); *U S Pub Health Eng Abstracts* 15, W, 10(Feb 16, 1935).—A gummy mat on the rapid sand filters of the S. Pittsburgh Water Co. caused much trouble but was finally corrected by lowering the pH of the coagulated water to 6.9, thereby bringing down the excess Al and Fe from the acid raw water.

**Emergency work during the New York State flood.** Anselmo F. Dappert *J. Am. Water Works Assoc.* 27, 1647-69(1935).—As a result of the flood many filter plants and an enormous no. of private wells were seriously damaged. Portable emergency chlorinators and emergency hypochlorite treatment plants were installed in 16 communities, most of them by department engineers. In addn. the general emergency work covered nearly a score of individual related matters. Also in *J. New Engl. Water Works Assoc.* 49, 376-94(1935).

**Aeration of water by air diffusion.** Frank C. Roe *J. Am. Water Works Assoc.* 27, 897-904(1935).—In the abstr. in *C A*, 29, 6338, the expression "perforated pipe or plates" should read "porous diffuser plates and tubes".

**The municipal water-softening plant at Glendive, Mont.** C W Eyer *J. Am. Water Works Assoc.* 27, 1704-7(1935).—The lime-soda process is used, but for the soda-ash supply water is available from a deep well carrying 7.2 grains free  $Na_2CO_3$  per gal and 35.2 grains  $NaHCO_3$ ,  $Ca(OH)_2$ , and Na aluminate are first added and then enough well water. A baffle mixing chamber and flocculator brings about quick settling in the clarifier. A portion of the sludge is recirculated, this saves an estd. 10 lb. lime per hr.

**Future problems of water works operators with regard to taste, odor and palatability.** Lewis O. Bernhagen *Proc. 17th Texas Water Works Short School* 1935, 135-7, *U S Pub Health Eng Abstracts* 15, W, 111(Oct. 26, 1935).—General methods of preventing offensive tastes and odors are detailed. Microscopic exams made at frequent intervals will serve to detect undesirable algae.

**Possible value of hydrogen ion in the forecasting of taste and odor periods by algae and decomposition of organic matter.** Wm. Veyan *N. Dak. Water and Sewage Works Conference* 1, No 6, 3-4(June, 1935), *U S Pub Health Eng Abstracts* 15, W, 110(Oct. 26, 1935).—Succ

algae consume the free and even half-bound  $\text{CO}_2$ , thereby raising the  $\text{pH}$  of the water, and since bacterial action on org. matter releases  $\text{CO}_2$ , lowering the  $\text{pH}$ , it is suggested that changes in the  $\text{pH}$  may forecast taste from either of the 2 causes.

C. R. Fellers  
Interference of algae with tests for residual chlorine  
E. W. Johnston and W. R. Edmonds *J. Am. Water Works Assoc.* 27, 1717-24 (1935).—See C. A. 29, 5953<sup>4</sup>

D. K. French  
Ammonia-chlorine in the tropics  
J. S. Boisser *Water and Water Eng.* 38, 24 5 (1936)

W. A. Moore  
Maintenance of chlorine equipment  
Raymond J. Faust *Mech. Eng. Expt. Sta. Bull.* No. 64, 27 30 (1935).—Phys. characteristics of  $\text{Cl}_2$  types of chlorinators, common troubles with chlorinators, need for maintaining chlorinator, lines and tanks at uniform temp., and solns for cleaning chlorinator parts are discussed. G. H. Y.

Bacteriological examination of water supplies  
Committee Rept. Ministry of Health (Engl.), *Rept. on Pub. Health and Med. Subjects* No. 71, 38 pp (1934), *U. S. Pub. Health Eng. Abstracts* 15, W, 16 (Mar. 9, 1935).—The plate count on agar incubated 3 days at  $20-2^\circ$  and 2 days at  $37^\circ$  is recommended. Results are interpreted as most probable nos. of coliform bacteria per 100 ml. This is det'd by a series of diln. tubes in MacConkey's liquid bile salt-lactose-neutral red medium incubated at  $37^\circ$  for 2 days. Differentiation into *Es. coli*, intermediates and *Aerobacter aerogenes* is recommended if the water contains 2-10 bacteria per 100 ml. The Committee regards the presence of *Aerobacter aerogenes* with far greater suspicion than is done in the U. S.

C. R. Fellers  
Comparative studies of media for the determination of the coli-aerogenes group in water analysis  
C. C. Ruchholtz *J. Am. Water Works Assoc.* 27, 1732-45 (1935), cf. C. A. 30, 1301.—In order of productivity the following media are listed from highest to lowest: buffered lactose broth, fuchsin lactose broth, methylene blue-bromocresol purple broth, brilliant green-blue broth, crystal violet broth, and formate-nicotinate broth. For inhibitivity to spore-bearing lactose fermenters, formate-nicotinate broth was the best. Addnl. work is being done with this media as well as brilliant green-blue broth, and MacConkey's broth in confirmation procedure, in comparison with standard procedure, on finished chlorinated water where coli-aerogenes det'n is difficult. D. K. F.

Weed growths in reservoirs and open canals  
G. E. Arnold *J. Am. Water Works Assoc.* 27, 1684-93 (1935).—Weeds are defined as aquatic growths having a root system whereby they are attached and not free floating. Weed growth increases with the temp., there being very little at temp. of  $40-50^\circ\text{F}$ . Silty, loam soil or sand is preferable.  $\text{CO}_2$  is consumed and  $\text{O}$  liberated by growing weeds. Covering a reservoir to eliminate direct light will prevent practically all weed growths.  $\text{CuSO}_4$  in various quantities is the best agent to prevent as well as to kill weed growth. Mature growing weeds are not always killed with  $\text{CuSO}_4$ , and cutting and pulling is probably the most effective means of growth control.  $\text{Cl}$  and  $\text{NH}_3$  and  $\text{Cl}_2$  are not successful. Varying the water level is frequently helpful. D. K. French.

Disinfection of water mains  
W. A. Gentner and M. S. Wellington *J. New England Water Works Assoc.* 1933, No. 2, 165; *U. S. Pub. Health Eng. Abstracts* 15, W, 13 (Feb. 23, 1935).—In Hartford, Conn., the practice of placing 225-g doses of solid  $\text{NaOCl}$  in the newly laid mains at 30-m. intervals is followed. The method is inexpensive and effective. At New Haven, brushing out the pipes and washing them with strong  $\text{NaOCl}$  soln before laying did not give satisfactory bacteriol. results. Sufficient  $\text{Cl}$  to give 1 mg. per l. of water in the pipe was allowed to remain in the pipe line for 5 days. The residual  $\text{Cl}$  concn. at that time was 0.3 mg. per l. The results were fully satisfactory. C. R. Fellers.

Chemical behavior of Eternit pipes  
L. Kaatz and H. E. Richter. *Gas- u. Wasserfach* 77, 119 (1934); *U. S. Pub. Health Eng. Abstracts* 15, W, 11 (Feb. 23, 1935), cf. C. A. 28, 5155<sup>3</sup>.—On contact with Eternit, the  $\text{pH}$

of Leipzig water increases, while the hardness decreases at first, then remains constant. A protective coating of  $\text{CaCO}_3$  forms on the pipe but this is sometimes destroyed by fresh aggressive water. Very aggressive water may cause asbestos fibers to become detached from the pipe. The changes are greater in the new pipe.  $\text{Ca(OH)}_2$  is washed out from the cement in the Eternit and reacts with, and removes, any free  $\text{CO}_2$  present in the water. There is no objection from a chem. viewpoint to the disinfection use of Eternit pipes for water. C. R. Fellers.

Chemistry of corrosion [in water pipes]  
George M. Crook. *Proc. 17th Texas Water Works School* 1935, 44-8, *U. S. Pub. Health Eng. Abstracts* 15, W, 122 (Nov. 9, 1935).—A review. C. R. Fellers.

The use of bitumen in the construction of water and sewage mains, plants, etc.  
Fr. Joedicke. *Tech. Gemeindeblatt* 38, 269-72 (1935). M. G. Moore.

Recent tendencies in the field of sewage disposal  
H. Bach. *Chem.-Ztg.* 59, 861-3 (1935). E. H.

Sewage treatment at Muskegon Heights (Mich.).  
R. A. Anderson. *Mech. Eng. Expt. Sta. Bull.* No. 64, 34-43 (1935).—Suggestions for design changes are based on operating experience with the new plant. Operating and cost data are tabulated for ready comparison.

G. H. Young  
Pretoria sewage-disposal works extensions  
G. F. Newby. *Surveyor* 86, 251 (1934), *U. S. Pub. Health Eng. Abstracts* 15, S, 70-1 (Aug. 31, 1935).—The dry-weather flow of sewage is 330,000 gal. per day. The works comprise a screening chamber and 4 units, each consisting of a grit channel, settling tank, 4 trickling filters and 2 sludge-digestion tanks. The new settling tanks have hopper bottoms and provide 2 hrs. retention with an upward velocity of 4.73-7.6 ft. per hr. The new digestion tanks will provide a capacity of 0.78 cu. ft. per head for a population of 100,000. Utilization of sludge gas for heating is not economical in Pretoria because of the very low elec. rates. The final effluent is discharged into the Apes River. C. R. Fellers.

Sewage disposal by subsoil irrigation  
E. Z. Weber. *Gesundh. Tech. Stadtechy.* 25, 145 (1933), *U. S. Pub. Health Eng. Abstracts* 15, S, 48 (Aug. 3, 1935).—Porous subsoil is an excellent medium for the disposal of sewage in settlements not connected with a sewerage system. The sewage should undergo treatment in a septic tank before irrigation in the subsoil. Russian regulations require that not more than 10-20 l. of sewage should be received by 1 sq. m. of surface per day. C. R. Fellers.

Sewerage and sewage disposal of Sittingbourne and Milton  
G. T. Cotterell. *J. Inst. Munic. Co. Eng.* 61, 290 (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 68-9 (Aug. 31, 1935).—This article describes in detail the 3 sewerage systems of the recently amalgamated districts of Milton Regis, Sittingbourne and Murston. Sewage flows through an inlet channel, screens, grit channel and a baffled aerated grease and oil interceptor to 3 two-story settling tanks; sludge from the double hopper-bottomed lower stories of the settling tanks is discharged into 2 digestion tanks which are equipped with heating coils and gas collectors. The sludge gas has a calorific value of 700 B. t. u. per cu. ft. Supernatant liquor from the sludge-digestion tanks is returned to the raw sewage. Digested sludge is dried on clinker drying beds without causing odor, fly trouble or ponding. Settling-tank effluent is treated in an activated-sludge plant which comprises a mixing channel, 6 aeration and re-aeration channels for diffused-air aeration, and 6 hopper-bottomed final settling tanks. Excess activated sludge is returned to the raw sewage. The final effluent is discharged into Milton Creek. C. R. Fellers.

Operation of the York Township sewage-disposal plant  
C. Chamberlain. *Can. Engr.* 69, No. 10, 25-7 (1935).—Weekly operating records are given for the 2.5 million gal. per day of sewage which is treated with  $\text{FeCl}_3$  and dewatered in vacuum filters. Also in *Eng. Contr. Record* 49, 936 (1935). Ann Nicholson Hurd.  
Operation of the Byholmen sewage-disposal plant at Helsingfors.  
R. Granqvist. *Gesundh.-Ing.* 58, 683-8

(1935), cf. C. A. 30, 552<sup>6</sup>—Details of construction and extensive tubular operation data are given. The plant, built to serve a population of 20,000, is overlaid to serve 55,000. Treatment is by simple settling (with partial sludge digestion at the bottom of these basins), activated-sludge treatment, subsequent settling and addnl sep. sludge digestion. The plant is so planned as to facilitate expul work, e. g., there are 4 sep. activated-sludge units, each built on distinctly different plan. M. G. M.

Operating experiences with sewage-treatment plants and the design and construction of new plants. Z. Weidlich *Gesundheitsfach* 26, 185 (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 71 (Aug. 31, 1935).—Sep. rather than combined sewerage systems are recommended for towns whose sewage undergoes treatment. Flow in sand traps should be at the rate of 0.3–0.5 m. per sec. For variable sewage flows, a double channel is recommended. Sep. sludge digestion is preferable to treatment in 2-story tanks. Hopper bottoms should be at least 6 m. deep and not more than 1.2 m. in diam. Digestion tanks should be heated in winter for best results. Fishponds or deep final settling tanks are most suitable for secondary treatment. C. R. Fellers

Sewerage works at Tunbridge Wells. H. P. Bishop. *J. Inst. Munic. Co. Eng.* 61, 169 (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 69 (Aug. 31, 1935).—The av. compn. of the sewage in parts per 100,000 is: free  $\text{NH}_3$ , 1.4;  $\text{O}_2$  absorption in 4 hrs. at 26.7°, 5.2; nitrite and nitrate N, 0.0; Cl 10.3; dissolved solids 53; suspended solids 36; and dissolved  $\text{O}_2$  absorbed in 5 days, 41.4. C. R. Fellers

A biozeolitic theory of sewage purification. Emery J. Thierault. *Ind. Eng. Chem.* 28, 85–6 (1936); cf. C. A. 29, 5560<sup>6</sup>—Previous studies and theories of the action of activated sludge are critically reviewed. The new theory offered holds that clarification by activated sludge (which is complete in 30 min.) is due to a base-exchange process with the aluminosilicate complex or sludge zeolite which surrounds the bacteria. Oxidation is explained partly as the result of primary decomposition by bacteria and partly by enzymic or purely chem. action. Applications to sewage treatment and comparison with corresponding methods of water treatment are considered. M. G. Moore

Adsorption by activated sludge. Emery J. Thierault and Paul D. McNamee. *Ind. Eng. Chem.* 28, 79–82 (1936).—Addnl. evidence is offered in support of the biozeolitic theory of sewage purification (cf. preceding abstract). Measurements on the adsorption and desorption (washing out) of various cations from their salt solns. by both ordinary activated sludge and that which had been previously dried at 100° (to destroy humus and eliminate any effect due to its presence) yielded data which followed the Freundlich formula satisfactorily even over a 20-fold range of soln. concns. The approx. analysis of activated sludge gives: zeolite 30%, bacterial cells 35%, adsorbed matter 20%, water 5% and carbon from humus 10%. M. G. Moore

Mechanical chemical methods of sewage treatment. H. C. H. Sinton. *Surveyor* 85, 375–7 (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 40 (July 20, 1935).—The use of vacuum filters enables the sludge to be de-watered to such an extent that it can be incinerated. In the Guggenheim process which has been on trial in New York and Chicago, the settled effluent after chem. pptn. is passed through a zeolite filter which acts partly as a straining filter and partly as an appliance for the removal of  $\text{NH}_4$  compds. There is an increasing tendency to regenerate and reuse the chemicals employed in water purification or sewage treatment. C. R. Fellers

Removal of hydrogen sulfide in sewage by aeration. W. S. Mahle. *Sewage Works J.* 7, 91–6 (1935); *U. S. Pub. Health Eng. Abstracts* 15, S, 52 (Aug. 3, 1935); cf. C. A. 28, 5157<sup>6</sup>—Expts. showed a marked increase in the  $\text{H}_2\text{S}$  in the Imhoff tanks at the Fort Worth, Tex., sewage plant, a decrease in the aeration tanks, and practically complete elimination on passage through the sprinkling filters. The most economical time for  $\text{H}_2\text{S}$  removal is approx. 150 min., using 0.25 cu. ft. of air per gal. Max.

$\text{H}_2\text{S}$  removal in raw sewage was obtained in 15 min. aeration with 0.4 cu. ft. of air per gal. C. R. Fellers

Susceptibility of certain sewage bacteria to the action of bacteriophages. Olof Sievers. *Z. Hyg. Infektionskrankh.* 116, 15 (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 72–3 (Aug. 31, 1935).—Filtrates from untreated water had a bactericidal action on 82 out of 150 strains of typhoid-colon bacteria from sewage while the filtrate of treated water affected only 47 strains. That is, treatment reduced the lytic action of the water. Bacteriophages play an important part in sewage treatment. Treated water contains more lysin-resistant bacteria than untreated water. The no. of lysin-sensitive bacterial strains is reduced during sewage treatment though they do not entirely disappear; this reduction is believed to be due to bacteriophages, especially in the activated-sludge treatment. C. R. Fellers

Disinfection of sewage with minimum amounts of chlorine. W. L. Mallmann and W. F. Shephard. *Misch. Eng. Exptl. Sta. Bull.* No. 64, 21–6 (1935).—Results are given of a plant-scale check on the work of Rudolph and Ziernba (cf. C. A. 28, 5561<sup>6</sup>). Samples were collected in thio-sulfate-treated bottles, chlorinated samples were collected after exposure for 7, 14 and 21 min.; control samples were collected at a point just before the effluent entered the chlorination tank. Cl demand and residual tests were made each time samples were collected. Av. bacterial count on the influent was 1,000,000 bacteria per ml., Cl demand on the influent averaged 5 p.p.m. Tentative conclusions drawn are: (1) With an excess of Cl, all exposed bacteria are killed and the excess Cl oxidizes org. particles surrounding other bacteria, so that the overall effect is to destroy all bacteria. The need of long detention to effect decided reduction in bacterial counts bears out this theory. (2) The higher the initial count the greater the percentage reduction in bacteria, even though the no. of surviving bacteria is const., accordingly, this method of presenting results gives an inaccurate picture. The safety of an effluent must be based upon the actual no. of bacteria escaping chlorination, not upon percentage reduction. (3) The data indicate that partial satisfaction of Cl demand is unsatisfactory for sewage sterilization. G. H. Young

Stabilization of settling-basin sludge by activated carbon at Sacramento. R. E. Mittelstaedt. *Taste and Odor Control* 1, No. 5, 1–2 (1935); *U. S. Pub. Health Eng. Abstracts* 15, W, 98 (Oct. 5, 1935).—Sludge decompos. in the settling basins imparted a slight taste and odor to the sewage effluent. Washing and scouring gave only temporary relief. Approx. 300 lb. of sludge, mostly alum and mud, is present in 1,000,000 gal. of water entering the basins. Lab. beaker tests translated into terms of plant practice showed that 2 lb. of activated C per 1,000,000 gal. of water was the min. effective dose. This amt. of added C effectively removed all odor from the sludge. C. R. Fellers

Septic tanks for hotels. E. A. Hepburn. *Dept. Pub. Health, State of Victoria, Health Bull.* 13, 1120–4 (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 77 (Sept. 7, 1935).—The capacity of a septic tank in gal. should equal 700 + 30 N, N being the total no. of persons served. Imhoff tanks are preferred to septic tanks where supervision can be ensured. C. R. Fellers

Annual report of the Ruhrverband, 1933. K. Imhoff. *Ruhrverband, Essen* 8 pp (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 65–6 (Aug. 24, 1935).—Ind. pollution in the Ruhr is decreasing. Various new purification plants and treatment plants for sewage are described. C. R. Fellers

Stream pollution with special reference to some South African conditions. M. Lundie. *Surveyor* 86, 205 (1934); *U. S. Pub. Health Eng. Abstracts* 15, S, 52 (Aug. 3, 1935).—The change in the  $\text{O}_2$  content of waters which run normally 0.7 parts per 100,000 is the best measure of the self-purification of polluted waters. At the crit. point increasing pollution will result in more rapid consumption than production of dissolved  $\text{O}_2$ . The degree of  $\text{O}_2$  ab-

sorption constituting safety has been given values varying between 0.25 and 0.5 part per 100,000. C. R. Fellers

River pollution control and its organization in Poland. Z. Rudolf. 4th Hydrol Conf Baltic States, Leningrad 1933, Sed. Underground Waters.—State Hydrol. Inst. and United Sci. Tech. State Editorial Office of People's Commissary for Heavy Ind., U. S. S. R. U. S. Pub. Health Eng. Abstracts 15, S, 22(Mar. 2, 1937)

C. R. Fellers  
Pollution and purification of liquids. M. Y. Khwaja. J. Inst. Sanit. Eng. 39, 43(1935). U. S. Pub. Health Eng. Abstracts 15, W, 149(Dec. 14, 1935).—In nature, self-purification of water is effected by bacteria, sedimentation, dilution, the action of sunlight and the chemical interaction of inorganic substances. Methods of water treatment are described in general terms.  $KMnO_4$  is useful in water disinfection especially during epidemics. C. R. F.

Summary of the recent investigation of swimming pools and bathing places in New Jersey. D. M. Dittmar, et al. Pub. Health News, N. J. Dept. Health 18, 53-8 (1934). U. S. Pub. Health Eng. Abstracts 15, Sw, 3 (May 11, 1935).—Of the 378 places from which samples were collected, fewer than 4% contained water showing *B. coli* present in less than 10 cc. Methods of pool treatment and management are discussed. C. R. F.

Operation of a small swimming bath in the tropics. M. G. Ionescu. Engineering 139, 207(1935). U. S. Pub. Health Eng. Abstracts 15, Sw, 7(Dec. 7, 1935).—The water after treatment with coagulant and alkali is filtered at a rate of 3000-4000 gal. per hr. in a pressure filter. Sufficient  $NaOCl$  solution is added to give a concentration of 1.2 p.p.m. A concentration of 0.8 p.p.m. of  $Cl_2$  did not adequately disinfect the water. Algal growths were controlled by the use of  $CuSO_4$  at a concentration of 1.3 p.p.m. and 0.2 p.p.m. each week afterward. The tank is refilled every 3 weeks. C. R. Fellers

Purification of bath water. A. A. Turner. J. Roy. Sanit. Inst. 55, 441-5(1935). U. S. Pub. Health Eng. Abstracts 15, Sw, 4(May 11, 1935).—At the Finchley pools the water is coagulated, filtered, ammoniated, aerated and sterilized. At the children's pool sterilization is accomplished by the Rayzone ultraviolet-ray system. Bacteriological exams show approx. 1 bacteria per colony on agar plates at 37° and the absence of *E. coli*, *Chloridium welchii* and streptococci in 100 cc. C. R. Fellers

Artificial alteration of the reaction of water reservoirs for the control of the larvae of the malaria mosquito. P. I. Pavlova. Med. Parasitology & Parasitic Diseases (Moscow) 1, 265(1934); U. S. Pub. Health Eng. Abstracts 15, W, 114(Nov. 2, 1935).—In the region near Moscow, larvae of the mosquito, *Anopheles maculipennis*, were destroyed when the bog waters were reduced to a pH value of 1.9 by the addition of  $H_2SO_4$ . In acid peat bogs and in pits lined with clay beds, the pH of the water remained fairly constant for several months, but in alkali peat bogs the acidity was soon lost. C. R. Fellers

Relation of bacteria and bacterial filtrates to the development of mosquito larvae. Lloyd E. Rozeboom. Am. J. Hyg. 21, 167-79(1935); U. S. Pub. Health Eng. Abstracts 15, Ma, 26(Oct. 20, 1935).—Autoclave sterilization of a medium for larvae renders it unsatisfactory for their development. The presence of bacteria seems necessary for larval growth. The bacteria furnish either an intra- or extra-cellular substance that acts as a stimulus to larval development. Bacterial suspensions in distilled water are not entirely satisfactory as larval food. Colloids or solutes were not used as larval nutrients. C. R. F.

Anthracene, a new mosquito larvicide. S. Kutcher. Med. Parasit & Parasitic Diseases (Moscow) 3, 141-8 (1934); U. S. Pub. Health Eng. Abstracts 15, Ma, 18 (May 25, 1935) (in Russian).—Best results were obtained by the use of refuse from coke-benzene plants (containing 12-15% anthracene) plus dust so that the resultant mix contains 10% anthracene. The mix is spread over the surface of the water by means of a pulverizer. The kill is practically 100% of all larvae. The effect on fish and water vegetation was not detrimental. C. R. Fellers

Use of chloropicrin as a mosquito larvicide. F. Yat-

senko. Med. Parasit. & Parasitic Diseases (Moscow) 3, 91-3(1934); U. S. Pub. Health Eng. Abstracts 15, Ma, 17(May 25, 1935) (in Russian).—Chloropicrin may be used both as a fumigant and as a stomach poison. For the 1st, the chloropicrin is mixed with fine road dust and paraffin oil in the proportion of 11 to 1 kg. of dust and 2 l. of paraffin oil and the mix is stored in a closed receptacle for 4 hrs. Just before use, the mix is again mixed with additional road dust (amt. not stated), placed in a pulverizer and sprayed over the surface of the water, this amt. is sufficient for 1 hectare. In 24 hrs. all eggs, larvae, pupae and almost all other aquatic organisms are destroyed completely. Fish are affected only when the depth of the water does not exceed 12-15 cm. Men and animals should not visit the dusted areas for 2 hrs. following treatment. When used as an intestinal poison, 100-150 g. of chloropicrin is mixed with 1 kg. of flour, 1 kg. of fine road dust and 100-150 g. of paraffin oil. This amt. is sufficient for 1 hectare. The mix is kept in a closed container for 24 hrs., after which it is spread by means of a pulverizer or by hand. All larvae are destroyed in 10-12 hrs., though all eggs and pupae are not killed. Both methods are very suitable for mosquito control. C. R. Fellers

Sanitary significance of the succession of coliform organisms in fresh and in stored feces. Leland W. Parr. Am. J. Pub. Health 26, 39-45(1936). J. A. K.

Regenerating fatty substances from waste waters of the wool industry (Sokolov) 27. Gutter and water supplies in Holland (Reith) 11G. Minimal threshold of the dental sign of fluorosis (mottled enamel) (Dean, Elvove) 11G. Sterilizing liquids (Brit. pat. 432,101) 13

Apparatus for purifying water by heating and centrifugal action. John S. Wallace. U. S. 2,027,501, Jan. 14. Various structural and operative details.

Apparatus (with superposed filtering beds) for purifying water. Pierre R. Gaudet (to Soc. d'étude pour l'épuration des eaux, "procédés G. et B."). U. S. 2,027,475, Jan. 14. Structural, mech. and operative details.

Reclaiming pure water by vacuum distillation from impure waters. Vincen P. McVoy. U. S. 2,027,395, Jan. 14. Various details of app. and operation are described.

Transformation products of carbonaceous materials. Établissements Phillips et Pain. Fr. 788,692, Oct. 14, 1935. Products obtained by treating wood, lignite, peat, cork, wood charcoal, coal, coke, etc., with concd.  $H_2SO_4$ ,  $CH_3SO_3H$  or  $SO_3$  or mixtures of these, are used as base-exchange substances for purifying water and treating salt solutions.

Reagent for purifying and softening water. Chem. Werke vorm. H. & E. Albert (Hans Huber and Hans Mengede, inventors). Ger. 622,294, Nov. 25, 1935 (Cl. 85b 101). Addn. to 595,072 (C. A. 28, 4146). The prepn. of a water-purifying reagent from Fe-Al phosphate by the method of Ger. 595,072 is effected at a raised temp. and pressure.

Apparatus for softening water. Établissements Phillips et Pain. Fr. 784,567, July 22, 1935.

Removing boiler scale. Ewald Franz. Ger. 619,570, Oct. 3, 1935 (Cl. 13e 7). The scale and also other non-metallic deposits such as soot or rust, is removed from metal surfaces by heating the metal with a fine-pointed flame of at least 2000°. The flame may be fed by vaporized lower alcohols or ketones and O.

Treating waste waters. Pierre L. Boucherie. Fr. 788,343, Oct. 8, 1935. Apparatus for aerating is described.

Apparatus for purifying sewage by blowing in air. O. F. T. A. (Office française de travaux d'assainissement). Fr. 788,360, Oct. 9, 1935.

Apparatus for aerating and clarifying sewage. Frank S. Currie. U. S. 2,027,370, Jan. 14. Various structural and operative details.

Sewage-sludge treatment. Edward D. Flynn (to Oliver United Filters Inc.). U. S. 2,026,969, Jan. 7, 1935. Solids and liquids of sewage are sepd. and the sepd. solids

are continuously rolled and tumbled in the presence of a sufficient quantity of hot gases and a comminuted combustible material such as coal dust to form pellets substantially capable of supporting their own combustion; these pellets are then burned, and the resulting gaseous products of combustion are used in the pelletizing App is described

Apparatus (with steam-heated tubes) for drying sludge such as sewage sludge. Laurence M. Keoughan U. S. 2,028,395, Jan. 21. Various structural and operative details

Apparatus for removing sludge from sewage settling tanks, etc Samuel Shafter, Jr (to Chain Belt Co) U. S. 2,028,094, Jan. 14. Mech. and operative details

## 15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

M S ANDERSON AND E D JACOB

Soil survey of Wales. Progress report, 1931-34 D O Hughes and W G D Walters *Welsh J Agr.* 11, 188-208(1935), cf C A 26, 4897 —Data are given on the percentages of org matter and available Ca, P and K in typical soils from Anglesey and Northwest Denbighshire. In general, the soils of North Wales have no natural reserves of CaCO<sub>3</sub>. P deficiency is widespread and many of the soils are deficient in K K D Jacob

The soil of Tagaytay Ridge, Cavite D Z Rosell and A S Argüelles *Philippine J Sci* 57, 409-20(1935) —Morphological and phys characteristics, the  $pH$  value and the N, P, K, Ca and Mg contents are given J O H

Study of soils XVI Study of two soils of Indochina. Displaceable acid radicals and  $pH$  Creation of clay improperly called "colloidal" J Clarens and J Lacroix *Bull soc chim* [5], 2, 1884-92(1935), cf C A 29, 7544 —Analyses, particularly in respect to the buffer power to both acids and bases, are given for 2 dissimilar soils of Indochina with suggestions for their phys and chem improvement. In both cases the soil could be improved by increasing its buffer action through the addn of CaO or CaCO<sub>3</sub>. These soils constitute examples of equal phenomena in which both the anions and cations take part, an equal which can be simply expressed by the  $pH$  of the supernatant soln of the soil sample

J R Adams  
Influence of parent material on soil character in humid, temperate climate R S Stauffer *J Am. Soc Agron* 27, 885-94(1935) —Where other factors concerned in soil formation, such as age of material, rainfall, temp, topography and vegetative cover, are very similar, soils may still vary widely in their properties because of differences in materials from which they are being formed. Glacial drift varies greatly even within short distances, and these differences are reflected in the soils formed from it. Parent materials will be responsible to some extent for the characteristics of all except the most highly weathered soils, and a knowledge of this parent material is important in any system of soil mapping J R Adams

Influence of the nature of the soil on the composition and baking value of wheat Em Midge *Compt rend acad agr. France* 21, 832-6(1935) —The gluten content and the quality of the gluten in the wheat increase as the percentage of fine material in the soil increases. An optimum soil constitution is indicated. These wheat values are also in proportion to the humus/clay ratio of the soil. The use of fertilizer caused considerable improvement in the quality and amt. of gluten in the wheat J R A

Solonetz soils of the state farm Delve (Uzbekistan) S A Kudrin and A N Rozanov *Pedology* (U S S R) 30, 371 91(1935) —Analyses—mech., chem (total), and exchangeable bases—of a series of gray semi-desert soils in various stages of solonchicity are given

J S Joffe  
The nature of the chemistry of the solonchak and solonetz of the Araxdyan steppe and methods of chemical amelioration R Kh Aldinyan *Pedology* (U S S R) 30, 392-404(1935). —A series of analyses of solonchak and solonetz soils and the influence of irrigation and gypsum addns on the base exchange complex are given

J S Joffe  
The solonetz and its cultivation in the Transbaikal region I I Gantimurov *Pedology* (U S S R) 30, 149-66(1935) —Chem analyses of a no. of solonetz, solonchetic and solonchak-solonetzic soils are presented

showing that on the true solonetz addns of gypsum were very effective in increasing permeability and decreasing dispersion and moisture-holding capacity. CaO + S and S alone were also effective in the same direction as gypsum J S Joffe

Fertility of marsh soils M. Popp. *Das Superphosphat* 11, 103-5(1935) —Acid-sol Ca in marsh soils varied from 0.85 to 2.00%, K varied from 0.40 to 0.60%, and P<sub>2</sub>O<sub>5</sub> from 0.15 to 0.25%. From 0.15 to 0.45% of N was found. In general the nutrient content of the soils varied with the clay content. The P<sub>2</sub>O<sub>5</sub> content of the marsh soil is dependent upon the age of the soil. With increasing age the P<sub>2</sub>O<sub>5</sub> content decreases. The phys nature of the soil also affects the P<sub>2</sub>O<sub>5</sub> content, the light marsh soils contg less P<sub>2</sub>O<sub>5</sub> than the heavy ones K. C. B

Minerals in soils C D Jeffries Pa Agr Expt Sta, Bull No. 308, 11-12(1934) —Preliminary studies of 4 typical soils showed outstanding mineral differences. Feldspars are notably lacking in Dekalb and Lackawanna soils, and the small amt present is plagioclase and not K-bearing. In Hagerstown and Volusia soils, large quantities of feldspars were present, notably microcline and a trace of plagioclase. Microcline is very insol and does not add materially to the K content of the soil. Mineral analyses of the soils are given and the application of the results to soil fertility and fertilizer studies is pointed out. C R Fellers

A procedure for the estimation of sand in soil samples Eduard v. Boguslawski *Z Pflanzenernähr., Düngung u Bodenkd* 41, 1-8(1935). —Boil a 10-g sample for 30 min with 75 ml 2% H<sub>2</sub>O<sub>2</sub>, to samples high in humus, add 25 ml 6% H<sub>2</sub>O<sub>2</sub> and continue the boiling, keeping the soil well stirred. Transfer the residue substantially freed of humus to a 300-ml Erlenmeyer flask with 0.01 N NaOH, almost filling the flask with the soln, and allow to stand overnight to ensure decompn of all excess H<sub>2</sub>O<sub>2</sub>. Stopper the flask and shake 2 hrs by machine or by hand, and then transfer the contents to a special cylinder bearing 2 marks exactly 15 cm apart, the upper at 300 ml vol, the lower 3-4 cm. from the bottom. Fasten a glass siphon with the lower end reversed, 7-8 mm internal diam, in the cylinder with a stopper, to draw off the suspension of particles smaller than 0.02 mm. to the lower mark, after settling for exactly 7 min. Refill the cylinder to 300 ml with 0.01 N NaOH, stir well, settle and siphon as before, repeating until material is entirely removed. Transfer the residue to a dish, evaporate and weigh as total sand, and further fractionate by suitable sieves C J S

Decomposition of organic matter in Norfolk sand. the effect upon soil and drainage water Charles E Bell *J Am Soc Agron* 27, 934-46(1935); cf C A 29, 6343\* —The addn of freshly ground, dried org matter to Norfolk sand reduced the  $pH$  of the soils at the beginning of the expt below that of the virgin soil. The application of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> further reduced the  $pH$  and the addn of complete fertilizer reduced the  $pH$  to a greater extent than did (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. There was a gradual increase of the  $pH$  from the beginning to the end of the expt. The addn of org materials caused a slight increase in the residual N in the soils after 1 yr. The addn of inorg nitrogenous fertilizer with these org materials in cropped soils caused a slight loss of N as compared with soils to which only org matter had been applied. When only inorg. nitrogenous fertilizers were added to cropped soils, there was an increase in the N over that found in the untreated check



The application of org. matter caused an increased residual org. matter content at the end of the expt. The addn. of org. matter to the soil apparently had little effect upon the amt. of drainage water lost over a period of a yr. More drainage water, total N and total bases were lost from fallow than from cropped soils, and the quantity of N lost depended upon the amt. added to the soil. The loss of total bases was increased by the addn. of org. matter, and applications of inorg. fertilizers increased their loss. The loss of bases was greatest from soils to which the greater quantity of N was added. The addn. of org. matter failed to show any favorable influence on the retentive power of Norfolk sand for bases. The addn. of org. matter increased the exchangeable Ca and Mg content in both the fallow and cropped soils. Soils to which  $(\text{NH}_4)_2\text{SO}_4$  was added contained less exchangeable Ca than those soils not treated with this material, and addn. of complete fertilizer to fallow soils increased the exchangeable Ca. Apparently, the presence of N reduced the amt. of exchangeable Ca in the soil. Fallow soils contained more exchangeable Na and K than did the cropped soils. The univalent ions were of greater quantity in the drainage water from the fallow soils than in the water from the cropped soils, but the quantity of bivalent Ca was about the same in both. As the proportion of Ca in the total bases in the drainage waters increased, the proportion of K decreased. J R Adams

The importance of soil type and mechanical composition for sensitivity of plants to chlorine. A V Sokolov. *Repts. Sci. Inst. Fertilizers and Insectofungicides, Leningrad, 3rd Intern. Congr. Soil Sci., Oxford 1935, 23-32* (in French).—From pot expts with cereals, flax, hemp and buckwheat on soil and soil-sand mixts., in which complete fertilizers are compared with sulfate and Cl, it is concluded that Cl in fertilizers is more harmful on podzols than on chernozems, on the former more Cl is absorbed by the plants. The cause is not lower  $pH$  developed by chlorides, nor is liming always an effective remedy. In general, potatoes and fiber plants growing on light soils are more adversely affected by Cl than when growing on compact soils. Nineteen references. C J S

[Soil] acidity studies. G H Poesch. *Ohio Agr. Expt. Sta., Bull. No. 548 (53rd Ann. Rept.), 56 (1935)*.—Alk. soils are undesirable for snapdragons, clarkia, pine, daisies, lilies, tulips and daffodils. Optimum growth for green color in these plants was at  $pH$  6-6.5. At  $pH$  7 and 8, distinct stunting and yellowing of foliage resulted. This was probably due to the unavailability of P, Fe and Mn. C R Fellers

Exchange cations in the soils of the U S S R. N P Remezov. *Repts. Sci. Inst. Fertilizers and Insectofungicides, Leningrad, 3rd Intern. Congr. Soil Sci., Oxford 1935, 7-22* (in English).—The importance of exchange cations in pedological processes is discussed, with special reference to the principal soil types of the U S S R. Analytical data from the examn., by methods outlined, of profile samples from 10 of these types, taken at agricultural expt. stations, are presented and discussed. C J S

The physical-chemical rules for the cation exchange in mineral soil. F. Alten and B. Kurnies. *Ernähr. Pflanze 31, 401-7 (1935)*.—See C. A. 29, 8197. J O H

The chemical character and importance of the colloidal fraction of the soil. J. V. Csiky. *Z. Pflanzenernähr., Düngung u. Bodenb., 41, 185-203 (1935)*; cf. C. A. 27, 547.—C. agrees with Mattson and Gustafsson (C. A. 29, 2272) in considering soil particles neither uniform, definite compds. nor simple mixts. of such, but gigantic amphotolytic ions which can combine mutually as well as with simple ions, and he explains exchange and hydrolytic reactions and the behavior of the soil's colloidal complex under acid or alk. conditions on this basis. Exptl. work with artificial "Al silicates," "protein-lignates" and natural soil colloids is discussed in support of these ideas. He believes that it is possible to develop a simple procedure, based on equil. between soil and salt soln. at definite  $pH$  values, and requiring detn. of ionic balance, which will completely characterize a soil's colloidal complex and enable comparisons to be made between soils of widely

varying nature. This, with special studies of the soil's content of nutrients and its H-O economy during the growing season, should be of great practical value. Twenty-six references. C J Schollenberger

Chemical nature of organic matter or humus in soils, peat bogs and composts. Selman A. Waksman. *J. Chem. Education 12, 511-19 (1935)*.—In the abstract in C. A. 30, 202, the statement "The lignins contain hemicelluloses, notably polyuronides and resins" should read "The lignins, certain hemicelluloses notably polyuronides and resins are resistant and tend to accumulate."

Irvin C. Feustel  
Characterization of humic acids from the standpoint of carboxyl and hydroxyl groups and nitrogenous compounds. S S Dragunov. *Repts. Sci. Inst. Fertilizers and Insectofungicides, Leningrad, 3rd Intern. Congr. Soil Sci., Oxford 1935, 83-95* (in German).—The chemistry of humus from soil, peat and lignite is reviewed, and expts. are described from which it is concluded that the estn. of COOH and OH groups by methods outlined is of value in soil studies. The humus from soil contained less of both than that from peat, and the transition from lignite to coal showed progressive decreases. Hydrolysis expts. with humic acid indicate that the N compds. are in part proteins and their decomposition products, easily made sol. by heating with acid or alkali, but another part of the N is firmly combined with humic acid and is not attacked by acid hydrolysis. Humic acid from various peats fixed 3-5-4% N on ammoniation. No exact relation between the degree of decompn. of a peat and its N content was discovered, but the higher the content of humic acids, the greater was the quantity of non acid-hydrolyzable N. In many cases, drying peat promoted hydrolysis of the N compds. Eighteen references. C J Schollenberger

Practical soil analysis. L. Schmitt. *Ernähr. Pflanze 31, 421-30 (1935)*.—The detn. of the fertilizer requirements of soils is discussed. With regard to lime requirement, the advisability of detg. values for exchange and hydrolytic acidity in addn. to  $pH$  values is discussed. When interpreting the results of the Neubauer method for available  $K_2O$  and  $P_2O_5$  in the soil, it is necessary to take into account the individual soil characteristics. John O. Hardesty

The determination of organic carbon in soils. László G. Kotzmann. *Mészgazdasági Kutatók 8, 333-7 (1935)*.—The dry combustion method of Dennstedt, and methods of oxidation in soln. by sulfuric and chromic acids and by  $KMnO_4$  were compared on nine soil samples. Two series of expts. were made, one without any previous treatment and another with samples treated previously with a strong soln. of  $H_2SO_4$ . Chromic acid oxidation gave an av. of 91.1%,  $KMnO_4$  oxidation 100.0% of the values obtained by the Dennstedt dry-combustion method. The  $KMnO_4$  method seems to be the best for quick serial work. The values obtained ranged from 90.6 to 113.2% of those obtained by the Dennstedt method. It is proposed to det. factors for the  $KMnO_4$  method on the basis of a set of many soil samples in order to eliminate the errors. S. S. de Finály

The carbide method of determining soil moisture. V. Sibirskii. *Pedology (U S S R) 30, 187-97 (1935)*.—See C. A. 29, 7545. J. S. Joffe

The influence of irrigation on solonetz soils. N. V. Orlovskii. *Pedology (U S S R) 30, 360-70 (1935)*.—Analyses—total and exchangeable bases—on the profiles of some solonetz soils are given. J. S. Joffe

The influence of absorbed magnesium on the physical properties of the soil. P. I. Shavruigin. *Pedology (U S S R) 30, 167-73 (1935)*.—Samples of chestnut brown soils, solonetz and chernozem were satd. with Ca, Mg and Na and subjected to a series of tests. The Mg soil did not filter as well as the Ca soil; its swelling, max. hygroscopicity and moisture capacity were higher than the Ca and lower than the Na soil. J. S. Joffe

Relations between surface, hygroscopicity and heat of wetting of soil. W. U. Behrens. *Z. Pflanzenernähr.,*

*Düngung u Boden* 40, 257-310(1935) —A critical review is given of theories and exptl work of others with description of procedures and calens. B's investigations on the heats of wetting ( $H_w$ ) of Na permute, kaolin and soils with  $H_2O$ , aniline, olive oil, toluene and paraffin oil indicate fairly regular decreases in the above order, i.e.,  $H_w$  decreases as the interfacial tension with  $H_2O$  increases (cf Patrick and Grimm, *C A* 16, 518). Wheat starch showed practically no  $H_w$  except with  $H_2O$ , but the other substances investigated had considerable  $H_w$  with all the liquids. With soils,  $H_w$  in cal/g multiplied by  $3.6 \times 10^4$  approximates surface in sq cm/g, the hygroscopicity over 10%  $H_2SO_4$  in g  $H_2O$ /100 g soil multiplied by  $2.7 \times 10^4$  gives a similar value for surface, calens indicate that the av thickness of the  $H_2O$  layer as above is 0.33  $\mu$  or 1.2 mol diams. Ninety-seven references.

C. J. Schollenberger

Granulometric (mechanical) composition as an index of the physicochemical properties of soils and subsoils V Bezruk *Pedology (U S S R)* 30, 202-18(1935) — Quartz sand was ground in a colloid mill, the fractions were sepd., and their properties with respect to swelling, hygroscopic moisture, max hygroscopicity and plasticity were detd. It is shown that the fractions less than 0.001 mm attain colloidal properties. The various quartz fractions were added to a morain loam (horizon B<sub>1</sub>), podzol loam (horizon B<sub>2</sub>), chernozem (horizon A), and solonetz (horizon B) in quantities sufficient to make them all of the same mech compn. On the mixts the detns made on the quartz fractions and resistance to compression and rate of wetting were also made. It was found that beside the mech compn the difference in the cation satn (Na for solonetz, Ca for chernozem and H for the podzol) has a marked effect on the properties examd and in road making these should be considered. J S Joffe

The influence of soil temperature upon development of plants J O Musso *Z Pflanzenernähr, Düngung u Boden* 40, 311-22(1935) —High soil temp. relative to that of the air is favorable to the development of above-ground parts and hastens ripening, underground parts develop better when the relation is reversed, with delay in maturity. The adaptability of soils to particular crops is influenced by factors affecting soil-air temp. differences, and artificial means of modifying the same, e.g., mulches, may be useful.

C. J. Schollenberger

Factors affecting the value of soil as a source of inoculation for leguminous crops H W Batchelor *Ohio Agr Expt Sta., Bull No 548 (53rd Ann Rept)*, 20(1935) —Four years' work shows that soils of  $pH$  8.0, 7.0 and 6.0 are of value in the order given as carriers of N-fixing bacteria. Soils contg added P were better carriers of the bacteria than unphosphated soils. Probably any soil contg 1 million symbiotic N-fixing bacteria per g is satisfactory to use as an inoculant. C R Fellers

Comparative investigations on ashed and unashed materials by electrometric titration G Demes *Z Pflanzenernähr, Düngung u Boden* 41, 74-82(1935) —The electrometric titration previously described (*C A* 29, 1561) has been applied to the detn of "alkalinity" or potential base content of leaves, etc., in forestry soil studies. Two series of detns were made on the same samples, one set merely suspended in  $H_2O$ , the others previously ashed, but the procedures are not clearly described. The detns on unashed materials were not in good agreement with those previously ashed, the latter procedure is evidently necessary as a preliminary treatment. C J Schollenberger

The relationship between the reaction of a soil and its phosphate content E G Doerell *Das Superphosphat* 11, 107-12(1935) —With an increase in the av yearly temp. from 4° to 10° over a period of 6 years there was a tendency for most of the soils examd to become more alk. As the rainfall increased from 500 to 1300 mm, the no. of soils poor in phosphate increased from 85 to 100% of the total studied. When the soils were classified as to  $pH$  and amt. of phosphate, it was found that the majority of those that were very poor in phosphate were in the range 5.3-6.5. Those soils that were deficient in phosphate were in

the range 6.5-7.5. Those soils classed as good were in the range 7.5-8.5. K. C. Beeson

Efficiency of soil and fertilizer phosphorus as affected by soil reaction Robert M. Salter and E E Barnes *Ohio Agr. Expt Sta., Bull No 553, 3-40(1935)* —A description is given of the effects of liming the naturally acid Wooster and Canfield silt loams upon the availability of soil P and upon the comparative efficiencies of different P fertilizers. Data from 6 field and 1 greenhouse study show there is a notable tendency for P response to decline as the soil reaction is changed from  $pH$  5.0 to 7.5 by repeated lime applications. Lime makes available the stores of soil P with a rise in  $pH$  value. With most crops nearly max yields were produced at the most alk. reaction without any added P. Soil P sol in dil. org. acids at const.  $pH$  also was lowest at  $pH$  5.0, increased markedly to  $pH$  7.5, and was higher at  $pH$  4.5 than at 5.0. Sudan grass in the greenhouse removed considerably more P from unphosphated soil at  $pH$  7.0 than at 6.0. Conclusion: By maintaining the reaction of these soils at  $pH$  7.5 satisfactory yields of crops may be produced with a min. investment in P fertilizers. Probably greater fixation of P took place at the higher  $pH$  values. Steamed bone meal showed an efficiency compared to superphosphate of approx. 80% for the cereals and timothy and equalled the superphosphate for clover on unlimed land, at  $pH$  5.0. As the soil reaction was increased to  $pH$  7.5 by repeated liming, the efficiency of the steamed bone decreased to 0 for corn, to about 20% for oats and wheat and to 30% for clover. Basic slag showed an efficiency compared to that of superphosphate of about 85% for the cereals on unlimed land and was about 40% superior to superphosphate for clover under the same conditions. With repeated liming the efficiency of the basic slag dropped to 0 for corn, to 70% for the cereals and to 85% for the clover. The P of rock phosphate had an efficiency of about 40% for both grain crops and clover on unlimed land. With repeated liming to a  $pH$  of 7.5 its efficiency dropped to 10% or less for the same crops. Its efficiency for sweet clover at  $pH$  7.0 was about 50%.  $NH_4H_2PO_4$ , with wheat at  $pH$  5.5 showed an efficiency, as compared to superphosphate, of 68%. This increased to 73% at  $pH$  6.0 and to 112% at  $pH$  7.0. The efficiency of all P fertilizers on these soils decreased as the soil reaction approached neutrality. C R Fellers

Availability and fixation of phosphorus in Hawaiian soils A Floyd Heck *J. Am. Soc. Agron.* 27, 874-84(1935), cf *C. A.* 30, 2061 —The quickly available P content of Hawaiian lateritic soils is more variable than that for most groups of soils. As many as 75% of these laterites have a P availability low enough that they should respond to P fertilization. Laterites with a  $pH$  value below 6.5 are usually low in available P. Of the 70 soils with a  $pH$  value below 6.5, 54% have a P availability of 25 p. p. m. or less, and only 9% are above 100 p. p. m. In the 24 soils with  $pH$  values of 6.5 or higher, 54.2% have an availability above 100 p. p. m. Native P in these laterites of low P availability is largely in the form of basic Fe phosphate with a soly. of P similar to that of dufrenite. When soil P is applied to these laterites, the majority of them fix over 80% of the applied P in slowly available form. Most of this P is fixed in the soil as basic Fe phosphates, with perhaps some Al phosphates, but there is seldom much of it as Ca phosphate. J R A

Further experiments on the influence of soil reaction and phosphonic acid on the growth of vines Karl Möhringer *Z Pflanzenernähr, Düngung u Boden* 41, 28-36(1935) —In a continuation of the work previously reported (*C. A.* 29, 1558), the effectiveness of Rhenania phosphate in promoting general vigor and root growth was confirmed, a small dose of  $CaO$ , raising the  $pH$  value from 3.9-4.1 to 4.3-4.5, caused a temporary improvement almost as great, although the final effect was less favorable. This indicated a need for  $P_2O_5$  as well as reduction of soil acidity. Observations on leaf color, etc., indicated some correlation with factors known to affect the vines, but it is concluded that the practical value of such foliar diagnosis is slight. Detns of  $P_2O_5$  and  $K_2O$  in wood and roots at the end of the expt. indicated nothing significant.

Differences in soil  $pH$  at the end were so slight that they were probably not decisive, unless indirectly through changes in availability of  $P_2O_5$ ; the substantial correctness of the acidity limit,  $pH$  5, for good growth of the vine still stands. Nine references. C. J. Schollenberger

The role of phosphates and lime on podzol soils of variable concentrations of hydrogen and iron. A. T. Kurnanov. *Podology* (U. S. S. R.) 30, 493-500 (1935). — Pot expts with barley on podzol show that P in the form of acid phosphate immobilizes the soil iron, an addition of lime accomplishes this result and increases the  $pH$ , the addition of  $Na_2HPO_4$  immobilizes the Fe and increases the  $pH$ . Mobile Fe decreases the P intake by plants. Thomas slag is very effective on podzols because of its activity similar to  $Na_2HPO_4$ . J. S. Joffe

The determination of the forms of inorganic phosphorus in soils. R. Anderson Fisher and R. P. Thomas. *J. Am. Soc. Agron.* 27, 863-73 (1935). — A method is proposed for distinguishing the different forms of inorg. P. An extractant of  $pH$  2 consists of a 0.02 N  $H_2SO_4$  and 0.3% of  $KHSO_4$  and another of  $pH$  5 consists of a buffered soln. of  $AcOH$  contg. 3.6 cc of concd.  $AcOH$  per l. and 19.04 g. of  $Na$  acetate. To two 750-cc. Erlenmeyer flasks contg. 2-g. samples of soil is added 400 cc. of the  $pH$  5 extractant and, similarly, to a third flask is added an equal vol. of the  $pH$  2 extractant. These solns were sufficiently buffered to maintain unchanged  $pH$  values during extn. of calcareous as well as acid soils. After 30 min. of shaking, the soln. of 1 of the  $pH$ -5 extn. flasks is filtered. The other  $pH$ -5 extn. soln. is filtered after 2 hrs. of shaking and the soln. with the  $pH$  2 extractant is filtered after 2 1/2 hrs. of shaking. In each case only the filtrate which has run through in 15 min. is used for analysis and of this the first 25-30 cc. is discarded. Regular colorimetric detns are made on these filtrates. From data on the solvent action of these 2 extractants, equations are set up for estg. the P present in the following 3 groups of materials: (A) amorphous and finely divided cryst. phosphates of Ca, Mg and Mn; (B) amorphous phosphates of Al and Fe; and (C) P absorbed upon hydrous oxides and that present in the form of apatite. This method of analysis placed 22 representative Maryland soils in practically the same order of P needs as did pot tests for response to  $P_2O_5$  fertilization. J. R. Adams

The study of the mutual reactions of phosphates and soils. II. Action of lime on monocalcium phosphate in the presence of anhydrous calcium sulfate. J. Clurens and H. Marrius. *Bull. sv. chem.* [5], 2, 1930-5 (1935); cf. C. A. 23, 3325. — The presence of  $CaSO_4$  modifies the pptn. of  $CaH_2(PO_4)_2$  by  $CaO$ . At first  $CaHPO_4$  is pptd. alone but the pptn. of  $Ca_2(PO_4)_2$  soon follows and the proportions of this salt rapidly increase. With sufficient  $CaO$  the  $P_2O_5$  is completely pptd. as tricalcium phosphate. The  $P_2O_5$  is first pptd. as the monocalcium salt and addnl.  $CaO$  merely changes this to the  $Ca_2(PO_4)_2$ . The application of these data to the use of superphosphate as fertilizer is shown. J. R. Adams

The relative feeding power of oaks and maples for soil phosphorus. H. L. Mitchell and R. F. Finn. *Black River Forest Papers* 1, 6-9 (1935). — Over the range of P applied to the soil in the form of finely ground Florida phosphate rock, red maple leaves contained (wt. for wt.) approx. twice as much P as did the leaves of red oak growing on the same site and receiving identical fertilizer treatments. The ratio between the P absorption of the 2 species was approx. the same with the P supplied as "soil phosphate" (unfertilized soil of control plot) or as soil P plus various supplements of rock phosphate. Likewise, the leaves of sugar maples contained approx. twice as much P as did those of chestnut and white oaks growing on the same sites. P absorption tended to vary between genera but not with species. K. D. Jacob

A method for determining the nutrient needs of shade trees with special reference to phosphorus. Harold L. Mitchell. *Black River Forest Papers* 1, 1-4 (1935). — Comparable 0.25-acre plots of an even-aged (about 40 years) stand of mixed hardwoods, principally red oak (*Quercus borealis* Michx.), chestnut oak (*Q. montana*

Willd.) and red maple (*Acer rubrum* L.), were fertilized uniformly with varying amts. of finely ground rock phosphate on May 23, 1934. On Oct. 1 of the same year, 5-6 leaves of the various species were taken from approx. the same location on each tree (from the ends of the branches near the top of the crown on the south side); after they were dried, the petioles were removed and the finely ground leaf material was analyzed for P. The results indicated a close relationship between the amt. of P applied to the soil and the quantity of this element in red oak, chestnut oak and red maple leaves. Preliminary results indicated that there is also a correlation between the N and K contents of the leaves of various deciduous trees and the quantities of these nutrients supplied by soils. Ground phosphate rock seems to be a good source of P for both coniferous and deciduous trees. K. D. Jacob

The assimilation of phosphorus by *Aspergillus niger* and *Cunninghamella* sp. F. B. Smith, P. E. Brown and H. C. Muller. *J. Am. Soc. Agron.* 27, 988-1000 (1935). — The wt. of *Aspergillus niger* mycelium in Nihilas medium (cf. H. Nihilas, *et al.*, *Ernährung Pflanze* 26, 97-103 (1930)) to which sol. was added was roughly proportional to the concn. of P in the medium over the range from 0 to about 60 p. p. m. of P. This growth of *Aspergillus niger* may be used to indicate roughly the amt. of P available to *Aspergillus niger*, but the application of such results to show quantitatively the amt. of available P in the soil is likely to lead to erroneous conclusions. It may serve as a qual. test if the technique employed is carefully standardized. The most satisfactory procedure for the soil used was 4 g. of soil in 90 cc. of medium in a 250-cc. extn. flask with a 1-cc. suspension of spores 6-10 days old for incubation and an incubation period of 6 days. J. R. Adams

The role of potassium and of phosphorus in the utilization of nitrate and ammoniacal nitrogen by plants. F. V. Turchin. *Repts. Sci. Inst. Fertilizers and Insect-Fungicides, Leningrad, 3rd Internat. Congr. Soil Sci., Oxford 1935, 33-49* (in German). — From yields and compn. of barley grown in pots of sand, variously fertilized and with N supplied in several forms, and other crops in field expts., it is concluded that with deficiency in K,  $NH_4$  is not efficiently utilized, but its accumulation in the leaves is toxic. Glucose also accumulates. When N is supplied as nitrate, lack of K is less serious, but the importance of P is greater.  $P_2O_5$  is essential for the normal utilization of nitrate, with an insufficient supply, nitrate accumulates in the leaves. The relatively greater importance of K and P therefore depends upon the form of N available. In the soils of southern U. S. S. R., this is mainly nitrate; hence K is not needed so much as for the acid northern soils in which  $NH_4$  is the source of N. These characteristics of the action of K and P also exert an influence upon the reduction-oxidation power of plant tissues, as shown by expts. P supplied in fertilizers increased the reducing power; this effect is explained as due to utilization of nitrate which requires a reduction. On the other hand, K increases oxidation power, required for the synthesis of amides from  $NH_4$ . Depending upon the source of N, there is probably an optimum reduction-oxidation potential in plant tissues, and a proper balance between K and P supply is favorable to its maintenance. Fourteen references. C. J. Schollenberger

Phosphorus content and buffer capacity of plant sap as related to the physiological effect of phosphorus fertilizers in fibrous low-moor peat. J. R. Neller. *J. Agr. Research* 51, 287-300 (1935). — Fresh-sap studies were made of the leaves, and in some cases of the stems, of 5 different plants growing under field conditions on the brown fibrous peat of the Everglades. The total sol. P of the saps of these 5 plants was much increased by soil dressings with sol. phosphates. Lime acted to reduce and S to increase the assimilation of P in all plants studied. Total acidity and the total amt. of inorg. P tended to vary directly with the concn. of sol. P in the sap. Active acidity of the plant sap, expressed as  $pH$  values, changed appreciably as a result of different phosphate treatments, whereas the buffer capacities were much greater in the saps of high P content. Neither the sp. concs. nor the total sol. solids

of the saps appeared to have any definite relation either to P concn or to physiol injury. The crops that responded unfavorably, if at all, to P were those of which the saps were relatively high in P in the absence of soil treatment with a phosphate carrier. Those that responded favorably contained sap relatively low in P. W. H. Ross

Migration of phosphoric acid during the course of the process of podzolization. Joret and Malterre. *Compt rend acad Agr France* 21, 943-6 (1935).—The infertility of podzolized soils is due not only to a removal of the bases but also to the removal of  $P_2O_5$  during podzolization. The removal of  $P_2O_5$  to the lower horizons is paralleled by the removal of  $Fe_2O_3$ . The removal of  $P_2O_5$  from the surface soil does not prohibit setting up a supply of  $P_2O_5$  in this soil but it puts a time limit on its availability. J. R. Adams

The influence of special cultivation and liming on forest soils. Hugo Lendle. *Forstwiss Centr* 57, 477-94 (1935).—A preliminary report is given on the sep and combined effects of liming and cultivation with a special implement. Expts were made over a 3-yr period on forest soils of low lime content. Humus was detd by loss on ignition, and  $pH$  colorimetrically and by the quinhydrone electrode. Methods for mech analysis, percolation and pore vol also are described. *Ibid* 513-29.—The cultivation markedly reduced the time of infiltration of  $H_2O$  into the soils while liming caused a slight increase in the time on the cultivated plots. The penetration resistance to a sounding instrument was increased by lime on plots low in humus but decreased on those higher in humus. The loosening effect of cultivation was most lasting on soils low in clay and humus content. *Ibid* 550-72.—Liming and cultivation caused a decrease in humus content.  $H_2O$  content in general was parallel to the humus content. The limed and cultivated soil of one plot showed a higher relative  $H_2O$  humus ratio than the plots cultivated or limed separately. The reverse was true on another plot, however. *Ibid* 695-608.—The  $pH$  of fresh soil was from 0.1 to 1.4 units higher than of air-dry soil from one plot but in others small differences were found. Liming was effective to a depth of 10 or 15 cm, and raised the surface  $pH$  from 5.5 to nearly neutral. Cultivation raised the  $pH$  of sandy soil but not of loam soil. *Ibid* 627-39.—A summary of preceding articles is given with a list of 62 references. Irvin C. Feustel

Lime and calcium sulfate plants, a contribution to the subject of soils and plants. O. H. Volk. *Ber. deut. botan. Ges* 53, 796-806 (1935).—Soils high in  $CaSO_4$  did not differ essentially from soils high in  $CaCO_3$  in the properties investigated. The plants found on  $CaSO_4$  soils are similar to those found on other soils that have an alk  $pH$  and are well aerated, provided other conditions are favorable. Lawrence P. Miller

Atmospheric additions of combined sulfur to arable soils. Gabriel Bertrand. *Compt rend acad. agr. France* 21, 1015-18 (1935), cf. *C. A.* 29, 8998<sup>1</sup>.—Careful detas of the combined S in rain water near Grignon showed that there was about 2.5 mg of the metalloid per l of water. If this was completely absorbed by the soil, it would be sufficient to cover the S requirements of the principal cultivated crops of that locality. J. R. Adams

Soil and fertilizer studies by means of the Neubauer method. S. F. Thornton. *Ind. (Purdue) Agr. Expt. Sta., Bull* No. 399, 3-38 (1935).—Soils of  $pH$  5.6-8.0 were examd by the Neubauer technic to det mineral nutrient deficiencies. Very acid soils are slightly more deficient in available P and K than those of neutral reaction, but soil reaction cannot be used as an indication of fertilizer needs. Subsoils are much lower in available P than surface soils. Nearly all subsoils are deficient in P. Values for available K are only slightly lower for subsoils than for corresponding surface soils. For 20 soils of  $pH$  values 4.5-8, P recovery varies from 4.4 to 56.8% with  $CaH_2(PO_4)_2$  and from 4.4 to 42.4% with  $Ca_3(PO_4)_2$ . The efficiency of sol  $CaH_2(PO_4)_2$  is detd largely by the fixation capacity of the soil, while that of the  $Ca_3(PO_4)_2$  is influenced by both fixation power and solvent power. K recoveries are much higher than those of P, varying from 29.2 to 100%.

1 On very acid Newton fine sandy loam, the addn of lime doubles the P recovery from  $CaH_2(PO_4)_2$  but has little effect on K recovery. With separates from 8 types of domestic rock phosphates, neither the Neubauer method nor pot tests show any significant influence of particle size on P availability. P recovery from 22 different P carriers is increased by addn of KCl, the av increase being 4% from both quartz sand and acid silt loam soil. For 21 different soils of  $pH$  4.5-8.0, the addn of KCl results in a consistent increase in the available P supply. The addn of Ca phosphates does not increase the available soil supply of K.  $NH_4NO_3$  gives a marked increase in recovery of P from  $Ca_3(PO_4)_2$ . Results of the analysis of soils from 6 continuous fertilizer expts running 10-20 years show no significant residual accumulation of available P or K as a result of the different treatments. Seventy-seven references. C. R. Fellers

3 Movement of fertilizer salts in soils by capillarity. G. M. McClure. *Ohio Agr. Expt. Sta., Bull* No. 548 (53rd Ann. Rept.), 18-19 (1935).—P supplied by rock phosphate did not move upward to any extent. That supplied by either  $(NH_4)_2HPO_4$  or  $NH_4H_2PO_4$  showed the greatest movement, followed by Na and K phosphates. The movement from treble superphosphate was appreciably greater than that from 20% superphosphate but less than that from the  $NH_4$ , K or Na phosphates. The presence of  $(NH_4)_2SO_4$ ,  $NaNO_3$ , or  $NaCl$  did not affect the extent of the movement of P from 20% superphosphate. N supplied by the  $NH_4$  phosphates did not move upward to so great an extent as did that supplied by  $(NH_4)_2SO_4$  or  $NaNO_3$ . The soils used were Chenango loam of  $pH$  4.6 and after liming 7.2, Wooster silt loam, Miami silty clay loam and a Brookston clay. The fertilizer was applied in a narrow zone about 1/4 in. above the bottoms of soil columns which were 6 in. deep and 3/4 in. diam. C. R. Fellers

5 Irrigation water and cultivation of citrus. A. Reilenberg. *Hadar* 8, 231-3 (1935).—Irrigation waters from various sources in Palestine contained bicarbonate (expressed as  $HCO_3$ ) 96-304, Cl 47-1784, sulfate (expressed as  $SO_4$ ) 6-189, nitrate (expressed as  $NO_3$ ) 6-57, Na 5-917, K trace to 57, Ca 39-265, Mg 7-102 and Fe trace to 14 p.p.m. The  $pH$  values were 7.3-8.6. Damage to citrus trees occurs when the Cl content of the water is 300 mg/l. or more, but complete infertility or death of the trees occurs only at much higher concn. K. D. J.

The requirements in water and fertilizers for the accumulation of nicotine in some types of *Nicotiana rustica* cultivated in the irrigated fields in the lower Volga region. A. Ya. Tolstoplek. *Tobachnaya Prom* 1935, No. 3, 20-2.—The yield of nicotine does not show any regularity when irrigation and fertilizing conditions are changed; the changes affect various types of *Nicotiana rustica* differently. A. A. Boechting

Further experiments on the fertilizing action of magnesium. M. Popp, J. Contzen and F. Nieschlag. *Z. Pflanzenenernähr., Düngung u. Bodenk* 40, 323-57 (1935).—Pot tests with grass and field expts with potatoes on peat and sandy soils, comparing  $CaH_2PO_4$  and  $MgHPO_4$ ,  $K_2SO_4$  and sulfate of potash magnesia and other fertilizers, as to effect upon yields and compn. of crops and analyses of the soils are reported. Conclusion. Although Mg salts are in many cases beneficial, their effect cannot be predicted from data obtained by HCl or  $NH_4Cl$  extn. of the soil. C. J. Schollenberger

Ammoniated peat—mechanism of formation of water-soluble nitrogenous constituents. L. B. Howard, L. A. Pinck and G. E. Hilbert. *Ind. Eng. Chem.* 27, 1508-9 (1935), cf. *C. A.* 29, 3447<sup>1</sup>.—Ammonia, urea, other amide, unclassified and total N were detd. on the cold water exts of a peat ammoniated 4 hrs at 180° at 35 kg per sq cm and of the same peat after drying and reammoniation for an addl 24 hrs. The formation of 75-90% of the water-sol constituents of ammoniated peat involves the production of  $NH_4$  salts of carboxylic acid which are transformed on continued heating (1) by decarboxylation to evolve  $CO_2$  which in the presence of excess  $NH_3$  is converted to urea and (2) by dehydration to

form other amides. This transformation of the  $\text{NH}_4^+$  salts prevents loss of  $\text{NH}_3$  by disson upon storage. Max. potential carboxylic acid content of the peat is desirable.

Colin W. Whittaker

Color of apples as affected by weather and cultural conditions. C. O. Rawlings. New Hampshire Hort. Soc., 24th Ann. Rept. 1934, 29-34.—Defoliation of apple trees was accomplished by spraying the trees with 5% solns of either  $\text{Ca}(\text{NO}_3)_2$  or  $\text{NaNO}_3$  2 weeks prior to harvest, the treatment materially improved the color of the apples without injuring them, although the Williams variety showed some fruit drop. Injection of  $\text{H}_2\text{BO}_3$ ,  $\text{HCl}$ , sugar or  $\text{NaNO}_3$  into the branches caused defoliation in some instances, but none of the treatments was successful, for one reason or another, although  $\text{H}_2\text{BO}_3$  markedly improved the color of the fruit.  $\text{NaNO}_3$  reduced the color and caused leaf injury. The color of the fruit was improved somewhat by ringing the trees to increase the sugar content of the apples. The color of the apples was increased markedly in some instances by applying sugar to the soil beneath the trees 3 weeks before harvest, and the fruit ripened 2 weeks earlier, direct injection of sugar into the branches had no significant effect. Application to the soil of S, lime, citric acid,  $\text{Fe}_2(\text{SO}_4)_3$ ,  $\text{MnO}$ ,  $\text{KCl}$  or superphosphate had no effect on the color. In general, late summer applications of  $\text{NaNO}_3$  did not affect adversely the color of the apples.

K. D. Jacob

Examination of vine leaves to determine deficiency of nutrients. László Salacz. Magyar Ampel. Értékönyv 9, 218-53(1935).—Analyses are given of vine leaves during the period of vegetation on plots untreated and fertilized with various combinations of plant nutrients. The N content decreased from 3.29-4.14% in May to 0.85-1.85% in October.  $\text{K}_2\text{O}$  content of dry matter was 1.64-1.96% in May and diminished to 0.73-1.23% in Oct.  $\text{P}_2\text{O}_5$  decreased from 0.66-0.79% in May to 0.08-0.20% in Oct. The results correspond to those of Lagatu (C. A. 22, 837) with the exception that in Hungary the K content of the leaves is not higher than that of nitrogen in a good harvest.

S. S. de Finály

Experiments to formulate practical rules of fertilization. László Salacz and István Kúhn. Magyar Ampel. Értékönyv 9, 254-7(1935).—Expts. made on different types of vine soils in Hungary from 1921 to 1934 are summarized. Yield produced by various mixes of fertilizers is noted. Best results seem to be obtained by using chiefly N fertilizers only. P and K salts are of less importance.

S. S. de Finály

Choosing the proper types of fertilizers on the basis of characteristics of vine soils. Desző Dicity. Magyar Ampel. Értékönyv 9, 257-66(1935).—A table based on theoretical investigations is given, by which fertilizers can be selected for vine soils according to their appearance, color, lime content and density.

S. S. de Finály

Hardiness [of apple trees] in relation to fertilizer application. Fred. S. Browne. Pomological & Fruit Growing Soc. Quebec, 41st Ann. Rept. 1934, 22-4.—The av. hardiness rating (resistance to winter injury) of Wealthy apples was 80.56 for trees receiving fertilizers contg. N, P and K. The av. rating was 26.25 for trees receiving only N and P, 26.46 for those receiving only N (applied as  $\text{NaNO}_3$ ) and 29.37 for unfertilized trees. Similar results were obtained with the Golden Russet variety, but fertilizers had comparatively little effect on the hardiness of the more resistant Fameuse variety.

K. D. Jacob

Fertilizer experiments on lucerne grass. Felix Frank. Erdk. Pflanze 31, 410-15(1935).—It is only when the crit.  $\text{H}_2\text{O}$  content of the soil has been detd. (Sekera's method) that allowance can be made for the mobility of the soil nutrients, and the Neubauer values corrected accordingly. This explains why good response of  $\text{K}_2\text{O}$  fertilizers is found on soils with high Neubauer values. Neubauer values are of themselves not a reliable criterion of the fertilizer requirements of the soil.

J. O. H.

The technology of humic phosphate fertilizers. K. K. Apushkin and E. A. Kurochkina. Mineral'nue Udo-

breniya Insektosungusidus 1, No. 4, 96(1935).—Humic phosphates contg. up to 40%  $\text{H}_3\text{PO}_4$  were obtained by repeatedly treating peat with technical  $\text{H}_3\text{PO}_4$ . In this process peat is partially hydrolyzed to sugars which can reduce Fe, forming with it complex compds., which in their turn inhibit the pptn. of Fe phosphates. The humic phosphates on being treated with liquid  $\text{NH}_3$  and  $\text{KNO}_3(\text{NO}_2)$  become ammonized, this permits the prepn. of humic ammonium nitrate phosphates with a ratio of N P of 1:1, with a humic ammonium nitrate phosphate content of up to 20% in the fertilizer (over 40% of N + P). The humic ammonium nitrate phosphates were obtained not only from peat but also from long-flame coals. Good results were obtained with these compds. in the field.

A. A. Boethlingk

The agricultural chemical characteristic of potash-ammonium nitrate. F. V. Turchin. Mineral'nue Udobreniya Insektosungusidus 1, No. 4, 68-72(1935).—The best fertilizer for areas requiring N and K is an equimol. mixt. of  $\text{NH}_4\text{NO}_3$  and  $\text{KCl}$ . The presence of Cl (22-23% of the weight of the fertilizer) in the potash-ammonium nitrate fertilizer does not have a detrimental effect on the plants.

A. A. Boethlingk

The antagonistic action between potash and alkaline earths for plant growth. Kisaburo Shibuya and Takashi Toru. J. Sci. Soil and Manure (Japan) 9, 411-24(1935).—Expts. were conducted with rice plants, sown and grown for 18 days in quartz sand according to the Neubauer method. Mixed salts in soln. together with lime and magnesia were added in several quantities to 0.05 N  $\text{K}_2\text{SO}_4$ , 0.1 N  $\text{KCl}$  or 0.01-0.05 N  $\text{KNO}_3$ . Results were: (1) K-Ca and K-Mg antagonisms were noticeable during the growth of rice plants. (2) Degree of the antagonistic action depended upon the kinds of cations and anions in the following order: K-Ca greater than K-Mg, and  $\text{SO}_4$  greater than Cl greater than  $\text{NO}_3$ . (3) The greatest effect of antagonistic action was observed in the soln. of salts of the same disson degree. (4) The antagonistic action caused an unfavorable effect upon plant growth in cases where the nutritive potash was present in less than adequate amt. The effect was not so unfavorable in the cases where potash was in excess. (5) The nitrates were observed to display only slight antagonism between cations owing to the fact that they changed the disson degree to no appreciable extent on being added to the soln.

Y. Kamoshita

Determination of fertilizer needs from root-soluble, citric acid- and water-soluble phosphoric acid. II. E. Knickmann. Z. Pflanzenernähr., Düngung u. Bodenk. 41, 208-24(1935).—A continuation of the study previously reported (C. A. 28, 1451), now including data on 4126 samples, indicates that a prediction as to the profitable use of a  $\text{P}_2\text{O}_5$  fertilizer based on a lab. examn. of a soil sample by any single test is of questionable value, and in any event requires careful consideration of local conditions rather than a rigid adherence to arbitrary rules. The application of 2 or more different tests may lead to more reliable conclusions. The indications as to root-sol and  $\text{H}_2\text{O}$ -sol  $\text{P}_2\text{O}_5$  agreed better than in the previous study, but  $\text{H}_2\text{O}$ - and citric acid-sol. data were again at wide variance.

C. J. Schollenberger

Compositions of nitrogenous fertilizer salts sold in the American market. Henry E. Cutts. Ind. Eng. Chem. 27, 1491-2(1935).—All constituents whose presence was suspected were detd. in 4 samples of com.  $(\text{NH}_4)_2\text{SO}_4$ , 3 of domestic and 6 of foreign  $\text{NaNO}_3$ , 1 of Ca cyanamide, 2 of Chilean and 2 of synthetic  $\text{KNO}_3$ , 1 of Ammono-Phos, 1 of  $\text{Ca}(\text{NO}_3)_2$  and 3 of  $\text{NH}_4\text{NO}_3$  (Calnitro). The analyses are believed to be representative.

C. W. W.

Comparative test of different bedding materials and chemical supplements with cow manure applied in a three-year rotation. T. E. Odland and H. C. Knoblauch. R. I. Agr. Expt. Sta., Bull. No. 251, 3-10(1935).—Straw, sawdust and wood shavings used as absorbents for cow excrement over an 18-year investigation showed no ill effects on crop yields when used as fertilizer. There was no significant change in the soil acidity of any of the plots receiving these materials. When these bedding materials

were supplemented with P and K, normal crop increases were obtained. C. R. Fellers

Further vegetation experiments with Kotka phosphate C. Krügel and C. Dreyspring. *Superphosphate* 8, 201-6, 221-6 (1935); cf. C. A. 28, 3512. —Kotka phosphate is a material made in Finland by treating phosphate rock with a deficiency of  $H_2SO_4$ , approx 55% of the total P is sol in water and approx 63% is sol in water and  $NH_4$  citrate soln. In expts with winter cereals on acid to slightly acid loamy sand soils and sandy loam soils the effect of Kotka phosphate and of basic slag in increasing the initial growth of the plants was inferior to that of superphosphate. As vegetation progressed the differences became less pronounced, and at the end of the period of growth the 3 materials gave practically the same results. When it was applied as a top-dressing, Kotka phosphate gave results decidedly superior to those obtained with basic slag but somewhat inferior to those obtained with superphosphate. K. D. Jacob

The conservation of liquid and stable manures with superphosphate W. Dix. *Das Superphosphat* 11, 105-7 (1935). —Ten-lb quantities of superphosphate were added 3 times during collection to a 5000-l pit of urine. By the addn of superphosphate total solids increased from 1.97 to 3.04%, P 0.26 to 0.58%, K 32.4 to 33.0%, and although the N content decreased from 15.43 to 13.7%, the amt of N per l increased from 3.05 to 4.16 g. Mixts of superphosphate and stable manure showed 40% more N than did the stable manure alone. K. C. Beeson

Can fertilizers be harmful to fish in ponds? P. Vivier. *J. agr. prat.* 99, 328-9 (1935); cf. C. A. 28, 4160. —While ammoniacal salts are much more toxic to the fish than K salts, the concns in the ponds arising from the normal use of these salts as fertilizer on the surrounding soil or in the ponds is so small that they are inoffensive to the fish.  $P_2O_5$  fertilizers are absolutely inoffensive. J. R. Adams

Evaluating liming materials M. Popp. *Deut. Landw. Presse* 62, 487-8 (1935). —The solubilities of several carbonates and quicklimes in  $AcOH$  (15 g per l) were detd. The particle size in each case was the same, and the time of contact with the acid soln in each case was 10 min. The 3 softest and most porous limestones were about 86% sol. The remaining limestones averaged 84% sol. Cryst. materials were not less sol than the noncryst. A dolomite showed a soly of 10.7% in 10 min, and after 60 min of contact with the acid soln, the soly reached 22.8%. Several quicklimes low in Mg were about 90% sol, while those contg Mg showed a wide variation in soly. (57.8 to 83.1%). K. C. Beeson

Rates of solution and movement of different fertilizers in the soil and the effects of the fertilizers on the germination and root development of beans Charles B. Sayre and Arthur W. Clark. N. Y. Agr. Expt. Sta., Tech. Bull. No. 231, 67 pp (1935). —Greenhouse and field expts show that different kinds of fertilizers with the same placement may cause vastly different results on the growing plant or germinating seed. When broadcast, fertilizers are less likely to be injurious than when applied in contact with the seed. Contact of highly ionized mineral fertilizers with the seed or plant causes injury by plasmolysis. Many org. fertilizers also show this injury when first applied to the soil. Apparently injury from org. substances is caused by such sol. compds as amino acids or sol. peptides. Org. fertilizers greatly stimulate mold growth in the soil. Possibly some parasitic molds are thus stimulated and attack the plant to a greater extent. Lateral movement of fertilizer in the soil was very limited. Except with  $CaCN_2$ , the movement did not exceed 0.5 in. Side placement of fertilizer is likely to give better results than direct placement on the row above or below the seed. N compds dissolved out of org. fertilizers more slowly than from inorg. compds. Urea was very injurious to roots when first applied to the soil. With most fertilizers beans could not be planted in the fertilizer band for 14 days after fertilizing without reduction of germination. Castor pomace, cottonseed meal, Milorgaite, fish scrap and dried blood greatly reduced the germination of seeds

planted in contact with the fertilizer band until after the fertilizer had been in the soil for 14 days. When first applied these 5 org. fertilizers were also very toxic to the roots. Animal tankage and garbage tankage did not inhibit root growth, but the animal tankage did reduce the germination of seeds placed in contact with it. No N fertilizer stimulated the root growth. Steamed bone meal did not reduce germination of seeds placed in contact with it, nor did it injure root growth. It had the slowest movement of P out of the fertilizer band of any P fertilizer. The superphosphate reduced germination of seeds when first applied, but after 2 days, there was no injury. The superphosphate had a decidedly stimulating influence on plant root growth, especially the fibrous roots. Ammoniated superphosphate and Ammo Phos A were injurious to germination of seeds planted in contact with the fertilizer until these fertilizers had been in the soil for 14 days. As soon as the toxic action of the sol. N in these compds had worn off (in a few days), there was a very stimulating effect on root growth. The carriers of K had no stimulating effect on root growth. They had a very rapid rate of soln and reduced the germination of seeds planted in contact with the fertilizer bands and also inhibited root growth until the concn of sol. salts had been sufficiently reduced by diffusion so that they no longer plasmolyzed the plant cells. After 2 days,  $K_2SO_4$  and KCl no longer injured germination of seeds or produced root injury. Kainite remained toxic for a longer time. Com. fertilizers showed germination and plant root injuries in accordance with the sep. actions of their ingredients. Fertilizers have a considerable perpendicular movement in the soil. The inorg. N compds. showed the greatest movement, org. N compds. next, and P compds. showed the least movement in the soil. Water-sol. K from the fertilizers was quickly changed to a water-insol. form in the soil. Several small applications of fertilizer are less injurious than a single large application. C. R. F.

Treating ordinary and acid superphosphates with ammoniacal solutions of ammonium nitrate L. Berlin, L. Gontskaya and A. Zasedateleva. *Mineral'noye Udobreniye Insektifungitsid* 1, No. 4, 21-37 (1935); cf. C. A. 29, 3094. —When ammoniacal  $NH_4NO_3$  was introduced into the superphosphates in batches, the following ratios of  $N:P_2O_5$  in the product were obtained: (a) Ordinary superphosphate from phosphorite and unconcentrated apatite,  $N:P_2O_5 = 0.5-1.2$ ; (b) ordinary superphosphate from floated apatite after storage for 20-30 days,  $N:P_2O_5 = 0.4-0.7$ ; (c) double superphosphate from Vyatka phosphorite,  $N:P_2O_5$  maximum 0.5; (d) double superphosphate from 35 and 40% apatite concentrates,  $N:P_2O_5$  up to 0.75. Continuous satn. in the lab yielded a sticky product. Commercial-scale expts were successful. A detailed description of the expts is presented. A. A. Bochtlingk

Microelements A. A. Khalizev and M. V. Katalumov. *Repts. Sci. Inst. Fertilizers and Insectofungicides, Leningrad, 3rd Intern. Congr. Soil Sci., Oxford 1935*, 51-65 (in German). —Pot expts with mustard in water and sand cultures with variations of Hellriegel's soln. of nutrient salts of single and double concn, with and without a special addn of microelements, viz., B, Mn, Zn, Cu, I and F, indicated in the latter case a greater yield of pods when N was supplied as  $NH_4$  salts, but this was due to traces of microelements from the impure chemicals which were still available in the physiologically acid soln. When the microelements were intentionally added, nitrate was the best source of N. Yields of oats, especially of grain, were greatest with the doubled concn of nutrients with a special addn of microelements, but in the absence of the latter, growth was best in the less concd. soln. B and Mn had the greatest effect in increasing yields. Field and pot expts indicated that the depressant effect of overliming podzols, a red earth and *Hypnum* peat, as well as an artificially acidified chernozem, is dependent upon deficiency in available B. When the latter was supplied in small amt, the same liming treatments increased yields. A similar expt with barley on the peat soil indicated deficiency in both B and Cu. On peat excessively limed, B

and Mn caused great increases in yields of oats, likewise with wheat upon an alkali soil treated with  $\text{CaSO}_4$ . Plants growing on soils deficient in B and Cu often accumulate great quantities of N and ash elements. Mustard is damaged by a light liming of sandy podzol deficient in B, flax does not grow well unless B is supplied, even when this soil is not limed, and similar observations were made on a degraded chernozem. Twelve references. C. J. S.

The effectiveness of organomineral fertilizers. Z. V. Logvinova and A. G. Ivanov. *Repts. Sci. Inst. Fertilizers and Insectofungicides, Leningrad, 3rd Intern. Congr. Soil Sci., Oxford 1935, 67-82* (in German).—"Ammonium humate" (I) was prep'd by treating peats with  $\text{NH}_3$ , which caused increases in N from 0.8-2.9 to 2.5-6.3%. Ammoniacal N increased from 1.3-6.3% to 28-50% of the total N. "Humamimophos" (II), ppt'd from  $\text{NH}_4\text{OH}$  exts. of the same peats by a  $\text{H}_3\text{PO}_4$  ext. of phosphate rock and ammoniated to neutrality, contained 8.4-44.1%  $\text{H}_2\text{O}$  and 5.0-10.0% N on the dry basis, of which 57-85% was in  $\text{NH}_3$ , with 7.6-16.1%  $\text{P}_2\text{O}_5$ . Pot exts in which these in combination with KCl, etc., were compared with  $(\text{NH}_4)_2\text{SO}_4$  and superphosphate for flax, hemp, barley and wheat indicated superior action of the humus preps., the N so supplied equaling that of  $(\text{NH}_4)_2\text{HPO}_4$  or  $\text{NH}_4\text{NO}_3$ . The products made from low-ash peat were higher in N and more effective. Some kinds of raw lignite had favorable effects in similar exts., others were of neg. value, but without exception the ammoniated products therefrom, similar to I and II, were superior to inorg. N and  $\text{P}_2\text{O}_5$  fertilizers, causing large increases in both yields and quality. The first crop of flax utilized 40-90%, of wheat 70-100%, of the ammoniacal N so supplied. Both I and II should be evaluated on the content of ammoniacal N; the  $\text{P}_2\text{O}_5$  of II is not inferior to that of  $(\text{NH}_4)_2\text{HPO}_4$ . The org. colloids of these materials are of special value on sandy soils. Twenty-three references. C. J. S.

The effects of fertilizers of various physiological character on Hungarian vine soil types. Dező D. Dencity and Gyula Ebsenpauger. *Magyar Ampelol. Évkönyv* 9, 240-7 (1935).—*Sinapis alba* in acid soils (pH 5.07-7.31) gave best yields on fertilization with Rhenania phosphate and either Chile or lime salt-peter or  $(\text{NH}_4)_2\text{SO}_4$ . In alk. soils (pH 7.65-8.13) Rhenania phosphate and superphosphate were best, combined with  $(\text{NH}_4)_2\text{SO}_4$ .

S. S. de Finily. An investigation into the influence of ammonium sulfate on stubble-sown oat crops in Victoria. D. Wark. *Proc. Roy. Soc. Victoria* 47, 1, 78-95 (1934).—The final yields of hay and grain were markedly increased by the application of  $(\text{NH}_4)_2\text{SO}_4$  at seeding. Marked variations occurred in the  $\text{NH}_3$  content of the soil throughout the growing period of the crop. The nitrate contents of the soil increased from June to August on the  $(\text{NH}_4)_2\text{SO}_4$  plots. The percentage N content of the crops in Oct was not affected by the different treatments, nor was there any difference in the N contents of the grain or straw at harvest. The total N removed by the crop on the  $(\text{NH}_4)_2\text{SO}_4$  plots was in all cases more than that removed on the corresponding no-N plots, but the difference in amts. of N removed by the crops was less than 1/2 the difference in the amts. of N applied in the corresponding fertilizer dressings.

Leopold Schellán. The place of nitrogen fertilizers in a pasture-fertilization program. D. R. Dodd. *J. Am. Soc. Agron.* 27, 533-62 (1933).—A review of the literature on the use of N fertilizers on pasture land under Ohio conditions is given, and the major conclusions are summarized. J. R. Adams.

Fertilization experiments with various nitrogen salts. László Salacz. *Magyar Ampelol. Évkönyv* 9, 203-22 (1935).—Expts. were made on fertilization of grapes with lime nitrogen, or Leuna salt-peter and Nitrophoska. Fertilization increased yield in each case. On heavy soils the sugar content of the must obtained generally increased; total acids almost always diminished. On sandy soils the yield was increased, the sugar content of the must decreased and acidity became higher.

S. S. de Finily. Fertilizer experiments with lime-nitrogen. László

Salacz. *Magyar Ampelol. Évkönyv* 9, 190-203 (1935).—Application of lime nitrogen generally increased the yields of grapes. The sugar content of the must from plots where a yield increase was observed somewhat diminished, the content in acids increased. S. S. de Finily.

Experiments on vines with potassium fertilizers. László Salacz. *Magyar Ampelol. Évkönyv* 9, 223-39 (1935).—The acid content of musts was decreased by fertilization of the vine, the sugar content sometimes decreased, other times increased. Soils poor in K gave good results on application of double doses of K salts, other soils showed better yield with single doses. S. S. de Finily.

Comparison of the effect of potassium fertilizers on grain cultures. V. N. Prokoshchev and E. M. Ustyuzhanina. *Mineral'nue Udobreniya Insektofungitsidus* 1, No. 5, 59-69 (1935).—Expts in great variety were made with sylvinit, carnallite (of various degrees of concn), KCl and mixts of these. The results depended on the soil, weather conditions and the type of plants and their rotation. A. A. Boethlingk.

Influence of aluminum sulfate on *Cyperus malaccensis*. K. Yamamoto. *J. Sci. Soil and Manure* (Japan) 9, 367-76 (1935).—In the cultivation of *Cyperus malaccensis* (a substance used in making Japanese mats) application of  $\text{Al}_2(\text{SO}_4)_3$  to the soil has a favorable effect upon growth, yield and quality of fiber. This effect has been considered due to the increase of soil acidity. A similar effect was obtained in another case by applying  $\text{H}_2\text{SO}_4$  instead of  $\text{Al}_2(\text{SO}_4)_3$ . In the water culture of the plant, however, the best growth was observed at pH 6 and the yields decreased gradually with decrease of pH, even though the plant grew normally at pH 4. Consequently, it may be concluded that the effect of acidity is indirect. The addn. of acid substance to the soils of paddy field caused an increase of  $\text{NH}_3$  in the soils. The N content in the crops from the plot with acid substance was not always high in spite of the fact that the color of the stems was always green. Y. Kamoshita.

Calcium silicate slags. Properties of quenched and unquenched slags and effects of their admixtures with phosphatic fertilizers. W. H. MacIntire, L. J. Hardin and F. D. Oldham. *Ind. Eng. Chem.* 28, 48-57 (1936).—Results of microscopical and chem. examn. of slags from phosphate-reduction furnaces are given. Expts with dry admixts. of slag and triple superphosphate, with and without the addn. of  $\text{NH}_3$  phosphates and  $\text{K}_2\text{SO}_4$ , showed no formation of citrate-insol.  $\text{P}_2\text{O}_5$  when measured by the citrate extn. method at the end of a 35-day period. There was no loss of  $\text{NH}_3$  from the dry mixt. and no appreciable fixation of  $\text{K}_2\text{O}$  in the "complete" mixt. Similar expts. with admixts. of moistened slag to triple superphosphate caused the complete disappearance of  $\text{H}_2\text{O}$ -sol.  $\text{P}_2\text{O}_5$  and the formation of considerable citrate-insol.  $\text{P}_2\text{O}_5$ . With the moistened mixts. contg.  $\text{NH}_3$  phosphates there was a partial decline in the  $\text{H}_2\text{O}$ -sol.  $\text{P}_2\text{O}_5$ , a negligible increase of citrate-insol.  $\text{P}_2\text{O}_5$ , some loss of  $\text{NH}_3$  and a definite fixation of  $\text{K}_2\text{O}$ . The influence of the slags on the solvent power and pH values of citrate sola, the factors of F content and silica value, and the inapplicability of the "official" method for citrate extn. of such mixts. are discussed. The admixts. are proposed for dilg. triple superphosphates to "standard" concn near the point of usage to improve drillability and to eliminate acidity and F toxicity. John O. Hardesty.

The influence of chloride upon the growth of tomatoes. A. V. Yur'ev. *Konservnaya Prom.* 1935, No. 5, 20-4.—Lab. and field observations establish a concn limit of NaCl of 0.2% by wt. of dry soil for cultivating tomatoes in a soil contg. little other salts. A concn of 0.03% NaCl in the same conditions stimulates their growth.

B. V. Shvartzberg. Influence of sodium salts on the crop of sugar beets in the presence of nitrogen derived from various sources. P. N. Ushkina. *Mineral'nue Udobreniya Insektofungitsidus* 1, No. 5, 98 (1935).—NaCl and  $\text{Na}_2\text{SO}_4$  had a favorable effect on the crop and the yield of sugar only in containers with nitrogen and phosphate or a complete fertilizer. The expts. are described. A. A. Boethlingk.

The effect of the organic and inorganic fertilizers prepared from peat Z V Logvinova *Mineral nuse Udobreniya Insklofungsudis* 1, No. 5, 69-81(1935) — Fertilizers prep'd by treatment of peat or coal with  $\text{NH}_3$  and  $\text{H}_2\text{PO}_4$  are better than  $(\text{NH}_4)_2\text{SO}_4$  and are better than, or equal to, humic ammonium phosphates and  $\text{NH}_4\text{NO}_3$  because of the presence of the org. substance in peat. They are higher in the total ammonia N when peat low in ash is used. Field expts are described. Twenty-three references.

Flax investigations J G Hutton and Clarence Strickland S Dak Agr Expt Sta, *Ann Rept* 1934, 20 (1934) — Applications of P trebled the yield of flax seed, the yield of oil was proportionately increased. The P also increased the wt of individual seeds. Both the vegetative and seed-producing stages were advanced 1-8 days where P was used. There was no correlation between oil content and I value of the oil.

Concentrations of mineral nutrients in the corn plant as affected by fertilizer treatment J D Sayre and V H Morris Ohio Agr Expt Sta, *Bull* No 548 (53rd Ann Rept) 25-61(1935), cf. C A 29, 80611 — From corn plants grown on soils with various fertilizer treatments the sap was expressed and examd for nitrate,  $\text{NH}_4$ , amino N, sol org N, inorg P and K. Inorg P showed no consistent increase in the stem tissue with fertilization. On the other hand, inorg N showed definite increases with added N fertilizers.

Spraying experiments on fruit trees B Husz *Bull Ec Hong Hort* 1, 8-22(1935), *Rev Appld Mycol* 14, 773 — Almost perfect control of red spot on plum varieties was given by 4 applications of 0.5 to 1% Bordeaux mixt., lime sulfur and a combination of lime sulfur and Ca arsenate. The best control of apple diseases (chiefly *Podosphaera leucotricha*) and pests was given by a combination of 1% lime sulfur with Pb arsenate paste (8.75% Pb) or for later applications with Ca arsenate (0.25% Arzola).

Control of the bean weevil and the cowpea weevil S Marcovitch J Econ Entomol 28, 796-7(1935) — The bean weevil (*Acanthoscelides (Mylabris) oblectus*) and the southern cowpea weevil (*Callosobruchus (Mylabris) maculatus*) were studied with reference to the effects of dust upon them. The latter species is the effects resistant, 1 part  $\text{Ca}(\text{OH})_2$  to 1 part cowpeas or 1 part Na-commodity. For protection from A. oblectus 1 part  $\text{Ca}(\text{OH})_2$  to 50 parts beans and 1 part  $\text{Na}_2\text{SiF}_6$  to 1000 parts beans are required.

Calcium arsenate as a control measure for the tobacco flea beetle and hornworm W W Stanley and S Marcovitch J Econ Entomol 28, 797-801(1935) —  $\text{PbH}_2\text{AsO}_4$ , Ca arsenate,  $\text{Na}_2\text{AlF}_6$  (natural and synthetic) and  $\text{BaSiF}_6$  did not seriously injure tobacco foliage. Paris green, today are vastly superior to those of 15 yrs ago. The fully controlled with dust applications of  $\text{BaSiF}_6$  or Ca arsenate, slightly better with Pb arsenate dust than with  $\text{PbH}_2\text{AsO}_4$  and  $\text{Na}_2\text{AlF}_6$  (synthetic) applied as sprays were about as effective as when used as dusts. Flea beetles were controlled with equal effectiveness by sprays of these 3 compds. Derris and pyrethrum were not satisfactory for the control of these insects. Ca arsenate was the most practical insecticide for these insects on tobacco.

Control of the pecan nut case bearer in the Southeast (United States) G P Mozzette J Econ Entomol 28, 791-4(1935), cf. C A 29, 59763 — *Acrobasis caryae* was controlled with sprays of summer petroleum oil emulsion nicotine sulfate (contg 40% nicotine) mixt. with fish oil nicotine sulfate mixt., and with Bordeaux-hat a viscosity (Saybolt) of 61-63 secs, unsulfonated residue 92.8-96.8%. The fish oil showed a sp gr of 0.929, free fat acid (calcd as oleic) 8.3, I value (Hanus)

145, sapon value 195. Petroleum oil sprays with time were less effective, and pyrethrum and det in sprays were valueless.

"Brown heart," a new disease of swede, control T. Whitehead *Welsh J Agr* 11, 235-6(1935) — The disease is characterized by a brown staining, flesh in more or less concentric zones at some from the rind of the swedes. Slices of the disease are translucent and the whole appearance is rem of "water core" in apples. A considerable degree of the disease was effected by the application of to the soil at the rate of 10 lb /acre; no further in ment was obtained by doubling the rate of appli.

The zinc sulfate treatment for mottle leaf of trees in the Sundays River Valley I Matthews *Grouer* 1935, No 41, 30-2 — The disease was cor by spraying the trees in the autumn or early spring mixt of  $\text{ZnSO}_4$  (contg 23-25% Zn) 10, hydrated and spreader 0.5 lb per 100 gal  $\text{H}_2\text{O}$ . The bet effect of the treatment was evident in 3-4 months.

The results of experiments carried out at the Enns factory to combat the cercosporiosis of the be (*Cercospora beticola* Sacc) in 1934 H Redlich *inst belge amelior betterave* 3, 275-93(1935); *Rev A Mycol* 14, 813 — Almost without exception the y foliage, roots, weight and sugar content of beets tr with Bordeaux mixt or  $\text{CuSO}_4$  dust against *Cercospora beticola* were higher than for untreated material. Bordeaux mixt proved the more reliable in most tests. A concn was inadequate but a 1% concn was adequate economical, whereas if a 2% concentrate were necessary cost would be too high.

The green peach aphid—further observations Ken M Ward J Dept Agr Victoria 33, 500-6(1935), cf. C A 28, 55801 — A considerable reduction in the incidence of the insect (*Myzus persicae* Sulz.) was obta by spraying the trees in winter with either 2-3% tar dilute, 5% red oil or 10% lime-S. The best results were tained with 3% tar distillate.

Economical amounts of nitrate of soda to apply in greenhouse for the growth of tomatoes Basil E Gil and Frederick R Pember. R I Agr Expt Sta, *E No* 252, 3 14(1935) — For greenhouse tomatoes the of  $\frac{1}{2}$  lb of  $\text{NaNO}_3$  and 7 lb of org matter per  $\frac{1}{2}$  sq ft gave optimum growth and yields. Spring crops of tomatoes grown on the same soil required approx dou these quantities. Cow manure was an excellent sou of org matter for tomatoes. During the short days winter the nitrate content of the tomato plant is high cause of lack of utilization of nitrate for elaborating carl hydrates. Under these conditions only small amts nitrate are required in the soil for optimal growth (C A 25, 2227).

Effects of arsenical sprays on grapefruit, orange tangenness, Temple oranges, limes and lemons Longfield-Smith Fla Dept Agr, Chem Div, *Rej Winter Haven Chem Lab* 1933-4, 3 70(1935); cf. C A 30, 2224 — In all cases where As sprays had been applie As was found in the rind, rag and juice. If 1 portion of citrus tree was sprayed, the fruit from the unsprayed portion contained only traces of As. There was only very slight translocation of As by the vascular system of the tree. Exptl sprays were made with As concn of 1.5 and 3 lb of Pb arsenate per 100 gal of water. Although the amt. of As present in the juices was negligible from health viewpoint, it reduced acidity from 5-20%, de creased sol solids in some cases, and greatly hastened maturity. Growers were enabled to evade the green fruit law and fulfil the maturity standards of acid sugar ratio, although the fruits were of inferior quality, lacked juiciness and acid, and were insipid. Oranges were adversely affected to a much greater extent than grapefruit. Complete data are presented in 38 tables.

Effects of spray applications of triple phosphate of lime to grapefruit and orange trees L Longfield-Smith Fla Dept. Agr., Chem. Div., *Rept. Winter Haven Chem*



*Lah.* 1933 4, 71-84(1935).—Where the strength of the phosphate soln. used was 20 lb. per 100 gal. and only 1 application in June was made, the acidity was reduced 10% in grapefruit. Where the phosphate concn. was 15 lb. per 100 gal. the acidity decrease was 4.1%. There was no decrease in acidity when the phosphate applications were 5 and 10 lb. per 100 gal., resp. For the 2 largest applications there was a very slight but not consistent increase in sugar content. This is the opposite effect to that of As spray treatment. On oranges, only the 20 lb. per 100 gal. application gave a small decrease in acidity of 2.0%. As in the grapefruit, the phosphate sprays increased the sugar content very slightly. C. R. Fellers

**Evaluation of vine sprays.** Sándor Ránky and Ágnes N. Osztrószky. *Magyar Ampelol Fisköny* 9, 135-45 (1935).—Adhesive factor was detd. according to Paes and Staehelin. For Bordeaux soln. contg. excess lime the adhesive factor was about twice that of a neutral soln. The suspensibility of agents was detd. by sedimentation in cylinders and the data obtained were compared with that of a 1% Bordeaux soln. Sedimentation velocity of agents should always be detd. in evaluating them. S. S. de Finlay

**Disinfection of vine shoots.** Bertalan Sztehló. *Magyar Ampelol Fisköny* 9, 65-6(1935).—Vine shoots were treated with 0.4 to 1.2% solns. of formaldehyde for 2 to 10 min.; 2.5-7.0% of the buds of shoots were destroyed. Thus treatment with CuSO<sub>4</sub> soln. is both cheaper and more effective. A 5-min. treatment in a 1% CuSO<sub>4</sub> soln. or fumigation with 20% formaldehyde is proposed. S. S. de Finlay

**Petroleum-kaolin emulsions in viticulture.** Sándor Ránky. *Magyar Ampelol Fisköny* 9, 119-51(1935).—Field expts. showed that petroleum-kaolin emulsions are suitable only for spraying small areas, since the dild. emulsion should be sprayed immediately after prepn. Where storage of the spray is necessary, soap emulsions are better. S. S. de Finlay

**Insecticidal effects of nicotine-containing vine sprays.** Sándor Ránky and Ágnes Németh Osztrószky. *Magyar Ampelol Fisköny* 9, 116-9(1935).—Expts. with various nicotine-contg. sprays proved that they act as tracheal and skin-poisoning agents. S. S. de Finlay

**Recent experiments in flax disinfection.** A. I. Iabel Nachr. *Schidl. Bekimpf., Leverkusen* 10, 70-3(1935). *Rev. Applied Mycol.* 14, 763.—Promising results in the control of wilt (*Fusarium lini*) and anthracnose (*Colletotrichum lini*) on flax seed have been obtained by dusting the seed with Cresan which reduced the no. of diseased plants from 191 out of 600 to 11 out of 500 and also increased germination 13%. Oden E. Sheppard

**Effect on transpiration of varying the copper-lime ratio in Bordeaux mixtures.** J. D. Wilson and H. A. Rannels. *Ohio Agr. Expt. Sta., Monthly Bull.* No. 177, 206-9 (1935); cf. C. A. 30, 212.—When *Coleus* plants were treated with Bordeaux mixts. in which the Cu and lime ratios were varied by 3 lb. each in 50 gal. of water, the max. effect on transpiration rate was caused by the 3 to 50 formula. As the CuSO<sub>4</sub> increased beyond 3 lb. in 50 gal. of water, the effect on transpiration decreased. This was also true to a lesser extent when the lime content was increased beyond 5 lb. Increases in the CuSO<sub>4</sub> content of Bordeaux mixt. which was applied to plants increased their water requirement slightly. Most plants were injured by the application of a 15 to 50 formula and some by a 12 to 50. Increases in the amt. of Ca(OH)<sub>2</sub> used in the spray formula resulted in a decrease in growth and an increase in water requirement. C. R. Fellers

**New observations on the action of boron in beet heart rot.** L. I. Fox and H. Burg. *vin. Compt. rend. acad. agr. France* 21, 979-82(1935). cf. C. A. 29, 2257.—The results confirm the value of H<sub>2</sub>BO<sub>3</sub> or Na borate as a control against beet heart rot. H<sub>2</sub>BO<sub>3</sub> should be used at the rate of 8 to 10 kg. per hectare for a first treatment and Na borate at the rate of 11-16 kg. The best results are obtained by intimately mixing with the fertilizer before spreading. A certain delayed protective action was observed. At the rates recommended there appears to be

no danger of toxicity caused by the accumulation of B in the soil. J. R. Adams

**Improving Bordeaux solution.** Sándor Ránky and Ágnes N. Osztrószky. *Magyar Ampelol Fisköny* 9, 131-5 (1935).—The sedimentation was slower, the adhesive power greater and the fungicidal effect stronger when the CuSO<sub>4</sub> soln. was poured into the dild. lime milk and not the latter poured into the CuSO<sub>4</sub> soln. as originally proposed by Millardet. S. S. de Finlay

**Pyrethrin, rotenone and nicotine.** D. Mann. *Sesfensieder-Zig* 62, 892-3, 913-5, 914-5, 954, 973-5, 991-3, 1010-17, 1015-7(1935), cf. C. A. 29, 5080.—A review of methods of prepn., testing and using rotenone and derris-root exts. Over 100 references. J. W. Perry

**A new source of sulfur for combating insects and agricultural diseases.** L. A. Pokrovskii. *Mineral'nue Udobreniya Insektofungusidus* 1, No. 5, 89-91(1935).—

An insecto-fungicidal prepn. was obtained by boiling S tallings (containing 30-55% S) obtained in the flotation of S ore. These tallings were boiled with Ca(OH)<sub>2</sub> and water to the appearance of a black color. The undissolved S was used in the following batches. The same tallings can be used for fumigating by supplying additional heat. A. A. Boethlingk

**"Ultra" sulfur.** V. P. Munchev. *Mineral'nue Udobreniya Insektofungusidus* 1, No. 4, 60-61(1935).—

"Ultra" sulfur was prepd. by spraying a heat-treated clay (passing a 200-mesh sieve) with liquid S (mixed with small amts. of I and  $\beta$ -naphthol, to lower its viscosity) at 140°, to give a product contg. 12.13% S. The reports obtained from field operators using the above prepn. as a fungicide were divided. A detailed description of the process is presented. A. A. Boethlingk

**A new method of producing Paris green.** Lotorev, P. M. and Artamannov, I. *gorov. Tekhnika* 1935, No. 50.—CuSO<sub>4</sub> soln. is mixed with AcOH, and metarsenite is added to the mixt. The product is filtered and the ppt. of Paris green is washed carefully. B. V. Shvartberg

**Comparative toxicity of anabasine and nicotine sulfates to insects.** Joseph M. Ginsburg, John B. Schmitt and Philip Granett. *J. Agr. Research* 51, 319-51(1935).—Anabasine sulfate equals or exceeds nicotine sulfate in toxicity to *Aphis rumicis*, *A. pomi* and *Macrosiphum rosarum* and is decidedly more toxic to *Rhopalosiphum rufumaculata* and *Macrosiphum rosae*. Anabasine sulfate is decidedly less toxic to grasshoppers and to silk-moth larvae as a stomach poison than is nicotine sulfate. W. H. Rows

**Controlling cattle grubs on dairy cows.** H. A. Herman and Geo. D. Jones. *Mo. Agr. Expt. Sta. Bull.* No. 340 (*Ann. Rept.* 1933), 45(1933).—Ox warbles were thoroughly controlled on cattle by the use of prepns. of derris. By working in the soln., contg. 0.5% derris plus a fatty base, into the warbles, a kill of 90-100% was obtained. C. R. Fellers

**Plot tests with sodium arsenite and sodium chlorate as soil sterilants in California.** A. S. Crafts. *Calif. Dept. Agr., Monthly Bull.* 24, 217-50(1935); cf. C. A. 30, 804.—From 2 to 8 lb. of Na arsenite per sq. rd. is an effective and practical chemical for controlling weeds in waste areas such as roadways and banks of irrigation ditches. This chemical is best applied as a spray to the top soil or vegetation. NaClO<sub>3</sub> is also effective against annuals for more than 1 year in heavy alluvial soils where rainfall is light. It is useful against deep-rooted perennials. A combination of 4 lb. of As<sub>2</sub>O<sub>3</sub> and 1 lb. of NaOH with 3 lb. of water is another effective weed killer which can be used as a dust. C. R. Fellers

**Factors influencing the effectiveness of sodium chlorate as a herbicide.** A. S. Crafts. *Hilgardia* 9, 437-57 (1935).—Chlorates may kill deep-rooted perennial plants either by rapid absorption and translocation within the plant or by absorption from the soil by the roots. Application of the same amt. of NaClO<sub>3</sub> to the soil killed morning glory, *Convolvulus arvensis*, and Russian knapweed, *Centaurea repens*, but merely injured hoary cress, *Lepidium draba*. Three years' work shows that proper

vertical distribution of  $\text{NaClO}_3$  within the soil by leaching of water rains is essential to success with the soil treatment method. A similar vertical distribution may be accomplished with irrigation water. Application of the  $\text{NaClO}_3$  to both soil and plant is more efficient than application to only the soil or to the plant. In many cases roots were destroyed 3 ft below the soil surface. A single spray was usually as effective as 3 light sprays in weed control. Continual leaching with irrigation water proved the best means of ridding the land of residual  $\text{NaClO}_3$ . Sterile areas leached with 36 in. of water were returned to cultivation within a season by this practice. Toxicity of sodium arsenite and sodium chlorate in four California soils. *Ibid.* 461-98.—Trivalent As, on a cost basis, was the most feasible chemical for soil sterilization. Toxicity of As was greatest in the Fresno sandy loam and lowest in the Yolo clay loam. Loss of toxicity with time and cropping was greatest in the Yolo soil and lowest in the Fresno sandy loam. Yolo clay had the strongest fixing power for As and held 4 times as much as the Fresno soil. The Stockton and Columbia soils held about twice as much As as the Fresno soil. The amt. of As held by all 4 soils was greater with increased concn. of the solns. applied. Even after leaching the Yolo soil with 160 surface cm. of water, the upper 4 in. of soil was sterile and the chemical had not descended below the 16-in. level. Similar leaching removed all the As from the Fresno soil. Soil application of  $\text{NaClO}_3$  so as to secure max. absorption by the roots gave optimum results with this chemical. Toxicity of  $\text{NaClO}_3$  was highest in the Stockton adobe clay and lowest in the Yolo clay loam. It was only slightly higher in the Columbia fine sandy loam and intermediate in Fresno sandy loam. The fixing power of the 4 soils was much less for chlorate than for Na arsenite. In the Yolo clay loam the chemical was fixed from a slowly moving soln. Rate of moistening had no effect in the Fresno sandy loam. The Stockton soil had little fixing power for  $\text{NaClO}_3$ . Chlorate toxicity was greater in soil columns previously moistened with distd. water than in those moistened only with the chlorate solns. Leaching with distd. water moved the chlorate downward in the soil columns. Forty surface cm. of water was sufficient to remove the chlorate from the Yolo and Columbia soils.  $\text{NaClO}_3$  was present in the top 20 in. of the soil from sterile areas in a field sprayed 3 yrs. previously to kill morning glory. A leaching with 36 surface in. of water removed the chemical sufficiently so that alfalfa made a good growth. C. R. Fellers

Analysis for fluoride—application to detn. of spray residue on food products (Hoskins, Ferris). 7. Oil of Tennessee red cedar [use in insecticides] (Huddle). 7. Decompos. of raw phosphates with  $\text{H}_2\text{SO}_4$  (Lehrecke). 18 App. for cooling and aerating ammoniated phosphate fertilizer (U. S. pat. 2,028,413). 1.

Reconditioning soil after killing weeds. I. G. Farber and A. G. (Udo Ehrhardt, inventor). Ger. 622,201, Nov. 25, 1935 (Cl. 42). 5. Soil to which a chlorate has been applied as a weed-killer is reconditioned by treatment with a substance which decomps. chlorates. The substance may be (a) a simple reducing agent, e. g., a ferrous salt or a hyposulfite, or (b) a reducing agent convertible into a fertilizer, e. g., a nitrite or hypophosphite, or (c) a substance which accelerates catalytically the decompos. of unstable chlorates, e. g.,  $\text{MgCl}_2$ .

Fertilizers. John A. Heskett. Brit. 435,763, Sept. 23, 1935. See Australia 16,704/34 (C. A. 29, 2654).

Fertilizers. Société d'études pour la fabrication et l'emploi des engrais chimiques. Ger. 619,834, Oct. 8, 1935 (Cl. 16.2).  $\text{CaHPO}_4$ ,  $\text{CaCO}_3$  and  $\text{NH}_4\text{Cl}$  are formed

by treating natural phosphates with dil.  $\text{HCl}$ , pptg. the  $\text{CaHPO}_4$  by  $\text{Ca}(\text{OH})_2$ , sepg. this and treating the  $\text{CaCl}_2$  liquor with  $\text{NH}_3$  and  $\text{CO}_2$  or  $(\text{NH}_4)_2\text{CO}_3$ . The  $\text{HCl}$  soln. used contains about 65 g.  $\text{HCl}$  and 255 g.  $\text{CaCl}_2$  per l., the  $\text{CaCl}_2$  being provided by adding sufficient of the filtered  $\text{CaCl}_2$  liquor. Examples are given. Cf. C. A. 30, 1172.

Ammoniated peat fertilizer. Royall O. E. Davis and Walter Scholl (dedicated to the free use of the Public). U. S. 2,027,766, Jan. 14. Anhyd.  $\text{NH}_3$  is brought into contact with peat in a closed autoclave, and the mixt. is heated to 180-300° for 8 hrs., the temp. is then allowed to drop to 50° and the uncombined  $\text{NH}_3$  is removed from the mixt.

Superphosphate fertilizer. Beverly Ober and Edward H. Wight (to Oberphos Co.). U. S. reissue 19,825, Jan. 14. A reissue of 1,947,138 (C. A. 28, 2456).

Anticryptogamic products. Compagnie de produits chimiques et électrometallurgiques Alsas, Froges et Camargue. Fr. 788,631, Oct. 14, 1935. Insol. chloro-cupric compds. of the formula  $\text{CuCl}_2 \cdot 3\text{CuO} \cdot n\text{H}_2\text{O}$  or  $\text{CaCl}_2 \cdot 3\text{CuO} \cdot n\text{H}_2\text{O}$  ( $n$  is about 4) concd. or diltd. by inert materials such as hydrated  $\text{CaSO}_4$  are made by the simultaneous reaction, in aq. soln. or suspension and at ordinary or raised temp., of 1 or more sol. Cu salts other than the chloride, with 1 or more alkali or alk. earth metal chlorides or any other simple or complex sol. chloride, the aq. solns. of which have a reaction less acid than solns. of  $\text{CuCl}_2$ , and with a base such as  $\text{CaO}$  or  $\text{MgO}$  or the corresponding carbonates. The ppt. is filtered, washed and dried. Cf. C. A. 30, 806.

Insecticidal sprays. Irwin Stone. U. S. 2,028,109, Jan. 14. To facilitate the removal of spray residues from fruits, there is incorporated in the spray compn. used a water-insol. material such as  $\text{ZnCO}_3$  which is inert to the active spray material but which reacts with acid wash solns. to form a gas which dislodges the spray residue film.

Destroying insects. Arthur Balzars. Fr. 787,963, Oct. 2, 1935. An atm. contg. chlorinated hydrocarbons, e. g.,  $\text{C}_2\text{H}_2\text{Cl}_4$  80 and  $\text{C}_2\text{H}_3\text{Cl}_3$  20%, is used.

Killing insects, etc., on plant products such as seeds of plants. Thomas J. Headlee (to Endowment Foundation). U. S. 2,027,976, Jan. 14. Plant products or plants are passed along on a conveyor while subjected to an electrostatic field between electrodes by a source of energy having a frequency preferably not less than 1,000,000 cycles and a voltage preferably of about 4000 v. per linear in. between the electrodes.

Seed disinfectants (inner anhydrides of mercurized phenols). Fritz Wolf (to Schering-Kahlbaum A.-G.). U. S. 2,021,277, Nov. 19. Inner anhydrides of mer-

curized phenols of the general formula:  $\text{C}_6\text{H}_4\text{X} \cdot \text{HgO}$ , where X represents either H or any substituted or unsubstituted alkyl, aryl, aralkyl or alicyclic radical, or any other substituent, such as halogen, the nitro, hydroxy and like groups, are good wet as well as dry disinfectants, for seeds. For their manuf., phenols which have been mercurized by treatment with mercuric salts are dissolved in  $\text{NaOH}$  soln., whereupon the alk. soln. is treated with  $\text{CO}_2$ , which ppts. the anhydrides; or the molten phenols are treated at a low temp. with mercuric oxide until the latter has been dissolved. The inner anhydrides obtained in this manner are sol. in phenols without cleavage of the anhydride bond, in contrast to their soln. in  $\text{NaOH}$  whereby the anhydride bond is changed and split open. The phenol acts as solvent, renders the inner anhydrides water-sol. and allows the application of these compds. as wet and as dry disinfectants. Several examples are given, including the production and use of the inner anhydride of mercurized 1,3,5-xyleneol and some similar compds.

# 16—THE FERMENTATION INDUSTRIES

C. N. FREY

The action of charcoal on alcoholic fermentation. A Reinhard and V. Obrastova. *Bell. soc. intern. microbiol.*, *Ses. ital.* 7, 331-5 (1935) (in French).—Powd. wood charcoal increases the alc. fermentation of the solns of sucrose and levulose, even if inactivated by heat for 10 min. at 50°, and stimulates the multiplication of saccharomycetes. G. A. Bravo

Boiling points in the distillation of alcoholic liquors. E. Jacobson. *Destillatør u. Lakorfabr.* 48, 605 (1935).—In distg. a mixt. of equal parts of Et and Am alcs.—The percentage of each is given for 7 fractions collected between 80° and 132°. K. Becker

The power alcohol problem and the sugar beet. O. Munerati. *Ird. soccar. ital.* 23, 488-96 (1935)

Alcohol losses during storage of bottled distilled liquors. C. Luckow. *Wein und Rebe* 17, 165-74 (1935).—When brandies and cordials were kept 6 months at room temp. in unopened corked bottles, no appreciable loss of alc. was found. The alc. losses of half-empty bottles kept 6 months varied from negligible to 2.05% by vol., depending upon the nature of closure and temp. of storage. K. Becker

Isolation and possible intermediary role of formaldehyde in the propionic acid fermentation. H. G. Wood and C. H. Werkman. *J. Bact.* 30, 652 (1935). J. T. M.

Honey vinegar. Fredereck W. Fabian. *Mach. Agr. Expt. Sta., Extension Bull.* No. 149, 3-14 (1935).—Waste or low grade honeys can be easily and profitably converted to vinegar. A satisfactory formula consists of extd. honey 40-45 lb., water 30 gal.,  $K_2C_2O_4$  2 oz. and  $(NH_4)_2PO_4$  2 oz. The distd. vinegar stock should contain approx. 15% sugar. The soln. is sterilized by boiling for 10 min., cooled, inoculated with yeast starter and allowed to ferment until the sugar has all been converted into alc. The soln. is then inoculated with *Acetobacter* which changes the alc. into AcOH. The optimum temp. for the fermentation is 24-28°. After the fermentation has been started the film of *Acetobacter* at the surface should not be disturbed. C. R. Fellers

Report on malt analysis during the months of March and April 1935. F. Ancker. *Wochschr. Brau.* 52, 166-7 (1935).—A summary of 236 barley malt and 9 wheat malt samples. S. Jozsa

Malting and mashing. Hugo W. Rohde. *Cereal Chem.* 12, 610-20 (1935).—A review. L. H. Bailey

Barley and malt studies. I. Developing new varieties of barley for malting and their properties. James G. Dickson, H. L. Shands, Allan D. Dickson and B. A. Burlhart. *Cereal Chem.* 12, 596-609 (1935).—The 6-rowed types of barley have predominated in the U. S. throughout the period of barley culture. Exptl. malting equipment is available at several locations for the survey of the malting quality of the barley varieties and the testing of new selections. The equipment at Madison, Wisconsin, is described. The most suitable procedure for the comparative malting of the barley samples is outlined. The development of new malting varieties of barley is in progress. L. H. Bailey

The determination of moisture in barley malt. A comparison of methods and apparatus for this purpose. D. A. Coleman and S. R. Sinder. *Cereal Chem.* 12, 621-45 (1935).—The vacuum-desiccator method, i. e., drying of the sample over anhyd.  $P_2O_5$  in a desiccator maintained at a pressure of 10 mm. of Hg or less until a const. loss in wt. has been reached, has been selected as the standard or referee method for making moisture detns. on malt because of the absence of caramelization or decomposition phenomena which commonly accompany the other methods used for moisture-dets. purposes. The 130° air-oven method gave results 0.63% higher than the standard method. A vacuum oven operated at 25 mm. pressure at 100° for 5 hrs. gave results 0.25% higher than

those of the standard method. The Carter-Simon method results were 0.5% higher than those of the standard method. The vacuum oven operated at 25 mm. pressure but at 70° for 18 hrs. produced results that were 0.1% lower than those of the standard method. Malt Analysis Standardization Committee method, i. e., heating for 3 hrs. at a temp. between 103° and 104° gave results practically equal to those by the standard method, being only 0.04% lower. Results by the water-oven method were 0.23% lower than those by the standard method. L. H. B.

Methods of wine analysis. C. H. McCharles and G. A. Pitman. *Ird. Eng. Chem., Anal. Ed.* 8, 55-6 (1936).—Methods of making wine analysis are given. The necessity of accurate sampling is pointed out. The use of the ebullioscope, pycnometer and hydrometer in detn. of alc. is discussed. Volatile acidity and total acidity detns. are described. Sugars can be detd. by the Lane and Eynon (cf. C. A. 22, 829), Munson and Walker, and Shaffer and Hartmann (cf. C. A. 15, 1327) methods, the latter a volumetric iodometric method. Analytical methods are given for metals,  $SO_2$  and tannin. C. N. Frey

Analyses of Hungarian and foreign natural wines and vermouths. Ottó Wolf. *Magyar Ámpel. Értékes.* 9, 458-64 (1935).—Analyses are given of 112 wines. Natural Hungarian sweet wines contained 7.20-17.74% alc. by vol., 0.58-1.32% total acids, 2.68-40.50 g. per 100 cc. ext., traces to 35.58 g. reducing sugars, 0.14-0.56% ash, 0.040-0.135% phosphoric acid, 0.95-2.40% glycerol, 17.2-57.2 ash alkali. The ratio alcohol glycerol ranged from 100:8.6 to 100:34.5. S. S. de Fmily

Examination of wine by means of a quartz lamp. István Szabó. *Magyar Ámpel. Értékes.* 9, 454-7 (1935).—A Hanau lamp was used to compare natural wines with wines from coned musts. The wine samples were (1) extd. with  $CHCl_3$  and the luminescence of the soln. was detd. by comparison with a glycerol soln., (2) decolorized according to Werder-Zach by means of carbo animalis and the filtrate examd. as previously. The published results seem to prove that on further modification this method will be available to show whether a wine has been prepd. by means of dried grapes or coned. must or has been produced of fresh grapes as natural wines. S. S. de Fmily

The determination of organic acids of wine. Ervin Wettstein. *Magyar Ámpel. Értékes.* 9, 429-39 (1935).—Treatment with  $BzCl$  at 153° for 2 hrs. forms insol. benzoate of malic and dibenzoate of tartaric acid, which can be sepd. Succinic acid is pptd. from the filtrate as a basic iron salt, filtered and weighed. AcOH can be detd. in the filtrate by titration after a prescribed purifying treatment. The benzoyl ester acids are then sepd. from benzoic acid after sapon with KOH, the potassium salts treated with  $H_2SO_4$ , the excess  $K_2SO_4$  is removed and the tartaric acid pptd. in an alc. medium as potassium tartrate. The filtrate contg. malic acid is worked up by a longer procedure and the acid detd. by titrating with 0.1 N alkali in presence of phenolphthalein. A table shows deviations from the theoretical contents ranging from 0.06 to 0.07%; thus the method is already more reliable than the present ones. S. S. de Fmily

Bromoacetic acid and normal bromine of wines. L. Chelle and G. Vitte. *Bull. soc. franc. Bordaux* 73, 179-87 (1935).—Wines suspected of contg.  $CH_2BrCO_2H$  as a preservative were assayed for their Br contents and the results were compared with results obtained with other wines with and without added KBr or  $CH_2BrCO_2H$ . Method: Evap. 10, 50 or 100 cc. on a water or sand bath and then heat in an oven. Calcine slowly over an alc. lamp and pulverize the carbonaceous mass. Calcine the powder in a Pt crucible at the edge of a muffle furnace. Wash the ash with hot distd. water and filter. Det. the amt. of Br by using the Deniges and Chelle reaction (cf. C. A. 7, 746) and comparing with known samples run at

the same time. Between 0.1 and 0.7 mg of Br occurred normally in the wines tested. S W G

Tartaric acid content and alkalinity of ash of Hungarian wines with special regard to the identification of wines of American vine types. Géza Requinyi and István Soós *Magyar Ampel Évkönyv* 9, 425-7 (1935) —Wine from vines of American types (Othello, Noah, Delaware, etc.) contained generally more tartaric acid and had more alk. ash than wines from other vines. Hungarian wine contained 0.071-0.665 g. per 100 cc. of tartaric acid and ash alkalinities ranged from 1.64 to 16.20 cc. N acid for 1 g. ash. S S de Finály

A report on fluorides in wine. Frederick F. Johnson and Louis Fischer. *Am J Pharm* 107, 612-14 (1935) —The ordinary volumetric determination of F, such as the titration with Th. nitrate or cerous nitrate, are not practical in the presence of so much organic matter. The following method is a modification of the proxonized Th. method (C A 28, 431<sup>12</sup>) which was designed chiefly for the determination of F in spray residues. Thirty-four g. of wine is alkalinized with NaOH and evaporated under steam until the alk. is removed. The residue is washed into a 270-cc. Claisen flask previously charged with a large no. of glass beads and 15 g. of NH<sub>4</sub> persulfate. Fifteen cc. of concd. H<sub>2</sub>SO<sub>4</sub> is added and an ordinary Willard and Winter distillation (C A 27, 181) is performed; the distillate is collected in 20 cc. of said K<sub>2</sub>MnO<sub>4</sub> in a 200-cc. volumetric flask. If any sulfate distills over, the operation must be repeated. At this stage an amt. of dil. HNO<sub>3</sub> must be added so that a 40-cc. aliquot, made to vol. in a 50 cc. Nessler tube, with TiCl<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, and Cu(NO<sub>3</sub>)<sub>2</sub> for the color reaction, will have a p<sub>H</sub> of 1.50 ± 0.02. Since there are different amts. of volatile organic matter present in different wines, there is a varying amt. of reduction of the K<sub>2</sub>MnO<sub>4</sub> with the formation of more or less of the alk. products as the case may be. This requires that the exact amt. of HNO<sub>3</sub> must be determined for each individual wine, although some indication can be gained from the amt. of ppt. in the distillate. On account of this difficulty it is preferable to evaporate and ash the samples in the presence of Al nitrate, providing that the temp. can be maintained below 600°. The distillate is then conducted as before. Results are about 10% higher by the ashing method. A series of 6 standard tubes are used containing 0, 1.0, 2.0, 3.0, 4.0, 5.0 mg. of F and of p<sub>H</sub> 1.50 ± 0.02. Above the 5.0 mg. tube colors are not satisfactory for comparison so smaller amts. of sample must be taken if necessary. However, if a 34.0 g. sample is used, mg. per sample tube will be equiv. to grains per lb. W G G

The ratio of dextrose and levulose in the grape, in the must and in the wine. István Szabó and László Rakcsányi. *Magyar Ampel Évkönyv* 9, 346-61 (1935) —Grapes at the beginning of ripening contained more dextrose, during ripening levulose increased and became dominant during drying and thickening of grape. During fermentation (1) under 17-20% sugar content dextrose decreases more rapidly, (2) at 20-25% sugar content both sugars ferment similarly, (3) at high sugar content of 25-30% or more, levulose decreases more rapidly. In natural sweet wines (1) at low sugar content the amount of levulose was 2-6 times as great as that of dextrose, (2) at higher sugar contents levulose was about the same in amount as dextrose and (3) above 25% sugar content levulose was less than dextrose. For wines improved with concd. must (1) if the added must had been fermented the ratio of dextrose to levulose was the same as in natural wines, i. e., more levulose was present, (2) wines improved without fermentation showed the same ratios as the concd. musts with which they had been mixed. S S de Finály

Experiments to conserve sweet wine and must. István Soós and László Rakcsányi. *Magyar Ampel Évkönyv* 9, 362-70 (1935) —A combination of SO<sub>2</sub> treatment and asbestos filtering conserved the products for 21 to 42 days. The best method consists of treating the wine or must with not too much SO<sub>2</sub> (300 mg.), then clarifying with K<sub>2</sub>Fe(CN)<sub>6</sub> and tannic acid gelatin, sedimenting, filtering through asbestos, and treating again with SO<sub>2</sub>. The must treated thus remained unfermented for about 4 months. S S de Finály

Conservation of sweet wines by filtering. István Soós. *Magyar Ampel Évkönyv* 9, 371-2 (1935) —Wine samples were improved with concd. must and fermented by means of special yeast cultures. After fermentation the wine was filtered and the procedure repeated until no further fermentation could be observed. Analyses showed that 15-50% of the original N and 11-45% of the original P<sub>2</sub>O<sub>5</sub> content could be removed by such treatment. Further expts. should prove whether this method is practically available. S S de Finály

The electro-cathodyn. treatment of wines. Gyula Pálinkás. *Magyar Ampel Évkönyv* 9, 373-97 (1935) —Expts. with various wines and musts show the practical availability of the treatment in cellars. S S de Finály

Fermentation of musts improved with concentrated must and sucrose. Géza Requinyi and István Soós. *Magyar Ampel Évkönyv* 9, 398-401 (1935) —Musts improved with sucrose fermented much more weakly when inoculated with special yeast cultures. Those treated with must concd. under high vacuum showed the strongest and most complete fermentation. This explains why sometimes improved wines begin later on to ferment again. S S de Finály

Clarifying wines fermented with pure yeast cultures. Géza Requinyi and István Soós. *Magyar Ampel Évkönyv* 9, 422-5 (1935) —Wines fermented by Hungarian pure yeast cultures could be well clarified. These yeasts seemed to have better effect than the Champagne Haut-willers pure cultures. S S de Finály

Elimination of the copper of musts and wines. J. Ribereau Gayon. *Compt. rend. acad. agr. France* 21, 862-7 (1935); cf. C A 29, 700<sup>12</sup> —A discussion of the removal of Cu from wines and grape juices through the use of a sulfide and reasons for the authorization of its use. Even in excess the sulfide has no other disadvantage than that of causing a cloudiness and disagreeable taste which soon disappears. J. R. Adams

Decacidifying wines beginning to turn vinegary. Géza Requinyi and István Soós. *Magyar Ampel Évkönyv* 9, 453-4 (1935) —A wine contg. 0.70 g. total acids as tartaric acid (of which 0.213 g. was acetic acid) was neutralized with CaCO<sub>3</sub> in 3 phases. In each step 0.2% acid was removed. Details of volatile acids showed that first tartaric and malic acids are neutralized, then acetic acid. Vinegary taste could not be removed by treatment with CaCO<sub>3</sub>. S S de Finály

Treatment of wine by ferrocyanide. Complexes of iron in wines. J. Ribereau-Gayon. *Bull. soc. pharm. Bordeaux* 73, 210-32 (1935) —A crit. discussion of the problems and defense of earlier results of the author's work. Bibliography. S W Goldstein

Diminishing the sulfur dioxide content of oversulfured wines and musts. László Rakcsányi. *Magyar Ampel Évkönyv* 9, 442-7 (1935) —Attempts were made to remove excess SO<sub>2</sub> by (1) 30% H<sub>2</sub>O<sub>2</sub>, (2) perhydrol preps. combined with urea, (3) hexamethylenetetramine and (4) 40% formaldehyde. (1) and (2) were effective and the sulfate originating from oxidized SO<sub>2</sub> did not rise above the 2 g. K<sub>2</sub>SO<sub>4</sub> per l. permitted by the German wine law. (1) caused no detectable changes in the compn. of wine or must. The urea introduced by (2) is analytically determinable but is not objectionable. (3) diminished the amount of free SO<sub>2</sub> only and was also observable chemically in the treated products. (4) had similar effects on the free SO<sub>2</sub> only but its use is prohibited by the authorities. The expts. were made in lab. scales only. S S de Finály

Sulfuration methods of wine. Géza Requinyi and István Soós. *Magyar Ampel Évkönyv* 9, 440-2 (1935) —Sulfuration of wines by means of sheets of elementary S, liquefied SO<sub>2</sub>, and K metabisulfite was examined. The fixation of SO<sub>2</sub> was the same in each case. The application of K metabisulfite increased the ash content by 5-6%. Also the alk. of the ash was somewhat higher. There was no difference in the tastes of the wines sulfured by the different methods. S S de Finály

Refermentation of wine to determine deficiency of nutrient salts for yeast. Géza Requinyi and István Soós.

which covers the liquid surface, the ferment layer is produced on a membrane pervious to the liquid, e. g., parchment paper, or filter paper impregnated with gelatin and nutrient materials. The membrane may be supported by a sieve of coarse mesh. Forms of app are indicated.

**Alcoholic fermentations** Usines de Melle (Soc anon) Fr 788,126, Oct 4, 1935. The fermentation of yeast is carried out in the presence of CO<sub>2</sub> under pressure, whereby higher yields are obtained and the development of organisms such as *Mycoderma* is prevented.

**Citric acid** Wilhelm Klapproth Ger 619,977, Oct 10, 1935 (Cl 6 16 02). A soln contg pure sugar and nutritive salts such as NH<sub>4</sub>NO<sub>3</sub>, K<sub>2</sub>PO<sub>4</sub>, and MgSO<sub>4</sub>, and a trace of FeSO<sub>4</sub> and ZnSO<sub>4</sub>, is fermented by the acid of mildew fungus, such as *Aspergillus*. The acid is pptd, as Ca citrate by adding CaO. The Ca citrate is worked up by the free acid by the usual methods.

**Fermented beverage** Marie Haller (nee Wagner) Ger 619,567, Oct 3, 1935 (Cl 6 21). A refreshing drink is made by roasting oats, wheat and barley, boiling the product with water, filtering, adding cane or sugar and grape sugar to the filtrate and finally adding tansy and yeast and allowing to ferment.

**Filter for wines, etc** Paul Pinel Fr 787,892, Sept. 30, 1935.

**Agung whiskey** Carroll A Hochwalt and Wm H Carmody (to Nelson S Talbott) U S 2,027,099, Jan 7. To a vessel contg green whiskey there are added finely divided heat-treated products of wood distn comprising a first component of active charcoal having substantial absorptive characteristics and free from substantial

quantities of extractable alc. solubles and a second roasted wood component of less active and absorptive character but contg a greater amount of alc. solubles (at least 90% of the first component being used), and the whiskey is heated with the added materials.

**Agung whiskey** Carroll A. Hochwalt and Charles A Thomas (to Nelson S. Talbott). U. S. 2,027,100, Jan 7. See Brit 428,518 (C. A. 29, 66983).

**Aging whiskey** Charles A. Thomas and Carroll A. Hochwalt (to Nelson S. Talbott) U. S. 2,027,129, Jan 7. Whiskey in the vapor phase, together with H<sub>2</sub>, is passed over a hydrogenating catalyst such as Ni on an inert carrier so that hydrogenation is effected while the whiskey is maintained in the vapor phase. App is described.

**Beer** René Rome Fr. 787,870, Sept. 30, 1935. Beer is aromatized by adding ext. of anise or oil of anise when the fermentation is almost complete.

**Beer-brewing kettle with two boiling zones** Rudolf Wiedemann Ger. 619,627, Oct 4, 1935 (Cl 6 12).

**Yeast** Wirtschaftliche Vereinigung der deutschen Hefenindustrie. Ger. 619,555, Oct 3, 1935 (Cl 6a. 17 02). App. with an ascending series of contiguous chambers for the breeding of yeast is described.

**Yeast** Nils Å Öström Fr. 788,370, Oct 9, 1935.

**Dry living yeast in powder form** is obtained by nourishing beer or pressed yeast after growth in a substratum poor in glucose and rich in N compounds, e. g., the residues of the fermentation which has served for the growth of the yeast. By this means living spores are formed.

## 17—PHARMACEUTICALS, COSMETICS AND PERFUMES

W O EMERY

**Comparative investigations of thyroid preparations in rats.** J. Freud and Ernst Lagueur *Nederland Tijdschr Geneeskunde* 80, 1, 25-32(1936).—Various thyroid preps. are standardized by the Abelin method. The metabolic activity is parallel to the total I content for preps. from all kinds of animals. Parenteral is more effective than oral administration. R. Beutner.

**Liquid used in the manometric determination of the oxygen index characterizing the degree of fermentation of yellow types of tobacco.** A. I. Smirnov. *Tabachnaya Prom* 1935, No 3, 4-5.—The O number, which is a criterion in the detn. of the degree of fermentation of tobacco, is obtained by measuring the vacuum produced by samples of tobacco in special glass containers. A kerosene fraction b 200-50° and particularly that b 250-60° with added coloring matter is a suitable manometric liquid. Special calibration of the manometer is required because of the difference in the sp. gr. between this kerosene fraction and the liquids ordinarily used for this purpose.

**Characteristics of the practical application of commercial scale fermentation [of tobacco]** P. G. Asmaev and S. I. Kramev. *Tabachnaya Prom*, 1935, No 3, 17-19.—The relative humidity of the air close to the tobacco should amount to 70-5% to assure a proper fermentation. An excess of humidity will promote the formation of mildew, while a lowering may retard the process of fermentation. Various expts are described. A. A. Bochtlingk.

**Determining the quality of tobacco goods.** G. Dükker. *Tabachnaya Prom* 1935, No 3, 30-34.—The quality of tobacco goods depends on the humidity, ash content (sand, clay, etc.), nicotine content, NII content (a lowering of the N no. indicates inferiority of tobacco), and amount of reducing substances (the amount of reducing substances is in direct proportion to the quality). The higher the ratio of the reducing substances to the N no. the better the quality of the tobacco goods. The quality of tobacco can also be detd. by the "Shmuk no.", i. e., ratio of carbohydrates to albumin (the higher the Shmuk no. the higher the quality of tobacco).

A. A. Bochtlingk

**Democratized cigarettes.** A. Koperina and S. Kalibab. *Tabachnaya Prom* 1935, No. 3, 34-35.—Up to 93% of the nicotine is absorbed by hygroscopic cotton wool when placed in the cardboard cigaret tube, 0.06 g. being required for one cigaret. A still better absorption is obtained by using two cotton-wool filters. Carbon is a poor absorber for nicotine from smoke. The absorption is very little affected by impregnating the cotton wool with picric acid soln. A. A. Bochtlingk.

**Forced fermentation of tobacco.** Vodop'yanov and Antonadi. *Tabachnaya Prom* 1935, No. 4, 25-26.—The forced fermentation, e. g., at 50°, yields the same quality of tobacco as that carried out under normal conditions, provided that the raw tobacco was of the proper quality. A. A. Bochtlingk.

**Colorimetric assay of bismuth pharmaceuticals.** C. S. Leonard and Alma Chaplin. *Compt. rend. Congr. Pharm. Liege* No 47, 197-204(1934).—The following products were assayed for Bi content by (I) the sulfide method of Treadwell and Hall, (II) the oxide method of Hillebrand and Lundell and (III) the authors' colorimetric method (C. A. 20, 3044): (1) compressed and powd. products of Bi subnitrate, rhubarb and soda, (2) Bi subgallate, (3) Bi subnitrate and soda, (4) pepsin, Bi and charcoal, (5) CaCO<sub>3</sub> compd. (contg. Bi subcarbonate), (6) bicreol cream and (7) Bi salicylate cream. A comparison of the methods shows that I gives a pos. error, II is about as accurate as III but less rapid and convenient.

H. M. Burlage

**Absorption from the human skin.** A. Richard Bursle, Jr. *Drug and Cosmetic Ind.* 37, 730-2(1935).—An elaboration of studies previously reported (C. A. 28, 6836). (A) Me salicylate (25%), (B) I (4%), KI (25%) and quinine-HCl (25%) in ointment form with each of the following ointment bases: petrolatum, lard and hydrous lanolin as well as Tincture of I U S P., and compd. soln of I, U. S. P. were applied by inunction on 43 people for 30 min 4 times a day with the exception of the tincture and soln which were applied in single doses of 15 cc. by means of a camel's hair brush. To avoid irritation the sites of application were the inner surfaces of the right and left

thighs and of the right and left arms. Urine samples were collected and tested for presence of the drugs during 72 hrs at intervals. (A) is absorbed the most rapidly and in greatest quantity; (B) is applied more rapidly and effectively as the tincture. The other compds. and forms were not absorbed in amts. to be detected in the urine. Results indicate that the properties and powers of the drugs rather than those of the base are the major factors in the absorption from the skin. The youngest subjects, females, those with soft and finer skin textures, especially blonds and fat individuals, showed more prompt and intense pos reactions.

**Astringency** E. G. McDonough *Drug and Cosmetic Ind.* 37, 733-4, 736 (1935).—The various astringents and their actions are discussed. H. M. Burlage

**Fundamentals of tablet making** Harry J. Sander *Drug and Cosmetic Ind.* 37, 737-8, 742 (1935).—The operations and ingredients are discussed. H. M. B.

**Water-in-oil emulsions with cetyl alcohol** Joseph Kalish *Drug and Cosmetic Ind.* 37, 739-40 (1935), cf. C. A. 30, 567<sup>4</sup>.—A discussion with formulas.

**Cholesterol** Its significance and value in creams and ointments Robert A. Kramer *Drug and Cosmetic Ind.* 37, 741-2 (1935).—A discussion with formulas. H. M. Burlage

**Trend in hand lotions** Thorpe W. Deakers *Drug and Cosmetic Ind.* 38, 37-8 (1936).—Advantages and disadvantages of the old soap-water lotions are discussed. The requisites of and methods of obtaining an ideal lotion are given. H. M. Burlage

**Drugs with volatile oils and their evaluation** L. Kofler. *Pharm. Monatsh.* 16, 209-12 (1935).—A previously described modification of the Dalfert method (*Pharm. Ztg.* 79, 972 (1934); C. A. 30, 568<sup>7</sup>) was used. The following exams are reported: (1) Nine samples of whole cinnamon bark from various com. sources showed volatile oil contents of 0.54-2.10% by wt; (2) 9 samples of calamus root 0.51-3.43% by wt.; (3) 9 samples of whole fennel 0.68-5.21% by wt; (4) 10 samples of cinnamon powder 0.05-0.59% by wt; and (5) 8 samples of calamus powder 0.48-2.23% by wt. Expts seem to indicate that the low contents of volatile oil in powd. drugs are not due chiefly to pulverization but to storage of the powders. A modification of the method is necessary for the detn. of the volatile oil in calamus. H. M. Burlage

**The estimation of the tannin-bearing drugs** O. Dalfert and M. Fleischer. *Pharm. Monatsh.* 16, 221-6 (1935); cf. C. A. 30, 235<sup>4</sup>.—The official drugs galls, granatum, hamamelis leaves, Koso flowers, hematoxylon, myrtillus fruit, quercus, tormentilla root, shaniya, willow and catechu are subjected to the following tests: (A) *Color reactions*—1 g. drug is boiled with 100 cc. H<sub>2</sub>O and treated with (1) solns. of Fe salts, FeCl<sub>3</sub>, Fe alum, FeSO<sub>4</sub>, and Br water, Mitchell's reagent and Fe NH<sub>4</sub> citrate, (2) Na<sub>2</sub>SO<sub>3</sub>, (3) NaNO<sub>2</sub>, (4) pine shaving reaction, (5) concd. H<sub>2</sub>SO<sub>4</sub>, (6) HCl, (7) NaOH, (8) NH<sub>4</sub>OH, (9) alum, (10) lime water, (11) dyeing of cloth strips stained or dipped in solns. of metal salts whereby the drugs are divided into 5 classes. (B) *Precipitation reactions*—(1) HCHO-HCl, (2) Pb(AcO)<sub>2</sub>, (3) Br water, (4) Cu(AcO)<sub>2</sub>, (5) gelatin, (6) antipyrine, (7) tartar emetic, (8) K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, (9) (NH<sub>4</sub>)<sub>2</sub>S, (10) strychnine-HCl. (C) *Capillary analysis*—A procedure for the detection of the tannin groups and individual tannins is outlined. H. M. B.

**Estimation of medicinally used tannin-bearing drugs** O. Dalfert and M. Fleischer. *Pharm. Monatsh.* 16, 231-6 (1935); cf. C. A. 30, 235<sup>4</sup>.—Quant. methods are divided into 2 groups: (A) direct methods by which the tannin compds. are sep'd from their solns. as insol. compds. and weighed or det'd directly and (B) indirect methods whereby from measured vols. of soln. the total tannin compds. are sep'd, and from the difference of certain values before and after the detanning of the soln. calcd. (A) includes (1) gravimetric methods with Al(OH)<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, and Ca(AcO)<sub>2</sub>, (2) measurement of the vol. of the ppt. by allowing it to settle or by use of the centrifuge, (3) by hide powder, (4) titration with TiCl<sub>4</sub> or with Cu salt

1 soln. and (5) colorimetric method with gelatin-Fe paper. (B) includes the shake, filter and Schröder's methods. Biol. methods are discussed. H. M. Burlage

**Preparations for the care of the hair.** Josef Augustin. *Reichstoff-Ind.* 10, 215-17 (1935).—Shampoo powders, liquid shampoos, shampoo soaps and soapsless foaming shampoos are discussed and formulas offered.

H. M. Burlage

**Little-known volatile oils** III Oil and extract of 2 *Tagetes pumila* Alfons M. Burger. *Reichstoff-Ind.* 10, 218 (1935), cf. C. A. 29, 8228<sup>1</sup>.—Steam distn. of the fresh plant yields an orange-red oil (0.25%) with a characteristic odor,  $d_4^{20}$  0.899 and  $n_D^{20}$  +3°. The plant extd. with low-boiling petr. ether yields an orange-brown concrete (0.7%), incompletely sol. in EtOH. The products appear to be of value in making lavender perfumes and cologne waters. H. M. Burlage

**Ipecacuanha decocta** Lajos Dávid *Magyar Gyógyszerészeti Társaság Értesítője* 11, 625-8 (1935); cf. C. A. 30, 569<sup>4</sup>.—It is recommended that 2 cc. citric acid should be dissolved in the water for each g. of drug used and that the mixt. should be boiled for 10 min. Exts. of 100% were thus obtained. S. S. de Finály

**Decocta and infusa** Pál Rózsa *Magyar Gyógyszerészeti Társaság Értesítője* 11, 629-42 (1935).—An Al flask 17 cm. high and 12 cm. in diam. is fitted with a stopper and thermometer and with a sieve 1.5 cm. above its bottom. Two 250-cc. glass bottles are put on this sieve contg. the drugs and the extg. liquid. Water (300 g.) is introduced into the flask (under the sieve) and kept boiling. Several infusa and decocta made by this method were much higher in active matter than those made by other methods. S. S. de Finály

**Detailed "acid hydrolysis method for opium analysis."** G. E. Mallory and Peter Valac, Jr. *Am. J. Pharm.* 107, 515-25 (1935).—A very accurate method of analysis is described step by step in great detail as to washing of solvents, etc. This method may be applied to any opium, any part of the opium poppy plant, and opium refuse and to most all pharmaceutical preps. contg. traces of morphine or large quantities of morphine. W. G. G.

**Determination of iodine in organic medicinals.** Leon Leclercq *J. Pharm. Belg.* 17, 545-50, 563-70, 585-90, 603-9, 631-5, 641-70, 647-51, 705-8, 721-5, 739-45, 758-62, 778-81, 798-803, 815-22, 837-40 (1935).—The various methods are critically reviewed and some changes are suggested. Ninety-six references. S. W. Goldstein

**Aqueous distillate of cherry-laurel.** L. van Itallie. *J. pharm. chim.* 22, 452-4 (1935).—The present official ratio of free and combined HCN (1.3:6) is claimed to be too low and a ratio of 1:4.5 is suggested as representative of a prep'n of good quality. The following test is suggested to det. whether a sufficient quantity of BaH is present. Add 3 cc. of NH<sub>4</sub>OH to 10 cc. of cherry-laurel water. The liquid should appear milky after 30 min. at most. Distinction between distd. cherry-laurel water and an artificial prep'n is impossible at present. S. W. G.

**Quinoline derivatives as a source of antimalarial products** IV. Compounds with long chains in the 8-position O. Yu. Magidson, O. S. Madavaev and M. V. Rubtsov. *J. Gen. Chem.* (U. S. S. R.) 5, 1506-16 (1935).—See C. A. 29, 7013<sup>1</sup>, where the 1st author name is incorrectly given. G. G.

**A critical study of the preparations of black mustard of the Farmacopea Argentina and a proposed method of analysis** Alfredo J. Bandoni. *Rev. centro estud. farm. biolum.* 25, 486-99 (1935). L. E. Gilson

**Mode of action of chemical disinfectants.** Atilio Roccatagliata. *Rev. centro estud. farm. biolum.* 25, 520-5 (1935).—Discussion. L. E. Gilson

**Study of lu-jung, the Chinese drug.** H. Historical study in Japan. Tetsuo Mineshita. *Folia Pharmacol. Japon.* 21, Opera Orig. 223-35, Breviaria 53 (1935); cf. C. A. 29, 2661<sup>1</sup>.—Historical review of lu-jung, a medicine made from young horns of deer and used as an energy builder and rejuvenator in China and Japan for many centuries. It is no longer valued in Japan.

G. H. W. Lucas

A new restorative F. Haefner, F. Schellong and W. Baetzner *Med. Klin.* 31, 1861-4 (1935) —  $N,N,N',N'$ -Tetraethylphthalimide is a new restorative which appears to have a marked stimulant action on the respiration as well as some action on circulation. G. H. W. L.

Effectiveness of hot hypochlorites of low alkalinity in destroying *Mycobacterium tuberculosis* S. M. Costigan, J. W. Yates, W. A. Hadfield and E. C. McCulloch *J. Bact.* 31, 6 (1935) — Hypochlorites in a concn to yield 50 parts of available Cl per million kill *Mycobacterium tuberculosis* in 2.5 but not in 1 min at 50°, in 60 but not in 30 sec. at 55°, and in 30 sec. or less at 60°. Solns contg. 200 parts per million kill in 60 but not in 30 sec at 50°, and in 30 sec. or less at 55° and 60°. J. T. M.

An improved method for determining the antiseptic and bactericidal powers of gases and vapors J. L. Posner, A. R. Toder and John T. Myers *J. Bact.* 31, 9 (1935) — Put the reaction mixt. in a 50-cc. Erlenmeyer flask, stopper and allow gaseous equl. to develop. Pass a pin through a cork so that it will project about 3.5 cm. into the flask. Cut disks of lens paper about 11 mm. in diam. with a cork borer, autoclave, soak in a liquid culture and dry between filter paper, suspend them centrally pierced on the pin and insert into the prep. flask. At timed intervals remove and drop one disk into a tube of broth and another on an agar plate for an impression inoculation. John T. Myers

Ukrainian St. John's bread H. Ya. Tropp *Farm. Zhur.* 1934, No. 6, 222-3 (1934), cf. following abstr. — The samples of St. John's bread contained alkaloids 0.10-2.3, fatty oils (acid no. 2-3) 18.24, moisture 5-8 and ash 3-4%. L. Nasarevich

Ukrainian St. John's bread Condition affecting its toxicity during preservation H. Ya. Tropp *Farm. Zhur.* 1934, No. 6, 223-7 — Ukrainian St. John's bread approaches the highest known content of alkaloids (0.23%). To preserve St. John's bread, it should be defatted and dried. The alkaloids are most sensitive to heat, 1 hr. at 100° being enough to decompose 40%. L. Nasarevich

Analysis of ichthyol S. M. Bolotnikov *Farm. Zhur.* 1935, No. 1, 21-3 — Ichthyol should be sol. in 2 parts of  $H_2O$ , 4 parts of alc., or 8 parts of ether-alc. mixt. It should contain no cresols, phenol derivs. or foreign sulfo acids. Total S should be over 11%, sulfate S not over 2.8%, thiophene fraction over 12%, dry residue over 55%. An analysis is given. L. N.

Preparation of saligenin I. M. Rotbart and D. H. Kolesnikov *Farm. Zhur.* 1935, No. 1, 27-8 — Phenol (1), formalin (1),  $CaO$  (0.5) and alc. (2.5 parts) react until HCHO odor is gone, the mixt. is acidified with  $AcOH$ , extd. with ether, dried, distd. off and recrystd. The crystals of *m*-hydroxybenzyl alc. are finally crystd. from  $H_2O$ . L. Nasarevich

Comparison of tinctures with 70 and 45% alcohol Yu. Petrovskii, K. Shulzhenko and I. Bidoshina *Farm. Zhur.* 1935, No. 2-3, 71-2 — Tinctures of *Digitalis purpurea* and *Adonis vernalis* are richer in glucosides and more active biologically if made with 45% alc. L. Nasarevich

Determination of magnesium in medicines D. S. Belyutskaya *Farm. Zhur.* 1935, No. 2-3, 76-8 — For medicinal mixts. the best methods are direct alc.-acid titration by Knöfner's or Berg's method, in which Mg is pptd. with hydroxyquinoline and brominated and excess of standard Br is titrated with KI and  $Na_2S_2O_3$ . L. N.

Determination of hexamethylenetetramine in medicines D. B. Yokhel'son *Farm. Zhur.* 1935, No. 5, 172-3 — Hexamethylenetetramine is treated with  $H_2SO_4$ , excess HCHO is added to the  $(NH_4)_2SO_4$  formed and the liberated  $H_2SO_4$  is titrated with  $NaOH$ . L. Nasarevich

Plasmoid I. T. Sirokov *Khim. Farm. Prom.* 1934, No. 6, 14-15 — To obtain a cryst. product instead of an oily mass, 2 moles of methylenedisulphic acid are used for 1 mole of 6-methoxy-8-( $\gamma$ -diethylamino)propylamine, which is dissolved in HCl (3.6 moles), the methylenedisulphic acid is dissolved in  $NH_4OH$ , enough just to neutralize the acid. L. Nasarevich

Determination of halogens in extracts I. Orlov and T. Ksenofontova *Khim. Farm. Prom.* 1934, No. 6, 22 — The color is removed by animal charcoal, then one aliquot is titrated (Volhard) for total halogens, the second aliquot is titrated after the removal of I, and in the third Br and I are removed by boiling with  $KMnO_4$ . L. Nasarevich

Synthesis of decamethylenebisguadimide (synthallin) K. S. Topchiev and L. N. Pavlov *Khim. Farm. Prom.* 1935, No. 1, 24-5 — Sebacia acid (made from castor oil and KOH) is dissolved in the picoline fraction of pyridine bases, treated with dry  $NH_3$  and  $POCl_3$ ; the aq. soln is extd. with  $CaH_2$ , evapd. on the water bath and the dinitrile of sebacia acid is distd. *in vacuo*. Decamethylene-diamine is obtained from the nitrile with 150  $AmOH$  and Na. Guanidine thiocyanate and the diamine are heated at 135°, poured into boiling 20% KOH and the hardened mass after cooling is ground, washed, dried, dissolved in abs. alc. and treated with HCl to yield decamethylenebisguadimide HCl. L. Nasarevich

Synthesis of acrinoline (2-methoxy-6-chloro-9-diethylaminoacridine) O. Yu. Magidson, A. M. Grigorovskii, V. I. Maksunov and R. S. Margolina *Khim. Farm. Prom.* 1935, No. 1, 26-34 — The synthesis is divided into 2 parts: (1) prepn. of 2-methoxy-6,9-dichloroacridine and (2) prepn. of 1-diethylamino-4-aminopentane and the condensation of the two (1) *p*-Nitrotoluene  $\rightarrow$  *o*-chloro-*p*-nitrotoluene  $\rightarrow$  *o*-chloro-*p*-toluidine  $\rightarrow$  2,4-dichlorotoluene  $\rightarrow$  2,4-dichlorobenzoic acid + anisidine  $\rightarrow$  *N*-*p*-amyl-4-chloroanthranic acid  $\rightarrow$  2-methoxy-6,9-dichloroacridine (2) Diethylaminoethanol  $\rightarrow$  diethylaminoethyl chloride  $\rightarrow$  1-diethylamino-4-pentanone  $\rightarrow$  1-diethylamino-4-pentanone oxime  $\rightarrow$  1-diethylamino-4-aminopentane. The condensation can be effected either by alkali or acid. L. Nasarevich

Properties of digitalis preparations S. I. Ordynskii *Khim. Farm. Prom.* 1935, No. 1, 49-55 — The digitoxin content varies from 0.240 to 0.643%. No const. ratio exists between the digitoxin content and the biol. action. Seventy % alc. produces the most effective tinctures. L. Nasarevich

Preparation of tripansin A. M. Lvov *Khim. Farm. Prom.* 1935, No. 2, 110-13 — Tripansin, the Na salt of tetrazo-*o*-toluidine-1,8-aminonaphthol-3,6-disulphonic acid is made from *o*-toluidine-HCl, which after tetrazotization is immediately coupled with H-acid in alk. soln. For avoiding decompos. of the tetrazo compd. the temp. should be low and the process not too long. Purification is effected with  $EtOH$ , varying amts. of impurities ( $NaCl$ ) being eliminated. L. Nasarevich

Colorimetric determination of adrenaline M. I. Shapuro *Farm. Zhur.* 1934, No. 4, 131-4 — The colorimetric reaction with either  $NaNO_2$  or phosphotungstic acid can be utilized for the detn. of adrenaline in the presence of  $ZnSO_4$ ,  $B_2O_3$ , antipyrine, procaine and cocaine. Detn. of adrenaline in ointments and in the presence of protargol is only approx. L. Nasarevich

Oil of sweet oranges from French Guinea Y. R. Naves *Parfums de France* 13, 298-308 (1935) (in French and English) — A review of the economic and tech. characteristics of the oil. Analyses carried out since those reported in 1932 (C. A. 26, 4912) and bearing on over 50% of the production in Guinea, indicate some slight modifications in the previously reported const., a frequently reaches 93°, the max. figure observed being 93°16'; a frequently reaches the min. figure of 1.4720  $\approx$  0.0002 in fresh oil; the aldehyde content frequently exceeds 1.5%, and the max. of frequently observed values should be raised to 2.4%. Oils from ripe fruit have the highest aldehyde and nonvolatile contents. From a discussion of the work of various investigators, it is concluded that the  $NH_4OH$  HCl method is the only one that gives accurate results for the detn. of aldehydes. A. P.-C.

Practical perfume formulas I. Tesdorpf, *Seifen- und Ztg.* 62, 787-9 (1935) — Recipes are given for the prepn. of perfumes of the types: quelques fleurs, juchten, toska and chypre. J. W. Perry

Alkaline or acidic cosmetics? Th. Ruemele, *Seifen- und Ztg.* 62, 837-8, 827-9 (1935) — A general discussion

of cosmetic powders and creams, shampoos, mouth washes, etc., leads to the conclusion that, as a rule, superior results are obtained when such products have an acidic reaction.

**Uses for isopropyl alcohol** Th Ruemele *Seifensieder-Zig.* 63, 21-22 (1936).—Although unsuitable for many products, e. g., mouth washes, isopropyl alcohol can often be employed advantageously in others, e. g., perfumes, nail polishes and other cosmetics.

**Contribution to the study of the composition of opium** A Labb *Riv. sci. essenze profums piante officinali* 17, 215-17, 260-1 (1935).—Numerous formulas are given.

**Camomile** Orlando Gulminelli *Riv. sci. essenze profums piante officinali* 17, 272-4 (1935). Roman camomile yielded 700-1000 g. per 100 kg. of oil of d. 0.905-0.915, contg. isobutyl isobutyrate, isobutyl angelate, amyl tiglate and angelaldehyde, a little strychnine and quercitrin. The oil was sol. in 8 parts of 90° alc. and in ether, solidified at 0°, depositing colorless crystals of pleasant balsamic odor and bitter burning taste.

**Glucose** Methods of use and therapeutic indications Agustín A. Salvati *Dia med.* 6, 939 (1934). *Annales assoc. quim. Argentina* 23, 23B. E. M. Symmes.

**Pill and lozenge making** K. Kromann Jensen *Farm Tidende* 2, 25, *Pharm. J.* 135, 650 (1935).—Formulas and modes of mfg. are given.

**New method for the determination of iodobismuthate of quinine** Lorenzo Bracaloni *J. pharm. chim.* 22, 49-52 (1935).—To a soln. of (BiI<sub>3</sub>), quinine 2Hl in acetone add aq. AgNO<sub>3</sub> in excess, AgI and basic Bi nitrate (A) are pptd., quinine nitrate in soln. is filtered off after the acetone is expelled by water-bath heat and the residual mixt. is treated with 95% alc., quinine is detd. by polarimetry. AgI and A are sep'd. by concd. HNO<sub>3</sub>, from A, Bi is pptd. by NH<sub>3</sub>, carbonate and weighed as Bi<sub>2</sub>O<sub>3</sub>. Results of detn. of Bi, quinine and I by this method show good agreement with those of B's previous method (cf. C. A. 25, 5952).

**Preparation of a sensibly neutral and permanent 0.1% adrenaline solution** Louis Julien *J. pharm. chim.* 22, 53-9 (1935).—The vague formula of the French Codex 1926 is revised by using a corrected relation between d. and percentage of NaHSO<sub>4</sub>, and replacing the no. of drops by cc. In the old and the corrected formulas, too much SO<sub>2</sub> is set free by HCl, resulting in *pu* 2-5-6 which is too high an acidity for the parenteric use of the prepn. Omission of HCl is recommended, reliance being placed on the small acidity of NaHSO<sub>4</sub> itself. Dissolve adrenaline (A) 1 g. and NaCl 7 g. in H<sub>2</sub>O contg. 5 cc. soln. of NaHSO<sub>4</sub> (d. 1.33 = 31.60 g. SO<sub>2</sub> per 100 cc.), add H<sub>2</sub>O sufficient for 1000 cc. This soln. has *pu* 6.2, similar to that of the H<sub>2</sub>O used, and keeps its content of A unchanged for more than 2 months. If 5 cc. of the NaHSO<sub>4</sub> soln. is reduced to 3 cc. (cf. Bndel, C. A. 17, 2169), *pu* = 6.6. The 3-cc. formula is recommended for a single ampoule dose, the 5-cc. formula, for use in larger flasks involving repeated renewal of air. If desired, the neutral formula can be modified by addn. of 5.45 cc. N HCl, i. e., the exact quantity to form A.HCl. The resulting *pu* = 3.8 is an extreme max. of allowable acidity (cf. *pu* 3.6, Dubouquet, C. A. 16, 2198).

**Alkalimetric determination of barbital** Ch. Morin *J. pharm. chim.* 22, 59-61 (1935); cf. Isnard, C. A. 18, 2944.—To avoid aq. hydrolysis of the K barbiturate formed in the method, dissolve 0.2 g. substance in 30 cc. neutralized acetone, add 4-5 drops of 0.1% thymol blue in 95% alc. and titrate with 0.1 N KOH soln. in MeOH from yellow through green to pure blue. Previously use the same indicator in titrating the KOH with 0.1 N H<sub>2</sub>SO<sub>4</sub>. With 6 samples of barbital, results varied from -3 to +0.05%; with neonal (6 samples) from -2 to +2%. Applied to com. tablets also including gardenal, rutilonal and dial, the method is more rapid than that of extn. and detn. by wt.; the accuracy is the same as with the barbital tablets.

**A new color reaction of chloral and its application to the identification of sirup of chloral.** M. Peser *J. pharm.*

*chim.* 22, 68-9 (1935); cf. C. A. 30, 1327.—To 2 cc. H<sub>2</sub>SO<sub>4</sub> (d. 1.84) in a test tube add 0.1 cc. (2 drops) of the KBr-resorcinol reagent, then 2 drops of chloral soln. and warm to 100°. The colors change from yellow-pink to violet at 90° then to blue at 100°. Upon addn. of 2 cc. H<sub>2</sub>O, the blue color turns to orange-red, when NaOH is added, a violet then reddish tint is produced. To detect chloral (A) in sirup of A (French Codex) by this test, ext. 1 cc. of the sirup with a mixt. of H<sub>2</sub>O (5 cc.) and Et<sub>2</sub>O (2 cc.), sep. and evap. off the Et<sub>2</sub>O completely, and apply the test to the residue. As tartaric acid (B) is insol. in Et<sub>2</sub>O, the test differentiates sirup of A from that of B.

**Iron in chemistry and pharmacy** I. Historical G. Malcolm Dyson *Pharm. J.* 133, 356 (1934). II The production of iron and steel *Ibid.* 729. —Historical and technical notes. III Compounds of iron *Ibid.* 135, 679 (1935). Phny's accounts of "shoemakers' black" (Fe<sub>2</sub>SO<sub>4</sub> used for blackening tanned leather) as an adulterant for verdigris, and various uses of it in medicine are quoted, also the accounts by Quincy (1720) on the prepn. of "Colicbatch's Stiptick powder," the "astringent saffron of iron," etc. "It has been recently suggested that the most assumable form of Fe is that obtained by the ingestion of raisins from grapes grown on ferruginous soil."

**Report on (the assay of) crude drugs** H. W. Youngken *J. Assoc. Official Agr. Chem.* 18, 515-18 (1935), cf. C. A. 28, 6527.—The phys. characteristics of roots of *Aconitum napellus* L., subspecies *neomanitatum* Wall, are described, together with the histology of the parent tuberous root and of the daughter tuberous root. Scrapings of both parent and daughter tuberous roots, when exam'd in water mounts, showed the starch grains to be simple, the individual grains were mostly spheroidal, plano-convex, angular-convex to pyriform, and up to 24.5 microns in diam. It is recommended that aconite root should be recognized in the U. S. P. as representing the dried tuberous roots of *Aconitum napellus* L. and its subspecies and varieties contg. aconitine.

**Oil of Tennessee red cedar [use in perfumes and medicine]** (Huddle) 27. **Bark of *Aspidosperma quadrangula* (Florim.) 11D** Emulsions [esp. cosmetics] (Thomassen) 2. **Halogen derivs. of procaine** (Trejka, Vymetal) 10. **Cyclic ketones [having a jasmine perfume]** (Fr. pat. 785,549) 10. **Removing halogens from fluids [products used as bandages for medicinal purposes]** (Brit. pat. 435,738) 13. **Unsatur. aldehydes and ketones [products used in pharmaceutical chemistry]** (Brit. pat. 435,194) 10. **Esters of etherified glycolic acids [used in the perfume industry]** (U. S. pat. 2,027,991) 10. **Oils [cosmetics]** (Ger. pat. 619,928) 27.

**Therapeutic compounds** Hanns John *Fr.* 786,888, Sept. 11, 1935. Esters of 2-phenylquinoline-4-carboxylic acid (I) its substitution products, isomers and homologs are prep'd. by causing glycerol or haloalcohols of glycerol to act on the acids, their salts, esters or halides. Examples are given of the prepn. of the glycerol esters of I, m 109°, of 2-phenylquinoline-4-carboxylic acid (II), m 110.5°, of 2-phenylquinoline-4'-carboxylic acid, m 113°, glycerol  $\alpha,\alpha'$ -diester of I, m 58°, glycerol  $\alpha,\alpha'$ -diester of 6-methoxy-I, m 92-4°, glycerol  $\alpha,\alpha'$ -diester of II, m 73-5°, glycerol  $\alpha,\beta'$ -diester of I, m 65-8°, glycerol triester of I, m 135°,  $\alpha$ -chloropropyl ester of I, m 109°, and  $\beta,\beta'$ -dichloropropyl ester of II, m 101°.

**Therapeutic solutions** I. G. Farbenund A-G (Carl Ludwig Lautenschlager, Max Bockmuhl and Rudolf Schwabe, inventors). Ger. 622,298, Nov. 25, 1935 (Cl. 30h. 2). Addn. to 446,290 and 447,161. Medicaments of low soly. in water are dissolved in water-sol. ethers of trihydric or higher polyhydric alcs., or in aq. solns. of such ethers. In typical examples, 3,4-diamino-7-ethoxyacridine (0.5 g.) is dissolved in diethylin (10 cc.), and acetylcholine acid (0.5 g.) is dissolved in dimethoxy-trihydroxyhexane (10 cc.).

**Therapeutic preparations liberating carbon dioxide**



Jean G Waldenmeyer Fr 788,108, Oct 5, 1935 The mixts of substances which by decompn yield  $\text{CO}_2$  are coated, separately, in fatty materials contg lipoids, e.g., lecithin. The presence of lipoids allows the passage and action of a catalyst while not impairing the keeping properties of the fat

Therapeutic mixture for oral administration in treating cornea, etc. Harold S Diehl (to Board of Regents of the Univ of Minn.) U S 2,027,722, Jan 14 A salt of codeine such as the sulfate is used in admixt with a salt of papaverine such as the hydrochloride

Local anesthetics Oscar H Stover and Edmund H Brigham (to Oleohessm Co.) U S 2,027,125, Jan 7 A procaine base is converted into a salt by reaction with an equimol proportion of a fatty acid contg at least 9 C atoms, such as pelargonic, capric, undecylic, lauric, myristic, palmitic, stearic, oleic or erucic acid. The product may be dissolved in various oils

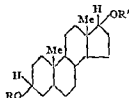
Thiazoline compounds (local anesthetics) Max Engelmann (to I du Pont de Nemours & Co.) U S 2,027,030, Jan 7 By the reaction of substituted phenyl isothiocyanates such as *p*-tolyl isothiocyanate with halo ethylamines such as bromoethylamine-HBr, products are obtained such as *p*-tolylmimodihydrothiazole, m 131°, hydrochloride, m 154° *p*-fluorophenylmimodihydrothiazole, m 152.3°, hydrochloride, m 134° *o*-butoxyphenylmimodihydrothiazole, m 68° *p*-hydroxyphenylmimodihydrothiazole, m 154°, hydrochloride, m 235-9° *p*-ethoxyphenylmimodihydrothiazole, m 140° *p*-hydroxy-*m*-methoxyphenylmimodihydrothiazole, m 168-9°, hydrochloride, m 211°. General mention is made of some other similar derivs and of their salts

Oxazoline compounds (local anesthetics) Max Engelmann (to F I du Pont de Nemours & Co.) U S 2,027,031, Jan 7 Various examples are given of the reaction of substituted phenyl isocyanates with halo ethylamines and further treatment of the resulting product or substituted phenylalkylhalo ureas to produce substituted phenyl dihydrooxazoles, and, as being new products, claim is made to compds such as 2-*p*-ethoxyphenyldihydrooxazole, 2-*p*-butyloxyphenyldihydrooxazole and the like (general mention being made of various similar compds and their salts)

Anthelmintic Wallace P Elmslie and Paul Caldwell (to Moorman Mfg Co.) U S 2,027,967, Jan 14. An anthelmintic ration suitable for treating swine or other livestock comprises an alimentary product such as wheat middlings mixed with a small proportion of a F-contg salt such as NaF or KF

Rectal narcotic Erich Goth (to Winthrop Chemical Co.) U S 2,027,905, Jan 14 An anhyd compn, stable to light and heating and liquid at room temps, comprises tribromoethyl alc together with a smaller quantity of a compd such as acetamide and methylacetamide or carbamic acid Me and Et esters, or tetramethylurea, etc.

Esters of polycyclic alcohols Schering-Kahlbaum A-G Fr 788,545, Oct 11, 1935 Esters of the general formula  $\text{C}_{18}\text{H}_{26}(\text{OR})(\text{OR}')$  and the constitutional formula



in which R is an acyl group and R' is H or an acyl group are prep'd by submitting esters of hydroxy ketones of the group of male sexual hormones  $\text{C}_{18}\text{H}_{26}\text{O}_2$  and  $\text{C}_{18}\text{H}_{26}\text{O}_4$  and their stereoisomers, to the action of agents capable of transforming the ketone group to a secondary alc group and acylation, if desired, of the alc. group. The transformation may be carried out by nascent H. The resulting

esters have the characteristics of the male sexual hormones

Aminoazo compounds Fritz Mietzsch and Josef Klarer (to Winthrop Chemical Co.) U S 2,022,921, Dec. 3 Compds suitable for therapeutic use in combating protozoa and bacteria and which have the general formula  $\text{RN}(\text{NR}'\text{N}(\text{X}')\text{X}'\text{N}(\text{X}')\text{X}')$ , where R represents an aromatic radical, X' stands for H or alkyl, X' represents a bivalent aliphatic hydrocarbon group which may be substituted by hydroxyl, and where the  $\text{C}_{11}\text{H}_7$  nucleus R! may be further substituted by alkyl, alkoxy or halogen, are obtainable by coupling aromatic, heterocyclic or aromatic-heterocyclic diazo compds with amines of the benzene series, substituted in one amino group by a basic radical and capable of coupling or by causing diazo compds of aminobenzene derivs contg, attached to one amino group a basic substituent to react with aromatic, heterocyclic or aromatic-heterocyclic compds capable of coupling. Or aromatic, heterocyclic or aromatic-heterocyclic nitroso compds are condensed with polyaminobenzenes, contg a primary reactive amino group as well as one amino group substituted by the basic radical or aromatic, heterocyclic or aromatic-heterocyclic amines can be caused to react with C-nitroso benzenes contg an amino group substituted by a basic radical. Various examples and modifications of procedures are described

Amino double compounds I G Farbenindustrie A-G Brit 430,008, Oct 3, 1935 Double compds. of alkali and alk earth metal halides with secondary and tertiary amines contg a hydroxyalkyl group are prep'd by interaction of the components, or of alkali and alk earth metal hydroxides with either the amine and a hydrohalic acid or the hydrohalide of the amine. The products are useful therapeutically in combination with acids, e.g., barbituric acids and 2-phenylquinoline-4-carboxylic acid. Among examples, compds are prep'd from (1) CaBr<sub>2</sub> and triethanolamine, and (2) CaI<sub>2</sub> and tripropanolamine. N,N'-Dichloroazodicarbonamidine Franz C Schmuckes and Henry C Marks (to Wallace & Tiernan Products, Inc.) Brit 430,093, Oct. 4, 1935 See U S 2,010,257 (C A 29, 8008'). The product possesses therapeutic properties

Cyclic amines Soc pour l'ind chim. à Bâle, Swiss 175,571-2, May 16, 1935 (Cl 116A). Addns to 172,873 (C A 29, 5603'). Civetone thioxosime is treated with a reducing agent to give a cyclic imine of the formula  $\text{CH}_2(\text{CH}_2)_8\text{CH}(\text{CH}_2)_8\text{NH}$  b<sub>2</sub>; 128° and m. 28-9° (175,571). Cyclohexadecanone thioxosime is reduced

to give the cyclic imine  $(\text{CH}_2)_{14}\text{NH}$  which b<sub>2</sub>; 126-7° and m. 58-9°. The substances have physiological action and are used in therapy. Cf C A 30,248°.

Guanyl and biguanyl compounds. I. G Farbenind. A-G Fr 788,429, Oct. 10, 1935. Guanyl and biguanyl compounds; the C atom of which is joined to a higher aliphatic radical by O or S as an ether, are prep'd by causing aliphatic alcs or mercaptans contg about 10-16 C atoms to react with cyanamide, dicyanodiamide, alkyl or aryl-alkyl ethers of isourea, guanylisourea, isothiourea or guanylisothiourea or their salts. They may also be prep'd by causing esters of higher aliphatic alcs with halogenated and sulfonic aromatic acids to react with guanidine, biguanidine, thiourea, guanylthiourea or their salts, or by causing  $\text{NH}_3$ , amines, or guanidines or their salts to react with cyanamides or rhodanides of high mol wt, contg an aliphatic group of 10-16 C atoms. The products have a strong bactericidal action and also have wetting, frothing and dispersing properties making them useful as disinfectant cleansing agents. Examples are given of the prep'n of dodecylisourea, m 78°, HCl salt, m 93°, dodecylguanidine, m 96°, dodecylbiguanidine, m 90°, S-dodecylisothiourea, m 82°, HBr salt, m 111°, HCl salt, m 133°, S-dodecyl- (m 140°) and S-ethyl-guanylthiourea-HBr, m 90°, S-dodecylmethoxythiourea-HCl, m 134°, dodecylthioethylguanidine-HBr, m 68°, and 3-biguandinephenyl dodecyl ether, m 175°, di-HCl

salt, m. 207° (from *m*-aminophenyl dodecyl ether, b<sub>p</sub> 204°, m. 63°, HCl salt, m. 115°).

**Pyrazolones.** I. G. Farbenind. A.-G. Brit. 433,053, Aug. 8, 1935. 1-Aryl-2-alkyl-5-pyrazolones that contain at least at 1 N atom a radical (Y) that is a partially or wholly hydrogenated cyclic hydrocarbon radical and have the formula  $RN.NR'.CR''CR'''CO$ , where R is Ph

or Y, R' is alkyl or Y, R'' is alkyl and R''' is H, alkyl or the group -N(X)X (where X and X' are H, alkyl, aralkyl or Y), are distinguished by good *seifbruge* and *sedative* action. They are prepd by (a) condensing an acylacetic ester with a hydrazine monosubstituted by Y and causing an alkylating agent or an agent for introducing Y to act upon the resulting product, (b) condensing an acylacetic ester with a symmetrically disubstituted hydrazine contg. at least 1 Y, (c) condensing a hydrazine monosubstituted by Y with an  $\alpha,\beta$ -unsatd carboxylic acid or an ester thereof, oxidizing the pyrazolidone thus obtained and causing an alkylating agent or an agent capable of introducing Y to act upon the resulting product, (d) nitrating or treating with HNO<sub>3</sub> a pyrazolone obtained by (a), (b) or (c), reducing the product in each case and, if desired, causing the resulting amino deriv. to react with an alkylating agent or with a halo substitution product of a wholly or partially hydrogenated cyclic hydrocarbon, (e) causing 1 molar proportion of a halo substitution product of a wholly or partially hydrogenated cyclic hydrocarbon to react with 1 molar proportion of a known 5-pyrazolone compd. substituted in the 1- and 2-positions and contg. a primary or secondary amino group in the 4-position, the product may be further alkylated, or (f) causing an aldehydesulfoxylate or aldehyde bisulfite to act upon the pyrazolones obtained according to (d) and (e) and contg. a replaceable H atom attached to N; soly. of the products in H<sub>2</sub>O is thus effected. Five examples are given. Cf. C. A. 29, 4134<sup>1</sup>.

**Barbituric acids.** I. G. Farbenindustrie A.-G. Brit. 435,450, Sept. 23, 1935. *N*-Mono- or di-alkylated barbituric acids substituted in the 5-position by Me and a group X, comprising a said cycloalkyl group, or a said alkyl group which may be substituted by a cycloalkyl residue, or an unsatd. alkyl group or halo deriv. thereof, provided the group contains at least 3 C atoms, are prepd by known methods. Methods involving the treatment of 1-cyano-2-iminobarbituric acids are excluded. The products have *therapeutic properties* and, in the case of the *N*-monoalkyl compds., may be converted into their salts with inorg. or org. bases. In examples, (1) butylmethylmalonic acid diethyl ester is treated with methylurea to give *N*-methyl-5,5-methylthylbarbituric acid, and (2) *N*-methyl-5,5-methylallylbarbituric acid is prepd. from methylallylmalonic acid diethyl ester and methylurea.

**Anthracenic products.** James Waddell (to E. I. du Pont de Nemours & Co.). U. S. 2,025,364, Jan. 21. Lipoid-contg. material such as cholesterol is heated to above 100° (suitably to 190° for 2.5 hrs. in a Cu pipe in the case of cholesterol) in the presence of a greater quantity of water, so as to increase the amount of activatable substances present without substantial injury to them, and the treated material is subjected to irradiation with ultraviolet rays.

**Pregnanolone.** Schering-Kahlbaum A.-G. Fr. 788,430, Oct. 10, 1935. Pregnanolone (I) and all-*l*-O or their derivs. are prepd. by transforming to tertiary carbinols, in the form of their esters, the corresponding 3-hydroxy- $\beta$ -methylcarbinolic acids or their derivs., in which the alk. OH group is replaced by halogen or is protected, e. g., by etherification or esterification, eliminating water by means of water-extg. agents between the tertiary OH and the neighboring tertiary H atom, and disoccr. the double bond thus formed in the lateral chain of the mol., by means of appropriate oxidizing means, thus forming the corresponding hydroxy ketones or their derivs. Cf. C. A. 30, 112<sup>2</sup>.

**Ketocyclopentanodimethyltetradecahydrophenanthrene.** Schering-Kahlbaum A.-G. Fr. 788,431, Oct. 10, 1935.

1 This is prepd. by transforming pregnanolones (I), by the action of organometallic compds., into a secondary-tertiary alc., causing an acylation agent to act on this, submitting the unsatd. ester obtained to an oxidation and finally sapon. the oxidation product. I are obtained from sterols or by the process of Fr. 788,430 (preceding abstr.). The product has an action similar to that of male sexual hormones.

2 **Corpus luteum hormones.** Société pour l'ind. chim. A. Bâle Brit. 435,530, Sept. 30, 1935. The hormones are obtained in cryst. form by causing crude preps thereof to react with reagents for the carbonyl group, e. g., bisulfite, NH<sub>4</sub>OH, N<sub>2</sub>H<sub>4</sub> or derivs thereof such as semicarbazide and phenylhydrazinesulfonic acid, sepp. the reaction products and decomp. them to liberate the hormones. Among examples, (1) an ext. prep. according to Brit. 426,166 (C. A. 29, 5601<sup>4</sup>) is dissolved in alc and treated with an aq. soln. of semicarbazide-HCl and AcONa, the crude ppt is washed with alc, dissolved in glacial AcOH and heated with dil. H<sub>2</sub>SO<sub>4</sub>, and (2) an Et<sub>2</sub>O soln. of a hormone prep. is shaken with aq. Na bisulfite and the aq. layer is sepd. and heated with H<sub>2</sub>SO<sub>4</sub> and the released hormone extd. with Et<sub>2</sub>O.

3 **Pituitary gonad-stimulating hormones.** Arthur E. Meyer (to Chappel Bros Inc.) U. S. 2,027,446, Jan. 14. A dil aq. soln. (suitably a 10% soln.) of a lower aliphatic alc. such as Me, Et, Pr, Bu or Am alc. is used as an extg. agent for extg. alc.- and acetone-insol. hormones from animal material contg. such hormones and which may have been previously treated with acetone.

4 **Bactericidal azo compounds.** I. G. Farbenind A.-G. Fr. 788,679, Oct. 14, 1935. Aromatic, heterocyclic or aromatic-heterocyclic diazo compds. substituted by a *p*-sulfonamide or disulfonamide group are coupled, by known methods, with compds. of the C<sub>6</sub>H<sub>5</sub>, C<sub>6</sub>H<sub>4</sub>, pyridine or quinoline series capable of coupling. The compds. contain a basic N atom fixed to the ring, which may be represented by the pyridic or quinolinic nuclear N atom, also one NH or OH joined to the ring and an acid group. The acid group may be a substituent of the NH<sub>2</sub> or OH group. Examples are given of the preps. of 4'-sulfonamide-2-aminophenyl (or acetylaminophenyl)-4-hydroxy (or amino)-azobenzene-5-sulfonic acid, 4'-sulfonamide (or sulfonamethylamide)-phenylazo-2-aminophenyl (or diethylamino)-5-naphthol-7-sulfonic acid, 4'-sulfonamide-2-aminophenyl-4-hydroxy (or amino)-azobenzene-*N*-methanesulfonic acid, 4'-sulfonamidediphenylazo-6-amino-2-naphthol-8-sulfonic acid, 4'-dimethylsulfonamidediphenylazo-8-naphthol-1-N-hydroxyethylamino-3,6-disulfonic acid and 4'-ethylsulfonamidediphenylazo-8-hydroxyquinoline-7-carboxylic acid.

5 **Compositions for cleaning or disinfecting the mouth.** Deutsche Gold- und Silber-Scheideanstalt vorm. Roesler (Wilhelm Schulenburg, inventor). Ger. 622,411, Nov. 27, 1935 (Cl. 30h. 13). Stabilized water-sol. compds. contg. active O are mixed with small proportions of Ag salts and preferably with acid substances. The compds. contg. active O may be stabilized by dehydration or addition of a known stabilizing agent, e. g., Mg silicate. Ag salt of org. or inorg. acids may be used, but salts of acids which have a disinfecting action are preferred. The proportion of Ag in the mixts. should be 0.5-3%. Org. acids are the preferred acid substances, but inorg. acid substances, e. g., H<sub>2</sub>BO<sub>3</sub> or an acid phosphate, may also be used. When an alk. substance contg. active O is used the proportion of acid substance is preferably less than that necessary for neutralization. A sp. compn. contains dehydrated NaBO<sub>2</sub>·H<sub>2</sub>O 30, tartaric acid 11.25 and Ag *p*-hydroxybenzoate 1.6 parts. Sweetening substances, perfumes, etc., may be added.

6 **Dentifrices.** Worcester Salt Co. Brit. 434,985, Sept. 12, 1935. See Fr. 775,290 (C. A. 29, 2666<sup>9</sup>). The suspension of NaCl may also be rendered stable by addn. of a concd. suspension of finely divided substance, e. g., Mg(OH)<sub>2</sub>, which may also contain a colloid, e. g., tragacanth.

7 **Dentifrice.** Bernard H. Jacobson (to Klipstein Chemical Processes, Inc.). U. S. 2,027,533, Jan. 14. Finely divided cryst. anthraquinone is used with other ingredients

such as glycerol, soap, cornstarch, Na benzoate, oil of peppermin and water

**Shaving preparations** Ralph H Fash. *Fr.* 788,572, Oct 12, 1935. The preps contain substances which protect the blade against oxidation by rendering the steel thereof neutral. Such substances include oxidizing compds such as chromic acid, chromates, di- and perchromates, perchlorates, perborates, persulfates, and nitrates of metals having, in soln, a tension lower than that of Fe or neutral steel. It is presumed that a very fine protecting film of oxide is formed on the steel.

**Cosmetics** I G Farbenindustrie A G. *Brit.* 435,811, Sept 30, 1935. Preps for protection against sunburn and allowing tanning of the skin consist of solns or dispersions of org compds that are colorless, free from phenolic OH groups, nonmucous, have a strong absorptn capacity for ultraviolet rays and have a slope in the absorption curve between 310 and 350 mμ corresponding to an increase of the logarithm of the molar extinction coeff by at least 1 within a range of 10 mμ. Suitable compds have the formula  $RX \cdot CR_2$ —in which R is an aromatic nucleus and X is N or the methine group, the substances

used preferably having the formula  $C_6H_5X \cdot CR_2Y$ , wherein Y is a member of 1 or 2 groups, e. g.,  $CH_3$ ,  $-CH_2CH_2-$ , O, S or NH. Specified classes of substances are aryl benzimidazoles, aryl benzothiazoles and 2-aryl indoles. In examples, the compds are (1) Na phenylbenzimidazolesulfonate, dissolved in  $H_2O$  and mixed with

starch, tragacanth, alc and glycerol, (2) 2-p-tolylbenzimidazole, dissolved in wool fat, and (3) 2-(p-methoxyphenyl)benzimidazole, dissolved in wool fat.

**Cosmetic preparations** Berta Hawck (nee Hosp). *Ger.* 622,372, Nov. 27, 1935 (Cl. 33h. 13). Cosmetic preps which are ordinarily applied as powders are made into pastes or liquid compns. with the aid of liquids which readily evaporate at the temp of the body. A mixt. of alc and  $CCl_4$  is suitable.

**Apparatus for irradiating wool fat or wool fat alcohols for use in cosmetic creams.** Leo-Werke G m b H. *Ger.* 622,378, Nov. 27, 1935 (Cl. 33h. 13).

**Hair cream** Nicolas Marchlone. *Fr.* 787,918, Oct 1, 1935. The cream contains henna 4, S 15, quinine 1.5, camphor 5, castor oil 1, formal 4.5 g, alc. of 38° 11 and a perfume.

**Hair-waving solution.** Frederic Maeder. *Brit.* 435,213, Sept 17, 1935. Hair is permanently waved by unpregnating with a soln of a sulfide, e. g.,  $NH_4HS$  2.5,  $H_2O$  97.5 parts, to which an alkali, e. g.,  $NH_3$ , is preferably added, and heating to a low temp. Org substances forming colloidal solns or suspensions, e. g., keratin, gelatin, agar agar, gum arabic, starch, resins, dextrin, shellac, viscose, may be added to the soln.

**Tobacco** Tabakforschungsanstalt für das Deutsche Reich. *Ger.* 619,680, Oct. 6, 1935 (Cl. 79c. 1). Tobacco is fermented in pressed bales containing insulating layer of cotton wool which may contain aromatic substances to flavor or scent the tobacco.

## 18—ACIDS, ALKALIES, SALTS AND OTHER HEAVY CHEMICALS

E. M. SYMMES

**Development of sulfuric acid production in Hungary** Laszlo Szathmari. *Magyar Gyógyászati és Társaság Értékezt.* 11, 634-63(1935).—Historical. S S de F. Conditions in the heavy-chemical industry in 1935. *P. Parish Chem. Age* 33, 587-60(1935). E. H.

**Active manganese dioxide from pyrolusite** V. V. Kozlov and T. I. Vol'fson. *J. Chem. Ind. (Moscow)* 12, 1107-71(1935).—The practical details for the extn of pyrolusite with  $HCl$ , pptn of  $Mn(OH)_2$  by  $NaOH$  and oxidation of the  $Mn(OH)_2$  with  $Cl_2$  to give  $MnO_2$  of high oxidizing power are described. H. M. Leicester.

**Determination of sulfur dioxide and sulfur trioxide in sulfur burner gases** L. Sokkela. *Finnish Paper and Timber J.* 1935, 1022.—By means of an aspirator, the gases are passed through 20 cc. of 0.1 N  $NaOH$  + 0.001 M  $SnCl_4$  as the absorption liquid. The  $SnCl_4$  prevents oxidation of the sulfites to sulfates. For a larger amt of gases 2 drops of methyl orange is added to the soln. The  $H_2SO_4$  formed from the  $SO_2$  is pptd with benzidine hydrochloride, filtered in a Buchner funnel and titrated in the usual manner with 0.1 N  $NaOH$ . For best results, gas washing bottles with glass filters should be used. Rubber tubing should be avoided, as  $SO_2$  is oxidized to  $SO_3$ , giving high values for the latter. I. E. Jukkola.

**The removal of fluoride and silica from technical phosphoric acid** M. O. Dormish and M. V. Sladkova. *J. Chem. Ind. (Moscow)* 12, 1160-1(1935).—Addn of double the theoretical amt of solid  $KCl$  to com.  $H_3PO_4$  ppts up to 96% of the  $H_2SiF_6$  present without harming the acid. The amt of ppt varies somewhat with the source of the acid. H. M. Leicester.

**The nitrogen industry in 1935** E. B. Marted. *Chem. Age* 33, 601-3(1935). E. H.

**Obtaining sulfur from pyrites containing carbon** S. L. Kuz'ko. *J. Chem. Ind. (Moscow)* 12, 1140-62(1935).—Semi-factory scale exps on the process of Postnikov, Kur'min and Kirillov (*C. A.* 28, 6233) show that the best temp. for burning the ore is 1050° and for reducing the  $SO_2$  to  $S$  is 700-900°, at a gas speed of 21-42 cu. m. per hr. Details required for planning a factory are given. H. M. Leicester.

**Burning Shor Su sulfur ores** Kh. B. Medunkin, G. I. Antonenko, M. D. Trusov and P. I. Balaban. *J. Chem.*

*Ind. (Moscow)* 12, 1152-5(1935); cf. *C. A.* 29, 300°.—Nonindustrial S ores, such as tripoli, can be burned in a shaft furnace without reference to particle size. Details of the construction and operation of such furnaces are given. H. M. Leicester.

**Treatment of langbeinite** Tadeusz Kuczyński. *IX Congr. intern. quim. pur. aplicada, Madrid* 3, 421-31(1934).—See *C. A.* 29, 6370°. G. G.

**Utilization of leucite** Gino Gallo. *Chimie & Industrie* 34, 763-6(1935).—An outline is given of investigations carried out over a period of 10 yrs with a view to obtaining very pure  $Al$  (or  $Al_2O_3$ ) and  $K_2O$  from Italian leucite. The process finally adopted, which has given entirely satisfactory results in semicom. tests, is essentially as follows: the leucite is treated with  $H_2SO_4$ , and the crude alum pptd from the soln is crystd. at low temp.; the alum soln is treated with sufficient  $KOH$  to produce sol.  $KAlO_2$ , the impurities (consisting of Fe and possibly  $CaO$  and  $MgO$ ) being pptd as insol hydroxides and completely removed by filtration, the aluminate is decomposed by a current of  $CO_2$ , which gives  $K_2CO_3$  and a very pure, insol, cryst.  $Al_2O_3$ , which filters easily, by concn. of the filtrate  $K_2CO_3$  is sepd and then treated with  $Sr(OH)_2$ , the  $K_2O$  which remains in soln is in the form of  $K_2CO_3$ . A. Papineau-Couture.

**The characteristics of botation talings and pyrite concentrates from the Degtyr region.** M. T. Lukina. *J. Chem. Ind. (Moscow)* 12, 1156-9(1935). H. M. L.

**Recent observations on the decomposition of raw phosphates with sulfuric acid** Hans Lehecke. *Tek. Tid. Uppf. & Kem.* 65, 81-5, 92-4(1935).—The reactions taking place by the treatment of raw phosphates with  $H_2SO_4$  in the manu. of  $H_2PO_4$  and phosphatic fertilizers are discussed. In the manu. of  $H_2PO_4$ , the extent to which the  $CaSO_4$  formed is hydrated depends on the temp. and concn. The less the  $CaSO_4$  is hydrated, the better its filtering properties. The conditions under which the hemihydrate is formed is expressed by  $t = -0.513 P + 0.0087 P^2 - 0.000333 P^3 + 107$ , where  $t$  is the temp. and  $P$  the concn. of  $H_2PO_4$  in percentage of  $P_2O_5$ . This equation is also shown graphically, the field below the curve representing  $CaSO_4 \cdot 2H_2O$ , the field above  $CaSO_4 \cdot \frac{1}{2}H_2O$ . At 0°  $P_2O_5$ , the curve intersects the ordinate at 107° which,

as established by van't Hoff, is the transition point between  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  and  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ . The  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  formed under these conditions is, however, readily hydrated by subsequent washing on the filter and the resulting product greatly retards the filtration. When the process is regulated so as to conform to the equation  $t = 6.032P + 0.0306P^2 + 254$  a hydration resistant  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  results. The corresponding curve, when extrapolated to the ordinate, indicates an ideal value at  $265.4^\circ$ . However,  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  is converted to nonhydrating anhydrite in water at  $190^\circ$  without initial formation of the stable  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ . It is, therefore, assumed that the presence of  $\text{H}_3\text{PO}_4$  renders  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  stable at the temps and concns indicated by the curve. At still higher temps.  $\text{CaSO}_4$  is obtained as a stable anhydrite, which also gives good filtration. The temp concn curve in this case follows that of stable  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  but is located  $15\text{--}20^\circ$  higher.  $\text{H}_3\text{PO}_4$  contg  $40\text{--}45\%$   $\text{P}_2\text{O}_5$  as obtained, suffices in strength for most purposes, but in view of the destructive action of this on the filter materials and the high heat consumption, operating at lower temps and concns. ( $30\text{--}32\%$   $\text{P}_2\text{O}_5$ ) may be more economical. Contrary to previous experience, it was also possible to obtain  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , which forms under these conditions in a filterable form when certain precautions were taken. Recent developments in the filtration and concn of  $\text{H}_3\text{PO}_4$  are described and illustrated. The mechanism of the reactions taking place by the mann of superphosphate (cf. C. A. 29, 3, 938, 4329) and the after-hardening of this are discussed. From detns of the water of crystn and Debye-Scherrer x-ray photographs of  $\text{CaSO}_4$  in superphosphate, it is concluded that  $\text{CaSO}_4$  is primarily present as anhydrite, which does not take up water. A minor amt. (max.  $5\%$ ) is also present as stable  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$  in the form of very small, poorly developed crystals. This could be expected in view of the concns and temps employed. A hydration of  $\text{CaSO}_4$  is, therefore, not the reason for the after-hardening of superphosphate. The x-ray photographs of fresh and of 6-yr.-old double superphosphate indicate that  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  originally is formed as an amorphous gel, which very slowly attains a cryst. form. The hardening is then assumed to originate from a progressive formation of a gel of  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  interspersed with  $\text{CaSO}_4$  crystals and a minor amt. of cryst.  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ , while the liquid phase diminishes as a result of a progressive reaction between primarily formed  $\text{H}_3\text{PO}_4$  and still undecomposed  $\text{Ca}_3(\text{PO}_4)_2$ . A brief review of recent developments in the mfg. methods of superphosphate is given. Cf. C. A. 29, 543, 4339, 6714. D. Thuesen.

The properties of zinc-chromium catalysts for the synthesis of methanol. K. I. Ivanov and V. I. Gusev. *J. Chem. Ind. (Moscow)* 12, 1143-6 (1935).—When a catalyst of  $8\text{ZnO} \cdot 1\text{Cr}_2\text{O}_3$  is first used, it is most efficient at  $370^\circ$ , but as its use is continued, the optimum temp. rises slowly to  $400^\circ$ . Very high rates of passing the gas over it favor MeOH formation, while low rates of passing the gas, or temps. above  $400^\circ$  begin to favor  $\text{CH}_4$  formation.

H. M. Leicester

Roasting and sintering dusts and powdery ores [phosphatic rocks] (Saint-Jacques, Poupert) 9.

Hydrogen halides. Albert M. Clifford (to Wingfoot Corp.). U. S. 2,028,687, Jan. 14. Equivalent proportions of a halogen such as Cl or Br are treated with  $\text{H}_2\text{S}$  in the presence of a S halide such as  $\text{S}_2\text{Cl}_2$  or  $\text{S}_2\text{Br}_2$  and the product may be washed with a solvent such as xylene. Various details of operation are described.

Nitric acid. Fredrik W. de Jahn (to Frank A. Bower). U. S. 2,027,578, Jan. 14. Acid gases under a pressure which may be about 7 atm. are passed into a plurality of sep. absorption chambers, through each of which acid is circulated, and the acid is cooled and maintained in surface contact with the gases under pressure, and acid is drawn off from the chambers. App. is described. This pat. was issued on an application filed Feb. 2, 1928.

Nitric acid. Nikodem Caro and Albert R. Frank. Ger. 351,166, Dec. 11, 1935 (Cl. 12). 26).  $\text{NH}_3$  or  $\text{HCN}$  is

1 catalytically oxidized with O at atm. or raised pressure at a temp. which is regulated in known manner by addn. of steam or H to the mixt. Water is then condensed as completely as possible from the products before the oxides of N are worked up in known manner to concd.  $\text{HNO}_3$ .

Concentrated nitric acid. Emil Lüscher (to Lonza Elektrochemische und chemische Fabriken A.-G. (Gampell)). U. S. 2,028,402, Jan. 21. In a continuous process of producing concd.  $\text{HNO}_3$  from water or dil.  $\text{HNO}_3$ , N oxides, and O at superatm. pressure, the downwardly flowing liquid reaction components and reaction products are brought into contact with upwardly flowing O and gases and vapors through a temp. gradient obtained by heating the acid accumulating at the lowest point of the path of the liquid enriched by  $\text{HNO}_3$  and contg. an excess of  $\text{N}_2\text{O}$ , to a temp. corresponding at the pressure used to the boiling temp. of an acid having the highest desired  $\text{HNO}_3$  concn and having a content of about  $5\text{--}20\%$   $\text{N}_2\text{O}$ , and the rising hydrous vapors are condensed by cooling at the highest point of the gas path, so that the reaction process is carried out in the form of a fractional distn., the lower N oxides are continually carried upward and  $\text{N}_2\text{O}$  remains in such quantity in the liquid trickling downward that fresh  $\text{HNO}_3$  is constantly formed in the downwardly trickling liquid and the liquid  $\text{HNO}_3$  having the desired highest content of  $\text{HNO}_3$  and a content of about  $5\text{--}20\%$   $\text{N}_2\text{O}$  is obtained at the lower end of its path and may be collected. App. is described.

Purifying hot burner gases in sulfonic acid manufacture. Bernard M. Carter (to General Chemical Co.). U. S. 2,028,318, Jan. 21. For eliminating deleterious impurities from hot burner gases in the contact- $\text{H}_2\text{SO}_4$  process, the hot gas contg. water vapor is cooled in stages to cause condensation of the water vapor together with impurities and  $\text{H}_2\text{SO}_4$  contained in the gas, the condensate from an initial and from a subsequent stage of the cooling being separately collected, initial condensate being discarded and subsequent condensate being retained so that impurities are eliminated with the initial condensate and  $\text{H}_2\text{SO}_4$  is separated with the subsequent condensate. An arrangement of app. is described. Cf. C. A. 29, 2074.

White alkali cyanide. Grangers Manufacturing Co. Ger. 619,823, Oct. 28, 1935 (Cl. 12b 11). The alkali cyanide, especially in cast sticks, is obtained by heating the cyanides to temps. above the m. p., e. g.,  $50\text{--}100^\circ$ , and casting. Thus NaCN from sugar-beet slops is heated to  $1200^\circ$  and cast at  $650^\circ$ .

Alkali dichromates. Ernst Hackhofer and Annemarie Beuther (to I. G. Farbenind. A.-G.). U. S. 2,027,477, Jan. 14.  $\text{CO}_2$  under superatm. pressure is caused to react with an alkali monochromate soln. at a temp. of at least  $80^\circ$  (suitably about  $80\text{--}85^\circ$ ) and the soln. is cooled down during the reaction and alkali bicarbonate is sep'd from the dichromate formed by filtration.

Alkali nitrates. I. G. Farbenind. A.-G. Fr. 788,821, Oct. 11, 1935. An alkali metal chloride is suspended in a liquid said with  $\text{NH}_4\text{Cl}$  and an alkali metal chloride and to the suspension is added, preferably with agitation and cooling,  $\text{NH}_4\text{NO}_3$  dissolved in a liquid of the same compn. By this means crystals of  $\text{NaNO}_3$  of 1-2 mm. grain size may be obtained.

Alkali titanates. Titanreselschaft m. b. H. (Joachim Rockstroh, inventor). Ger. 619,568, Oct. 3, 1935 (Cl. 12a, 59).

Trialkali phosphates and aluminas. Chem. Werke vorm. H. & E. Albert A.-G. (Ferdinand Bornemann and Hans Huber, inventors). Ger. 622,229, Nov. 22, 1935 (Cl. 12a 31). Crude material contg.  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ , with or without  $\text{Fe}_2\text{O}_3$ , is dissolved as far as possible in concd.  $\text{H}_2\text{PO}_4$  soln. and after sep'n. of undissolved matter the soln. is treated with sufficient alkali carbonate to form trialkali phosphate and alkali aluminate. The soln. is then evap'd. and the residue is calcined and dissolved in water. The soln. is filtered and trialkali phosphate is recovered by crystn.  $\text{Al}_2\text{O}_3$  is recovered in known manner from the alkali aluminate in the mother liquor.

Copper salts. Dambrover Kupferhütte and I. G. Farbenind. A.-G. Fr. 787,927, Oct. 1, 1935. Solns.,

prepd. by leaching residues of roasted pyrites, calcined after the addn. of NaCl and contg. Cu are treated so as to ppt. the Cu in the form of a salt more difficultly sol. than the corresponding salts of other metals in the soln. Such salts are  $\text{Cu}(\text{OH})_2$ ,  $\text{CuCl}_2$ ,  $\text{CuCl}$  and a double sulfate of Cu and Na.

**Aluminum chloride.** Ruhrchemie A.-G. Fr. 788,120, Oct. 4, 1935. The attack of Al by dry gaseous HCl is carried out in the presence of  $\text{AlCl}_3$  in media capable of the Friedel-Crafts synthesis. The  $\text{AlCl}_3$  may be present as addn. compds.

**Carbon disulfide.** Michael J. Leahy. U. S. 2,026,840, Jan. 7. A mixt. contg. finely divided C and S together with  $\text{CS}_2$  is heated in the absence of free O to a temp. sufficiently high to form addn.  $\text{CS}_2$  from the C and S.

**Copper sulfate.** Marcel Serciron. Fr. 788,242, Oct. 7, 1935. A liquor contg. HCl in soln. or an appropriate chloride and  $\text{NH}_4$  ions is caused to attack Cu in the presence of air or O and the Cu salt is afterward converted to sulfate or the attack may be carried out in the presence of  $\text{H}_2\text{SO}_4$ .

**Hydrogen peroxide.** B. Laporte Ltd., Isaac E. Weber and Wm. S. Wood. Brit. 439,401, Sept. 17, 1935. The stability of  $\text{H}_2\text{O}_2$  solns. is increased by adding  $\text{HPO}_3$  or a salt thereof and an org. stabilizer, e. g., acetanilide, phenacetin, salicylic acid, Na salicylate.

**Stabilizing hydrogen peroxide solutions.** Joseph S. Reichert (to E. I. du Pont de Nemours & Co.). U. S. 2,027,838, Jan. 14. A small proportion of a pyrophosphate (suitably about 0.15% or less of  $\text{Na}_2\text{P}_2\text{O}_7$ ) is added and the acidity of the soln. is adjusted to a pH between 1 and 7. U. S. 2,027,839 relates to purification of a  $\text{H}_2\text{O}_2$  soln. by pptg.  $\text{Sn}(\text{OH})_4$  in it at a pH of above 14. Cf. C. A. 29, 5230<sup>a</sup>.

**Hydrogen sulfide.** Raymond F. Bacon. Ger. 619,693, Oct. 10, 1935 (Cl. 12: 18). App. for obtaining  $\text{H}_2\text{S}$  by treating metal sulfides, especially  $\text{FeS}$ , with HCl is described. The  $\text{FeCl}_2$  soln. produced is evapd. to dryness, the steam produced being heated and passed over the dry  $\text{FeCl}_2$  to give  $\text{FeO}$  and HCl, the HCl is used to treat further  $\text{FeS}$ .

**Iron chlorosulfate.** Röhm & Haas A.-G. Fr. 788,094, Oct. 3, 1935. Cl is introduced up to satn. into a ground mixt. of solid anhyd.  $\text{FeSO}_4$  and solid cryst.  $\text{FeSO}_4$  in a closed vessel at normal temp. and slightly increased pressure, the proportion of the anhyd. and cryst. salts being so calcd. that the resulting  $\text{FeSO}_4\text{Cl}$  contains 6 mols. of water of cryst.

**Basic magnesium carbonate.** Samuel A. Abrahams (to Plant Rubber & Asbestos Works). U. S. 2,027,714, Jan. 14. Basic Mg carbonate for insulation is prepd. by adding  $\text{Na}_2\text{CO}_3$  and  $\text{NaHCO}_3$  to a Mg salt soln. such as a buttern in amts. suitable for pptg. an initial basic Mg carbonate ppt. which is transformable to an altered form and then introducing steam into the mixt. of ppt. and assoc. liquid until its temp. is about 70-83° for transforming the initial ppt. to a relatively light strongly bonded form having increased volume per unit of wt. and increased bonding properties.

**Syngentate, potassium sulfate.** Henry E. Z. Raczowski (to Palestine Potash Ltd.). Brit. 435,772, Sept. 27, 1935. See Fr. 776,937 (C. A. 29, 3789<sup>a</sup>).

**Sodium azide.** Dynamit A.-G. vorm. Alfred Nobel & Co. Ger. 619,753, Oct. 7, 1935 (Cl. 12: 30). In prep.  $\text{NaN}_3$  from  $\text{NaNH}_2$  and  $\text{N}_2\text{O}$ , an excess of mixed  $\text{NaN}_3$  and NaOH is added to stimulate the mass action.

**Sulfur dioxide.** Metallgesellschaft (Conway Freherr von Gursewald, Hans Weidmann and Gerhard Roesner, inventors). Ger. 621,529, Nov. 8, 1935 (Cl. 12: 21). Addn. to 606,447 (C. A. 29, 1594<sup>a</sup>). See Fr. 771,452 (C. A. 29, 895<sup>a</sup>).

**Sulfur trioxide.** Charles F. Silsby (to General Chemical Co.). U. S. 2,028,416, Jan. 21. In making  $\text{SO}_3$  by the roasting of sulfide ore and conversion of the resulting  $\text{SO}_2$  to  $\text{SO}_3$  by the contact process, the sulfide ore is permitted to fall in a continuous stream through the roasting zone countercurrent to a rising stream of air supporting its combustion and the supply of air admitted is regulated so

that the concn. of free O in the  $\text{SO}_2$  gas is 5-9% and the ore is caused to fall through an active oxidation zone becoming gradually richer in O in the direction of fall of the ore particles. App. is described.

**Titanium dioxide.** Reginald H. Monk and Archibald S. Ross (to American Zinc, Lead & Smelting Co.). U. S. 2,028,292, Jan. 21. A soln. of a hydrolyzable salt of Ti such as the sulfate is added with water which may contain about 0.5% of a colloid such as a complex polysaccharide, e. g., dextrin, followed by heating to convert the polysaccharide into sugar by boiling.

**Zinc chloride.** I. G. Farbenind. A.-G. (Hans Dohse and Fritz Spoun, inventors). Ger. 619,694, Oct. 5, 1935 (Cl. 12: 6).  $\text{ZnCl}_2$  is obtained by treating sal ammoniac slag or similar slag contg. Zn and  $\text{NH}_4$  salts, with Cl at high temps. Thus, slag contg. 41.3%  $\text{ZnCl}_2$ , 27.4%  $\text{ZnO}$ , 17.9%  $\text{Zn}(\text{OH})\text{Cl}$ , 10.0%  $\text{NH}_4\text{Cl}$  and 3.0%  $\text{SiO}_2$  and Fe compds. is suspended in water, heated to 100° and treated with Cl to give a soln. of  $\text{ZnCl}_2$ .

**Zinc sulfide.** Samuel Kremen. U. S. 2,027,440, Jan. 14. An intimate mixt. of a dil. 7n amalgam with S is heated until reaction is completed, the Hg is distd. off, and the residue is heated until any  $\text{HgS}$  formed is driven off from the  $\text{ZnS}$ .

**Basic alums.** Kalunite Co. Ger. 622,230, Nov. 23, 1935 (Cl. 12: 7). See U. S. 1,914,175 (C. A. 27, 435<sup>a</sup>).

**Colloidal solutions of cerium salts.** Chem. Fab. von Heyden A.-G. (Carl Hermann v. Hoessle, inventor). Ger. 622,160, Nov. 21, 1935 (Cl. 12: 9). Water insol. or sparingly sol. Ce salts are pptd. in the presence of a protective colloid and the ppt. is ground in water with the addn., if necessary, of a further amt. of the protective colloid.

**Apparatus for producing hydrogen by reaction of hydrocarbon gases with steam.** Wm. V. Hanks, Geo. H. Freymuth and Geo. Potts (to Standard Oil Development Co.). U. S. 2,028,326, Jan. 21. Various structural details.

**Iodine from brines.** Leonard C. Chamberlain and Geo. W. Hooker (to Dow Chemical Co.). U. S. 2,023,099, Jan. 14. Iodine is liberated in a natural I bearing brine or the like and is blown out with a current of air, adsorbed by active carbon while maintaining the current at a temp. above its dew point, and the I is extd. from the carbon with a hot soln. of an alkali such as KOH. An arrangement of app. is described.

**Krypton and xenon.** Vereinigte Glühlampen und Electricitäts A.-G. Fr. 788,304, Oct. 8, 1935. Kr and Xe are extd. from gases contg. them by washing with a liquefied gas which has a vapor tension lower than that of Kr and Xe, these gases being more sol. in the washing liquid than the other constituents of the gaseous mixt.

**Active carbon.** Andrew McCulloch and Robert E. Hargraves (to Carbo-Norit-Union Verwaltungs G. m. b. H.). U. S. 2,027,695, Jan. 14. See Fr. 765,716 (C. A. 28, 636<sup>b</sup>).

**Active carbon.** Robin G. W. Farnell. Brit. 435,345, Sept. 19, 1935. Active C of low apparent d. is obtained by leaching the black ash residue resulting from the digestion of esparto grass and contg. mainly pectocellulose with  $\text{H}_2\text{O}$  until it has a pH of 9-10, heating to 700-1000° in the presence of limited amts. of air or O and continuing the heating with excess of air or O at 400-500°. To increase the d. of the product, a proportion of wood, wood charcoal, coconut shell or charcoal therefrom, corozo nuts, apricot stones, etc., may be added to the black ash residue.

**Active adsorbent silica.** Herbert J. Wollner and John V. Shinn (to General Chemical Co.). U. S. 2,027,948, Jan. 14. See Can. 352,787 (C. A. 29, 825<sup>a</sup>).

**Catalyst for reduction of sulfur dioxide.** Mautland C. Boswell. U. S. 2,026,819, Jan. 7. A hydrated sulfide of a metal of the Fe group or a mixt. of hydrated sulfide material with hydroxides or oxides is partly dehydrated, in the absence of air, and the partly dehydrated material is subjected to the action of O to increase its susceptibility to the action of  $\text{SO}_2$ ; it is then subjected to the action of  $\text{SO}_2$  at a temp. above room temp. (suitably about 100-

200°) and the material is finally subjected to the action of H and SO<sub>2</sub> at a higher temp. (suitably about 200–300°) to prepare the catalyst.

Crystalline mercurous sulfate suitable for use as a catalyst. Ronald J. Baird. U. S. 2,027,364, Jan. 14 A soln. contg. about 2% H<sub>2</sub>SO<sub>4</sub> and 10% NaNO<sub>2</sub> is brought into contact with Hg and treated with a d. c. while using the Hg as anode and maintaining a c. d. at the anode of about 0.5–1 amp per sq. decimeter. App. is described.

Regenerating spent tungsten oxide catalysts. Ralph I. Brown (to Atmospheric Nitrogen Corp.) U. S. 2,027,855, Jan. 14 Spent catalysts which are relatively insol in aq. NH<sub>3</sub> and which may have been used in HOAc

1 synthesis from MeOH and CO are dissolved in a soln. of a strong base such as NaOH to form a sol. tungstate and the soln. thus obtained is acidified (suitably with HNO<sub>3</sub>) to ppt. W oxide; the ppt. is dissolved in NH<sub>4</sub>OH and the soln. thus formed is acidified to ppt. a catalytically active W oxide.

Vanadium catalyst for promoting oxidation reactions. Edward S. Johnson (to Calco Chemical Co.). U. S. 2,027,316, Jan. 7. A catalyst suitable for promoting reactions such as the production of SO<sub>2</sub> from SO<sub>3</sub> comprises a V compd. homogeneously incorporated in gelatinous curds of hydrous SiO<sub>2</sub>, produced by acidification of a silicate soln. under conditions alk. or neutral to Congo

## 19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. E. BARTON AND C. H. KERR

Glass making in Southern California. Roy L. Swain. *Glass Ind.* 16, 333–7 (1935).

Thuringian glass industry. David H. Buffum. *Glass Ind.* 16, 351–4, 357 (1935).

Performance history of glass tanks. J. W. Romig. *Glass Ind.* 16, 271–2 (1935).—Tabulations of the properties and behavior are given to aid in the analysis of glass tanks as production units.

Physical chemistry of glass. Pierre Gilard. *Glass Ind.* 17, 17–21, 27 (1936), cf. C. A. 30, 585.—A general statement of the present status of glass technology.

Microchemical analysis of glasses. VIII. The determination of arsenic in glasses. W. Geilmann and O. Meyer-Hoissen. *Glastech. Ber.* 13, 420 (1935), cf. C. A. 29, 3794.—In detg. the As in glasses it is recommended to volatilize it as arsine in the usual way and to det. the latter. Very small ams. up to 0.025 mg. are obtained colorimetrically with HgCl<sub>2</sub> paper, while for ams. from 0.025 to 10 mg. gravimetric detn. of the reaction product of the arsine with HgCl<sub>2</sub> soln. is useful. Total As is obtained by alk. decompn. of the glass, while quinquavalent As can be detd. by decompn. of the glass with HF-H<sub>2</sub>SO<sub>4</sub> mixt. and fuming off of the As<sub>2</sub>O<sub>3</sub>. The trivalent As is obtained by difference.

Influence of composition on the color of sulfur amber glass. K. Litzow and G. Brooks. *Glass Ind.* 17, 12–13 (1936); *Sprechsaal* 69, 51–3 (1935).—The reducing action of C, Zn, Si and Al on Na<sub>2</sub>SO<sub>3</sub> to produce colors in glass is studied. A definite C/Na<sub>2</sub>SO<sub>3</sub> ratio was observed for each strength of color produced. Al sticks worked as well as powd. Al, while Zn gave no color.

Fining of soda-lime magnesia glasses. A. E. Badger. *Glass Ind.* 16, 369 (1935).—Substitution of lime for part of the dolomite produced a marked improvement in the fining of the glass melted in closed pots of 2000-lb. capacity.

Clays. J. LeBraz. *Glaces et verres* 1935, No. 45, 2–8.—Their use in glass manuf. is discussed. H. S. W. Structure of phosphorescent glasses. A. Schloemer. *Glastech. Ber.* 13, 424–5 (1935); cf. C. A. 27, 4046.—The view of D. Dobischek (*Ibid.* 13, 370 (1935)) that the phosphorescence of certain glasses examd. could be ascribed to a certain cryst. fine structure is untenable, since nothing is known of P in the vitreous condition.

Defects of glass. Th. Ziehner. *Glas u. App.* 16, 237–8, 247–8 (1935).—The factors influencing the mech. strength of glass are discussed, as well as the causes of failures in operation and use of glass. Examples illustrate such defects.

Survey of insulation practice. C. W. Parmelee and A. E. Badger. *Glass Ind.* 16, 274–7 (1935).—The results are published of a questionnaire to continuous glass tank works to det. the value of insulation in the several parts of the system.

The situation of safety glass made with cellulose as the intermediate layer. Fritz Ohl. *Nitrocellulose* 6, 120–3 (1935).—A review.

Laminated safety glass. J. W. H. Randall. *Chemist* 12, 367–73 (1935).

Old Brussels faience. H. Nicaise. *Verre silicates* 16, 457–60 (1935).

Solid reactions at 1000° to 1200° between MgO or BeO and Ni, Fe, Cr, Mn and their oxides. Louis Navias. *J. Am. Ceram. Soc.* 19, 1–7 (1936).—The elements Ni, Fe, Cr and Mn and their oxides NiO, Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub> and Mn<sub>2</sub>O<sub>3</sub>, in finely divided state, were brought into contact with granular, fused MgO in loose form and in pressed disks and also into contact with slabs of high-fired MgO and BeO. Max temps. were 1000°, 1100° and 1200° in air firings. The degree of reaction was noted by the extent of discoloration of the MgO and BeO near the contact surface. Ni showed the least reactivity, Fe and Cr next, and Mn the most. The elements in oxidizing atm. caused more reaction than their oxides in all cases. There was less reaction with BeO than with MgO with corresponding contact material. Firings were also made with these elements and oxides in contact with pressed disks of fused MgO in an atm. of H<sub>2</sub>. Ni and Fe showed no reaction, Cr very slight, and Mn slightly more. NiO and Fe<sub>2</sub>O<sub>3</sub> were reduced to Ni and Fe without reaction, whereas Cr<sub>2</sub>O<sub>3</sub> did not reduce and gave no reaction, and Mn<sub>2</sub>O<sub>3</sub> became semi-sintered and gave a very slight reaction. When air or N<sub>2</sub> was passed, dry or moistened with H<sub>2</sub>O, through a mixt. of fused MgO with Mn, MnO<sub>2</sub> or Cr powders at 1200°, most reactivity was noted when the wet air was used.

Jars for preservation of food. A. Shtern. *Konservnaya Prom.* 5, 24–5 (1935).—It is proposed to manuf. jars from a mixt. of different slags and refractory clays. The heat conduction is high. Meat and fish preserved in slag jars were as good as those preserved in tinware. Although the jars are resistant to acids, glazing is necessary. A semifactory production has been started.

Effect of a tremolitic talc in whiteware bodies. Donald Hagar. *J. Am. Ceram. Soc.* 19, 14–23 (1936).—Wall-tile bodies of low moisture expansion and high resistance to delayed crazing can be developed by using tremolitic talc with other body materials. Clay, pyrophyllite and flint are best for use in such bodies. Sometimes limited ams. of feldspar may be advantageous. Bodies of lowest moisture expansion can be developed at cone 10, with either intermediate (20–40) or high (over 60) percentage of talc, but bodies matured at cone 10 require special glazes to fit them. Bodies of high crazing resistance, low shrinkage, good strength and long firing range can be produced at cones 1 and 6. While such bodies do not have moisture expansions as low as those matured at cone 10 they are easier to manuf. and more resistant to crazing than the usual com. wall tile.

Raw leadless glazes for pottery and tile at cone 2. C. W. Merritt. *J. Am. Ceram. Soc.* 19, 23–5 (1936); cf. C. A. 29, 3478.—The base glaze chosen for the color series was ZnO 0.30, K<sub>2</sub>O 0.20, CaO 0.25, BaO 0.10, MgO 0.15, Al<sub>2</sub>O<sub>3</sub> 0.22, SiO<sub>2</sub> 2.50, B<sub>2</sub>O<sub>3</sub> 0.375 equiv. Colemanite was the chief flux, the batch being calcined

ZnO 24.3, Buckingham feldspar 113.2, colemanite 51.5,  $\text{BaCO}_3$  19.7, steatite 18.9, flint 60.0,  $\text{Ca}(\text{NO}_3)_2$  2.9 lb. The results with many coloring materials and their blends are given. C. H. Kerr

Red glazes and underglaze red by reduction. C. M. Harder. *J. Am. Ceram. Soc.* 19, 26-8 (1936).—Various formulas are given and requirements in the firing procedure. Consistent development of the red color is dependent upon uniform evenly circulating kiln gases and a proper relation of firing procedure to glaze fusibility. C. H. Kerr

Recent developments and trends in refractory processes and materials. Louis J. Trostel. *Iron Age* 136, No. 21, 18-21, 90-4, No. 23, 26-9, 95 (1935).—See C. A. 29, 6024. F. G. Norris

Advanced and refined technic in the petrographic study of crystalline refractories. R. B. McCormick. *J. Am. Ceram. Soc.* 19, 7-13 (1936).—The new technic for the determination of minerals, as developed by R. C. Emmons, in C. A. 23, 794, is described. There is a discussion of immersion media and a description of the double-variation app., the modified universal stage, and the smithsonite refractometer. C. H. Kerr

Semiacid refractory materials. S. A. Zhukharevich and G. L. Kogan. *Sovetskii Rekonstruktsiya Nauka* 1935, No. 5, 164-5.—Brick for lining open hearth furnaces made of a mixt. of cryst. gravels and plastic refractory clays of low bonding power stood more fusions than the usual refractory brick. B. V. Shvartzberg

Refractories for the electrothermic zinc industry. Winfield B. MacBride. *Bull. Am. Ceram. Soc.* 14, 89-93 (1935).—A low  $\text{Al}_2\text{O}_3$  refractory of low porosity has given the best service thus far in the electrothermic smelting furnaces at Josephstown. Other types tried included high  $\text{Al}_2\text{O}_3$ ,  $\text{SiC}$  and magnesite. After extensive tests it was decided that the refractories should meet the following conditions: (1) low porosity to prevent Zn penetration, (2) high refractoriness to prevent softening, (3) high elec. resistance at high temps., and (4) high resistance to spalling and gradual disintegration due to slag, vapor of gas. C. H. Kerr

Improvements made and desired for glasshouse refractories. W. L. Fabian. *Glass Ind.* 17, 5-8 (1936).—A survey of 28 refractory plants showed marked improvement in quality of products as well as in mfg. methods. Various specifications for tank blocks for glass furnaces are summarized. H. F. K.

Chemicals in the enameling industry—changes in the demand for opacifiers. W. Astles. *Chem. Trade J.* 97, 527-8 (1935).—The most common opacifiers are  $\text{SnO}_2$ , as a mill addn.,  $\text{ZrO}_2$  and silicate as mill and melt addn., and  $\text{Sb}_2\text{O}_3$  as a melt addn. There is no difference in actual sp. gr. of the medium and the light  $\text{Sn}$  oxides. The higher oxides of  $\text{Sb}$  have little, if any, toxicity. Na. Zr silicate increases the acid-resisting properties of enamels. W. H. Boynton

Opacifying enamel ware. Richard Aldinger. *Glashutte* 65, 639-42 (1935).—Studies of fluorides as opacifiers in glasses yielded valuable information. Cryolite and NaF are the most stable and are therefore suitable for any type of enamel. Fluorspar and especially Na fluosilicate are more suitable for enamel batches rich in alkalis and having a low m. p., their use is discussed and examples are given. M. V. Kondody

Melting enamel. L. Vielhaber. *Enamelauren-Ind.* 12, 313-15 (1935).—The melting of enamel in crucibles and tank furnaces, and factors affecting it are discussed. M. V. Kondody

Working enamel batches. L. Vielhaber. *Enamelauren-Ind.* 12, 306-8 (1935).—A discussion of the working of raw materials for enamels and of the effect of structure and moisture of raw materials, grinding, mixing, drying, firing, etc. M. V. Kondody

Feldspar in enamel. L. Vielhaber. *Enamelauren-Ind.* 12, 290-1 (1935)., cf. C. A. 29, 2674.—Evaluation of feldspar by chem. analysis is misleading. Feldspars differing only slightly in compn. may have very different

mineralogical compns. and different thermal expansions. M. V. Kondody

Cerium and zirconium enamels for bathtubs. L. Vielhaber. *Enamelauren-Ind.* 12, 289-90 (1935).—Comments on the paper by Paquet (C. A. 29, 6007) dealing with the use of Ce and Zr in enamels for bathtubs. M. V. Kondody

Yellow pigments in the enamel industry. L. Stuckert. *Glashutte* 65, 611-12 (1935).—The no. of yellow pigments is limited. Cadmium yellow and Naples yellow cannot be used in ware for cooking. Yellow pigments are often used to change the white color;  $\text{SnO}_2$  or another opacifier is added to the enamel batch. Naples yellow consists essentially of pyroantimonate of Pb contg. some ZnO and  $\text{Al}_2\text{O}_3$ . It is obtained by heating 2 mol.  $\text{Pb}(\text{NO}_3)_2$  with 1 mol.  $\text{Sb}_2\text{O}_3$  and from 0 to 1 mol. ZnO, ZnO may be partially replaced by  $\text{Al}_2\text{O}_3$ . The color tone and refractoriness vary according to compn., but the disccn. temps. are never reached in enamel practice. Heating is done in an oxidizing atm. to obtain the oxidation of Sb which otherwise would produce black spots. The yellow orange pigment thus obtained has a limited coating power, and therefore the addn. to the mill amounts to 6 or 8%. The yellow enamel is often applied on a strongly opacified white enamel. The cryst. form, color tone and stability of cadmium yellow depend on the method of production.  $\text{CdS}$  pptd. by  $\text{H}_2\text{SO}_4$ , when cold is the  $\alpha$ -modification and is used for light tones, when pptd. hot it is the  $\beta$ -form and is more stable. It is necessary to eliminate the free  $\text{H}_2\text{SO}_4$  and the  $\text{CdSO}_4$ . The sulfide in the enamel is a colorant and an opacifier at the same time, 2.5% is added to the mill. It is usually applied on a white enamel. Oxidation during the firing of enamel contg. Cd must be avoided. Vanadium yellow is produced by heating  $\text{PbO}$  and  $\text{ZrO}_2$  with V salts. It is the only yellow of high refractoriness used under glaze. It coats well and is resistant to chem. agents. When various proportions of Se are added to cadmium yellow, a complete gamut of the intermediary color tones is obtained. Cadmium yellow can be mixed with various blue pigments also. M. V. Kondody

Effect of light and moisture on enamel. L. Vielhaber. *Enamelauren-Ind.* 12, 260-1 (1935).—Enamels did not change color, while laquer tested under the same conditions was greatly affected by moisture. M. V. Kondody

Determining thermal conductivity of enamel. L. Vielhaber. *Enamelauren-Ind.* 12, 305-6 (1935).—A discussion of the studies of Dawhl (C. A. 29, 7603). It was found that thermal cond. of enamel depends first on the kind and size of pores and secondly on the chem. compn. of the enamel. A series of factors with the help of which thermal cond. can be calcd. from the chem. compn. is given. The value of 0.0027 can be taken as a mean value for ware enamel at 40°. M. V. K.

Fitted or unfitted ground-coat enamel. Hans Hardwiger. *Glashutte* 65, 738-40 (1935).—It is still impossible to use all unfitted ground coat for sheet iron enamels because of the chem. reactions occurring during firing. However, good results are obtained by properly mixing a fitted portion with an unfitted portion and increasing the time of firing. M. V. Kondody

Oxidation control in firing ground-coat enamels. Wesley G. Martin. *Ceram. Ind.* 25, 60, 62, 64, 72 (1935). Herbert S. Willson

Enamel for water boiler. L. Vielhaber. *Enamelauren-Ind.* 12, 258-61 (1935).—Impurities in boiling water have little or no effect on some enamels while others are greatly affected. This is probably due not to ordinary reactions between acids and bases, but to exchange processes. M. V. Kondody

Wavy and net-like structure of enameled ware. L. Vielhaber. *Enamelauren-Ind.* 12, 235 (1935).—Enameled surfaces sometimes show a reticular structure with slightly uneven surfaces of different color tones. This defect can be partly removed by slightly shaking or vibrating the ware just enameled. However, if the ground coat has such a structure, this will not help. The formation of uneven

surfaces is sometimes due, especially in Sb enamels, to a sepn. of opacifying constituents. M. V. Kondody

How the continuous enameling furnace affects copperheads H. L. Cook *Bull. Am. Ceram. Soc.* 14, 393-5 (1935) —Box-type furnaces seal over the metal more rapidly than do continuous furnaces and the ware thus fired has fewer copperheads. Firing conditions are different with a continuous furnace and new problems arise. Pickling practice used for box furnaces is not satisfactory for continuous furnaces. Copperheading is minimized by using proper frits. Often a treatment of 30 sec in the acid pickle or etching the acid pickle will greatly decrease copperheading. A Ni bath following the pickle is now used as standard practice. C. H. K.

Cleaning and preparation of metal parts Martin H. Kipper. *Ceram. Age* 27, 274-5 (1936). H. S. W.

De-enameling L. Viehhaber *Emailwaren-Ind.* 12, 300-1 (1935). —De-enameling is especially suitable for cast-iron ware notwithstanding repeated firing, and is easily done by sand blasting. The more resistant the enamel is to acids, the more difficult it is to remove it. The enamel can be treated by acids, alk. solns or fused alkalis. HCl,  $H_2SO_4$ , chromic acid and HF are used. The latter dissolved acid-stable enamels but its use is dangerous. Cold HCl and  $H_2SO_4$  dissolve soft enamels.  $MgCrO_4$  (40% soln.) is very effective. Hot NaOH (50%) dissolves enamel in several hrs. An alk. bath at 540° dissolves the enamel in several min. without attacking the metal. It is possible also to de-enamel by steam or solns under pressure. M. V. Kondody

De-enameling enameled ware Walter Obst *Emailwaren-Ind.* 12, 233-4 (1935). —Various patents and methods are discussed. M. V. Kondody

App. for heating glass sheets for cutting (Brit. pat. 435,343) 4. Applying refractory linings to rotary kilns (Ger. pat. 622,305) 20

Glass I. G. Farbenind A.-G. (Paul Huppert and Hans Georg Grimm, inventors). Ger. 622,346, Nov. 26, 1935 (Cl. 32b. 1). Strongly refracting glass contg. 20-49% of  $TiO_2$  is obtained in an uncolored form by adjusting the compn. of the initial mixt. in such a manner that the product contains 20-35% of alkali, the remainder being  $SiO_2$ , with or without  $B_2O_3$ . The alkali is added wholly or in part as nitrate, and the mixt. is fused in an oxidizing atm. at a temp. not above 1300°.

Gas-burner system for heating glass-melting tanks Glaswerke Ruhr A.-G. Ger. 622,234, Nov. 22, 1935 (Cl. 32a. 5).

Apparatus for manufacture of glassware such as bottles Alexander F. McNally. U. S. 2,026,694, Jan. 7. Various mech. and operative details.

Glass objects. Georges Delacroix and Henri Horast 1r. 788,409, Oct. 10, 1935. The primary materials used are mixed dry, then malaxed with a liquid agglutinant composed of an aq. soln. of one or more of the chem. substances entering into the compn. of the glass or one or more of the primary materials, so as to obtain for the fusion, agglomerates of any form, e. g., granules or briquets.

Press for making ply-glass. Adolf Kampler and Adolf C. Plötze. Ger. 619,943, Oct. 10, 1935 (Cl. 39a. 17.50).

Apparatus for making sheet glass Libbey-Owens-Ford Glass Co. Ger. 622,468, Dec. 2, 1935 (Cl. 32a. 23.01). See Brit. 409,189 (C. A. 28, 5949).

Apparatus for making sheets of glass. Pittsburgh Plate Glass Co. Fr. 788,454, Oct. 10, 1935.

Apparatus for rolling sheet glass. Schliesische Spiegelglas-Manufaktur Carl Tietzsch G. m. b. H. Ger. 622,467, Nov. 28, 1935 (Cl. 32a. 18). The rolled sheet, while still plastic, is passed between supplementary rollers having their axes inclined to the axes of the main rollers.

Bending glass sheets Wm. Owen (to Pittsburgh Plate Glass Co.) U. S. 2,021,197, Nov. 19. Various details of app. and operation are described.

Form for use in bending glass sheets Henry J.

Galey (to Pittsburgh Plate Glass Co.). U. S. 2,021,180, Nov. 19. Structural details.

Apparatus for transporting glass sheets from a rolling machine to a position in front of an annealing lehr. Glaceries de la Sambre Soc. anon. Brit. 435,655, Sept. 25, 1935. Divided on 425,514 (C. A. 29, 63829). In app. comprising a roller-way conveyor fitted, between the rollers adjacent the lehr, with members which are raised to lift the sheet from the rollers during the charging stroke of the pusher, each member is formed with narrow spaced sheet-engaging surfaces.

Roller apparatus for conveying sheet glass to cooling lehrs Les Glaceries de la Sambre Soc. anon. Ger. 622,235, Nov. 23, 1935 (Cl. 32a. 34).

Apparatus for tempering glass Pilkington Bros. Ltd. Fr. 787,904, Oct. 2, 1935.

Glass-annealing furnace Corning Glass Works. Ger. 619,557, Oct. 3, 1935 (Cl. 32a. 29).

Vertical tunnel apparatus for annealing sheet glass Paul L. Geer (to Amco, Inc.). U. S. 2,026,781, Jan. 7. Structural, mech. and operative details.

Muffle lehr for annealing glassware such as bottles Leon J. Houze, Sr. (to L. J. Houze Convex Glass Co.). U. S. 2,026,737, Jan. 7. Structural and operative details.

Apparatus for charging glass-annealing lehrs Wm. Green & Co. (Ecclesfield) Ltd., Ernest Warren, King, Taudervin & Gregson Ltd. and John H. Webster. Brit. 435,488, Sept. 23, 1935. In a "stacker" of the kind comprising a transverse conveyor from which the ware is transferred to the lehr conveyor by a member which is reciprocated to have an operative stroke in the opposite direction to and an idle stroke in the same direction as the travel of the transverse conveyor, the speed of the idle stroke is greater than that of the conveyor and means is provided for delaying the next operative stroke.

Etching lamp bulbs Frederick Kallus U. S. 2,027,093, Jan. 14. A bulb is subjected to an etching fluid and then immediately, without washing, is treated with a limited quantity of steam, and then after an interval of time is washed out by flooding with addnl. steam. App. is described.

Laminated glass Geo. B. Watkins (to Libbey-Owens-Ford Glass Co.). U. S. 2,026,717, Jan. 7. Two sheets of glass are united with an interposed sheet of nonbrittle material such as a cellulose ester compn. treated with a mixt. of esters of phthalic acid such as diamyl and dibutyl phthalates. Cf. C. A. 30, 832<sup>1</sup>.

Laminated glass. James G. McNally and Sterling S. Sweet (to Eastman Kodak Co.). U. S. 2,026,987, Jan. 7. Glass sheets are joined with an intervening sheet such as one of cellulose acetate compn. bonded to the glass by a film such as one of cellulose acetate acid succinate dissolved in a plasticizing solvent such as benzyl alc.

Apparatus for laminated sheet-glass manufacture. Willard C. McDaniel (to Libbey-Owens-Ford Glass Co.). U. S. 2,026,692, Jan. 7. Various structural, mech. and operative details.

Conditioning clay for ceramic ware manufacture Minor F. H. Gouverneur (to Locke Insulator Corp.). U. S. 2,026,782, Jan. 7. The clay is extruded under pressure and simultaneously subjected to vibration in the direction of extrusion. App. is described.

Ceramic products. Grandes Tuileries de Nemours Fr. 788,194, Oct. 5, 1935. Extra-hard ceramic products are made from sedimentary rocks and volcanic rocks, to which is added about 5% of a decreasing element such as silica or quartz or an alk. flux. The ground ingredients are wetted, mixed, molded and baked at about 1200°.

Apparatus for pressing semi-dry ceramic material under high pressure as in the manufacture of refractory articles Dwight B. Hendryx (to Harbison-Walker Refractories Co.). U. S. 2,026,940, Jan. 7. App. and various operative details are described.

Silica suitable for coating stone, brick, sand molds, etc. Arthur B. Ray (to Carbide and Carbon Chemicals Corp.). U. S. 2,027,931, Jan. 14. For producing a



form of silica capable of adhering to material such as stone, brick, concrete, etc., the material is treated with a soln contg a silicic acid ester of a polyhydric alc such as a glycol or glycerol ester and silica is deposited by hydrolysis. U S 2,027,932 relates to sand molds adapted for use with alloys of high m. p. and having a smooth coating of adherent unfused silica deposited on their surface.

Apparatus for production of shaped ware such as tubing from fused vitreous material. Philip K. Devers (to General Elec. Co.) U S 2,027,155, Jan 7. An elongated stock is drawn from a silica fusion through a die submerged in the fusion (various details of app. and operation being described).

Casting porcelain and stoneware. Giovanni Fuschl and Maurizio Korach. Brit 434,494, Aug 27, 1935. Articles free from air bubbles are cast in plaster molds by circulating the fluid slip continuously through the mold by means of a circulating pump until the necessary thickness has been deposited. App. is described.

Pyroxyl thermometer cases, etc. Glenn A. Cooper (to Cruver Mfg. Co.) U S 2,027,152, Jan 7. Various details of app. and operation.

Refractory products. Compagnie de produits chimiques et electrometallurgiques Alais, Fröges & Camargue (M. Kortschak, applicant in Austria). Fr 788,492, Oct. 10, 1935. Bricks, linings and protective layers for furnaces, etc., are made without baking by intimately mixing powd. clay, grog,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , calcined quartzite, zircon, bauxite, kieselsäure, etc., with a binder consisting of substances of high mol. wt., such as residual lye from the sulfite manuf. of cellulose, and a sol. metasilicate, if desired with the addn. of  $\text{FeSO}_4$  and crystd

graphite and with or without water. The mixt may be treated with steam to bring it to the required consistency for molding.

Recrystallized chromite suitable for refractory products. Gilbert E. Seil (to E. J. Lavino and Co.). U S 2,028,017, Jan 14. See Fr. 781,276 (C. A. 29, 6384).

Refractory material for glass furnaces. The Carborundum Co. Fr. 787,931, Oct. 1, 1935. See Can 351,679 (C. A. 29, 6722).

Abrasive grinding wheels. Duane E. Webster (to Norton Co.). U. S. 2,027,132, Jan 7. Structural details.

Abrasive sheets. Orello S. Buckner (to Behr-Manning Corp.) U. S. 2,027,047, Jan. 7. A sheet of material such as paper or cloth having an adhesive surface is placed in a magnetic field, so that the lines of force pass approx. perpendicularly through the sheet and magnetically coated abrasive grains such as grains coated with iron or steel are showered onto the surface. App. is described.

Abrasive coatings on materials such as paper or cloth. Elmer C. Schacht (to Behr-Manning Corp.). U. S. 2,027,307-8-9, Jan. 7. Various details of app. and operation are described for applying abrasive particles such as sand to material such as paper or cloth and controlling their deposition by the action of an elec. field.

Tunnel kilns. Bernard J. Moore. Brit. 435,452, Sept. 23, 1935. A kiln for the manuf. of pottery, etc., comprises 3 or more side by side tunnels through which the goods are moved in opposite directions.

Apparatus for propelling a train of abutting trucks through a tunnel kiln. W. G. Allen & Sons (Tipton) Ltd and Wm. H. Jones. Brit 434,377, Aug. 30, 1935.

## 20—CEMENT AND OTHER BUILDING MATERIALS

### J. C. WITT

Special cements for use in mass concrete. H. Elser v. Gronow. Zement 24, 747-9(1935).—The desirable properties and methods of their prep. are discussed generally. H. F. K.

Theory of hardening of lime-pozzuolanic cements. V. N. Yung. Stroitel Material 1935, No. 8, 3-18.—Lime pozzuolanic cements harden very slowly as the result of a slow reaction at ordinary temp. of amorphous silica with  $\text{Ca(OH)}_2$ . Adsorption systems of an indefinite compn. are formed and these are converted into amorphous gelatinous hydrated silicates varying from  $\text{CaO} \cdot \text{SiO}_2 \cdot n\text{H}_2\text{O}$  to  $2\text{CaO} \cdot \text{SiO}_2 \cdot n\text{H}_2\text{O}$ . In water hardening the cementing mass consists of gelatinous hydrated silicates. In air hardening the strength ceases to increase as a result of drying and begins to fall on carbonation, owing to formation of a powd. mixt. of  $\text{SiO}_2$  and  $\text{CaCO}_3$ . In lime-sand brick subjected to the influence of steam under pressure are formed concretions of cryst. hydrated silicates that pass over when carbonated into cryst. concretions of calcite without any decrease of the strength of the brick with quartz grains. The hardening of lime-basic slag cement is followed by a colloidalization of slag under the influence of the  $\text{Ca(OH)}_2$  soln. The increase of strength ceases on account of early drying, sufficiently hardened in moist condition, lime-slag cements are much less subjected to carbonation on account of the density of colloidal formations as compared with those of lime-pozzuolanic cements. C. L. Stefanowsky.

Magnesian portland cement. I. L. Lefand and V. V. Serov. Zement 3, No. 7, 7-18(1935), cf I. L. Lefand and V. V. Serov. Ibid. 2, No. 1, 5-21(1934).—Lab. and plant investigations demonstrated the possibility of obtaining cement-vol. magnesian portland cement. The presence of free  $\text{MgO}$  (5-6%) in the clinker in quantities leading to inconstancy of vol. can be disclosed by the accelerated method of investigation in an autoclave during 3 hrs. under a pressure of 14 atm. The usual method does not give the required results on account of the slow hydration of free  $\text{MgO}$ . The excess over 5%

of  $\text{MgO}$  occurs as  $4\text{CaO} \cdot 2\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3 \cdot \text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{CaO} \cdot \text{Fe}_2\text{O}_3 \cdot \text{MgO}$  and  $\text{CaO} \cdot \text{MgO} \cdot \text{SiO}_2$  and as  $\text{MgO}$  in a solid soln. in these compds.; thus is assured constancy of vol. of the cement up to a content of 11% of  $\text{MgO}$ . The clinker can be burned in rotary kilns. The optimal conditions are 25-30% of volatile matter in the charge and a moderate calorific value of the fuel. The output and fuel consumption are equal to those in the usual portland cement production. The setting time of the cement with a 2% addn. of gypsum is normal, with 30% of hydraulic additions it does not decrease in the first period of setting as in ordinary portland cement. E. E. Stefanowsky.

The action of magnesium salts on pozzuolanic portland cements. V. V. Kind. Zement 3, No. 7, 42-53(1935).—Solns. of Mg salts attack pozzuolanic cements the more, the higher the percentage of hydraulic addns. Toward dil. solns. pozzuolanic cements are sufficiently resistant, at concns. of over 1% the resistance decreases in a degree depending on the kind of cement. The action of Mg salts is based on the decompn. of Ca silicates and aluminates. Free H ions liberated in the partial hydrolysis of Mg salts can also enter into the action. The slow deterioration of portland cement is explained by the presence of free lime, forming  $\text{Mg(OH)}_2$  with  $\text{MgSO}_4$  and  $\text{MgCl}_2$ , this takes place also in the case of a small pozzuolanic addn., leaving a considerable amount of free lime. E. E. Stefanowsky.

Influence of the temperature of drying of blast furnace slag on the hydraulic properties of slag cement. V. Ya. Eremenko. Zement 3, No. 6, 36-40(1935).—In slag contg. 46-7% of  $\text{CaO}$  drying temps. of 800° to 1000° decreased the mechanical strength of the cement, but the rate of decrease falls with the increasing temp. The decrease takes place only during the early hardening period and nearly disappears in 28 days. The setting time of the cement decreases only slightly. A drying temp. of 800° is recommended for the case investigated. E. E. Stefanowsky.

The calculation of the raw materials for cement. V. N. Yung. *Tzement* 3, No. 8, 4-18(1935).—The formula for the satn. of clinker with lime in ordinary portland cement is suggested on the basis of a detailed analysis of the chemistry of clinker formation  $100(\text{CaO} - \text{CaO}_{\text{bound}} - 0.75\text{SiO}_2) - 2.8(\text{SiO}_2 - \text{SiO}_{\text{bound}}) + 1.65\text{Al}_2\text{O}_3 + 0.35\text{Fe}_2\text{O}_3$ . In the production of high-grade cement high in lime, the calcn must be based on close analysis of all factors involved in the conditions of the given plant.

E. E. Stefanovsky

Calculation of the mixture of raw materials for the production of portland cement clinker. V. A. Kind. *Tzement* 3, No. 8, 19-24(1935).—The existing formulas for calcn are critically reviewed. The following is a modification of the Kuhl formula  $x = \frac{[(2.8\text{SiO}_2 \times \text{KH} + 1.65\text{Al}_2\text{O}_3 + 0.35\text{Fe}_2\text{O}_3) - \text{CaO}]}{[(2.8\text{SiO}_2 \times \text{KH} + 1.65\text{Al}_2\text{O}_3 + 0.35\text{Fe}_2\text{O}_3)]} \times \text{clay/lime}$  ( $\text{KH} = \text{coeff. of satn.}$ ).

E. E. Stefanovsky

The stability toward mineral salts of pozzuolanic portland cement with additions of burnt clay. V. V. Kind. *Tzement* 3, No. 8, 42-7(1935), cf. *C. A.* 29, 2327<sup>a</sup>.—With increase of burnt clay from 20 to 50% the stability increases. Cement contg 20% of burnt clay is less stable than pure portland cement, with 30% clay it is equal to, and with 40-50% clay considerably surpasses, portland cement in stability, but is less stable than the product obtained by addn. of Bryansk diatomite rock. The low stability is probably caused by the formation of Ca sulfoaluminate through the action of  $\text{CaSO}_4$  formed from free lime and  $\text{Na}_2\text{SO}_4$  of the cement.

E. E. Stefanovsky

Tshernometchenskii portland-cement plant. I. E. Bashmarchnikov and Ya. G. Sokolov. *Tzement* 3, No. 8, 25-32(1935).—The following are characteristic features of the plant. Wolff slurry filters reduce the fuel consumption and increase the output; economizers for the utilization of the flue gases have been installed, solid crust, limestone and shale clay are used. High-grade cement is obtained.

E. E. Stefanovsky

Automatic shaft kilns. A. S. Pantelev. *Tzement* 3, No. 8, 32-6(1935).—To obtain high-grade cement clinker in automatic shaft kilns it is necessary to use 1300-1400 cal. of fuel for 1 kg. of clinker and an excess of air. Cylindrical briquets 3-4 cm. in diam. must be used. The mixture of raw materials must be charged continuously and must be homogeneous.

E. E. S.

The stability of the lining at the Leningrad cement plant. Yu. S. Lur'e and S. I. Vorotuntzev. *Tzement* 3, No. 8, 37-8(1935).—High-grade cement is produced from lime tuff, clay and roasted pyrites. The grog brick lining of one kiln was replaced by a clinker-concrete packed lining in the drying and sintering zones. After 3 months, the kiln works normally.

E. E. S.

Fuel requirements for rotary cement kilns. A. S. Dokudovskii. *Tzement* 3, No. 6, 5-13(1935).

E. E. Stefanovsky

Ash from powdered brown coal as a raw material for the production of cement. V. V. Surovitzev and A. S. Rasorenov. *Stroitel. Material.* 1935, No. 7, 63-71.—The finely divided ash together with lime can be used for the production of pozzuolanic cements. The mechanical properties of the cement can be improved by adding about 2% of dil. solns of  $\text{CaCl}_2$  or  $\text{NaCl}$ .

E. E. S.

Strength reduction of heated mortar. Heinrich Luftschütz. *Zement* 24, 749-51(1935).

H. F. K.

The hardening of concrete. S. A. Mironov. *Tekhnika* 1935, No. 45.—In the hardening of hydraulic materials swelling is greater at lower temps. Slower formation of the  $\text{SiO}_2$  gel delays the formation of the crystals that effects hardening; thus hydration is more complete. Freezing the cement-sand mixt. increased the stability of the cements. It is a mistake to begin the elec. heating of concrete earlier than 1-1.5 days after it is laid and thus must be taken into account also during accelerated lab. tests.

B. V. Shvartzberg

Strength and impermeability of concrete and porous rocks as increased by treatment with siliceous solutions. M. Stamatii. *Rev. ind. minérale* No. 360, 597-602

(1935).—Concrete, sand, calcareous materials, etc., were injected first with a soln. of Na or K silicate, then with a pptg. soln. (dil.  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ ;  $\text{MgSO}_4$ , Ca, Mg or Al chloride, or Mg, Zn or Al fluosilicate). The specimens were then tested at intervals for strength in compression, for permeability, and for resistance to corroding agents. Best results in all cases are obtained when the 1st soln. is 30-38% B $\bar{e}$  Na silicate and the 2nd 33-41%  $\text{CaCl}_2$ .

C. D. West

The viscosity of road tars. J. G. Mitchell and A. R. Lee. *J. Soc. Chem. Ind.* 54, 407-117(1935).—Measurements were made with a modified Ostwald viscometer (*C. A.* 28, 3221<sup>a</sup>). The relationship of Evans and Pickard  $\eta T^a = k$ , where  $\eta$  is viscosity,  $T$  Fahr. temp. and  $n$  and  $k$  are constants was confirmed. The relative values of  $\eta$  occurring in the above equation were not affected by low-temp. distn.  $\eta$  was linearly related to  $\eta$  at 25° for distn. residues from a tar having  $\eta$  from 10<sup>4</sup> to 10<sup>6</sup> poises. The effect of oiling black pitch and of loss of oil from tar by distn. was detd. A working method for estg. amt. of cutting back required to produce tars of specified  $\eta$  was given.

P. S. Roller

Crystalline masses from Tshardzhii loesses for the production of paving blocks. P. S. Kuten. *Stroitel. Material* 1935, No. 7, 43-5.—Loess of the compn.  $\text{SiO}_2$  49.26,  $\text{Al}_2\text{O}_3$  11.25,  $\text{TiO}_2$  0.53,  $\text{Fe}_2\text{O}_3$  6.62,  $\text{Mn}_2\text{O}_3$  0.38,  $\text{CaO}$  12.75,  $\text{MgO}$  3.34,  $\text{SO}_3$  0.19,  $\text{K}_2\text{O}$  2.07,  $\text{Na}_2\text{O}$  1.27, ignition loss 12.95%,  $m$  1140°, was heated at 1350-1400° and cast mechanically into metal molds. In order to obtain a cryst. structure of the block, it was treated after casting in an annealing furnace at 500° to 1000° during 10.5-12.5 hrs. The block had a crushing strength of 5400 kg./sq. cm. and other good mechanical properties.

E. E. Stefanovsky

Roasting and sintering dusts [clinkering cement mixts.] (Saint-Jacques, Poupet) 9. Silica for coating stone, brick [concrete], etc. (U. S. pat. 2,027,931) 19. Bin for homogenizing cement raw meal (U. S. pat. 2,027,697) 1. Wood preservation (Remington) 26.

Cement. Charles H. Breerwood (to Valley Forge Cement Co.). U. S. 2,028,313, Jan. 21. In treating lime-bearing cement raw materials const. an undesirable quantity of at least one constituent such as  $\text{CaCO}_3$  and in which the  $\text{CaCO}_3$  value varies from time to time as received for treatment, the sands are sepd. from the fine particles of each material, an addnl. quantity of the coarser particles is sepd. from the fines and combined with the sands, the combined coarse particles are subjected to froth flotation so conducted that the concentrate is maintained substantially at a desired  $\text{CaCO}_3$  value and the rejects include the undesirable quantity of constituents originally present such as  $\text{CaCO}_3$  and the concentrate from the flotation operation is combined with the untreated fines to provide a mixt. of desired  $\text{CaCO}_3$  value which is maintained substantially const. by varying the quantity of the coarser particles sepd. from the fines and added to the sands, so that the combined quantity is varied inversely as the  $\text{CaCO}_3$  value of the materials varies in the materials as received. An arrangement of app. is described. Cf. *C. A.* 30, 594<sup>a</sup>.

Cement. Alton J. Blank (to Cement Process Corp.). Can. 353,934, Nov. 5, 1935. One part material const. lime in a form reactive to  $\text{Si}$  and  $\text{Al}$  is mixed with 2 parts of an argillaceous material, e. g. diatomaceous earth, and with portland cement in the presence of a 1% aq. soln. of  $\text{NaCl}$ . A temp. of 150° is maintained to produce mutual reactions between the lime and the siliceous and aluminous components of the argillaceous material. The tensile strengths for test samples under standard conditions for portland cement was increased considerably.

High-silica cement. Fritz A. B. Finkeldey. U. S. 2,028,386, Jan. 21. Addnl. siliceous and calcareous materials such as crushed shale and limestone are mixed with hot cement clinker as it comes from the clinkering kiln at a temp. of about 700-1050° and maintained in

contact therewith for a time sufficient to effect calcination of the calcareous material, the proportion of the added siliceous material being such as to equal up to about 24% of the combined materials when finished and the calcareous material to yield an added CaO content up to about 6% of the combined finished materials. An arrangement of app is described.

**Cement mixture containing calcium arsenate** Johan B. Stalhane (to Bolidens Gruvaktiebolag) U. S. 2,023,420, Jan. 21. About 20% or more of Ca meta- or pyroarsenate is used with lime, Ca silicate, portland cement or aluminate cement, and serves to give a low soly.

**Sintering cement, agglomerating ores** Metallgesellschaft (Bernhard Young, inventor) Ger. 622,146, Nov. 21, 1935 (Cl. 80c 11). In processes for sintering cement or agglomerating ores by burning a mixt. of the material with a fuel on a grate through which air is blown, the proportion of fuel is adjusted so that a part of the material is insufficiently burnt, and this part is added to the next batch of material.

**Rotary cement kilns** Johan S. Fasting Brit. 434,609, Sept. 6, 1935. A preheater for a kiln comprises a rotary perforated drum encircling the inlet end of the kiln, a fixed wall, having an outlet at its lower part, being provided to cause the waste gases from the kiln to pass downwardly through the lower portion of the drum and the layer of charge material fed thereto.

**Applying refractory linings to rotary kilns for burning cement, etc.** Didier-Werke A.-G. Ger. 622,305, Nov. 25, 1935 (Cl. 80c 14 01). Manipulative features are described.

**Rotary inclined drier and kiln installation suitable for use in cement manufacture** Ray C. Newhouse (to Allis-Chalmers Mfg. Co.) U. S. 2,027,039, Jan. 7. Various structural, mech. and operative details.

**Light concrete** Léon J. Walter Fr. 785,715, Oct. 15, 1935. A soln. for maintaining air in the form of independent small cells in suspension in a milk of cement during the setting of the cement contains anhyd.  $\text{Na}_2\text{CO}_3$ , brown saponin 20 g., water 100 and cement 21.

**Road making substances** Maschinenfabrik Komnick G. m. b. H. Ger. 619,973, Oct. 10, 1935 (Cl. 80b 25 09).

**A filling material for the spaces between bituminous blocks for roads, etc.**, consists of dry fine sand mixed with a small amt. of bitumen or tar and a small amt. of  $\text{Ca}(\text{OH})_2$ . Thus, 100 parts of fine sharp sand is dried by heating to 150–200° and mixed with 2–3 parts of hot bitumen or tar and 0.5–0.75 parts of  $\text{Ca}(\text{OH})_2$ .

**Ceramic paving stones, etc.** Henry Behrens Baustoffhandels, m. B. H. Ger. 619,899, Oct. 9, 1935 (Cl. 80b 12 07). Clay and ground unburned stone are mixed and then molded and fired. Thus, brick clay is mixed with sand and basalt, pressed and fired.

**Paving blocks** Fried. Krupp A.-G. Fr. 788,557, Oct. 12, 1935. Blocks are made of a layer of good supporting power, such as a mixt. of ground cryst. blast-furnace slag, hardening in gases contg.  $\text{CO}_2$ , and granulated blast-furnace slag, hardening in an alk. soln., and a layer resistant to wear composed of a mixt. of cement and mineral fragments, e. g., blast-furnace cement and fragments of slag.

**Paving** Hermann Plauson (to T. R. C. Corp.) U. S. 2,026,988, Jan. 7. A rolled ballast road is formed with ballast stones united and with their interstices filled with a hydraulically set bituminated mortar prepd. from an initially pulverulent intimate mixt. of a hydraulic binder such as a mixt. of gypsum with lime or cement and not more than 7% the wt. of the binder of a bituminous material such as asphalt, tar or pitch. U. S. 2,026,989 relates to forming a pavement of similar materials to which water is added to cause the binder to set after the pavement is compressed by rolling *in situ*.

**Covering material suitable for use on walls, etc.** Abraham B. Miller (to Paraffine Cos., Inc.) U. S. 2,027,744, Jan. 14. A body such as paper, cloth, etc., has a preformed sheet of material such as uncured linoleum against it so as to form a bond between the materials which exceeds the bonding between the particles of the sheet, and the sheet is then peeled from the body so that surface portions of the sheet are split off and retained on the surface of the body.

**Fabricated sheets of mica suitable for wall coverings, etc.** Francis C. Atwood U. S. 2,027,082, Jan. 7. App. and various operative details are described.

## 21—FUELS, GAS, TAR AND COKE

A. C. FIELDYER AND ALDEN H. EMERY

**Coals from the Chelyabinsk deposits** N. G. Titov *Khim. Tverdogo Topliva* 5, 679–83 (1934).—General characteristics of the Chelyabinsk brown coals are presented.

**A control chart for interpretation of coal sampling data** T. W. Guy *Combustion* 7, No. 6, 29–32 (1935).—A method is given for prep. and using a coal chart that will indicate whether the quality of a given coal is within required limits.

**Errors of data of washability curves in coal dressing.** G. Tarján *Roy. Hung. Palatin-Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met.* 7, 397–424 (1935).—Errors detd. in washability curves amounted in general to 0.1%. Calcs. showed that at an ash content of 30% in coals the probable error reaches 0.1004%. For smaller ash content it decreases somewhat. Also the probable errors of moisture detns. are discussed.

**Method for the proper evaluation of effects of coal dressing** G. Tarján *Roy. Hung. Palatin-Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met.* 7, 378–90 (1935).—Calc. coal output and efficiency of coal dressing from ash contents does not give reliable values. A modification of present calcn. methods is proposed by transforming the "ash" basal curve into the "gang" curve, which latter should serve as a basis for the calcn.

**Direct simultaneous microdetermination of carbon, hydrogen and oxygen** IV. Analysis of bituminous coals and derived products. W. R. Kurner. *Ind. Eng. Chem.*

*Anal. Ed.* 8, 57–61 (1936).—The methods previously described, C. A. 28, 6394<sup>1</sup>, 30, 459, 205<sup>1</sup>, are adapted to the examn. of coal and allied products. With the knowledge available from the previous work on pure compds. contg. N, certain deductions can be made with respect to the probable way in which the N is combined. It would seem that the linkage in bituminous coal is not that of an ammo or amido group but more likely that of a heterocyclic compd. In the case of nitro humic acid, evidence supports the view that an isonitroso-keto linkage is present. The values obtained directly for O are, as a rule, higher than the values obtained by difference, except with coke where the opposite is true.

**Purification of coal** Vieux. *Rev. ind. minérale* No. 359, 581–9 (1935).—A review of the subject under 3 headings: washing, treatment of fines and slimes and dry methods.

**Low-temperature carbonization of Chelyabinsk coals** II. I. B. Rapoport and Z. E. Kosolapov. *Khim. Tverdogo Topliva* 5, 702–10 (1934), cf. C. A. 28, 6274<sup>1</sup>.—Carbonization at 450°, 500° and 550° yielded 2.5–4% primary tar and 62.4–63.2% semicoke. The sp. gr. and the contents of  $\text{NH}_3$  and phenols increases in the tar waters with increase in the carbonization temp. The procedure is described and the results are tabulated.

**The determination of volatile substances in anthracites** M. P. Lonskaya. *Khim. Tverdogo Topliva* 5, 690–5 (1934).—A weighed amount (1 g.) of air-dry, ground anthracite is placed in a tared crucible and then moistened with kerosene (1–1.5 cc.). The mixt. is heated with a

Bartell alc. burner having a flame of 18 cm., while the distance between the flame and the crucible is maintained at 8 cm., for 3 min. It is claimed that results accurate within 0.3% can be obtained. Kerosene is evaporated and partly decomposed, creating the proper atmosphere in the crucible, whereby undesirable chem. reactions are eliminated.

**A A Boethlingk**  
Rapid determination of nitrogen in coal R D Sheinman and A Z Yurovskii *Khim. Tverdogo Topliva* 5, 696-702(1934)—The method proposed by the authors differs from that of Kjeldahl-Wittacker in that a stronger oxidizing medium is used ( $KMnO_4 + CuO$ ). The amount of substance used in the analysis is 0.2-0.3 g., this is titrated with 0.05 N acid for the detn. of  $NH_4$ , the flask being heated with specially adapted elec. heaters so as to accelerate the oxidation of coal. A detailed description of the method is given.

**A A Boethlingk**  
Experiments on a small coal panel at the Lischansk mine (on underground gasification) I P Kirichenko *Podzemnaya Gazifikatsiya Ugles* 1935, No 7, 8, 312, cf. C. A. 29, 321\*—This is a continuation on a larger scale of previous expts. on underground gasification of coal by the "drift-gas-generator" method, consisting in forcing air and steam through bore-holes into a system of drifts through the coal body. The points investigated were: (1) max. distance between drifts, (2) effect of length of drifts on the process of gasification, (3) effect of incline of drift to coal layer, (4) effect of forcing air and steam through the same or different bore-holes and (5) effect of the regime of alternating air and steam blow on the process. Forcing the air and steam through separate bore-holes gave a better control of the process. The water gas produced was of low grade having a heat value of 800-1200 cal. per cu m.

**S L Madorsky**  
More about underground gasification of coal by means of bore holes S I Buyalov *Podzemnaya Gazifikatsiya Ugles* 1935, No 6, 2-6, cf. C. A. 30, 1207\* S L M.

Underground gasification of coal as a basis for direct reduction of iron ore L O Trautman *Podzemnaya Gazifikatsiya Ugles* 1935, No 7-8, 18-23—A discussion.

**S L Madorsky**  
Synthesis of liquid fuel from gases obtained in underground gasification of coal. B Dolgov *Podzemnaya Gazifikatsiya Ugles* 1935, No 5, 10-18—Review. Nineteen references.

**S L Madorsky**  
Calculation of heat losses due to heating a layer of damp coal under conditions of underground gasification. A V. Lukov and A. A. Pomerantsev *Podzemnaya Gazifikatsiya Ugles* 1935, No 9, 3-14.

**S L Madorsky**  
Bitumens and humic acids present in coals G L Stadnikov and A. A. Fal'kovskaya *Khim. Tverdogo Topliva* 5, 6-3-9(1934), cf. C. A. 29, 5630\*—The bitumen and humic acid contents of various Russian coals are tabulated.

**A A Boethlingk**  
Brigetting experiments with Hungarian brown coals II. J Finkey. *Roy. Hung. Palatin-Joseph Univ. Tech. Econ. Sec., Sopron, Pub. Dept. Mining Met.* 7, 183-200 (1935); cf. *Ibid.* 5, 1 (1933) and C. A. 28, 3320\*—The original method was modified by using a Värpalö lignite ground in the dry state to a max. grain size of 0.073 mm. as a binder. This has previously been dried and mixed with the briquet coal. The ratio of the colloidal binder was varied from 20 to 40%. Värpalö, Komló, Dúsgyőr and Kisterenye brown coals can well be used for brigetting.

**S. S. de Finily**  
The chemical technology of the utilization of peat II Segeberg. *Chem.-Ztg.* 59, 961-3(1935).

**E. H.**  
Three-zone furnace with internal heating for the coking of peat B K. Klimov and V. A. Danilenko *Khim. Tverdogo Topliva* 5, 711-19(1934)—Construction and operation details are given. Repeated recirculation of the gases preliminarily stripped of the valuable fractions is effected.

**A A Boethlingk**  
Humic substances VII. Berginization and oxidation of humic acids of peat. N. A. Orlov, V. V. Trishchenko and P. M. Tarasenkova. *J. Applied Chem. (U. S. S. R.)* 8, 501-4(1935); cf. C. A. 29, 6035\*—One hundred g. of the humic acid was hydrogenated in a rotating 3-l.

autoclave heated to 410° for 2-2.5 hrs. in the presence of  $MoS_3$  at a cold H<sub>2</sub> pressure of 100 atm. The yield of liquid products amounted to 34% of an oil product composed of phenols, and mainly hydroaromatic hydrocarbons. On oxidation the humic acids of peat yielded a mixt. of benzene-carboxylic acids in addition to volatile acids; no mellic acid was produced.

**A A Boethlingk**  
Production of city gas and gas for synthesis from brown coal by the continuous-current gas process W. Allner. *Z. Ver. deut. Ing.* 79, 1487-92(1935), cf. C. A. 29, 6731\*—Structural details.

**Joseph H. Wells**  
Making city gas nontoxic by means of catalytic oxidation of carbon monoxide with steam Fritz Schuster *Wärme* 58, 854-6(1935)—Heating values are calculated; coal gas is compared with water gas. Removal of  $CO_2$  after oxidation of CO is discussed.

**W H Bruckner**  
Content of free oxygen in a fuel gas and its explosiveness A S Kuznetsov *Podzemnaya Gazifikatsiya Ugles* 1935, No 5, 22—Fuel gas contg.  $CO_2$  1.5,  $O_2$  1.0,  $CO$  9.17,  $CH_4$  0.37 and  $H_2$  33.72%, was mixed with air, 5 to 25% by vol., and tested for explosiveness by igniting it in a closed vessel by means of a heated Pt wire. Pressure varied from 4.8 to 14.4% above atm. Signs of explosion begin to appear at 6.42%  $O_2$  content.

**S L Madorsky**  
Water gas at Lischansk coal mine Preliminary report I P Kirichenko *Podzemnaya Gazifikatsiya Ugles* 1935, No 9, 34-5—The method consisted of blowing air and steam through bore-holes into drifts along the coal bed and removing the products of combustion through other bore-holes. In one expt. air alone was used. The gas had a compn. of  $CO_2$  11.0,  $O_2$  0.2,  $CO$  12.0,  $H_2$  12.0,  $CH_4$  5.0 and  $N_2$  59.8% and a heat capacity of 0.30 cal. per cu m. In another expt. air and steam were blown alternately. Compn. of the gas was  $CO_2$  23.8,  $O_2$  0.0,  $CO$  14.0,  $H_2$  41.5,  $CH_4$  5.8 and  $N_2$  11.8%, calorific value, 2075 cal. per cu m.

**S L Madorsky**  
Burning of anthracite plates for the purpose of studying production of water gas A I Semenov, I S Galunkin and V V Kondakov *Podzemnaya Gazifikatsiya Ugles* 1935, No 7-8, 23-30—Anthracite plates measuring approx.  $200 \times 250 \times 2650$  mm. were placed in a large pit in the ground, in such a way as to form channels through the pile. The pile was ignited and  $O_2$ -enriched air and steam were blown alternately through the coal pile. The optimum results were obtained by following a regime of 10 min. air (contg. 60%  $O_2$ ) blow and 20 min. steam blow. The water gas thus obtained had a heat value of 2160 cal. per cu m.

**S L Madorsky**  
Odorization of gas M M Wilson *Western Gas* 11, No 12, 10-12(1935)—Methods of adding odorous gas are discussed and illustrated.

**W H Bruckner**  
The Fadeev-Cheryatnikov new gas analyzer P T. Kolesnikov *Podzemnaya Gazifikatsiya Ugles* 1935, No 5, 20-7—A special app. adapted to analysis of gases produced in underground gasification of coal is described. A complete analysis can be made in 30 min.

**S L Madorsky**  
The Southern California Gas Co. calcium chloride brine dehydration plant B M Lauther *Western Gas* 11, No 12, 18-19(1935).

**W H Bruckner**  
The reaction between methane and water vapor. Kinzo Kafuku and Toyoyuro Ogura. *J. Chem. Soc. Japan* 56, 1145-50(1935)—The yield of H<sub>2</sub> and CO from  $CH_4$  and water vapor was best by using Ni as catalyst with low pressure and high temp. (5 mm. and 1000°). Use of Ni with  $Al_2O_3$ ,  $MgO$  or pumice was more effective.

**K Kutsuta**  
Cellophane and Kodapak for pipe-line protection Lee Holtz *Western Gas* 11, No. 10, 22-3, 5(1935).—Their use in conjunction with other materials such as asphalt, kraft paper, etc., is reviewed. A table is shown of materials required for pipe of various diams. and 100 ft. long. The use of the wrapper prevents entry of moisture and penetration of tree roots into coating.

**W H B.**  
The sulfur compounds of natural gas and natural gasoline of the Oktyabr district S M Gabrielyantz and S A. Isaeva *Grozneniskii Neftyanik* 5, No 1-2, 49-52 (1935).—The comparatively large amounts of elementary

S were formed from  $H_2S$  through oxidation, etc. Compns. of the gas and the gasoline are given. A. A. Boehlingk.

Removing sulfur dioxide from flue gases. B. G. Gaberman. *Khim. Tverdого Topliva* 5, 730-40 (1934). — Up to 76.5% of  $SO_2$  (present in an artificial flue-gas mixt.) was absorbed by passing the gas through a flushing system operated with  $MnO_2$  and  $Mn$  ions suspended in  $H_2O$  and passing the gas at a velocity of 20.51 per min through one l. of water at  $20^\circ$ , with a final yield of  $H_2SO_4$  of 41-42%. Thus 0.11 ton of  $H_2O$  was needed to absorb  $SO_2$  produced by burning one ton of peat contg. up to 8.42% S. A. A. Boehlingk.

Destructive hydrogenation of crude anthracene for the purpose of producing light aromatic compounds. II. V. N. Khadzhinov. *J. Applied Chem. (U. S. S. R.)* 8, 889-902 (in German 902) (1935), cf. *C. A.* 29, 6727. — Hydrogenation of anthracene in the presence of  $MoS_3$  at 480-500° gives a 32% yield of fractions b below  $180^\circ$ . A yield of 55% can be obtained by recycling fractions b above  $180^\circ$ . At  $400^\circ$  and less, up to 65% of a fraction b 230-300°, resembling kerosene, is obtained without recycling. In the first case almost exclusively aromatic compds. are produced, while in the second case hydroaromatic compds. and naphthenes in addn. to aromatic compds. were found. The  $MoS_3$  remained active up to 32 hrs. The following products were sepd:  $C_6H_6$ ,  $PhMe$ ,  $PhEt$ , naphthalene, tetralin, methyl-naphthalenes and their hydrides, ethyl-naphthalenes, biphenyl, methylcyclopentane, cyclohexane, methylcyclohexane, decalin and products of its isomerization, bicyclohexyl and products of its isomerization and perhydrides, and probably a mixt. of anthracene and phenanthrene perhydrides. Forty-one references. A. A. Boehlingk.

Investigating the chemical composition, properties and methods of treating primary tars from Chelyabinsk brown coals. I. Destructive hydrogenation of the primary tar from Chelyabinsk brown coals. A. V. Lozovoi and M. K. D'yakova. *J. Applied Chem. (U. S. S. R.)* 8, 488-99 (in French 500) (1935). — See *C. A.* 29, 1908. II. The chemical composition of the light fraction from the primary tar from Chelyabinsk brown coals and the stabilization of the hydrocarbon part of the "benzine". M. K. D'yakova, A. V. Lozovoi and S. I. Chertkova. *J. Applied Chem. (U. S. S. R.)* 8, 695-705 (in French 706) (1935). — See *C. A.* 29, 7045. A. A. Boehlingk.

Sulfur compounds that have thus far been found in brown-coal tar. Gerhard Free. *Chem.-Ztg.* 59, 975-7 (1935). E. H.

Sinaia strata of Introsura Buzau, Roumania [occurrence of coal] (Zaharesco) 8. Foundry coke (Mordecai) 9. Gravity sepn. app. [for coal-washing plant] (Brit. pat. 435,080) 1. Polynuclear org. compds. [from coal tar] (Brit. pat. 435,254) 10. Cracking and hydrogenating tars and coal (Fr. pat. 787,780) 22. Flow-controlling device for gas wells (U. S. pat. 2,026,844) 22. Cracking tar oils, bituminous schist oils or primary oils from low-temp. coal distn. (U. S. pat. 2,027,464) 22.

Motor fuels. Johanna M. H. K. Classen (nee Bischoff). Ger. 622,087, Nov. 19, 1935 (Cl. 235 4 01). Liquid fuels of high b. p. are mixed with up to about 25% of 2-furaldehyde. Liquid fuels of low b. p., e. g., benzene or alc., may be added to the mixts. Cf. *C. A.* 29, 8294.

Fuel for Diesel engines. Wilhelm Wilke, Robert Stadler and Franz Lappe (to I. G. Farbenind. A.-G.) U. S. 2,028,308, Jan. 21. To a hydrocarbon oil heavier than naphtha, an addn. is made of about 0.5-20% of a fluid aliphatic hydrocarbon material contg. more than 2 C atoms and having more than one multiple C bond, such as diacetylene to reduce the pressure at which spontaneous combustion of the oil would normally occur.

Device for mixing air with gaseous internal-combustion-engine fuel. Harrison V. Snodgrass. U. S. 2,027,492, Jan. 14. Structural and operative details.

Acetylene. Herbert P. A. Groll and James Burgin (to Shell Development Co.). Can. 353,604, Oct. 15. A gaseous mixt. of hydrocarbon and O is subjected to in-

complete combustion in an enclosed app. at a linear gas velocity not greater than the flame velocity of the mixt. The linear gas velocity is subsequently increased to exceed the flame velocity throughout at least the greater part of the flame while combustion is maintained by part of the flame proper.

Destructive hydrogenation. Friedrich Uhde. Brit. 435,457, Sept. 20, 1935. In the hydrogenation of carbonaceous materials by treatment with nascent H obtained by the interaction of finely divided metals and  $H_2O$ , with or without accelerators, as described in Brit. 339,317 (*C. A.* 25, 2557) and 405,371 (*C. A.* 28, 4867), the reacting materials are pumped continuously to the reaction vessel from which the solid products are withdrawn, sepd. from the vaporous products, subjected to reduction to reform the metals and returned to the reaction vessel in a continuous cycle. App. is described.

Breaking up coal and other minerals. Daniel Hilgenstock. Ger. 619,663, Oct. 5, 1935 (Cl. 50c 17 40). The formation of slack splinters, etc., is avoided by carrying out the breaking operations for coal, or other minerals, under a liquid such as water.

Apparatus for separating coal and like fragmentary material to be separated into classes. Kenelm C. Appleyard, Alfred Flint and The Birtley Co. Ltd. Brit. 435,263, Sept. 12, 1935.

Apparatus for separating dust from coal before washing. Westfalia-Dinnendahl-Gröppel A.-G. Fr. 788,232, Oct. 7, 1935.

Device suitable for separating and washing coal, etc. Wm. C. Menzies and Francis H. Blatch (Blatch to Wilmot Engineering Co.). U. S. 2,026,903, Jan. 7. Various structural, mech. and operative details.

Rotary-drum drier suitable for fine coal or other granulated materials. Henry F. Hebley and Klaas Prins. U. S. 2,027,977, Jan. 14. Various structural, mech. and operative details.

Furnace for low-temperature distillation of schists, lignite, coal and tar. Igor Soubbotin. Fr. 788,408, Oct. 10, 1935.

Coal distillation. Carl Stull G. m. b. H. Ger. 622,455, Nov. 28, 1935 (Cl. 10a 19 01). Means is described for effecting a gas-tight junction between a channel in the charge of a coal-distn. retort or chamber oven and a tube for withdrawing gases.

Coal-distillation furnaces. Slammit-Indugas Gaswerks-ofenbau-G. m. b. H. Ger. 619,846, Oct. 8, 1935 (Cl. 26a 1 02). Details of the heating arrangements of small furnaces are given.

Pressure-controlling means for coal-distillation retorts. Archibald A. Macintosh. Ger. 622,175, Nov. 21, 1935 (Cl. 26a 16). This corresponds to Brit. 390,167 (*C. A.* 27, 5568).

Recovering oils from coal-distillation gases. Carl Stull G. m. b. H. Ger. 622,884, Nov. 19, 1935 (Cl. 26d 10 01). Addn. to 617,355 (*C. A.* 29, 8297). Gases withdrawn by suction from the interior of a coal-distn. charge are treated as described in Ger. 617,355 to recover light oils, which are then improved in quality by treatment first with a polymerizing agent of limited activity, e. g., moderately concd. or dil.  $H_2SO_4$ , and then with concd.  $H_2SO_4$ . The first treatment may be effected at atm. temp. and the second at 30-35°.

Gas-collecting main valve assembly for coal-distillation plants. Charles V. McIntire (to Smet-Solvay Engineering Corp.). U. S. 2,027,548, Jan. 14. Various structural and mech. details.

Burning coal containing iron and sulfur. Lawrence P. Creelius. Can. 354,035, Nov. 12, 1935. To prevent  $FeS$  from fusing and hardening on the grates and boiler-tube surfaces, a compn. is added to supply Cl in the fuel bed during the burning. The Cl attacks the  $FeS$  and forms  $FeCl_3$  and  $SO_2$ ,  $SO_2$  passes up the flue. The  $FeCl_3$  decomposes under heat and forms  $HCl$  and  $Fe_2O_3$ . The  $Fe_2O_3$  passes into the ash, and the  $HCl$  is broken up into Cl and  $H_2O$  by catalytic agents such as  $MnO_2$  or  $FeO_3$ . Thus Cl is always present. A suitable compn. is of  $CaCl_2$  with small amts. of  $K_2Cr_2O_7$  and  $MnO_2$ .

**Cracking and coking coal and oil mixtures.** Edgar W. Brocklebank, Wm. B. Milford and Cecil H. Lander. Brit. 435,187, Sept. 9, 1935. Addn to 421,556 (C. A. 29, 45322). The process of Brit. 421,556 is modified in that a mixt. of coking and noncoking coal is used that contains over 10% ash and is ground to a fine powder to distribute the ash finely throughout the mixt. The ground material is incorporated with an equal proportion of oil having a b. p. above 200° by passing it while suspended in air into the oil maintained at 65°. The mixt. is then carbonized in a retort as described in Brit. 393,601 (C. A. 27, 5943).

**Organic products from coal.** I. G. Iarbenud-A-G (Carl Krauch and Mathias Pier, inventors). Ger. 619,739, Oct. 12, 1935 (Cl. 120 105). Useful org. products are obtained by treating coal-distn. or -extrn. products with H<sub>2</sub> or reducing gases (except H<sub>2</sub>S) in a streaming current at a pressure of 150 atm. or over, and in the presence of catalysts consisting of heavy metal sulphides. Thus, powd. Fe is mixed with a small amt. of Al<sub>2</sub>O<sub>3</sub> and alkali, and fused in a current of O<sub>2</sub>. The product is treated with H<sub>2</sub>S at 450°. The resulting catalyst is heated to 450-500° and a current of H<sub>2</sub> and crude cresol vapor from coal tar is passed over at 150 atm. The cresol is reduced or hydrogenated to give hydrocarbons. Other examples are given.

**Plant for cooling lignite briquets.** Humboldt-Deutschemotoren A-G. Ger. 619,917, Oct. 9, 1935 (Cl. 104 902).

**Industrial heating apparatus.** Alexander T. Stuart. Can. 354,059, Nov. 12, 1935. Surplus power from a central elec. station is used for the electrolysis of H<sub>2</sub>O. Part of the O produced is used to gasify fuel and another part to enrich the air for the combustion of the fuel gas.

**Steam generator of the water-tube type.** Arthur Lieberherr (to Sulzer Gebrüder Akt. Ges.) U. S. 2,027,443, Jan. 14. Structural and operative details.

**Steam superheaters.** John Johnson. Brit. 434,465, Sept. 2, 1935.

**Steam superheaters.** The Superheater Co. Brit. 435,883, Oct. 1, 1935.

**Steam superheater with temperature-regulating device.** Schmidt'sche Hesselampf-Ges. m. b. H. Ger. 622,032, Nov. 18, 1935 (Cl. 124 1102). This corresponds to Brit. 404,125 (C. A. 28, 38759).

**Nonpoisonous fuel gas.** Non-Poisonous Gas Holding Co. Ltd. Brit. 435,338, Sept. 26, 1935. See Fr. 783,151 (C. A. 29, 70567).

**Purifying gases.** N. V. Machinefabriek-en Apparaten Fabrieken "Meaf." Brit. 434,678, Sept. 6, 1935. CS<sub>2</sub> is removed from gases contg. H<sub>2</sub> and H<sub>2</sub>S and free from O<sub>2</sub> by passing the gas at 500-600° over Cu or CuO to form CuS, the gas being kept in contact therewith to convert the CS<sub>2</sub> to CH<sub>4</sub> and H<sub>2</sub>S, the latter being removed in a subsequent stage. Alternatively, CuS may be present initially, in which case the presence of H<sub>2</sub>S is not essential.

**Purifying flue gases.** Lodge-Cottrell Ltd. and Jens O. Roving. Brit. 435,560, Sept. 23, 1935. Oxides of S are removed from the effluent gases of boiler and like power plants by bringing said gases, without previous moistening, at about 400-625° and substantially at atm. pressure directly into contact with dry solid CaO, Ca(OH)<sub>2</sub>, or CaCO<sub>3</sub>. Catalysts, e. g., FeO<sub>3</sub>, may be mixed with the Ca compounds.

**Coal gas.** Heinrich Koppers G. m. b. H. Ger. 619,847, Oct. 9, 1935 (Cl. 24 J 9 04). H<sub>2</sub>S and other impurities are removed from gases such as coal gas by alk. washing solns. contg. compds. of the Sn group, e. g., S-Ac-O compds., and a small amt. of Mn or Cu compds. as catalysts. Cf. C. A. 29, 10274.

**Purifying coal gas.** Ges. für Linde's Gasmaschinen A-G (Paul Schulten, inventor). Ger. 619,809, Oct. 9, 1935 (Cl. 24 J 12 01). A process is described for purifying coal-distn. gas by cooling to below 0° and bringing the cooled dry gas into contact with further warm moist coal gas.

**Air gas producer.** Aldo Curioni. Ger. 619,930, Oct. 10, 1935 (Cl. 24 C 3). App. for producing a combustible gas from air and heavy liquid fuel is described.

**Carbureted water gas.** Owen B. Evans (to United Gas Improvement Co.). U. S. 2,026,577, Jan. 7. An

ignited fuel bed is air-blasted, the blast gases are burned with secondary air introduced marginally adjacent the upper edge of the fuel bed, and the flow of the blast gases is divided and they are passed in two streams through two heat-exchange vessels so that the amt. of heat stored in each vessel is controlled. Steam and oil are introduced to the fuel bed and the oil is vaporized and the blue water gas formed is carburated by the heat stored adjacent the upper part of the fuel bed. The water gas and oil vapors thus produced are led in by-pass relation to one of the heat-exchange vessels, oil vapors are fixed by heat stored in the other heat-exchange vessel, and the gas is led off. A back run is made through the fuel bed with steam passed through the by-passed heat-exchange vessel and heated by the remaining portion of the heat recovered from the blast gases. App. is described. Cf. C. A. 29, 34984.

**Apparatus and process for making carbureted water gas.** Reginald P. Oliveres (to Somet-Solvay Engineering Corp.). Brit. 435,727, Sept. 26, 1935. In making the gas by passage of oil and steam through an incandescent fuel bed, the back-run or down-run is divided into 2 portions, in the 1st of which all or most of the oil and a small portion of the steam is passed through the fuel bed and in the 2nd the greater part of the steam and little, if any, oil is passed through.

**Gas producers.** Robert Falconer and Eduardo Colignon. Brit. 435,976, Oct. 2, 1935. A combustible gaseous mixt. is obtained by supplying liquid fuel to an open-ended retort which extends into the fuel bed of a gas producer, the lower end of the retort reaching a point above the reduction zone.

**Tubular feed device for gas generators.** Wehrle-Werk A-G. Ger. 622,038, Nov. 18, 1935 (Cl. 24 E 9). Addn to 621,343 (C. A. 30, 12149).

**Means for removing ash from rotary-grate gas generators.** Hermann Gochitz. Ger. 617,364, Dec. 10, 1935 (Cl. 24 E 11.03).

**Demulsifying the sealing oils of waterless gas holders.** Hans Dellmann (to Maschinenfabrik Augsburg-Nürnberg A-G). U. S. 2,027,965, Jan. 14. Emulsion-forming impurities such as metal dust, other dust, linseed oil, etc., are removed from used sealing liquids substantially free from sulfonated hydrocarbon material by slightly heating the water-oil emulsion which has formed in use, adding at atm. pressure a mixt. contg. soap and water-glass at least 1% and allowing the mixt. to stand and sep. into layers, water and impurities forming an upper layer which is sep'd.

**Removing hydrogen sulfide from gaseous mixtures.** Paul E. Kuhl (to Standard Oil Development Co.). U. S. 2,028,336, Jan. 21. In removing H<sub>2</sub>S from a gaseous mixt. by carrying out a chem. reaction in 2 reaction zones between H<sub>2</sub>S and another reactive gas such as NH<sub>3</sub> or SO<sub>2</sub>, the gas mixt. contg. these mutually reactive gases is passed through the first reaction zone concurrently with a liquid medium contg. gases absorbed in the 2nd reaction zone, and in the 2nd reaction zone the gases are subjected to countercurrent treatment with fresh liquid medium such as water. App. is described.

**Removing hydrogen sulfide, etc., from gaseous mixtures.** Joseph A. Shaw (to Koppers Co. of Del.). U. S. 2,028,124, Jan. 14. Gas such as coal gas, oil gas, etc., is scrubbed with a corrd. aq. soln. of an alkali metal salt of phenol or an alkylated phenol substitution product, the unabsorbed gas is sep'd and the total liquid product from the absorption step is heated substantially to the b. p. to expel H<sub>2</sub>S and the aq. liquor is returned to the scrubbing step. Various details of app. and operation are described. U. S. 2,028,125 relates to a process in which a soln. which may contain about 10% of NaOH and 15% of tar acid is used for treating gas for the selective sep'n and recovery of H<sub>2</sub>S. App. is described.

**Removing hydrogen sulfide and other acid compounds from gases containing ammonia.** The Koppers Co. of Delaware. Ger. 622,042, Nov. 18, 1935 (Cl. 24 J 9 04). See Brit. 392,544 (C. A. 27, 5519) and U. S. 1,922,307 (C. A. 28, 6159).

**Recovering benzene from coal gas.** Carl Stull G. m. b.

11 Ger 622,370, Nov 27, 1935 (Cl 26d 930) Oil used for exg. benzene from coal gas is purified by washing the hot, benzene satd. oil with hot water. The oil is then sepd. from the water and from tar and like heavy impurities by sedimentation at a temp. above 100°. A continuously operating app. is described. Cl C A, 29, 341.

**Coking coal in a continuous internally heated retort.** Arthur W. Warner (to Isbell Porter Co.) U S 2,025,923, Jan 7. A column of coal in a retort is ignited at one end to form a combustion zone, and O bearing gases are forced through the combustion zone to maintain it and coke the coal. Coke is introduced into the central portion of the column above the combustion zone so as to form a core for the column, gases generated in the column are removed from its top, these gases passing through the coal column, and the residue is removed from the bottom of the column, so that an upstanding plastic film of conical shape of melted coal is formed and maintained vented at the apex of the coke core. App. is described.

**Coke-quenching car.** The Wellman Smith Owen Engineering Corp. Ltd. and Geo T. Purves Brit 435,127, Sept 16, 1935.

**Coking process.** H. A. Brassert & Co. Ltd. and Herman A. Brassert Brit 434,737, Sept 9, 1935. Coal, etc.,

is coked by being charged into a horizontal oven which is heated mainly through the floor, liquid hydrocarbons being sprayed onto the surface of the charge from the roof of the oven.

**Coke ovens.** C. Otto & Co. G. m. b. H. Brit 435,512, Sept 23, 1935. To assist the withdrawal of distillates, holes, which communicate with the space above the charge, are made in the charge by mandrels shortly after the coking seam has reached such a depth from the surface that during further carbonization the shape of the holes is maintained.

**Coke ovens.** C. Otto & Co. G. m. b. H. Brit. 435,842, Sept 30, 1935. The walls surrounding the coking chambers and heating flues are made of clay bound chamotte bricks in which the content of binding clay does not exceed 10%.

**Coke ovens.** H. A. Brassert & Co. Ltd. and Tom P. Colclough Brit 435,938, Sept 30, 1935. In an oven heated by parallel floor flues, adjacent ones of which are fired from opposite ends, the waste-gas flues leading to the exhaust main are arranged between flues supplying air for combustion.

**Coke oven.** Carl Stull G. m. b. H. Ger 618,740, Sept 14, 1935 (Cl 10a 36 03). A device is described for installation into coke ovens for producing distn. or middle-temp. coke. Cl C A 30, 844.

## 22—PETROLEUM, LUBRICANTS, ASPHALT AND WOOD PRODUCTS

W. F. PARAGHER

**The French petroleum market.** Ch. Berthelot *Chimie & industrie* 34, 1225-33 (1935). A. Papineau-Couture.

**Petroleum products and their use.** A. J. Partanen *Teknillinen Aikakauslehti* 25, 375-83 (1935).—A discussion of the production and manuf. of petroleum products, properties, research methods, international trade in oils and the significance of large companies in the production and marketing of petroleum products.

L. E. Jukkola.

**The technical principles of cracking.** Raphael Fossing *Mot. grasses* 27, 10465-6, 10497-600, 10627-8 (1935). Cl C A 29, 3415. —A brief review. A. P. C.

**Interfacial tension in the oil industry.** F. Vellinger *Petroleum Z.* 31, No. 34, 17-21 (1935).—Measurement of interfacial tension is of importance in following the removal of impurities in refining and for detg. the aging or the origin of oxidation products. The du Nouy method is described. Plotting the tension against the  $p_n$  value of the aq. soln. gives 2 types of curves, one which is nearly parallel to the  $p_n$  abscissa and another which falls rapidly at high  $p_n$  values. The light fractions of crude oil distillates follow the 1st type of curve, the heavy fractions the 2nd. The surface tension is independent of the mol. wt. but is a function of the degree of refining. Measurement of interfacial tension is of value in controlling acid treatment, washing, neutralization and bleaching action. Aged oils contg. oxidation products have greatly decreased interfacial tension. This is important in studying lubricating oils for turbines and transformer oils.

C. B. Jenni.

**The relation between the antiknock properties of light motor fuels and their physical properties.** Richard Heinze and Maximilian Marder *Angew. Chem.* 48, 770-9 (1935). Cl C A 29, 5411. —Twenty-five natural gasolines, 7 cracked gasolines, 1 synthetic gasoline, 6 gasolines from lignite and 4 cracked lignite gasolines were investigated, and the following properties are presented: dia. surface tension, sp. paracher,  $n_D^{20}$ , sp. refraction, corrected boiling no., octane no. (O. N.) (C.R. Motor), O. N. from sp. paracher O. N. plot, O. N. obtained from  $d_{40}^{20}$ -O. N. plot, O. N. obtained from  $n_D^{20}$ -O. N. plot and O. N. corrected to a boiling no. of 110°. The results agree with and amplify the earlier data, and show that approx. detns. of the O. N. can be made from relationships with phys. consts. other than the paracher.

Karl Kammermeyer.

**Synthetic acids of petroleum hydrocarbons and their uses.** Rob. Neublyum *Nitrocellulose* 6, 141-3 (1935). —

A review. F. M. Symmes.

**The separation and utilization of  $\alpha$ - and  $\beta$ -nitrotoluene from mononitrotoluene prepared from the gasoline fraction of Sykkökö crude oil.** I. Separation of  $\alpha$ - and  $\beta$ -nitrotoluene. Masakichi Mizuta *J. Soc. Chem. Ind., Japan* 38, Suppl. banding 576-7 (1935), Cl C A 28, 3017. — $\alpha$ -NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>3</sub> was sepd. from the mixed mononitro compds. by vacuum distn. with a 300-cc. flask, with a column 13 mm. in diam. and 1360 mm. long, filled with Al. Leasing rings, having an air-heated jacket and a reflux controlling device.  $\alpha$ -Nitrotoluene of 98.2% purity was obtained, amounting to 89.2% of the  $\alpha$ -compd. present. The  $\beta$ -compd. was sepd. by repeated crystals from the alc. soln. of the distn. residue with a purity of 98.8% (m. 51°) and a yield of 85%. K. K.

**Classification of lubricating oils.** Lya Neyman *Petroleum Z.* 31, No. 30, *Motorentrieb u. Schmierung* 8, No. 9, 2-6 (1935). —The quotient of the mol. wt. divided by the  $d$  gives a no. which is characteristic and useful for purposes of classification. By reason of the fact that the viscosity of oils of the same class of hydrocarbons increases with increasing mol. wt. and the observed influence of the mol. wt. on the temp. viscosity curve, the formula  $\frac{1}{d} = \frac{M}{M_0} [d_0 - \alpha]$  was established. This value is believed to characterize the oils. It depends only on the  $d$ , the mol. wt. and the viscosity of the oils investigated at 2 temps. C. R. Jenni.

**Investigation of lubricants with oil-testing machines.** Theodor Rabinovitch *Petroleum Z.* 31, No. 30, 14-17 (1935). —Types of oil-testing machines used are pendulum machines, friction scales, discharge machines, instrumentation machines, bearing testing machines and miscellaneous. Fundamental principles and examples of each class are discussed. C. B. Jenni.

**Regeneration of special and lubricating oils.** Maurice Van Rysselberghe *Ind. chim. belge* 6, 471-8 (1935), Cl C A 25, 3314. —A review of work done in countries other than Belgium on the regeneration of elec. insulating and lubricating oils. A. Papineau-Couture.

**The surface tension of asphalt bitumen.** R. N. J. Saal *Chem. Beekblad* 32, 440-8 (1915). —Detns. of the surface tension of 7 different bitumens of different origins and penetration values, natural and blown, gave at temps. 110°, 120° and 140° values all decreasing linearly with

temp. The du Noy app. was used. Detns by Nellensteyn with the Jaeger bubble method (*Z. anorg. allgem. Chem.* 101, 1 (1917)) did not give straight-line decrease, but the  $\sigma$ -T curve had a sharp bend at temps between 117° and 145°. It is concluded that Nellensteyn's values (N. and Loman, *Asphalt bitumen*, etc., Amsterdam (C. A. 26, 1763)) at temps below the bend are fictitious and due to the method used. The latter is unsuited for  $\sigma$  detns at viscosities of more than 15 poises. This was checked by detn of the total surface energy  $L(= \sigma - T d\sigma/dT)$  of the different asphalts and of road tar. The du Noy  $\sigma$  values indicated E values of about 50 ergs per sq cm for asphalts, 70 for tar, this agrees well with known E values of aliphatic compds of around 50, and of aromatic compds around 70 (Harkins, *C. A.* 11, 1558). Nellensteyn's  $\sigma$  values would indicate E up to 150. Conclusions of N. regarding the "semisolid" state of bitumen below the kink in the curve are obviously unjustified.

B. J. C. van der Hoeven

Obtaining camphor from oven turpentine B. N. Rutozskii, V. N. Karacheva, T. P. Andreeva, O. M. Klepikova and L. N. Mogilevskina *J. Chem. Ind. (Moscow)* 12, 1177 83 (1935)—Oven turpentine contg 40% of pinene gives very low yields of camphor by the pinene hydrochloride or the tetrachlorophthalic acid methods, although in the latter case, doubling the amt of the acid used raises the yield somewhat. The isomerization by the (bornyl) acetate method is satisfactory and gives yields of camphor nearly equal to those from natural turpentine.

H. M. Leicester

Geology of Texas Panhandle oil and gas field (Rogatz) 8 Bituminous shales of Saulx-de-Vesoul (Longchamps) 8 Synthesis of TiOH from artificially prep'd gases (Oil Inst. in Baku) 10 Containers of treated paper for holding lubricating oils (U. S. pat. 2,027,390) 13 Cracking and coking coal and oil mixts (Brit. pat. 435,187) 21. Appar. for testing lubricants (U. S. pat. 2,027,903) 1

Purifying petroleum oils Leo D. Jones (to The Sharples Specialty Co.) Brit. 435,826, Sept. 30, 1935 See Fr. 771,988 (C. A. 29, 1617)

Apparatus for treating petroleum vapors, etc. Horace M. Weir (to Atlantic Refining Co.) U. S. 2,027,410, Jan. 14. A bed of solid particles such as clay or fuller's earth is supported on a contacting layer of steel wool (various structural details being described).

Electrical dehydration of materials such as petroleum emulsions of the water-in-oil type Paul W. Prutzman (to Socony-Vacuum Corp.) U. S. 2,027,615-16, Jan. 14. Various details of app. and operation are described.

Purifying petroleum distillates The Standard Oil Co. of Ohio, Ir. 788,470, Oct. 10, 1935. Light petroleum distillates are mixed with an aq. soln of alkali, the aq. soln. is sepd. from the distillate and mixed with a soln of S in an org. solvent which is insol. in water. The aq. soln. of alkali is sepd. from the soln. of S and mixed with the petroleum distillate. By this means mercaptans are removed from the distillate. The mixt. of the aq. soln. and the distillate takes place countercurrently.

Desulfurizing petroleum distillates Vladimir Kalichevsky (to Standard Oil Development Co.) U. S. 2,028,335, Jan. 21. A distillate such as a West Texas cracked distillate is treated with a substantially anhyd. mixt. of NaOH and KOH contg about 1.25 lb NaOH per lb. of sour S in the distillate and about 0.33-0.67 lb KOH per lb. of corrosive S in the distillate.

Tube still and furnace suitable for use in pyrolytic treatment of petroleum oils John S. Wallis and Stephen B. de Bence (to Alco Products, Inc.) U. S. 2,028,305, Jan. 21. Various details.

Pyrogenic decomposition of hydrocarbon materials such as those of crude petroleum. Ovid E. Roberts, Jr. U. S. 2,027,552, Jan. 14. A plurality of hydrocarbon fractions of different b. ps. are decomposed in the vapor phase by flowing them through a reaction zone in countercurrent contact with a liquid metal catalyst (such as one contg.

1 Pb 90 and Na 10%), each fraction being separately introduced into the reaction zone to bring the liquid catalyst into contact at points which afford the optimum conditions of pyrogenic decomp. and coupling necessary for proper stabilization. App. is described.

Refining products of petroleum cracking Jacques C. Morrell (to Universal Oil Products Co.) U. S. 2,027,648, Jan. 14. A lighter gravity liquid product is first treated with a mixt. of HNO<sub>3</sub> and AcOH and then with concd H<sub>2</sub>SO<sub>4</sub>.

Cracking hydrocarbon oils Lester Kirschbraun (to Universal Oil Products Co.) U. S. reissue 10,818, Jan. 7. A reissue of original pat. No. 1,824,215 (C. A. 26, 292).

Cracking hydrocarbon oil Victor Stapleton (to Texas Co.) U. S. 2,026,803, Jan. 7. Oil such as a heavy gas oil is heated to a cracking temp. while passing through a heating zone such as a pipe coil and resulting products

3 are discharged into a sepg. zone from which vapors and residual liquid are separately removed. At least a portion of the vapors is passed through a high-pressure fractionating zone to effect condensation of constituents heavier than gasoline. The residual liquid is flashed into a zone of lower pressure to liberate lower boiling constituents in vapor and the liberated vapors are passed through a relatively low-pressure fractionating zone to condense constituents heavier than gasoline, and gasoline condensate

4 is sepd. from the vapors and gases remaining uncondensed in a low-pressure sepg. zone, remaining vapors and gases are compressed and commingled under the increased pressure with vapors leaving the high-pressure fractionating zone, combined vapors and gases are cooled under a pressure in excess of that of the low-pressure sepg. zone to effect a condensation of gasoline constituents and the gasoline condensate thus obtained is sepd. from asocd gases under superatm. pressure. App. is described. Cf. C. A. 30, 12,04.

Cracking hydrocarbon oil Norris Goodwin and Robert W. Ponder (to Industrial Process Co.) U. S. 2,027,802, Jan. 14. A continuous process involves preheating a stream of oil such as a refinery residual oil and passing the preheated oil into a revolving still forming a single chamber the diam. of which is at least twice that of the stream while being preheated, to diminish the velocity

6 of flow, subjecting the oil in the chamber to a cracking temp. in the presence of metal balls which may be formed of Ni or Fe and which together with the oil about half fill the chamber, continuously withdrawing all the products of cracking in a single stream from a point near the liquid level in the chamber at a rate of from 2 to 3 gal. of gasoline and (or) 2 to 3 lb. of C per sq. ft. of inner heated surface of the chamber per hr., so that incrustation is prevented and the C is kept in suspension, so that the rate of withdrawal balances the rate of supply and there is maintained in the chamber hydrocarbon oil unpreheated with C particles to accelerate the cracking action. App. is described.

Cracking hydrocarbon oils Gustav Egloff (to Universal Oil Products Co.) Can. 354,136, Nov., 1935. The oil is heated to cracking temp. under pressure, and sepd. into vapors and an unvaporized oil free from coke. The vapors are dephlegmated and reflux condensate is formed. The unvaporized oil is flash-distd. by lowering the pressure, and the residuum combined with portions of the condensate. The resultant mixt. is passed through a second heating zone and heated to cracking temp. The heated mixt. is discharged into an enclosed zone and sepd. into vapors and a coke-like residue.

Cracking hydrocarbon oils Gasoline Products Co., Inc. Ger. 622,016, Nov. 21, 1935 (Cl. 23b. 1.04). See Brit. 377,510 (C. A. 27, 4067).

Cracking hydrocarbon oils Deutsche Erdöl-A.-G. (Friedrich Schick and Eugen Emilius, inventors). Ger. 622,402, Nov. 27, 1935 (Cl. 23b. 1.04). As a contact mass for use in the vapor-phase cracking of hydrocarbon oils, use is made of coke or semicoke which has been prep'd from coal or brown coal by low-temp. carbonization and which has been activated by steam at a temp. up to about 650° and then freed wholly or in part from ash,



e.g., by successive extns. with HCl and water. Preferably, coke or semicoke contg. more than 5% of basic compds. of alkali or alk. earth metals, a part of which may be added if necessary, is activated and then de-ashed for use in the process.

**Cracking and hydrogenating oils** Gutehoffnungshütte Oberhausen A-G Brit 435,809, Sept 30, 1935 Hydrocarbon oils are cracked and (or) hydrogenated by passage together with H and O or O-contg. gas mixts. over catalysts, the reaction proceeding exothermally and without external heating due to the reaction of the H with the O in the presence of catalysts promoting oxidation, e.g., V, W, Mo, Pt, Pd. App. is described.

**Cracking and hydrogenating oils, tars and coal** I. G. Farbenind A-G Fr 787,780, Sept 29, 1935 The starting materials are treated, before the heat treatment, with an acid so dild. (10% HCl) that only the N bases are dissolved out while the olefins remain intact. A much higher yield of low boiling compds. is obtained from the subsequent cracking or hydrogenation.

**Cracking oils such as gas oil** Frank H. Edson (to Standard Oil Development Co.) U. S. 2,027,355, Jan 7. A stream of oil from a source of supply is divided into parts, one of which is passed under pressure into a conversion chamber into which oil is introduced from a heating zone after being heated to a cracking temp., vapors and liquid are separately withdrawn from the conversion chamber, the vapors are condensed, the liquids are brought into contact with the remaining part of the initial heavy oil, the mixt. is fractionated and fractions of different b. ps. are separately recovered, a lower- and a higher-boiling fraction are subjected to pressure, and the lower-boiling fraction of the mixt. is passed in a stream through the heating zone, the higher-boiling fraction is passed into the stream of the lower-boiling fraction after it has been heated to a cracking temp., and the mixt. is heated and passed into the conversion chamber. App. and various auxiliary details of operation are described.

**Cracking oil for gasoline production** Otto Behmer (to Texas Co.) U. S. 2,027,014, Jan 7. Oil to be treated is forced through a coil in which it is heated to a decomp. temp. without substantial cracking and thence into an enlarged drum under pressure in which decomp. of the oil is effected, while any return to the coil of oil remaining unvaporized in the cracking drum is avoided. App. is described.

**Cracking tar oils, bituminous schist oils or primary oils from low temperature coal distillation** Louis Boulanger and Ferdinand Emsens U. S. 2,027,464, Jan 14. The oils are oxidized by dild. O<sub>2</sub>, washed with lime water and treated in a dehydrator, vaporized in a retort and the vapors are superheated at a temp. above 800°, the major portion of the phenols is condensed from the vapors, they are mixed with O<sub>2</sub> and N monoxide, treated with lime water and magnesia, then passed through a soln. of KOH to retain remaining phenols, and benzene is condensed. App. is described.

**Fractionally condensing mixed hydrocarbon vapors such as those from oil cracking** Luis de Florez (to Texas Co.) U. S. 2,027,334, Jan 7. Numerous details of app. and operation are described.

**Apparatus for hydrolyzing acid sludge from hydrocarbon oil treatment** Harry W. Thompson and John T. Rutherford (to Standard Oil Co. of Calif.) U. S. 2,026,714, Jan 7. Various structural and operative details are described of an app. in which flow of tar to a tar-withdrawal outlet is permitted substantially only from the interface between acid and tar layers.

**Refining hydrocarbon oil vapors** Richard J. Dearborn and Wm. M. Stratford (to Texas Co.) U. S. 2,028,100, Jan 14. Vapors contg. unstable unsatd. constituents are passed horizontally through a bed of solid adsorptive catalyst such as fuller's earth promoting polymerization of such constituents, at a temp. below that of cracking, and the polymers formed are condensed and removed from the lower portion of the bed after they have condensed and flowed downwardly through it. App. is described.

**Conversion of hydrocarbon oils** Luis de Florez (to

Texaco Development Corp.). Can 353,990, Nov 5, 1935. The color of a condensed sample of a portion of vapors from which the cycle condensate for the cracking process is formed is used as a criterion of the cleanliness of the condensate. The extent of fractionation is controlled in response to variations in color of the sample in order to obtain a condensate without detrimental coke deposition.

**Conversion of hydrocarbons** Henry Dreyfus U. S. 2,020,731, Jan 7. For producing hydrocarbons contg. a lower percentage of H from satd. hydrocarbons b. below 150° (such as a mixt. of C<sub>12</sub>H<sub>22</sub> and C<sub>14</sub>H<sub>30</sub>), the said hydrocarbons in the gaseous phase are heated to 350-900° in the presence of liquid or semiliquid compns. contg. more than 80% of free C in a finely divided form, such as a heavy hydrocarbon oil bath contg. free C in suspension.

**Heating-chambers for hydrocarbons** H. A. Brassett & Co. and Herman A. Brassett. Brit 434,948, Sept 9, 1935. The floor of a chamber for heating solid, plastic or fluid hydrocarbons, which seps. the heating flues from the chamber proper, is formed of a series of shaped blocks adapted for relative movement so that under expansion the floor may flex.

**Destructive hydrogenation of middle oils** Mathias Pier, Walter Simon and Walter Kroenig (to Standard-I. G. Co.) U. S. 2,028,348, Jan 21. A middle oil is subjected to the action of H at a temp. of about 440° and under a pressure of 200 atm. in the presence of a catalyst consisting of Mo bromide and W sulfide.

**Aqueous dispersions of mineral oils, etc.** Leonard G. Gabriel, John F. T. Blott, Wm. L. Peard, Lawrence W. G. Firmin, Louis Meunier and Johan L. van der Munne (to Flintkote Co.) U. S. 2,027,584, Jan 14. A crude petroleum oil, oil fraction or distillate or residue, pitch, bitumen or the like is combined with water-sol. substances such as NaCl or glycerol in sufficiently small proportions that when the material is dispersed in water there are produced within the dispersed particles osmotic pressures which are mutually greater than those exhibited by the continuous phase so that absorption of water from the continuous into the dispersed phase is effected (various details of procedure being described).

**Products from mineral oils** N. V. de Bataafsche Petroleum Maatschappij Fr 788,676, Oct. 14, 1935. Kerosenes, gas oils, lubricating oils as well as solid resinous products and film-forming oils are obtained by causing mineral oils contg. aromatic compds. or mixts. of hydrocarbons contg. aromatic compds. and derived from mineral oils, to react in the presence of AlCl<sub>3</sub> or like condensation catalysts with acetylenic hydrocarbons or gaseous mixts. contg. them, the reaction mixt. being worked up afterward to obtain the desired products.

**Moderate temps. (30-50°) are used.**  
**Refining mineral oils** Edwin R. Burkholder (to Atlantic Refining Co.) U. S. 2,026,012, Jan 7. Acetophenone is used as a selective solvent for sepg. paraffinic and naphthenic hydrocarbons.

**Refining light oils** Walter A. Patrick, Jr. Brit 435,194, Sept 16, 1935. This corresponds to Fr 770,874 (C. A. 29, 919).

**Flow-controlling device for use at the top of oil or gas wells** James L. Minahan U. S. 2,026,844, Jan 7. Structural and operative details.

**Motor fuel** Nicolaas Max (to Shell Development Co.) U. S. 2,023,372, Dec 3. For rendering it "non-knocking" a fuel of the gasoline type is treated with about 0.025-0.05% metal of the Cu salt of hydroxymethylene camphor or other suitable metal compd. of the general formula RCOC(CH<sub>3</sub>)X'R', in which X represents OH or NH<sub>2</sub>, R represents an O-substituted hydrocarbon radical and R' represents H or a member of the org. radical represented by R.

**Motor fuel** Carbo-Norit-Union Verwaltungs-Ges. m. b. H. Fr 787,920, Oct. 1, 1935. Crude gasolines and benzenes are treated with anhyd. FeCl<sub>3</sub> before or during distn. so as to reduce the content of resin forming, odoriferous and coloring substances therein. The FeCl<sub>3</sub> may be used in alc. soln.

**Stabilization of motor fuel** Richard G. Clarkson and Claude G. Wolff (to Canadian Industries Ltd.). Can. 353,560, Oct. 15. A small amt. of a gum inhibitor is added to the fuel after cracking and before the final distill. The gum inhibitor has a vapor pressure above 50 mm. at the h. p. of the motor fuel.

**Stabilizing cracked gasoline.** Edward W. McMullan (to Gasoline Antioxidant Co.). U. S. 2,027,304, Jan. 14. Stabilization against gum formation is effected by adding a small proportion (suitably about 0.1% or less) of azobenzene, azoxybenzene or hydrazobenzene.

**Stabilizing cracked gasoline.** Eugene Ayres and Mark L. Hill (to Gulf Refining Co.). U. S. 2,027,462, Jan. 14. A small proportion (suitably about 0.01% or less) of a crude condensation product formed by reaction of di- $\alpha$ -naphthylamine with cresol is added to the gasoline to inhibit gum formation.

**Treating cracked gasoline to effect stabilization.** Wayne L. Benedict (to Universal Oil Products Co.). U. S. reissue 19,804, Jan. 7. A reissue of original pat. No. 2,014,923 (C. A. 29, 7636).

**Gasoline.** N. V. de Bataafsche Petroleum Maatschappij. Brit. 435,230, Sept. 17, 1935. See Fr. 789,916 (C. A. 29, 6042).

**Gasoline of high octane number.** Oceola P. Cottrell (to Fdeleann G. m. b. H.). U. S. 2,024,121, Jan. 14. In converting substantially all of a straight-run gasoline into high octane number gasoline, the gasoline is sep'd into a low-boiling and a higher-boiling fraction and the latter is treated with a solvent such as liquid  $\text{SO}_2$ , the raffinate and ext. are sep'd, the solvent is removed, the raffinate is cracked, the cracked raffinate is recycled by mixing it with the straight-run gasoline stock entering the cycle, and the ext. is blended with the low-boiling fraction to produce the high octane number gasoline.

**Refining benzine.** I. G. Farbenund. A.-G. (Hans Häuber, inventor). Ger. 622,017, Nov. 10, 1935 (Cl. 23b 1.03). Benzine is freed from unsat'd resin-forming hydrocarbons by treatment at about 50° with a previously prep'd. double comp'd. of  $\text{AlCl}_3$  and an olefin. The double comp'd. may be prep'd by treating  $\text{AlCl}_3$  suspended in an org. solvent with  $\text{C}_2\text{H}_4$  or  $\text{C}_3\text{H}_6$  at 40–60° Sp. processes are described.

**Ceresin from ozocerite.** Peter J. Wievech (to Standard Oil Development Co.). U. S. 2,029,307, Jan. 21. Ozocerite is treated at a temp. above its m. p. with a light hydrocarbon solvent comprising a major proportion of hydrocarbons contg. 3–5 C atoms such as liquefied  $\text{C}_3\text{H}_8$  and under a pressure sufficient to retain the light hydrocarbon material in the liquid phase at the working temp., undissolved material is sep'd, and the soln. thus formed is heated, without substantial intermediate cooling, to a temp. (suitably about 90°) close to the crit. temp. of the solvent so that a portion of the ozocerite is ppt'd. and carries with it a substantial proportion of the colored material present in it, the ppt. is removed, the remaining soln. is filtered through a bed of finely divided clay, and the ceresin is recovered from the filtered soln.

**Dewaxing hydrocarbon oils.** Francis X. Gowers (to Texaco Development Corp.). Can. 354,130, Nov. 12, 1935. The oil is mixed with a solvent comprising 28–35% of an aliphatic ketone such as acetone and  $\text{MePtCO}$ , 45–60% benzene and 45–60% toluene by vol. The proportion of toluene is sufficient to prevent sepn. of the oil from the mixt. at –10° F. The mixt. is chilled and the ppt'd. wax removed. Cf. C. A. 29, 4931.

**Removing paraffin from oils.** Aktiebolaget Separator-Nobel. Brit. 435,648, Sept. 25, 1935. This corresponds to Fr. 778,880 (C. A. 29, 4931).

**Removing paraffin from oils.** The Sharples Specialty Co. Fr. 785,324, Oct. 8, 1935. Paraffin is removed by adding 1,3-dichloro-2-methylpropane to the oil as solvent, cooling to about –22° and filtering.

**Dewaxing mineral lubricating oils.** Bernard Y. McCarty and Wm. E. Skelton (to Texas Co.). U. S. 2,027,340, Jan. 7. The oil is treated with a solvent such as a mixt. of propylene dichloride with ethyleneglycol ethyl ether which at a temp. of about –22° is a selective

solvent for the oil but not the wax, and the mixt. is chilled to ppt. the wax, it is filtered out of the oil with the use of filter-aid material and the solvent is removed from the filtrate to produce a low pour-test oil.

**Lubricating oils.** John M. Harris, Jr. (to Standard Oil Development Co.). U. S. 2,025,387, Dec. 21. A wax-contg. hydrocarbon oil is treated to produce a lubricating oil which is resistant to oxidation and has low haze-forming and sludge-forming tendencies, by subjecting the oil to the action of O-contg. gases at a temp. of about 65–200° for a time insufficient to oxidize more than about 5% of the oil, and then dewaxing the oil. App. is described.

**Lubricating oil.** Erwin R. Lederer. U. S. 2,027,734, Jan. 14. For producing a –18° cold test distillate fraction of paraffin base lubricating oil from the neutral fraction normally produced as part of a paraffin base long residuum in dewaxing it to –19° cold test, a portion of the undewaxed residuum is mixed with the neutral fraction, the mixt. is chld. (suitably with naphtha) and after chilling to about –30° to –50° the ppt'd. wax is removed from the chilled oil, the diluent is removed and the neutral and residual fractions are sep'd so as to provide a –18° cold test distillate fraction having a viscosity of 100° to 300° at a temp. of 34° and a –18° cold test residuum fraction stock having a viscosity of 50° to 200° at a temp. of 99°.

**Lubricating oil.** John V. Starr and David F. Edwards (to Standard Oil Development Co.). U. S. 2,023,361, Jan. 21. A lubricating oil stock is agitated with a selective solvent such as phenol, aniline or furfural and the ext. layer is sep'd from the raffinate layer, the raffinate layer is subjected to fractional distn. in the presence of its absorbed selective solvent and in the presence of an adsorbent clay and the adsorbent clay is removed from the distn. residue. App. is described. Cf. C. A. 29, 1622.

**Lubricating oils.** International Hydrogenation Patents Co. Ltd. Fr. 788,260, Oct. 7, 1935. Products b. above 300°, from the hydrogenation under pressure of lignite, are treated to remove asphalt and resins, the paraffin is removed by adding a diluent such as an oil of lower b. p. and gradually cooling, and the remainder is treated catalytically with H under pressure and at a high temp. to produce a lubricating oil.

**Hydrocarbon lubricating oil.** Ulric D. Bray and Claude E. Swift (to Union Oil Co. of Calif.). U. S. 2,026,720, Jan. 7. A process for the fractionation of a hydrocarbon oil mixt. contg. paraffinic and nonparaffinic constituents comprises commingling the oil with a light-hydrocarbon diluent such as liquid propane and a selective solvent such as  $\text{PhNO}$ , and forming a raffinate-oil phase comprising paraffinic-oil constituents and a portion of the nonparaffinic constituents dissolved in the light-hydrocarbon diluent and an ext. phase comprising nonparaffinic constituents dissolved in the selective solvent. The diluent is removed from the raffinate phase and the diluent-free raffinate-oil phase is re-ext'd. with selective solvent and further seps are effected. An arrangement of app. is described. Cf. C. A. 29, 5648.

**Petroleum lubricating oil.** John D. Fields. U. S. 2,027,770, Jan. 14. Lubricating stock is thinned with a light hydrocarbon solvent to form a mixt. of low viscosity, and the substantially anhyd. mixt. is brought into contact with a solid alkali metal in a comminuted state and at not substantially above room temp. until impurities have been coagulated, oily materials and coagulated impurities are sep'd from each other before chem. reaction of any substantial proportion of the alkali metal with hydrocarbon compds. is effected, and the light hydrocarbon solvent is dist'd. from the lubricating stock.

**Nondestructive distillation of easily decomposable materials such as petroleum lubricating oils.** Warren K. Lewis (to Standard Oil Development Co.). U. S. 2,028,340, Jan. 21. The material is evap'd in a distn. zone whence gases are withdrawn through a restricted outlet, and a stream of vaporized high-boiling hydrocarbon material such as gas oil substantially free from constituents of high vapor pressure is discharged through the restricted

outlet so that the distn. zone is maintained at a sub-atm. pressure, and the vaporized hydrocarbon is condensed. App. is described.

Apparatus (with a light-sensitive cell) for determining the lubricating quality of oils such as used engine crank case oils. Mead Cornell and Charles C. Curtis (to Charles C. Broadwater). U. S. 2,027,518, Jan. 14. Various structural and operative details are described.

Separating paraffinic and nonparaffinic hydrocarbons in refining oils such as lubricating oil fractions. Louis A. Clarke (to Texas Co.) U. S. 2,027,354, Jan. 7. A nitro-alc. such as nitrobenzyl alc. is used as a selective solvent for sepn. of nonparaffinic constituents.

Filter suitable for treating lubricating oils. Robert H. Pennebaker U. S. 2,027,876, Jan. 14. Various structural and operative details.

Oil filter containing filter screens and filter clay and suitable for use with engine lubricating oil. Robert H. Pennebaker U. S. 2,027,870, Jan. 14. Structural and operative details.

Lubricants I. G. Farbenindustrie A.-G. Brit. 435,548, Sept. 17, 1935. Lubricants are obtained by mixing with lubricating oils or greases a lubricant prep. synthetically in presence of S or of such org. S compds. as are antioxidants, e. g., thioacetdehyde, thiazole, thiocresol, thionaphthol, thiobenzamide, dimethylthiophene, aminothiophene, Et mustard oil, dihydroxypropyl sulfide, diphenylthiourea. The synthetic may be prep'd. by "volatilization", polymerization or condensation, e. g., of olefins, mineral or tar oils, waxes, fats, high-mol. acids or alcs., in presence of  $AlCl_3$ ,  $ZnCl_2$ ,  $FeCl_3$ , etc.; or hydrogenation of rubber, resins or other polymers of high-mol. wt. In examples, (1) hard paraffin 100, ceresin 20 and S 0.5 lb. are treated for 45 hrs. at 80° and 5-10 mm. pressure with silent elec. discharges of 7000 v. and 1000 periods, then freed from unchanged paraffin and low-boiling constituents by vacuum distn. and 1 lb. of the product is added to 100 lb. of a petroleum machine oil to stabilize said oil and reduce its setting point, and (2) chlorinated paraffin 100 is condensed at 30-40° for 24 hrs. with  $CH_4$  5 and  $Ph_3S$  10 lb. in presence of kerosene,  $AlCl_3$  and  $ZnO$ , the org. materials, diluent and low-boiling products are removed and 3 lb. of the synthetic is added to 100 lb. machine oil. In 435,567, Sept. 17, 1935, divided on 435,548, synthetic processes for the manuf. of lubricants are carried out in the presence of added org. O compds. that are antioxidants and contain a cyclic radical and at least 1 O atom directly connected with

1 2 C atoms, e. g., aromatic ethers, diphenylene oxide, alkyl derivs. thereof, and naphthyl ethyl ether. In 435,598, Sept. 17, 1935, divided on 435,548, the synthetic processes specified above, except "volatilization," are carried out in presence of added nitriles, amides or other org. bases, e. g.,  $PhNH_2$ , naphthylamine, phenyl-naphthylamine, aminodiphenylbenzene, aminoanthraquinone, aminobenzaldehyde, aminobenzophenone, benzylamine, pyridine, quinoline, carbazole, naphthotriole, acetaminole. Cf. C. A. 30, 282<sup>2</sup>.

Hydrocarbon lubricant mixture. Mathias Pier and Friedrich Christmann (to I. G. Farbenind. A.-G.). U. S. 2,028,349, Jan. 21. A lubricant which has a lowered setting point contains paraffin wax together with a small amt. (suitably about 1-5%) of a hydrocarbon product having a mean mol. wt. of at least 1000 and capable of increasing the viscosity and flattening the temp. viscosity curve of hydrocarbon oils, it includes polymerization products of unsatd. hydrocarbons capable of forming polymeric homologous series, their hydrogenation products or isomerization products, hydrogenated rubber, hydrogenated resins sol. in high-boiling oils, hydrogenated balata or gutta-percha, together also with a small amt. of another material for lowering the setting point such as high-mol.-wt. volatilization products of waxy hydrocarbons rich in H, condensation products of paraffine hydrocarbons having a high mol. wt., with or without cyclic hydrocarbons or condensation products of high-mol.-wt. aliphatic O-contg. compds. Cf. C. A. 30, 608<sup>13</sup>.

Aqueous bituminous dispersions. Jacob M. Fain (to Patent and Licensing Corp.). U. S. 2,027,582, Jan. 14. At least about 50% of bitumen is used with a mixt. of sol. soap and a mineral powder colloid such as bentonite as the primary emulsifying agent, and not less than 25% of water as the continuous phase, the soap and mineral powder colloid constituting less than 1% of the dispersion.

Bituminous emulsions suitable for subjection to low temperatures without breaking. James B. Small (to Glidden Co.). U. S. 2,027,494, Jan. 14. Material such as a Mexican petroleum asphalt is emulsified by use of a soap of rosin such as the Na or K soap heat-treated at about 250-315°.

Asphalts. Planktököll chemische Fabrik G. m. b. H. and Johannes B. Carpsow. Fr. 788,105, Oct. 4, 1935. See Brit. 434,925 (C. A. 30, 853<sup>9</sup>).

Thickening wood oil. Badenol G. m. b. H., Ger. 622,390, Nov. 27, 1935 (Cl. 224. 2). Wood oil is treated with  $O_2$  and then heated to about 150°.

## 23—CELLULOSE AND PAPER

CARLETON E. CURRAN

Identification of cellulose derivatives by chemical means. Harold A. Levey. *Chemist Analyst* 25, No. 1, 14-17 (1936).—A general discussion of methods for the identification of cellulose and cellulose products by the use of solvents or by the behavior toward dyestuffs, etc.

W. T. H.

The refractivity of cellulose as a function of the degree of swelling. Madeline Meyer and A. Frey-Wyssling. *Helv. Chim. Acta* 18, 1429-35 (1935).—In detg. no. of cellulos subject to swelling, an immersion method (in which the entire fiber is encased with, but not penetrated by, the immersion liquid) must be used. The imbibition method (i. e., the procedure in which the fiber is soaked for 2 days in mixts. of known no.) was found unsuitable. Abs. immersion without penetration cannot be attained, but it can be approximated by shortening the periods of imbibition as much as possible. As the swelling increases, the  $n_{\parallel}$  (parallel to the fiber axis) and the  $n_{\perp}$  (perpendicular to the fiber axis) of cellulose, decrease steadily, although very slightly. In ramie fiber, starting with 0%  $H_2O$  and ending with 24.4%  $H_2O$ , the  $n_{\parallel}$  changes from 1.5062 to 1.4960, and  $n_{\perp}$  from 1.5316 to 1.5231. These results are discussed in the light of previous data, especially those of Kanamaru (C. A. 29, 2737<sup>1</sup>).

Louis E. Wise

Adsorption of caustic soda by cellulose. S. M. Neale. *J. Phys. Chem.* 39, 1247 (1935); cf. C. A. 29, 3151<sup>1</sup>. Bancroft and Calkin (C. A. 29, 2737<sup>1</sup>) do not emphasize the fact that the concn. of the adsorbed soln. removed falls markedly as centrifuging proceeds. This was shown by N. from values derived from B. and C.'s own data, and would follow if the concn. of free OH ion in the adsorbed soln. is det'd. by the Donnan equation (cf. Neale, C. A. 24, 3110<sup>9</sup>).

Louis E. Wise

Cellulose wool and its utilization. M. Loescher. *Z. ges. Textil-Ind.* 38, 698-9 (1935).—Of the 3 grades of cellulose wool, the acetate cellulose wool is considered better than the Cu or the viscose cellulose wool, although each of these grades has its particular advantages. On account of their differences in properties the 3 grades must not be intermingled in manuf. operations. AcOH dissolves acetate cellulose wool and not the others. Conc'd.  $H_2SO_4$  turns the viscose and Cu cellulose wool brown and yellowish, resp. Various uses and directions are given.

Leopold Scheffan

The influence of moisture content of cotton on acid consumption in pyroxylin manufacture. N. L. Kostetzkii. *Voenaya Khim.* 1935, 11; *Nitrocellulose* 6, 109 (1937); cf. C. A. 29, 675<sup>9</sup>. E. M. Symmes

A method for isolating a cotton fiber-like material from rice straw. Munenari Tanaka. *J. Chem. Soc. Japan* 56, 1525-6 (1935); cf. C. A. 29, 7639<sup>1</sup>.—A method of purification of cellulose by treating the straw in alkali, vegetable glue and resinous substance, etc. K. K.

Lignin in Douglas fir—composition of the middle lamella. A. J. Bailey. *Ind. Eng. Chem., Anal. Ed.* 8, 52-6 (1936); cf. C. A. 29, 337<sup>1</sup>.—A sample of middle lamella (wt 0.521 mg) removed by using a chisel-pointed sewing needle contained 71.4% lignin (The micro-method for lignin is given in *Mikrochemie*, in press) Samples of wood rays, springwood and summerwood, weighing approx 3 mg each, yielded, resp. 41.1, 35.2 and 31.6% lignin The mech microprocedures are fully described and errors are discussed 25 photographs and 25 literature references Louis E. Wise

The problem of motive power in rayon plants. Otto Th Kornting. *Deut. Wollen-Gewerbe* 67, 803-5 (1935).

Power and heat in rayon plants. Otto Th Kornting. *Deut. Wollen-Gewerbe* 67, 923-5, 935-6 (1935).

Drying sulfite pulp, with examples from the Scandinavian pulp industry. F. A. Ernstler. *Zellstoff u. Papier* 15, 394-6 (1935).—The drying of pulp on machines and in drying chambers is discussed S. I. A.

Cooking beech pulp by the sulfite process. Hans G. Klein and Egon Kurzhaas. *Zellstoff u. Papier* 15, 486-7 (1935).—Earlier reports that pulp produced from beechwood by the sulfite process was dark and unsuitable, were in error The beech bark must be completely removed prior to cooking A cooking liquor of as low as 11% CaO content was found satisfactory Beechwood needs more impregnation time than conifers, because of its smaller pore size and hardness This increase in impregnation time is compensated for by a decrease in total cooking time The color, adhesiveness or odor of the black liquor give no sure indication of the progress of cooking A very characteristic odor is produced, however, at the point when the pulp begins to turn dark Titration with I<sub>2</sub> gives no definite indication that the cooking is progressing satisfactorily Examin. of sample blows is the best method of detg. the progress of cooking The hard beech chips in the digester change very rapidly into a soft pulped mass Av. good yields are obtained The cooked knots are easily bleached S. I. A.

Preparation of sulfite pulp with selenium-containing cooking acids. Erik Haglund, C. H. Bäckström, Mladen Karanović, Lars Runquist and Olof Vincent. *Zellstoff u. Papier* 15, 482-7 (1935); 16, 12-15 (1936).—See C. A. 30, 856<sup>1</sup> S. I. Aronovsky

Improved methods for the examination of paper. H. N. Lee. *Tech. Studies Field Fine Arts* 4, 93-100 (1935); cf. *Ibid.* 4, 3-14 (1935).—These methods are adapted to the examn. of papers in books or works of art. A mixt contg. 5 vols. CaCl<sub>2</sub> (satd. soln.), 10 vols. H<sub>2</sub>O and 4 vols. of aq. 2% KI satd. with I gave the following color reactions when applied to dry fibers: red (with cotton, linen, bleached hemp and paper mulberry); orange-pink (with purified, bleached coniferous sulfite pulp); very pale lavender (with bleached coniferous sulfite pulp); pale reddish violet (with purified, bleached hardwood sulfite pulp and with the long-fibered parts of esparto pulp); blue-violet (with bleached coniferous sulfite pulp), grayish blue (with bleached and unbleached soda pulp and with the shorter part of bleached esparto), blue (with bleached straw and bleached bamboo); deep yellow (with groundwood pulp, jute and lignified fibers generally); yellows of various shades (with unbleached sulfite and some other unbleached pulps). Formulas are given for other staining solns. more complex, but which give similar colors. Without resorting to an admixt. of acids, a soln contg. 10 g. KI and 10 g. I in 5 cc. H<sub>2</sub>O serves to differentiate between various fibers. All turn brown at first, but after washing, rag turns red, soda pulp blue, sulfite pulp violet, groundwood yellow, while unbleached pulps remain brown These colors are ephemeral Simple tests are also given for resin, starch, loading materials, glue, casein and formogelatin. By the

use of a hitherto unpublished technic in sectioning, applied by F. J. Hloxie to cotton yarns, the study of paper structure is facilitated The dry material is soaked in 2 changes of Am acetate, then in 3-4 progressively stronger solns of cellulose nitrate in Am acetate, the final one being highly viscous After removal, the fibers are hardened in CHCl<sub>3</sub>, stored and cut in Phil, and then mounted in castor oil Sections may then be cut by microtome or common hand method Permanent papers should have a pH of not less than 5 Louis E. Wise

Determination of the acidity and alkalinity of paper. Kurt Berndt. *Zellstoff u. Papier* 15, 487-91 (1935); 16, 15 18 (1936).—B discusses methods of measuring the acidity or alkali of paper, the uses to which papers of varying degrees of acidity are put, and the effects of acidity and alkali on the permanence of the papers The acidity of paper is detd by the use of indicators on aq. exts of the paper, with or without a colorimeter, the use of indicators on the surface of the paper, and by titrating the aq. exts., hot or cold, with a standard alkali The rating, pH according to surface measurements, and types of these papers, resp., are given as follows: (1) strongly acid, 3.2-3.6, hard sized and parchmentized papers, (2) normal acid, 3.6-4.5, writing, printing, and carton papers, (3) weakly acid and practically neutral, 4.5-7.0, metal wrapping papers, and (4) alk and strongly alk, 7.0+, unsized and coated papers The effects of pH on the strength and permanence of paper are briefly reviewed S. I. Aronovsky

The mechanical extension of paper in the printing press. Julius Beck. *Zellstoff u. Papier* 15, 493-4 (1935).—A brief discussion of the mech. extension brought about by the pulling and stamping requirements of the printing rolls and the compression strain in multicolor printing S. I. Aronovsky

The effect of loading materials upon the light fastness of dyed papers. S. Pestalozzi. *Zellstoff u. Papier* 15, 496-8 (1935).—Loading materials affect the light fastness of dyes in paper to a considerable extent. Talk, kaolin, blanc fixe, satin white, zinc white, chalk and TiO<sub>2</sub> were used by P. on a series of yellow dyes, i. e., brilliant yellow, sulfate yellow extra, paper yellow G C concentrate, and solarflavin R (Sandoz). Papers loaded with these fillers and tinted with the various dyes were subjected to direct sunlight The different fillers caused varying degrees of fading, dependent to some extent on the alkali of the filler The pH of filled or unfilled papers is generally reduced by exposure to sunlight. Papers of lower pH are more easily faded. Dyes which are fast to light are generally acid-resistant. S. I. Aronovsky

Determination of viscosity of small samples of oil from oil-impregnated paper. Method and apparatus. H. F. Schneider, Jr., and T. A. McConnell. *Ind. Eng. Chem., Anal. Ed.* 8, 28-9 (1936).—An inclined tube viscometer is described applicable to small samples The range of viscosities covered is about 0.70 to 25 poise. Calibration consisted of comparison with known oils. A. S. S.

Monel metal in the rayon industry of Japan (Arita) 9. Lignin derivs. (Can. pat. 353,896) 13. Proofing agents (for paper) (Ir. pat. 788,057) 13. Treating paper, etc. (Ir. pat. 787,950) 25. Evapg. black liquor from cellulose manuf. (U. S. pat. 2,027,115) 1

Bleaching cellulosic material. Maurice C. Taylor and James F. White (to Mathieson Alkali Works). Can. 353,906, Nov. 5, 1935 The following is one of several examples given: Heat 2500 parts of kraft pulp to 80°, add 152 parts of 50% NaOH, 570 parts of glacial AcOH and 2000 parts H<sub>2</sub>O, again heat to 80°, add 231 parts of Ca(ClO<sub>2</sub>)<sub>2</sub> and 4500 parts of H<sub>2</sub>O, after 3 hrs. sep. the bleach liquor, wash the pulp, add water to a d. of 4% and 50 parts of 50% NaOH, agitate at 17-20° for 10 min., and wash the pulp

Cellulose derivatives Robert Haller and Alphonse Heckendorn (to Soc. pour l'ind. chim. à Bâle). U. S. 2,025,660, Dec. 24. New cellulose derivs. of the probable

general formula  $C_{2H}O_2(ON)_x(OY)_z$ , where X stands for an alkyl or an acyl group, Y for the grouping  $=C=N-$ , which grouping, on the one part, is united by the ether linkage  $-O-$  to the  $C_{2H}O_2$  radical and, on the other part, belongs to the cyanuric chloride nucleus, and where Z stands for H, and p, q, and r represent numbers whose sum is equal to 3, p and q being greater than 0 and r being 0 or greater, which products are sol in the solvents for alkylated or acylated cellulose and differ from the parent materials by an enhanced stability toward alkali are produced by treating cellulose derivs. such as acetylcellulose or "commercial" ethylcellulose with heterocyclic compds. which contain at least one  $-N=C$  (halogen)—group such as cyanuric chloride or tetrachloropyrimidine. Various other examples and details of procedure are given.

**Cellulose derivatives** Camille Dreyfus. Fr 788,614, Oct 14, 1935. A soln. of a cellulose deriv., e.g., the soln. obtained by acetylating cellulose with  $Ac_2O$  in  $AcOH$ , is dispersed by passing a gas or vapor, e.g., dry steam, therethrough, and is then subjected to the action of a pptg. agent such as water.

**Cellulose esters** Soc. pour l'ind. chim. à Bâle. Ger. 560,036, Dec. 12, 1935 (Cl. 120 6). Cellulose esters not contg. S are obtained by treating cellulose xanthates or cellulose xanthic acids with org. acid anhydrides. The reaction may be effected at a temp. up to  $140^\circ$  in the presence or absence of a solvent and a catalyst. The process may be applied to filaments, films, etc., of cellulose xanthates or cellulose xanthic acids under such conditions that the materials retain their shape. Thus, it may be applied to filaments, etc., prepd. by coagulating a viscose soln. without regenerating cellulose therefrom, or to cotton which has been treated with alkali and  $CS_2$  under such conditions that its fibrous structure is retained. Sp. processes are described.

**High viscosity mixed esters of cellulose** Carl J. Malm (to Eastman Kodak Co.). U. S. 2,026,986, Jan. 7. A high viscosity cellulose acetate propionate or acetate butyrate contg. at least 40% of the total acyl as propionyl or butyryl is prepd. by pretreating cellulose such as cotton linters with a mixt. of  $HOAc$  and propionic or butyric acid, contg. at least 20%  $HOAc$ , and then esterifying in a bath contg. a catalyst such as  $H_2SO_4$  and a mixt. of propionic or butyric anhydride with at least 20% of  $Ac_2O$ . Cf. C. A. 30, 1230<sup>4</sup>.

**Cellulose ester solutions** I. G. Farbenind. A.-G. (Rudolf Hofmann and Ludwig Lock, inventor). Ger. 619,931, Oct. 9, 1935 (Cl. 295 3 03). See Fr. 773,380 (C. A. 29, 1635<sup>5</sup>).

**Apparatus for slitting lengths of foil such as that of cellulose acetate** Charles E. Stafford (to Celanese Corp. of America). U. S. 2,026,754, Jan. 7. Structural, mech. and operative details.

**Cellulose triacetate solutions** I. G. Farbenind. A.-G. Fr. 785,036, July 31, 1935. Cellulose triacetate is dissolved in acetone or a mixt. of acetone and dioxane at below  $0^\circ$  and afterward heated to above  $0^\circ$ . The solns. obtained are used for making rayon, films, varnishes, etc.

**Cellulose "bulking agent" suitable for use in foods** John Campbell and Robert G. Quinn (to International Paper Co.). U. S. 2,026,865, Jan. 7. Regenerated cellulose scrap is comminuted, treated to remove impurities (suitably by use of acids and alkalis or water and alc. or ether) and then dried at a temp. below that at which cellulose decomps. products are formed.

**Nitrocellulose compositions suitable for films, etc.** Lucas P. Kyrides (to Monsanto Chemical Co.). U. S. 2,028,399, Jan. 21. A neutral ester of a dicarboxylic acid contg. a cyclohexylcyclohexanol residue in at least one ester group, such as a phthalate ester of hydrogenated phenyl phenol is used as a plasticizer in nitrocellulose compds.

**Removing water of gelation from cellulose films** Milton J. Shoemaker (to Carbide and Carbon Chemicals Corp.). U. S. 2,028,296, Jan. 21. A film such as one of regenerated cellulose which has been coagulated from an aq. soln. and which contains nonreentrant water of gla-

tion is immersed in an aq. refrigerating soln. of a salt such as  $NaCl$  or  $CaCl_2$  which is at a temp. below the freezing point of water and is kept in the liquid until the nonreentrant water of gelation freezes and diffuses into the refrigerating soln.

**Transparent films** Friedrich Eichmann and Herbert Nerad. U. S. 2,027,682, Jan. 14. See Fr. 760,626 (C. A. 28, 4191<sup>1</sup>).

**Pxyrolyn films, etc.** Merlino M. Brubaker (to E. I. du Pont de Nemours & Co.). U. S. 2,027,466, Jan. 14. Flexible and nonbrittle films and coatings are formed of a cellulose deriv. such as nitrocellulose and an oily reaction product such as a phthalate of hydrogenated castor oil or the reaction product of hydrogenated castor oil with trimelic, chlorophthalic, trimellitic, naphthalic or diphenic acids, etc. U. S. 2,027,467 relates to the prepn. of such oily reaction products of polycarboxylic acids and hydrogenated castor oil.

**Apparatus for conveying long webs or sheets of material such as films of foil undergoing processing** James H. Rooney and Robert J. Ward (to Celanese Corp. of America). U. S. 2,027,451, Jan. 14. Mech. and operative details.

**Films, threads and similar shaped articles** I. G. Farbenindustrie A.-G. Brit. 435,864, Sept. 26, 1935. In partic. shaped articles from solns. of plastic masses, more particularly cellulose derivs., in org. solvents, a haloalcohol of a polyhydric alc. in which all the free OH groups are esterified with an aliphatic carboxylic acid contg. at least 8 C atoms in an unbranched chain or with a naphthene acid is employed as a softener for the plastic mass. Among examples, polyvinyl chloride contg. 63-65% Cl 300 and ethylenechlorohydrin naphthenate 60 are dissolved in a mixt. of  $C_2H_5$  350 and  $Me_2CO$  350 g. Cf. C. A. 30, 1229<sup>5</sup>.

**Artificial filaments, films, etc.** Henry Dreyfus. U. S. 2,027,419, Jan. 14. In forming products such as those of cellulose acetate, the material after extrusion is stretched while in plastic condition by the action of a moving body of liquid contg. a solvent or swelling agent and which moves in the same direction as the material. App. is described. Cf. C. A. 29, 4581<sup>1</sup>.

**Artificial filaments or threads** Henry Dreyfus. U. S. 2,026,730, Jan. 7. For the production of voluminous filaments of a cellular or tubular character, an org. cellulose deriv. such as cellulose acetate is dissolved in a solvent contg. at least 2 volatile constituents such as alc. and methyl formate the b. ps. of which differ by at least  $40^\circ$  and one at least of which is itself a solvent of the cellulose deriv. (the lower-boiling constituent being 50-75% of the total solvent mixt.), and dry spinning is effected at a temp. near to but not substantially above the b. ps. of the higher-boiling constituents.

**Apparatus for extrusion of sheets, films, etc., such as those formed from viscose or cellulose acetate** Edward Kinsella and Robert J. Ward (to Celanese Corp. of America). U. S. 2,026,740, Jan. 7. Various structural and operative details.

**Apparatus for stapling a band of continuous artificial filaments** I. G. Farbenindustrie A.-G. Brit. 434,250, Aug. 28, 1935.

**Artificial staple fibers and yarns spun therefrom** British Celanese Ltd. Brit. 434,858, Sept. 10, 1935. Continuous artificial filaments, threads, yarns, etc., are formed from spinning solns. having a basis of org. derivs. of cellulose and contg. wax-like compds. that are formed by reaction between higher fatty acids, e.g., stearic, palmitic, oleic, and di- or poly-hydric alcs., alkylolamines or  $NH_3$  and which are readily dispersible in aq. media. The filaments, etc., are converted into staple fibers and crinkled and delustered by treatment with hot aq. media. The last-mentioned treatment must be more vigorous, e.g., at a higher temp., than for mere delustering. In an example, diethylene glycol stearate is dissolved in heated  $Me_2CO$  and added to an  $Me_2CO$  soln. of cellulose acetate (I) to form a soln. contg. 5% of the stearate based on the wt. of the I. The soln. is dry spun to form filaments which are crinkled by treatment in 0.2% aq. soap soln. at  $95^\circ$  for 30 min. and then stapled.

Apparatus for production of rayon by the dry-spinning process. Robert P. Roberts and Edgar B. Johnson (to Celanese Corp. of America). U. S. 2,027,450, Jan. 14. Structural and operative details.

Guide funnels for centrifugal machines for spinning rayon. Kirkless Ltd and Arthur Bennett. Brit. 434,954, Sept. 12, 1935.

Rayon. Friedrich Lappe (to North American Rayon Corp.). U. S. 2,027,047, Jan. 7. Extruded filaments are passed to and over a take-up device (of a described app.) thence to a guiding device and back to the same take-up device, such as a spool for collection.

Rayon, etc. Glanzstoff-Courtaulds G m b H Brit. 435,948, Oct. 2, 1935. Rayon, films, etc., of modified luster are produced by adding styrene to the soln. to be spun or otherwise formed. The styrene may be added as such or as a di- or poly-styrene, and is preferably first dissolved in  $\text{CCl}_4$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ , xylene, dipentene, pineoil, etc.

Rayon production by the stretch-spinning process. Gotthard Rummel and Ewald Knehe (to American Bemberg Corp.). U. S. 2,027,895, Jan. 14. After forming a thread preliminarily by the cuprammonium stretch-spinning process in a stream of  $\text{ppg}$  liquid, the thread is subjected to considerable stretching and is wound upon a rigid winding device under reduced tension. App. is described. (This pat. was granted upon an application filed Nov. 12, 1928, based upon an application filed in Germany Nov. 21, 1927.) Cf. C. A. 29, 4940.

Desulfurizing viscose rayon. Alfred Stein Ger. 622,403, Nov. 27, 1935 (Cl. 295 3 (2)). See Fr. 766,577 (C. A. 29, 3599).

Creped rayon. Zellstofffabrik Waldhof and Hans Rade- stock Fr. 788,291, Oct. 7, 1935. Creping is obtained by subjecting the thread, during winding on the bobbin in the course of the spinning operation, by stretching and contracting the free length of thread in rapid successive movements.

Wood pulp suitable for esterification. Raphael L. Stern (to Hercules Powder Co.). U. S. 2,028,083, Jan. 14. A sheet of felted wood pulp is sheared in one direction and the material is split in a direction transverse to the direction of shearing (by a described app.).

Screen for wood pulp. Joseph J. Moravec. Can. 334,031, Nov. 12, 1935. Structural details.

Pulp shredder. John J. Warren. Can. 333,905, Nov. 5, 1935. Structural details.

Refined pulp from straw of graminaceous plants. Kenta Kodama. U. S. 2,028,000, Jan. 7. Straw such as that of rice, wheat, etc., is digested in an acidic soln. contg.  $\text{NaHSO}_4$  and  $\text{HCl}$  obtained by adding  $\text{H}_2\text{SO}_4$  to sea water or brine so that decomposition may take place in part of pectin substance and other impurities, and the material is then digested with  $\text{NaOH}$  soln. to remove remaining impurities, and the cellulose is sep'd. and collected.

Apparatus for the continuous treatment of fibrous materials as in cooking cotton linters with soda solution. Luke H. Sperry (to Hercules Powder Co.). U. S. 2,028,419, Jan. 21. Various structural and operative details.

Treatment of d-ester liquor. Albert D. Merrill (to Chemupul Process Inc.). Can. 333,568, Oct. 15. Structural and operative details.

Recovery of ammonia from pulp liquor. Roger W. Richardson (to Canadian Industries Ltd.). Can. 333,924, Nov. 5, 1935. Hot cooking liquor of the  $\text{NH}_4\text{HSO}_4$  paper-pulp process is alkaliized and passed countercurrent

to an inert gas, such as air. The resulting gas is cooled to about  $25^\circ$ . Part of the condensable  $\text{NH}_3$  is absorbed in the condensed liquid. The uncondensable  $\text{NH}_3$  is recovered by scrubbing the uncondensed gas free of  $\text{NH}_3$ .

"Freeness detector" for testing paper stock or pulp. Harold D. Wells. U. S. 2,027,060, Jan. 14. Various structural and operative details.

Multi-knife machine (Jordan engine) for refining paper stock and pulp. Walter H. Hermann and Ludwig H. Wesel (to Hermann Mfg. Co.). U. S. 2,027,041, Jan. 7. Various structural and mech. details.

Paper. Nikolaus J. Nils. U. S. 2,027,611, Jan. 14. Various details of app. and operation.

Paper. Ernest Poirier. Can. 334,033, Nov. 12, 1935. The cross-sectional area of a stream of stock to the machine is varied so as to maintain an approx. const. rate of delivery of solid constituents notwithstanding variations in the consistency of the stock.

Mousteining device for paper. Jagenberg-Werke A.-G. Ger. 619,836, Oct. 4, 1935 (Cl. 55e 3). Addn. to Ger. 616,773 (C. A. 30, 6169).

Suction box for paper-making machine. Rohrbacher Lederfabrik Jos. Poeschl's Söhne A.-G. Ger. 622,283, Nov. 28, 1935 (Cl. 55d 16 01). This corresponds to Austrian 133,780 (C. A. 27, 4923) and 135,816 (C. A. 28, 2349).

Drying cylinders for paper-making machines. Firma J. M. Vouth. Ger. 619,482, Oct. 4, 1935 (Cl. 55d 28 30). Addn. to 608,512 (C. A. 29, 5272).

Steam-heated drier roll for use in paper-making. Adam E. Bridge (to The Black-Clawson Co.). Brit. 433,689, Sept. 25, 1935.

Dispersing materials in special papers or the like. Win. W. Carter (to Braxton Morton, as trustee). U. S. 2,027,000, Jan. 7. Sep'd. particles of added material such as a gel contg. abrasives, etc., are first attached to fibers so that the ends of fibers project from the particles, and the material thus prep'd. is then mixed with a liquid suspension of fibers such as a paper pulp and the mixt. is formed into paper or the like.

Paper suitable for labels, seals, etc. Gov'n D. Mackrell. U. S. 2,027,741, Jan. 14. Portions of paper contain fibers crushed more than in other portions and the crushed fiber portions are treated with glycerol so that indicia such as identifying designs are visible when the paper is moistened and invisible when the paper is dry.

Toweling paper. Milton O. Schur (to Brown Co.). Can. 333,339, Oct. 15. The paper contains about 3% of tanned glue solids, and has a preservative against the putrefaction of the glue soln. Cf. C. A. 29, 5274.

Waterproof and greaseproof paper. Clifford S. Johnson (to International Latex Processes, Ltd.). Can. 334,114, Nov. 12, 1935. Paper is treated with an aq. dispersion of rubber contg. a minor proportion of protein material such as casein. A protective material, such as cellulose acetate lacquer is applied, and the solvent evap'd. The rubber deposit is then vulcanized. Cf. C. A. 29, 4942.

Apparatus for waxing paper so as to leave wax-free stripes. Rosenstiel & Cie. Ger. 619,900, Oct. 10, 1935 (Cl. 55f, 11 01).

Fibrous product suitable for fiber-board manufacture. Geo. H. Ellis (to Insulate Co.). U. S. 2,027,581, Jan. 14. A mass of vegetable fibrous material has distributed through it an insecticide and fungicide comprising a toxic agent such as creosote which has been mixed with melted resin with subsequent fine division of the resulting mixt.

## 24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE AND C. G. STORM

Advances in the explosives industry in 1934. Konrad Nitrocellulose 6, 123-4, 143-5 (1935).—A review.

E. M. SIMMONS

New nitrocellulose propellant powders and explosives. Oscar Kausch. Z. ges. Schuss-Sprengw. 30, 361-4 (1933).—A review of patent literature. C. G. Storm

The stability of certain explosives toward heat. Mario Tognetti. *Chimica industriale* (Italy) 17, 517-21 (1935).—By the Talami method (C. A. 29, 6661) the stabilities of a no. of explosives, such as gun cotton, picric acid, T. tetra and TNT, were det'd. by measuring the pressure developed on heating to  $120^\circ$ ,  $125^\circ$  and  $130^\circ$ . T. and

mixts of T, with TNT are more stable than pentrite or its mixts A. W. Conti

The explosive effects of hexogen and pentrite Ph Naoum *Nitrocellulose* 6, 8-8(1935) — Polemical with Stettbacher (C. A. 29, 3841)

The mechanism of explosive reactions K. K. Andreev *Bull. soc. chim.* 15, 2, 2128-34(1935) — An attempt is made to apply the theory of chain reactions in explaining the behavior of explosives The ability of detonating explosives to detonate by simple ignition and their marked sensitiveness to mech. influence such as shock are considered as consequences of the high value of the relation of the energy of the reaction to the energy of activation, and as favoring the development of chains The phenomena of the "dead pressing" of detonating explosives and reduction in sensitiveness to detonation caused by increase in cubic d., common to all explosives, are explained as the result of the existence of intermediate reactions and "wall effect" C. G. Storm

The effect of temperature on the inflammability limits of mixtures of air with combustible vapors Marius Briand *Ann. combustibles liquides* 10, 112-85(1935) — The lower (lean mixts) inflammability limit is detd. between 100° and 300° for mixts of air with 100-C<sub>4</sub>H<sub>10</sub>, C<sub>4</sub>H<sub>8</sub>, C<sub>3</sub>H<sub>8</sub>, C<sub>2</sub>H<sub>6</sub>, PH<sub>3</sub>, PhMe, cyclohexane, cyclohexene, MeOH, i-PrOH, PrOH, Me<sub>2</sub>CO and some of their binary and ternary mixts The variation with temp. is linear, and the mixts. follow the laws of Le Chatelier

Chemical and mechanical incendiary devices Alfred Stettbacher *Nitrocellulose* 6, 202-4, 220-5(1935) — A review I. M. Symmes

Inflammation of firedamp by incandescent lamp filaments Eugene Cotté *Compt. rend.* 201, 1454-5 (1935) — An incandescent lamp (4 v) was placed in an explosive mixt. of coal gas and air When a small hole, not more than 2 mm. diam., was made therein no explosion occurred, but with a larger one it did so, more and more regularly and invariably with a hole 12 mm. diam. With a still larger hole explosion occurred less certainly, and not at all if the lamp were completely smashed, the cooling effect of the air on the filament preventing it The spark on breaking the filament of the 4-v. lamp does not cause explosion C. A. Silberrad

Rocket propulsion Willy Ley. *Aircraft Eng.* 7, 227-31(1935) — Review of the theory of rocket propulsion shows the use of high exhaust velocities of the gases generated is more important for high speed than ejection of large quantities of gases The fuels required for ascension against gravitational force are reviewed, modern fuels have adequate power O<sub>2</sub> carriers such as KClO<sub>4</sub>, KNO<sub>3</sub>, HClO<sub>4</sub>, etc., are ruled out because of poisonous, corrosive or explosive conditions assoc. with their use, and the only possible oxidants considered are O<sub>2</sub> or O<sub>3</sub> A list of the possible fuels is given, and the greatest power is seen to be obtainable with a 1.8 mixt. of H<sub>2</sub> and O<sub>2</sub> Expts. are reviewed in the matter of devices to cool the rotor and to inject the fuel Thermal efficiency of powder rockets is about 57%, and when power is supplied by burning of H<sub>2</sub>, efficiency rises to 80% L. believes that this latter efficiency will be increased to a greater efficiency than that of internal combustion engines, however, about 50% of the thermal energy of a powder rocket is converted into kinetic energy Diagrams and photographs are given for the various types of rockets Materials of construction are reviewed The most difficult problem is the combination of strength at high temp. and oxidation resistance for the metal which forms the exhaust nozzle W. H. Bruckner

The critical explosion limits in the oxidation of carbon oxysulfide and silicon hydride Henz Gutschmidt and Klaus Clusius *Z. physik. Chem.* B30, 265-79(1935) — The crit. explosion limits were studied for mixts of COS

and O between 190° and 490° in quartz vessels and in glass vessels of various diams. For the lower limit, the results agree with the theories of Hawn and of Thompson, Howd and Cairns (C. A. 27, 2302) The upper limit is somewhat indefinite and does not agree with the theory of Semenov (*Chem. Kinetics and Chain Reactions*, Oxford, 1935, cf. C. A. 29, 1706) which is not applicable over a wide temp. range The exptl. results obey the relation  $(p_1 + p_2)d^m = C$ , where  $p_1$  and  $p_2$  are pressures of COS and O,  $d$  is the diam. of the vessel,  $m = 0.7$  and  $C$  increases from 75 at 200° to 251 at 190° For SiH<sub>4</sub>, the expts. were concerned only with the lower explosion limit and the Semenov expression,  $p_1 p_2 d^m = \text{const.}$ , was not obeyed The results indicate this to be due to 2 different end products obtained in the explosion Deviations from the ideal case of Semenov are apparently found in a dependence of the reaction path on the concn. The single general method so far discovered for identifying nonstationary chain reactions is the fact that such explosions occur without change in the no. of moles G. M. Murphy

Content of free O in a fuel gas and its explosiveness (Kuznetsov) 21.

Explosives Harcourt T. Simpson *Brit.* 435,588, Sept. 24, 1935 An explosive specially suitable for mining and quarrying comprises a mixt. of Al or Al alloy, which may be coated with a waterproofing material, e. g., paraffin, a metallic oxide and NH<sub>3</sub>, K or Na nitrate One example contains NH<sub>4</sub>NO<sub>3</sub> 90, Al to 5 and MnO<sub>2</sub> 3.5%

Explosives Imperial Chemical Industries Ltd. *Brit.* 436,002, Oct. 9, 1935 NH<sub>4</sub>NO<sub>3</sub> explosives are made from NH<sub>4</sub>NO<sub>3</sub> of low bulk d. not exceeding 0.75, which may be prep'd. in various ways Thus, an NH<sub>4</sub>NO<sub>3</sub> soln. may be atomized and caused to crystallize quickly in a current of warm air, or a concd. NH<sub>4</sub>NO<sub>3</sub> soln. may be introduced quickly with stirring into alc., or NH<sub>4</sub>NO<sub>3</sub> may be treated in a colloid ml.

Smokeless powder. Fredrick Olsen, Gordon C. Tibbitts and Edward B. W. Kerone (to Western Cartridge Co.). U. S. 2,027,114, Jan. 7. A smokeless powder base such as nitrocellulose is agitated in a nonsolvent vehicle such as water with a solvent such as EtOAc which is substantially immiscible with the vehicle, to form globules of dissolved base, and the solvent is removed from these globules to form solidified droplets of gelatinized smokeless powder base. An arrangement of app. is described Various details of compns. and procedure are described

Priming mixtures for ammunition Geo. H. Jacobs (to Peters Cartridge Co.). U. S. 2,027,825, Jan. 14 Zr 2-30% is used in admixt. with nitrates and chromates such as Ba(NO<sub>3</sub>)<sub>2</sub>, Pb chromate, Pb (CNS)<sub>2</sub>, ground glass and Hg fulminate.

Blasting-charges James Taylor and Imperial Chemical Industries Ltd. *Brit.* 436,144, Oct. 7, 1935. In cartridges comprising a casing contg. a mixt. of an alkali metal nitrite with 1 or more NH<sub>4</sub> salts, with or without other ingredients, said mixt. being adapted to produce gas which is released when it has acquired sufficient pressure, as described in *Brit.* 431,935 (C. A. 30, 617), granules of such a compn. are mixed with an incombustible compn. of the kind described in *Brit.* 430,258 (C. A. 29, 833) in powd. or granular form, the proportions being such that the latter compn. is substantially decompd. when the charge is ignited in a confined space

Ignition composition suitable for use in delay detonators Charles F. Sosson (to Imperial Chemical Industries Ltd.). U. S. 2,027,208, Jan. 7. Zr 50-95 is used with a Pb salt of a nitrophenol such as basic Pb 2-nitroresorcinate 50-55%

Miners' and other safety lamps Ernest A. Hallwood *Brit.* 434,164, Aug. 27, 1935, divided on 419,594 (C. A. 29, 3164), 434,812, Sept. 6, 1935

## 25—DYES AND TEXTILE CHEMISTRY

L. A. OLNEY

Some recent developments in the application of sulfur dyestuffs John L. Crist. *Am. Dyestuff Repr* 25, P 11-13(1936).—The use of  $\text{Na}_2\text{S}_2\text{O}_4$  in connection with  $\text{Na}_2\text{S}$  and  $\text{NaOH}$  gives beneficial results with certain leuco types of S blues.  $\text{NaHSO}_3$  may be used in place of  $\text{Na}_2\text{S}_2\text{O}_4$ , it acts as a reducing agent and lowers  $pH$ . Recent org. derives from sugars and sugar alcs are employed in the dyeing of cotton.  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{NaHSO}_3$  have given satisfaction as exhausting agents.  $\text{NaBO}_3$  has been used for finishing off S color dyeings but their wash fastness is usually impaired by this usage. W. H. Boynton

A new group of dyes [The immediate leuco dyes] George Rudolph. *Z. ges. Textil-Ind* 39, 24 5(1936). The immediate (I) leuco (L) dyes can be used for all plant fibers including rayon and are particularly useful in those cases which should correspond in fastness to those produced with I or sulfur dyes. The new dyes are water-sol., faintly alk. and can be absorbed in a nearly cold bath. Dyeing directions are given. All IL dyes may be mixed with each other. The fastness of some of the dyes to light may be improved by after-treatment with  $\text{CuSO}_4$  or  $\text{K}_2\text{Cr}_2\text{O}_7$ . This after-treatment has an effect upon the shade. The following dyes are available in this group: IL yellow 3GT, IL orange RT, IL brown 5G, IL brown BR, IL brown T, IL bordo R, IL violet Beta, IL blue B, IL blue FGL, IL blue CL and IL green FFG. This new group of dyes can be employed for the dyeing of mixed fabrics from cotton and viscose rayon. Leopold Scheflan

Indigoid dyes Sisir Kumar Guha. *J. Indian Chem. Soc.* 12, 659-64(1935), cf. C. A. 28, 6315. The condensation of 5-methyl-3-hydroxythiophenone (I) with aldehydes readily takes place with the production of crystalline solids which are easily sol. in  $\text{H}_2\text{SO}_4$  with characteristic coloration. On addn. of  $\text{H}_2\text{O}$  the substances are reprecipitated in a finely divided condition suitable for dyeing wool from an acid bath. They are not suitable for the dyeing of cotton from an alk. hypochlorite vat. The addn. of 0.244 g. of glyoxal- $\text{NaHSO}_3$  in 5 cc.  $\text{H}_2\text{O}$  to a soln. of 0.324 g. of I in 25 cc. of hot alc. and treatment of the mixt. by shaking with 5 cc. of concd.  $\text{HCl}$  gave deep red fine silky needles of bis(5-methyl-3-thiophenyl)ethyleneindigo,  $\text{C}_{12}\text{H}_{10}\text{S}_2\text{O}_2$ , m. above  $300^\circ$ . Condensation of I with  $p$ -Me- $\text{NC}_6\text{H}_4\text{CHO}$  similarly yielded shining red prisms of 2-(4'-dimethylaminobenzylidene)-5-methyl-3-thiophenone,  $\text{C}_{12}\text{H}_{12}\text{NOS}$ , m.  $198-9^\circ$ . A similar condensation with  $m$ - $\text{H}_2\text{NC}_6\text{H}_4\text{CHO}$  gave brownish yellow needles of 2-(3'-aminobenzylidene)-5-methyl-3-thiophenone,  $\text{C}_{12}\text{H}_{11}\text{NOS}$ , m.  $157^\circ$ . Condensation of I with  $p$ - $\text{O}_2\text{N}$ ,  $m$ - $\text{O}_2\text{N}$ ,  $p$ -Cl,  $p$ -MeO,  $p$ -HO,  $m$ -HO,  $p$ -Me and  $m$ -MeO- $p$ -HO derivs. of  $\text{BzH}$ , piperonal, 3,4-(HO) $\text{C}_6\text{H}_3\text{CHO}$  and PhCH $_2$ CHO gave the corresponding dyes, m.  $204^\circ$ ,  $233^\circ$ ,  $178^\circ$ ,  $157^\circ$ ,  $232^\circ$ ,  $200^\circ$ ,  $159^\circ$ ,  $194^\circ$ ,  $248^\circ$ ,  $231^\circ$  and  $184^\circ$ , resp. Their colors with concd.  $\text{H}_2\text{SO}_4$ , shade given on wool from acid bath, shade on cotton and solubilities are tabulated. They are all sol. in  $\text{O}_2\text{NPh}$ , PhNH $_2$ , pyridine, AcOH and AcOH and all, except the HO compds., are insol. in concd. NH $_4\text{OH}$  and caustic alkalis. In boiling glacial AcOH, acanthraquinone (C. A. 27, 1759) and I yielded an indigoid dye, 2-(5-methylthiophenyl)-1'-acanthraquineneindigo,  $\text{C}_{20}\text{H}_{14}\text{S}_2\text{O}_2$ , m. above  $310^\circ$ , whose properties of dyeing wool in orange-brown shades from an acid bath and cotton in light red ochre from a yellow vat have been compared with those of 2-(5-methylthiophenyl)-acanthraquineneindigo. C. R. Addinall

Influence of position isomerism (structural differences) in azo dyes on their fastness to light and washing. Marion F. Griffith. Ohio Agr. Expt. Sta., Bull. No. 548 (53rd Ann. Rept.), 80-1(1935); cf. C. A. 28, 7021. The influence of the position of sulfonic acid groups in the diazotized nucleus of isomeric naphthol-sulfonic acid dyes on the properties of these sulfonic acid dyes was detd. The presence of a sulfonic acid group in the  $\text{C}_6\text{H}_4$  nucleus of phenylazo-1- or -2-naphthol dyes has a decided influence upon the exhaustive properties of the dyes. When the

sulfonic acid group occupies the  $o$ -position the degree of exhaustion is the greatest. Substitutions in the naphthol ring have little effect on the exhaustive properties. The presence of a sulfonic acid group in the  $\text{C}_6\text{H}_4$  nucleus of phenylazo-1- or -2-naphthol dyes increases the fastness of the dyes to washing. The position of the sulfonic acid group has no effect on fastness or washing. Substitutions in the naphthol nucleus do not greatly affect fastness to washing. The combined effect of fading and darkening by exposure to fadometer light is least with the  $o$ -sulfonic acid dyes. The  $m$ -substitution has decidedly the greatest effect on the fastness to light. In general, the dyes having an OH group in the 2 position are superior to those in which the OH group is in the 1 position. The exceptions to this generalization are 1-naphthol-4-sulfonic acid which is comparatively fast to light, and 2-naphthol-3,6-disulfonic acid which is fugitive. The combined effects of light and washing indicate that the  $o$ -sulfonic acid dyes are the least affected.  $p$ -Sulfonic acid dyes are more affected than  $o$ -sulfonic acid dyes but less affected than  $m$ -sulfonic acid dyes. The presence of 2 sulfonic acid groups in the naphthol nucleus does not decrease the fastness of the dyestuff. C. R. Fellers

Color tolerance—its measurement and specification for dyed textiles Robert D. Nutting. *Am. Dyestuff Repr* 25, 1-4, 21-5(1936). The Universal method of color specification was applied to the practical problem of color tolerance in the study of numerous com. matches from woolen and cotton mills. Data show that no dyeings are ever matched "exactly," but it is possible to guarantee a color match "exact" within a specified tolerance limit. The adoption of such a universal method of specifying color would aid materially in creating a more satisfying situation for the dyer and for his customers.

The behavior of artificial textile fabrics with respect to dyes Herbert Brandenburger. *Monatsh. Textil-Ind* 50, Trade Issue III, 90-2(Nov., 1935)—A survey.

Absorption of dyestuffs by cellulose. VI. Effect of modification of the cellulose, and a theory of the electrolyte effect J. Hanson, S. M. Neale and W. A. Stringfellow. *Trans. Faraday Soc.* 31, 1718-30(1935); cf. C. A. 29, 347. Rates of absorption of dyestuff (Sky Blue FF, Color Index No. 618) by cotton cloth, mercerized cotton, hydrocellulose, cotton cloth, after HBrO $_3$  oxidation, Cellophane, viscose, rayon and cupra rayon were studied. Increased rate of motion of the sample relative to the dye bath has a profound effect on the rate of absorption by yarns and fabrics, but little effect in the case of the sheets. Mercerized cotton takes up dye more rapidly than natural cotton in a ratio unaffected by NaCl concn. Oxidation of cellulose reduces dye absorption, an effect more pronounced at low salt concn. The Donnan-type equilibrium is used to explain the action of electrolytes and the effect of oxidation. An equation was developed relating dye absorption and concn. of dye and salt from theoretical considerations, and was found to agree satisfactorily with exptl. results. C. E. P. Jeffreys

Progress in the coloring and finishing of textile materials A. J. Hall. *Chem. Age* 33, 597-8(1935).

E. H. The spray dyeing of silk and rayon fabrics. Remington C. Cayser. *Deuts. Wollen-Gewerbe* 67, 1465(1935). Various precautions are given. The solns. used must be thin fluid. The following substances may be used for the prepn. of spraying liquids in the ratio of 5 to 50 per 1000 parts of water on silk and rayon fabrics: tragacanth, glue, gelatin, carrageen, flaxseed, agar-agar and glycerol. Very few patented preps. are suitable on account of their irritating effect on the dyer. Only high-grade gum tragacanth may be employed. Powdered glue facilitates the prepn. of the soln. and yields better results. Glycerol increases the wt. without any noticeable injurious influence on the luster. Leopold Scheflan



The stripping and dyeing of reclaimed wool H Feld *Monatsh. Textil-Ind* 50, 206-7(1935).—A review.

Leopold Scheflan  
The dyeing of clothing H Feld *Monatsh. Textil-Ind* 50, 222-3(1935).—A discussion dealing with the effect of light on textile fabrics, the choice of suitable dyes, simple chemical methods for detecting the kind of textile fiber at hand and the proper cleaning. Directions are given for removing the color from faded textile fabrics previous to the dyeing.

Leopold Scheflan  
The dyeing of Vistra and Vistra products Georg Rudolph *Monatsh. Textil-Ind* 50, Trade Issue III, 89-90(Nov., 1935).—Directions for dyeing are given.

Leopold Scheflan  
Is it possible to dye Vistra or a mixed yarn from cotton and Vistra fast with Turkey old red? Heinrich Rantisch *Monatsh. Textil-Ind* 50, Trade Issue III, 92(Nov., 1935).—This dyeing operation can be carried out to utilize old red oil, a water-sol product obtained from cactor oil and rank olive oil. Detailed directions are given.

Leopold Scheflan  
Printing cotton—an outline. S L Parker *Am. Dyestuff Repr* 25, 16-10(1935). W H Boynton

The German textile industry in 1935. A retrospect of the past year A Niemeyer *Z. ges. Textil-Ind* 39, 2(1935).

Leopold Scheflan  
The inventor's activity in the textile industry during the last 50 years F C Boettcher *Monatsh. Textil-Ind* 50, 212-14(1935).—A study of the no. of German textile patents shows maximums in 1910 and 1920 and a min. in 1919. The no. of patents has been steadily decreasing since 1920.

Leopold Scheflan  
The origin and meaning of the names of [textile] fabrics A Hamann *Spinner u. Weber* 53, No 13, 3-5, No 14, 4-7(1935).

Leopold Scheflan  
Textile materials which are called for by the present fashion. Otto Pennekamp *Deut. Wollen-Gewebe* 67, 869-2(1935).—An alphabetical survey of modern textile raw materials and yarns with particular emphasis on the German products.

Leopold Scheflan  
From laboratory and practice. [Four cases of chemical examinations of improperly reacting commercial materials.] Kehren *Z. ges. Textil-Ind* 39, 26-8(1935).—The chem. examn. of the following cases is discussed in detail: (1) the occurrence of brittleness in a rubberized cotton fabric during vulcanization, (2) the hardening of a rubberized fabric, (3) the hardening of the paper roller of a calendar and (4) the excessive incrustation of the gravel filter of a lime-soda water-purification installation.

Leopold Scheflan  
From practice and science [in the textile industry]. H Russina *Z. ges. Textil-Ind* 39, 2-4(1935).—Nageli's melle theory is described in its applications to starch, cellulose and various textile fibers. Difference in the quality of German and English wool and the fact that the character of the wool can be varied by raising previous to drying are explained by analogy to the colloid chemistry of gelatin.

Leopold Scheflan  
A review of recent analytical methods with special applications to textile analysis. Irvine W. Grote *Am. Dyestuff Repr* 25, 114-16(1935).—Several semimicro-methods of analysis are outlined. Adsorption indicators, elec. cond. or resistance methods for analytical operation, the refractometer, the glass electrode *pn* outfit and oxidation and reduction indicators and their possible applications in the textile analysis field are discussed.

W H Boynton  
Fiber surface studies. Can even the slightest damages in wool fibers be made visible? H Reumuth and H. Schwerdtner *Z. ges. Textil-Ind* 39, 12-18(1935).—Microscopic methods of examn. of fiber are described and an improved method is developed. Photomicrographs show the results obtained by this method on a variety of wool fibers.

Leopold Scheflan  
Methods and apparatus for the determination of the uniformity of threads and yarns. H. Ottensmeyer *Monatsh. Seide Kunststoffe* 40, 372-8, 411-14(1935).—A review dealing with optical, volumetric, weighing, strength

and elongation methods and the app. employed. Many illustrations are shown.

Leopold Scheflan  
The determination of stiffness in individual yarns Irving J. Sasi *Textile Bull* 49, No 20, 8-9, 28(1935).—A method for measuring stiffness has been developed that consists essentially of detg. the force necessary to bend a piece of yarn, held at one end, through various angles. A new instrument designed for this purpose is used in conjunction with an analytical balance. Some older testing methods and stiffness testers are briefly described and results obtained by this new method are given.

L. S.  
Preservatives for raw materials of the textile industry Justin Hausner. *Spinner u. Weber* 53, No 3, 10-11(1935).—CuSO<sub>4</sub> has in general only very slight preservative power and cannot be employed in liquors which are alk. or which contain soap or glue. ZnCl<sub>2</sub> and MgCl<sub>2</sub>, being strongly acid owing to hydrolysis, will attack cotton, linen and rayon. ZnCl<sub>2</sub> cannot be used in alk. or soap-contg. laths. Formalin cannot be employed in the presence of glues or gelatins. Phenol and salicylic acid are unsuitable for preserving alk. and Fe-contg. materials.

Amurcol is more efficient in neutral or acid baths than in alk. baths, is more effective than salicylic acid and does not harm proteins. Amurcol CB is effective for preserving neutral and acid materials. Tests are described for detg. the amt. of preservative necessary for a particular purpose.

One kg. of potato starch paste will be preserved for the same length of time by 10 g. ZnCl<sub>2</sub> as by 0.6 g. Amurcol. Size from carob-bean flour can be preserved more easily than that from rye flour. A size contg. 0.65 g. Amurcol CB per kg. of carob-bean flour was still satisfactory after 70 days, while a similar mixt. of potato-starch size developed molds after 9 days.

Leopold Scheflan  
Defects and sources of defects [in textiles], where they are and how to recognize and avoid them. Günt. *Deut. Wollen-Gewebe* 67, 893-901(1935).

Leopold Scheflan  
New methods for the treatment of wool, [a discussion of] solvents and nonfatty assistants for the washing operation. E. Meissner. *Deut. Wollen-Gewebe* 67, 143-4(1935).—A survey.

Leopold Scheflan  
The era of artificial wool. Preussische Textilfachschule Forst (Lausitz). *Z. ges. Textil-Ind* 39, 4-5(1935).—A discussion dealing with the utilization of artificial wool in the German textile industry and with chem. and mech. technological exams of a mixed fabric.

L. S.  
Artificial-wool manufacture. A. Kauerz *Deut. Wollen-Gewebe* 67, 751-3(1935).—A review of the chem. and mech. operations.

Leopold Scheflan  
Vistra and Wollstra. Georg Rudolph. *Deut. Wollen-Gewebe* 67, 463-5(1935).—A review, giving directions for washing, bleaching and dyeing. Numerous dyes are listed that can be used on Vistra and Wollstra (a mixt. of Vistra and wool).

Leopold Scheflan  
New finishes for knit goods from rayon Alexander Prior. *Monatsh. Textil-Ind* 50, Trade Issue III, 84-6(Nov., 1935).

Leopold Scheflan  
Stockings from rayon. H. Engel. *Monatsh. Seide Kunststoffe* 40, 420-5(1935).—A review of the manuf., washing, dyeing, finishing and drying.

L. Scheflan  
Partially substantivized acetate rayon and its utilization for the production of various effects in fabrics. Erich Wagner and Paul-August Koch. *Z. ges. Textil-Ind* 39, 18-22(1935).—A cellulose fabric can be dyed in either of 2 ways both of which depend on the fact that partly or completely esterified cellulose is affected differently by dyes than is pure or hydrated cellulose. (1) Cellulose is immunized to the action of direct dyes by slight esterification. When completely or locally immunized cotton fibers are mixed with untreated cotton fibers and the mixt. is treated with substantive cotton dyes the immunized fibers will appear colorless while the others will have been dyed in the normal manner. The dyeing properties of such yarn can be altered in such a way by modification that it can be dyed with acid and basic dyes. (2) Acetate rayon threads are sponified (substantivized) locally but completely and mostly only at the surface up to hydrate cellulose. The chem. fundamentals of the acetate formation of cellulose are reviewed together with the 3 theoretical stages of

acetylation. The phys., optical and dyeing properties of acetate rayons are discussed Leopold Scheffan

The sizing of rayon yarns. R. Hünich *Monatsh. Seide Kunststoffe* 40, 409-500(1935); cf. C. A. 29, 7665f.—Rayon threads are sized with drying oils instead of starch and glue. On application of the oil size in solution in a volatile organic solvent the oil may become coned, on the exterior of the yarn layer after the evaporation as a result of capillary action and diffusion. This irregular distribution can be avoided by applying a solution of the oil and then adding a second liquid which is miscible with the first solvent but is not a solvent for the oil. Particularly good results are obtained with drying oils which in contact with air, O<sub>2</sub>, ozone or other suitable agents at elevated temperatures oxidize and polymerize sufficiently to render them insoluble in the volatile paraffin hydrocarbons. Such products are still very soluble in acetone, benzene, pyridine, dioxane, etc., but precipitate on the addition of paraffin hydrocarbons. Three examples are described in detail Leopold Scheffan

The delustering of rayon. A. Prior. *Monatsh. Seide Kunststoffe* 40, 415-20, 456-8, 460, 462, 500-3(1935) —A detailed survey dealing with the Foulard delustering method, the black-dull finishing operation, historical development of the technique, spinning and piece delustering, luster as a factor in the textile industry and the patent literature Leopold Scheffan

Strength and stretch in cotton yarns. H. Pomfret *Textile Bull.* 49, No. 18, 6, 9(1936) —The effects on the strength and stretch of a yarn of the type and quality of raw cotton used, the fineness of the yarn, and the turns per inch are discussed. The numerical magnitudes of strength and stretch depend in part upon the length of specimen tested, the rate of loading of the specimen, and the moisture contents of the sample. Sizing increases the strength of the yarn and protects the surface against the raising of entangling hairs before the thread is formed into cloth. Sized cotton is more extensible at higher than at lower humidities. Leopold Scheffan

A survey of the finishing operation for textile goods made from cotton. R. Kling. *Spinner u. Weber* 53, No. 9, 26-8, 30-2, 34-6(1935) —The survey covers singeing, desizing with water, enzymes, alkalis or acids and with oxidizing agents, boiling in lye, bleaching, mercerization, starching, fulling, drying and stretching. L. Scheffan

Cotton-yarn testing as an indication of weaving qualities. David Atkinson *Textile Bull.* 49, No. 14, 5-6(1935) —Physical tests are discussed. L. Scheffan

Cotton-cleaning installations. A. Koltermann. *Deut. Wollen-Gewerbe* 67, 1011-12, 1014(1935). L. S.

Striations in the warp of acetate textiles delustered in boiling baths. Emilio Debernede. *Boll. reparto fibre tessili vegetali staz. sper. ind. carta fibre tessili vegetali* 30, 88-104(1935) —Cloth with acetate warps had light and dark warp striations of 1 mm to some cm in width due to fine point cavities in the filaments causing opaqueness. The cavities were produced by boiling delustering soap and phenol baths. The striped effect was due to tension differences and irregular sapon. All acetate threads had deficient elasticity, different with the light and dark threads. Color discharging removed the striations completely from some parts and partially from others; this indicates superficial sapon. R. Sansone

Bleached fabrics that turn yellowish, spotty and streaky, with particular reference to surgical, bale, padding and absorbent cotton. H. Rehmman. *Monatsh. Seide Kunststoffe* 40, 265-6(1935) —Medical cotton will turn yellowish in time if it has been bleached too little or excessively, boiled, rinsed or washed insufficiently, and if it contains residues of fats, Cl, acids, soaps or Ca compounds. The discussion deals with the proper bleaching of medical cotton and the purity of the water employed. Various other factors which cause a spotty and streaky appearance are pointed out. L. S.

Practical tests of the wearing quality of porous mixed fabrics which had been rendered impervious to moisture by impregnation. Fritz Ohl. *Spinner u. Weber* 53, No. 49, 12, 14, 16-17(1935) —The tests were made on mixed fabrics of wool and cellulose (1:1) which had been impregnated with alumina (A), paraffin preps (B), gelatinous

plant extracts (C) and similar preps. (D). By the use of 4 different methods the samples were tested before and after they had been subjected to ordinary wear. Wearing lowers the waterproofness of porous fabrics no matter what impregnation medium had been used. An increased stress, such as exposure to rain or creasing, decreases the waterproofness still further. Washing with soap flakes, benzene, an O-contg., an enzymic and a synthetic washing agent all greatly diminish the waterproofness of the fabrics. The chemical nature of the washing agent is an important factor because of its relation to the chemical properties of the impregnation medium used. Leopold Scheffan

The resistance to tear of cloth for uniforms. Peer. *Spinner u. Weber* 53, No. 6, 8-11(1935). —Calcs are given and various factors are discussed which affect the tear resistance. Leopold Scheffan

The testing of materials in the ribbon-weaving mill. E. Peuster. *Deut. Wollen-Gewerbe* 67, 1055-60(1935)

Discussion of various methods dealing with (1) the moisture contents of the yarn (conditioning), (2) the uniformity, strength, elasticity and torsion of the yarn and (3) the strength and elongation of the fabric in its raw and finished state. Leopold Scheffan

The impregnation technique [for textiles]. Fritz Ohl. *Spinner u. Weber* 53, No. 15, 8-13(1935) —A critical review of com. preps and suggested methods for waterproofing textile fabrics. Leopold Scheffan

The impregnation (of cellulose) with ammoniacal copper oxide. Gustav Durst. *Monatsh. Seide Kunststoffe* 40, 267-9(1935), cf. C. A. 29, 1940f. —Detailed directions are given for prep. ammoniacal Cu oxide on a large scale and for carrying out the impregnations. The value of this impregnation lies in the very shiny surface produced, its stiffness and its powerful resistance to putrefaction and the action of termites, which is due to its high Cu content. Disadvantages are the rapid fading of the color in the sun and the rapid etching of the Cu by weak acids. L. S.

The prevention of the precipitation of lime by means of Calgon. C. Steiner. *Spinner u. Weber* 53, No. 39, 13-14(1935). —Calgon (Na<sub>2</sub>PO<sub>3</sub>) prevents the pptn of insol. soaps by formation of sol. complex Ca and Mg salts. The formation of ppts was avoided with (1) hard water and soda of 6° and 15° hardness by the use of 0.5-1.0 and 1.0-2.5 g./l., (2) 6° and 15° hard water and soap with 1.0-2.5 g./l. (while a clear soln. required 2.5-5.0 g./l.) and (3) 6° and 15° hard water, soap and soda with 1.0-2.5 and 2.5-5.0 g./l. of Calgon. Ca soaps already pptd. could be dissolved by Calgon. Leopold Scheffan

What is meant by calgonizing? Anton Volz. *Z. ges. Textil-Ind.* 38, 563-4(1935). —To calgonize means to treat a textile fabric with Calgon in order to obtain certain improvements of the quality. Calgon increases the absorptive capacity of the fabric and gives it a better feel and color. Calgon is not suitable for general water softening purposes as the well-known softening methods are more economical. It is used to neutralize residual hardness formers in softened waters and to prevent the formation of ppts in various baths at any time and without any app. Leopold Scheffan

Methods for the determination of the degree of mercerization. Kann Schulze. *Monatsh. Seide Kunststoffe* 40, 472-8(1935) —Numerous chem. and phys. methods are briefly described and references are given. Only very few methods of detg. the degree of mercerization are quantitative and no one method can be used alone to det. with certainty either this value or the cause of mercerization defects. Some of the industrially valuable properties of mercerized cellulose fibers, as luster and heightened strength and better feel have very little to do with the processes used for the detn. of the degree of mercerization. Therefore, the correct detn. of this value gives no guarantee of the outcome of industrial mercerization. L. S.

The felting operation. W. Haussner. *Spinner u. Weber* 53, No. 2, 8-10(1935) —A review covering the use of fats, soaps and acids and discussing various causes of defects. Leopold Scheffan

Warp sizing. II. Paul Seydel. *Cotton* 99, 44-6(Dec., 1935); cf. C. A. 30, 619f. —The details of the

process and the dextrins and starches employed are discussed. W. H. Boynton

**Desizing agents and their action.** Fritz Ohl. *Spanner u. Heber* 53, No. 20, 8-10 (1935).—Desizing agents are classified as (1) Oxidizing agents (a) those containing perborate as *Astral powder*, *Alston powder*, *Perborat Cembra Superlat*, *Terhid C*, (b) organic chloride compounds as *Alkalin*, *Amidonit*, (2) diastatic agents (a) malt diastases as *Diastase*, *Diastase*, *Diastase*, *Diastase*, *Gabulu*, *Konings-Diastase*, *Maloforin*, *Maloforin*, (b) pancreatic diastases as *Adresin*, *Delavan*, *Degomma*, *Novoforin*, *Novoforin*, *Novoforin*, (c) bacterial diastases as *Biostase*. The diastatic desizing agents are most commonly used in cotton operations on account of their efficiency. The removal of linseed oil and its preps. from textiles is reviewed as well as the use of various desizing agents which contain soap or other constituents that take the place of soap. The employment of textile acids which avoid the formation of lime soaps is discussed. Various hints are given on the proper desizing procedure. Leopold Scheffan

**Textile oils.** Hans G. Meyer. *Algem. Oel- u. Fett-Ztg* 32, 443-4 (1935).—A review of the type of oils used in the textile industry. M. M. Piskur

**Hot-air drying machines and oxidation machines.** Their importance today for the printer and dyer. Richard Golbs. *Z. ges. Textil-Ind.* 38, 556-7, 558-9 (1935).

**Is soap coming into its own again?** Anton Volz. *Monatsh. Textil-Ind.* 50, Trade Issue III, 93-4 (Nov., 1935).—A literature review shows that the action of soap is important for numerous textile purposes and that soap cannot be entirely displaced by Ca-resistant detergents. This is particularly true for the degumming of natural silk, the dissolving of linseed oil size, the alk. felting of wool and a variety of washing operations in the textile industry. Leopold Scheffan

**The use of higher sulfonated fatty alcohols in the laundry.** A. Bresser. *Z. ges. Textil-Ind.* 38, 505-9 (1935).—The manifold use of sulfonated alcs. are described. The following washing and cleaning agents were found to be very useful to the textile industry: *Sopadon*, *Mersapal*, *B* and *Mersapal* of unknown sulfonate constitution, "CFD 1931" containing a fatty alc. sulfonate, *Perpapon* and *Superbion* TS containing a fatty alc. sulfonate and a solvent and *Purion* containing a fatty alc. sulfonate and a bleaching agent. Leopold Scheffan

**Injuries to textile fabrics due to the application of fireproofing impregnations.** Hans-Joachim Henk. *Monatsh. Textil-Ind.* 50, 223-4 (1935).—Most fireproofing materials cause a damage when the impregnated fabric is exposed to an elevated temp.  $Al_2(SO_4)_3$  attacks various dyes and damages white curtains during ironing.  $NH_4Al$  sulfate hydrolyzes, forming  $H_2SO_4$  which causes damages.  $(NH_4)_2HPO_4$  is unstable, forming  $NH_4H_2PO_4$ , which is injurious by reason of the presence of the free acid radical and in addition the salt ppts. on the surface of the fabric.  $(NH_4)_2SO_4$  forms  $NH_3$  and  $H_2SO_4$ , when both which destroy the fiber and the dye.  $MgSO_4$  acts similarly. Boric acid and Na borate have a gradual damaging action. Pb silicate should not be used on account of its low efficiency and its very poisonous character.  $ZnCl_2$  damages the fiber. Fireproofing impregnations which do not affect the fiber and dyes and which have a good and permanent action are obtained with Na tungstate, Na vanadate, Na stannate, titanate and  $NH_4Br$ . Na tungstate is used either alone in 10-20% solns. or dissolved together with Na phosphate (20% Na tungstate + 4% Na phosphate). In order to avoid the suffening of the fabric by impregnation with the other fireproofing agents it is advisable to use addns. of salts of low hygroscopicity as, e.g., lactates. When a fabric is treated with Na stannate it is first dipped into a 25% soln. of this salt, then dried and finally treated with Zn acetate soln. Titanate acid ppts. from its compds. as an insol. solid in the fiber. The effect can be increased by impregnations with Na tungstate or stannate. Leopold Scheffan

**Effect of loading materials on the light fastness of dyed**

11A. Regenerating fatty substances from waste waters of the wool industry (Sololov) 27. Cleansing agents (for textiles) (Brit. pat. 435,445) 23. Mono-N-alkyl derivatives of aromatic diamines and polyamines (U. S. pat. 2,022,245) 10. App. for drying cloth (U. S. pat. 2,027,317) 1. Reaction products of substituted methylenethiuronnes (Ger. pat. 619,246) 10. Lecithin preps. [used in the textile industry] (Fr. pat. 788,632) 27. Wetting agents [for making dye pastes and rinsing dyed textiles fast to rubbing] (Brit. pat. 435,451) 13.

**Dyes.** I. G. Farbenindustrie A.-G. (Georg Kramlein, Hans Schliebmacher and Ludwig Schornie, inventors). Ger. 619,521, Oct. 3, 1935 (Cl. 22e 3). See U. S. 2,016,022 (C. A. 29, 5663) where the word "aldehyde" should be "anhydride".

**Dyes.** John D. Kendall. Brit. 432,628, July 23, 1935. Divided on 431,141 (C. A. 29, 7541). Comps. containing an ethylene linkage or a polymethylene chain are prep. by causing an org. compd. containing a ketomethylene (CO-

$CH_2$ ) group and of formula  $=C(CH_3)COX$  or  $=C(CH_3)COCH_2X$ , where X is C, S or O, to react with a

quaternary salt of a heterocyclic N compd. containing a reactive group an aryl, alkyl or aralkyl thio ether or seleno ether, an amino-, arylamino-, acetamido- or acetylaminovinyl or halo group. Alternatively, the org. compd. containing the ketomethylene grouping, the heterocyclic base and an alkyl salt are mixed together and heated to effect quaternary salt formation simultaneously with the condensation reaction. The products are also obtained by condensing with a heterocyclic quaternary  $NH_4$  salt having a reactive group, the condensation product of the compd. containing the ketomethylene group and a formamidine or a formimino ether. Among examples, (1) 1,3-indandione is condensed with diphenylformamidine in  $Ac_2O$  and the product with 1-methyl-2-pyridylmethyl Et  $p$ -toluenesulfonate, lepidine-Mel and guanidine EtI to give products having a dimethenyl linkage, indandione and heterocyclic radicals, and (2) 1-hydrindone is condensed with the product of 1-methylquinoline-2-thione and ethyl- $p$ -toluenesulfonate to give N-methyl-2-hydroquinolyl 2,2'-indan-1-one.

**Dyes, intermediates.** Societe pour l'ind. chim. à Bale. Brit. 433,574, Aug. 16, 1935. Anthraquinonecarbazole dyes are prep. by treating with a diazotizing agent a compd. of formula 1-(ANH)-2(H,N)C<sub>6</sub>H<sub>3</sub>, where A is anthraquinonyl or a nuclear substitution product thereof where at least 1  $\alpha$ -position to the  $NH_2$  is occupied by H and X is H or a univalent substituent, and the product is heated to above 100°, followed, if desired, by treatment with a halogenating or nitrating agent. On diazotization, an anthraquinone-azimide is probably formed and this loses N on heating to form a carbazole. Among examples, (1)  $\alpha$ -nitroanthraquinone (prep. by the reaction of 1-aminoanthraquinone with  $\alpha$ -nitrochlorobenzene) (II) is reduced by hypophosphite, diazotized and heated in  $PhNO_2$  in presence of 1  $NH_2$ , the product may be nitrated in  $PhNO_2$ . (2) 1-aminoanthraquinone is condensed with nitro- $p$ -dichlorobenzene, the product reduced, diazotized and boiled in diphenylamine (II); bromination of the product in  $PhNO_2$  yields a yellow vat dye, (3) 4-amino-1,1'-anthranilide is condensed with I, the product reduced, diazotized and boiled in II to give a gray dye.

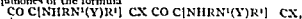
**Dyes of the anthraquinone series.** Klaus W. Wenzel and Ernst Kalkbrenner (to General Aniline Works). U. S. 2,027,658, Jan. 14, 1935. Anthraquinone compds. of the general formula  $A(NHRSO-alk)_n$ , where A stands for a radical of the anthraquinone series, R stands for a radical of the benzene series, alk stands for an alkyl group, and n stands for one of the numbers 1 and 2, and where at least one sulfonic acid group is present in the molecule, being in form of their alkali metal salts generally crystalline dark metallic lustrous powders, easily sol. in water and

dyeing wool from an acid bath generally bluish to greenish shades, are prep'd by various methods, e. g., there may be first prep'd. anthraquinone-sulfonic acids contg one or two substituents which can be replaced by radicals of amines, such as halogen atoms or hydroxy groups. These substituted anthraquinone-sulfonic acids are then caused to react with an amine of the general formula:  $\text{NH}_2\text{R}^1\text{SO}_2\text{R}^2$ , wherein R means a radical of the benzene series, or the said substituted anthraquinone-sulfonic acids are condensed with an amine of the general formula  $\text{NH}_2\text{R}^1\text{SR}^2$ , alkyl, R meaning a radical of the benzene series, in which case an oxidation of the sulfide to the sulfoxide must be carried out, this oxidation can be performed either simultaneously with the condensation process or afterward in a separate step. Various examples, details and modifications of procedure are described.

**Dyes of the anthraquinone series** Donald P. Graham (to I. du Pont de Nemours & Co.) U. S. 2,024,140, Jan 14. Dyes producing yellowing dyestuffs are obtained when 1-benzoylamino-6-aminoanthraquinone or other similarly reacting, 1-acylamino-6-aminoanthraquinone is treated with benzotrichloride and S or with other thiazolizing reagents to convert it to the 1-acylaminoanthraquinone-5,6-thiazole, which may then be hydrolyzed to give the new intermediate, 1-aminoanthraquinone-5,6-thiazole. This primary amino comp'd may then be condensed with alkyl or aryl acid chlorides or chloro comp'ds of the anthraquinone series to give a series of 1-aryl-aminoanthraquinone-5,6-thiazole and 1-arylaminoanthraquinone-thiazoles, and the latter class may be converted into other dyes by treating them with acid-condensing agents. Several examples with details of procedure are given.

**Dyes of the dibenzanthrone series** Alexander J. Wuerz (to E. I. du Pont de Nemours & Co.) U. S. 2,024,117, Jan 14. When diketodibenzanthrone, dihydroxydibenzanthrone or the substitution products of the latter are reduced in alk. suspension by boiling with finely divided metals which liberate H, the dye is brought into soln, while the org. and inorg. impurities are put in or remain in an insol form, whereby they may be sepd by filtration. When the same comp'ds are vatted with alk. hyposulfite, there is no such selective action, and much of the org. material is brought into soln. The org. impurities removed by the use of Zn or equiv. metals, after being freed from such metal, are sol in alk. hyposulfite, and give very dull bluish green dyestuffs. The dyestuffs obtained from the purified product are materially increased in purity and brilliance. Several examples with details of procedure are given.

**Dyes of the oxazine series** Georg Kranzlein, Heinrich Greune and Max Thiele (to General Aniline Works) U. S. 2,026,092, Dec 31. 2,5-Di-(N-ethylcarbamoyl)-3'-amino-3,6-dichloro-1,4-benzoquinone and other benzoquinones of the formula



where X stands for H or halogen, Y for H or alkyl and R and R' for aromatic groups of the benzene or naphthalene series, one of the R's being an aromatic group of the benzene series, or R, R' and N' together stand for a carbazole ring system, yield, when being treated as such alone or with the addn. of an oxidizing agent and, if necessary, of a catalyst with high boiling solvents, bluish condensation products, from which, by sulfonation, water-sol dyes are obtained which dye blue, bluish gray or bluish green tints. For the reaction there may be used as high-boiling solvents nitrobenzene, trichlorobenzene or the like, as oxidizing agents  $\text{K}_2\text{FeC}_2\text{O}_8$ ,  $\text{MnO}_2$ ,  $\text{FeCl}_3$  or similar products and as catalysts there may be used  $\text{PbCl}_2$ ,  $\text{SbCl}_3$  and others. Instead of first condensing and then introducing the sulfo group it is also possible to introduce the sulfo group before the oxazine has been formed, by starting for instance from diaryl-amino- or amino-carbamoyl-sulfonic acid. The products have good fastness to light, are suitable for dyeing silk and when treated with bases as described in U. S. 1,500,299 (C. A. 25, 3495) yield blue, bluish gray or bluish green pigments suitable for

use in varnishes and lacquers. U. S. 2,026,093 relates to various pigment dyes, similarly produced, suitable for coloring wall papers, rubber, sugar, varnishes and printing compns. Several examples with details of procedure are given. Cf. C. A. 29, 83401\*, 30, 620\*

**Pigmentary dyes** I. G. Farbenindustrie A.-G., Brit. 435,817, Sept. 30, 1935. See I. r. 787,453 (C. A. 30, 1237\*)

**Lakes** Gustave Widmer (to Soc. pour l'ind. chim. à Bâle) Brit. 435,767, Sept. 27, 1935. See I. r. 771,423 (C. A. 29, 039\*)

**Complex metal compounds of dyes** I. G. Farbenindustrie A.-G. Brit. 435,108, Sept. 9, 1935. These comp'ds are made by treating the disazo dye humidine (I)  $\Rightarrow$  a naphthol or a hydroxynaphthol acid arylide with a heavy metal salt of an org. carboxylic acid of high mol. wt., e. g., a higher fatty acid, resin acid or naphthenic acid. Org. solvents or substances that depress the m. p. of the salt may be added. The products from stearates may be used for coloring candles. Among examples, (1) a mixt. of the dye I  $\Rightarrow$   $\beta$ -naphthol, Cu stearate and xylene is boiled, the blue product is suitable for printing-varnishes, and the dye of (1) is boiled with Cr stearate and trichlorobenzene (bluish green)

**Azo dyes** Max Raack (to General Aniline Works) U. S. 2,019,830, Nov. 5. Dyes of the general formula  $3,5-(\text{O}_2\text{N})_2-2-(\text{HO})\text{C}_6\text{H}_3\text{N} \cdot \text{NC}_6\text{H}_4\text{X}-2,4-(\text{NYZ})_2$ , where X means H, halogen, an alkyl or alkoxy group and Y and Z stand for H, a hydroxyalkyl or polyhydroxyalkyl group, at least one Z however being a hydroxyalkyl or polyhydroxyalkyl group, being dark powders which are easily sol in water, and which dye wool in the presence of a Cr mordant reddish brown to Bordeaux tints are obtained from components such as diazotized 1-hydroxy-2-amino-4,6-dinitrobenzene and 4-chloro-1-amino-3-( $\beta, \gamma$ -dihydroxypropyl)-benzene, etc. (several examples being given).

**Azo dyes** Heinrich Clingestem and Karl Dolmayer (to General Aniline Works) U. S. 2,019,844, Nov. 5. Various examples are given of the production of dyes of the general formula  $(\text{HO}-\text{alk})\text{XNO}-\text{SR}^1\text{NR}^2$ , where "HO-alk" stands for a hydroxyalkyl group, X stands for H, alkyl, aryl, hydroxyalkyl or hydroaralkyl, R stands for the radical of a coupling component suitable for producing an azo dye which has been coupled in an alk. medium, said coupling component contg. as substituent at least a sulfonic or carboxylic acid group, and where the benzene nucleus R' may be substituted by substituents selected from the group consisting of alkyl, alkoxy, hydroxy, a substituted amino group, the nitro group and halogen. In the form of their alkali metal salts the dyes are generally yellowish to orange to red to brown powders, sol in water, dyeing animal fibers generally yellow to orange to red to brown shades of good fastness to fulling.

**Azo dyes** Carl Taube and Josef Hülger (to General Aniline Works) U. S. 2,026,920, Jan. 7. Disazo dyes generally dyeing cellulose fibers red to violet shades of good fastness to light are produced from components such as a diazotized 2,4-dihaloaniline b-sulfonic acid, m-toluidine and 2-benzoylamino-5-naphthol-7-sulfonic acid or the like.

**Azo dyes** Gerald Bonhôte (to Soc. pour l'ind. chim. à Bâle) U. S. 2,027,897, Jan. 11. Dyes which when prep'd on the fiber produce vivid scarlet to red tints are formed of components such as diazotized 2-amino-4-acetyl-1,1'-diphenyl ether and 2,3-dihydroxy-naphthoic acid-5'-chloro-2'-methylamide or the like.

**Azo dyes** I. G. Farbenindustrie A.-G. Fr. 785,638, Aug. 14, 1935. 2-(Alkoxy- or halo-aminoaryl)arylenethiazole

comp'ds of the formula  $4-(\text{H}_2\text{N})-3-\text{XC}_6\text{H}_4\text{C}_2\text{N} \cdot \text{C}_6\text{H}_4\text{S}$ , in which X is alkoxy or halogen and the  $\text{C}_6\text{H}_4$  rings may contain other substituents, are diazotized and coupled with coupling components. Thus, 2-(4'-amino-3'-methoxyphenyl)-6-methylbenzothiazole-sulfonic acid (by condensing 4-nitro-3-methoxybenzoyl chloride with 4-amino-3-mercaptotoluene, reducing and sulfonating) is diazotized and coupled with 2-phenylamino-5-naphthol-7-sulfonic acid. Reddish violet shades are obtained on cotton with this dye. Other examples are given.

**Azo dyes** I G Farbenund A-G Fr. 787,733, Sept. 27, 1935 Dyes are prep'd by coupling diazotized compds of amines of the formula  $3\text{HO}\cdot\text{C}_4\text{H}_3\text{N}_2\text{C}_6\text{H}_4\text{NH}_2$ , (X is  $\text{CONH}$  or  $\text{SO}_2\text{NH}$ , and the benzene ring may contain other substituents) with halogenated 1-aryl-5-pyrazolones and treating the dyes in substance or on the fibers by agents furnishing Cr Thus, 3-(*m*-aminobenzamido)-5-sulfo-6-hydroxybenzoic acid  $\rightarrow$  1-(*o*-chlorophenyl)-3-methyl-5-pyrazolone gives yellow dyes on wool which are little changed by chroming. Other examples are given.

**Azo dyes** J R Geigy S A. Fr. 788,247, Oct. 7, 1935 Tetrazotized diamino bases of the formula  $\text{H}_2\text{N}-\text{RSR}-\text{NH}_2$  (R is an aromatic radical) are coupled with para substituted phenols, neither of which contain solubilizing groups. The dyes may be used for coloring cellulose ester lacquers, etc. In an example tetrazotized thioaniline is coupled with 4-hydroxy-1-methylbenzene and the dye formed colors cellulose esters pure yellow shades. Cf. C. A. 30, 872<sup>1</sup>

**Azo dyes** Soc. pour l'ind. chim. à Bale. Fr. 787,483, Sept. 23, 1935 Fast dyes are prep'd in substance or on the fiber by causing diazo compds of the formula 2-X-5-X-4-( $\text{ROCH}_2\text{CONH}$ ) $\text{C}_6\text{H}_4\text{N}_2\text{OH}$  (R is an aromatic ring of the  $\text{C}_6\text{H}_4$  series and X is alkoxy) to react with arylides of *o*-hydroxy carboxylic acids coupling in position ortho to the OH, operating in a medium the pH of which does not pass 6.5 and is not appreciably below 5. Thus 4-(1'-methylphenoxy)acetamido-2,5-dihydroxyaniline  $\rightarrow$  2,3-hydroxynaphthoic acid naphthalide gives a bright blue on cotton and 4-(3'-methylphenoxy)acetamido-2,5-dimethoxyaniline  $\rightarrow$  2,3-hydroxynaphthoic acid 4'-ethoxyanilide a bright violet shade.

**Azo dyes** Soc. pour l'ind. chim. à Bale. Fr. 788,349, Oct. 8, 1935 Dyes are prep'd by causing 1-nitroaminonaphthalene, in which the para position to the nitroamino group is free, to react with diazo compds which are not or only very slowly modified by dil. alkalis, e.g., 1-diazo-2-ethoxy (and methoxy)benzene.

**Azo dyes** I G Farbenund A-G (Hans Schindhelm, inventor) Ger. 618,810, Sept. 16, 1935 (Cl. 22a 13) Stilbene dyes are obtained by condensing nitrostilbene compds of the general formula 4-X-2-HO $\cdot\text{SC}_6\text{H}_4\text{CH}=\text{CH}\cdot\text{C}_6\text{H}_4\text{SO}_2\text{N}_2\cdot 2'\cdot\text{NO}_2\cdot 4'$ , where X represents a  $\text{NO}_2$ , arylazo or arylazoxy group, with aminostilbene compds of a similar general formula, the  $\text{NO}_2$  being replaced by  $\text{NH}_2$  and the X representing  $\text{NO}_2$  or arylazo or arylazoxy groups different from those in the nitrostilbene compd. The condensation takes place at the  $\text{NO}_2$  and  $\text{NH}_2$  groups and is effected by heating the compds with alkali. In an example Na 4,4'-diaminostilbene-2,2'-disulfonate is heated with Na 4-nitro-4'-aminostilbene-2,2'-disulfonate in 10 N NaOH to give a dye. The shades of the dye can be brightened by af. treatment with Cl<sub>2</sub>. Other examples are given. Cf. C. A. 29, 4930<sup>1</sup>

**Azo dyes** I G Farbenund A-G (Karl Holzach and Ludwig Neumann, inventors) Ger. 622,409, Nov. 27, 1935 (Cl. 22a 1) The diazo compds. of aromatic monoamines not contg.  $\text{SO}_3\text{H}$  groups are coupled with 2,4-dihydroxyquinolinesulfonic acids or with the sulfonic acids of N-alkyl-4-hydroxy-2-quinolones. Yellow dyes, particularly useful for printing acetate rayon, are obtained. Sp. dyes are described.

**Azo dyes** I G Farbenund A-G Brit. 432,353, July 18, 1935 Dyes are made by coupling a diazotized amine of formula  $\text{AXNH}_2$ , in which A is the residue of an aminobenzene, B is the residue of an *o*-hydroxybenzoic acid and X is CO or  $\text{SO}_2$ , with a pyrazolone contg. the residue of an *o*-hydroxybenzoic acid and corresponding to the formula (a)  $3\text{HO}\cdot\text{C}_4\text{H}_3\text{N}_2\text{C}_6\text{H}_4\text{R}$  (R =  $-\text{N}\cdot\text{C}_2\text{H}_5$ ), (b)  $5\text{Y}\cdot 2\text{C}_4\text{H}_3\text{N}_2\text{C}_6\text{H}_4\text{R}$  (R =  $-\text{C}_6\text{H}_4\text{R}$ ), (c)  $m\text{-(3-HO}\cdot\text{C}_4\text{H}_3\text{N}_2\text{C}_6\text{H}_4\text{R})_2$ , (d)  $m\text{-(3-HO}\cdot\text{C}_4\text{H}_3\text{N}_2\text{C}_6\text{H}_4\text{R})_2$ , in which Z is alkyl, aryl, COOH, COO-alkyl, CONH<sub>2</sub> or CONHR (R is an org. radical) and Y is H,  $\text{SO}_3\text{H}$ ,  $\text{CH}_3$ , Cl or  $\text{NO}_2$ , the OH being attached to 1 of the *o*-positions to the COOH in the nucleus marked A. The Cr compds. of the dyes (prep'd in substance or on the fiber by after-chroming the

dyeing or by dyeing chrome mordanted fibers) have a good leveling power and dye animal fibers yellow shades that are fast to light, washing, fulling and carbonizing. Among dyes and their Cr compds. prep'd in examples are (1) 1-hydroxy-4-(4'-methyl-3'-aminobenzoyl)aminobenzene-2-carboxylic acid, 1-hydroxy-4-(3'-aminobenzoyl)aminobenzene-2-carboxylic acid-6-sulfonic acid, 1-hydroxy-4-(1'-aminobenzene-3'-sulfonyl)aminobenzene-2-carboxylic acid (I) or 1-hydroxy-4-(4'-or 3'-aminobenzoyl)aminobenzene-2-carboxylic acid  $\rightarrow$  1-(2'-hydroxy-3'-carboxy-5'-sulfophenyl)-3-methyl-5-pyrazolone, (2) 1 or 1-hydroxy-4-(3'-aminobenzoyl)aminobenzene-2-carboxylic acid  $\rightarrow$  1-(5'-sulfon-4'-hydroxy-3'-carboxy-2'-diphenyl sulfone)-3-methyl-5-pyrazolone.

**Azo dyes, dyeing** Imperial Chemical Industries Ltd and Arthur H. Knight. Brit. 435,807, Sept. 30, 1935 Monoazo dyes are made by coupling a diazotized *o*-nitroaniline with a *N*-sulfatoethyl deriv. of a *p*-coupling amine of the  $\text{C}_6\text{H}_4$  series. They give yellow and orange colorings on acetate rayon, wool, silk and leather and are suitable for printing rayon. In examples dyes are prep'd from *o*-nitroaniline, 3-nitro-4-toluidine, 4-chloro-2-nitroaniline; the *N*-sulfatoethyl derivs. of *o*- and *m*-toluidine, PhNH<sub>2</sub> and 3-amino-4-methoxytoluene. Cf. C. A. 29, 7669<sup>1</sup>.

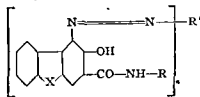
**Azo dyes; dyeing, intermediates** I G. Farbenindustrie A-G Brit. 435,711, Sept. 26, 1935 H<sub>2</sub>O-insoluble dyes are made by diazotizing a base of formula 3(or 4)- $\text{H}_2\text{N}\cdot\text{C}_6\text{H}_4\text{NHCOR}$ , where R is an alphabetic radical, particularly an alkylene group, and R' is alkyl, alkoxy-alkyl, aralkyl or a hydrogenated aromatic radical and in which the  $\text{C}_6\text{H}_4$  nucleus may contain nonsolubilizing substituents, e.g., alkyl, alkoxy, halogen, and coupling in substance or on a substratum, particularly on vegetable fibers, with an arylamide of an *o*-hydroxy carboxylic acid that couples in *o* position to the OH. Among examples, cotton is impregnated with the arylamide from aminohydroquinone dimethyl ether and 2,3-hydroxynaphthoic acid and developed with diazotized 1-amino-4-methoxyacetyl amino-2,5-dimethoxybenzene to yield a clear reddish blue. The amines of above formula are made by condensing a *p*- or *m*-nitroamine of the  $\text{C}_6\text{H}_4$  series free from solubilizing groups with a carboxylic acid of formula  $\text{HOOC}\cdot\text{R}'\cdot\text{OR}$ , or a functional deriv. thereof, e.g., a halide or ester, and reducing the  $\text{NO}_2$  group, or by acylating an amine of the  $\text{C}_6\text{H}_4$  series free from solubilizing groups and subsequently introducing the amino group into the *m*- or *p*-position to the arylamino group by nitration and reduction.

**Blue azo dyes** Gerald Bonhote (to Soc. pour l'ind. chim. à Bale) U. S. 2,027,764, Jan. 14 Various examples are given of blue dyes formed by diazotizing an initial material such as 4-(4'-methylphenoxy)acetyl amino-2,5-dihydroxyaniline and coupling the diazo compd thus formed with an arylide of 2,3-hydroxynaphthoic acid which is itself obtainable by condensing 2,3-hydroxynaphthoic acid with an analog or homolog of aniline which can be regarded as an ortho substitution product thereof, such as *o*-toluidine, *o*-aminophenol, *p*-xylidine, *m*-xylidine, cresidine, chloroaniline ( $\text{NH}_2$ ,  $\text{OCH}_3$ , Cl = 1, 2, 5), amino hydroquinone di-Me ether, 4-chloro-2-toluidine, 2,4-dimethoxy-5-chloroaniline, 1-aminotetrahydroquinoline, or amines which are substituted in *o*-position to the amino group by an *O*-alkyl group whose alkyl groups contain more than one C atom, e.g., *o*-phenetidine, further the mixed ethers from *o*-aminophenol and aliphatic alcohols such as Pr, Bu, Am, alc, or also ether alc, such as glycol mono-Bu ether, glycol mono-Et ether, glycerol di-Bu ether, or the like. The dyes may be formed on the fiber. Cf. C. A. 29, 8333<sup>1</sup>

**Substantive azo dyes** I G. Farbenund A-G. Fr. 788,104, Oct. 4, 1935 The monoazo dye prep'd by coupling 1 mol. proportion of 1-amino-8-naphthol-3,6-disulfonic acid (I) and 1 mol. proportion of a diazotized mononitroaromatic amine in acid medium is coupled with 1 mol. proportion of the same or another diazotized mononitroaromatic amine in alk. medium and the diazo dye obtained is reduced to a diamino-diazo dye which is tetrazotized and coupled with 2 mol. proportions of *m*-aminophenols which

contain 1 atom of halogen in the position para to the  $\text{NH}_2$  group. The nitroarylamines may be replaced by monoacylated diamines and the acyl group afterward sepd. by sapon. The dyes give black shades fast to acids on cotton and rayon. Thus, the dye *p*-nitroaniline  $\rightarrow$  I  $\rightarrow$  *p*-nitroaniline is reduced, tetrazotized and coupled with 2 mols of 6-chloro-3-aminophenol.

**Water-insoluble azo dyes** Friedrich Muth (to General Aniline Works) U S 2,026,908, Jan 7. Numerous examples are given of dyes (dyeing brown to black shades) which have the general formula



where X stands for a S or an O atom, R and R' for aromatic radicals and n stands for one of the nos 1 and 2 Cf C A 28, 5249.

**Ethylene azo dyes** Robert Wizinger (to I G Farbenindustrie A.-G.) Brit 435,449, Sept 16, 1935. The dyes, or acid addn products thereof, are prepd by coupling with diazonium salts ethylenes of formula  $\text{R}(\text{R}')\text{C}(\text{CH}_3)(\text{R}')_2$ , where R and R' are aryl groups substituted by at least 1 group imparting a pos. character, e. g., MeO, HO,  $\text{NH}_2$ ,  $\text{NMe}_2$ , PhNH and R' is a univalent atom or group, or acid addn products thereof. In examples, (1) 2-naphthylamine-6,8-disulfonic acid is diazotized and coupled with tetramethyldiaminodiphenylethylene, the product dyes wool Bordeaux-red shades from an alk. bath and, on acidifying, an intense blue shade is obtained, and (2) *p*-nitrophenyldiazotized is coupled (acid) with diaminylethylene.

**Pyrazolone azo dyes** Walther Benade (to General Aniline Works) U S 2,026,861, Jan 7. Monoazo dyes dyeing wool by the single bath process in the presence of a chrome mordant orange to red tints of good fastness are made from components such as diazotized 4-nitro-6-chloro-2-amino-1-hydroxybenzene and 1-(2'-naphthyl-6'-sulfo)-3-methyl-5-pyrazolone or the like.

**Azo dyes containing metals** I G Farbenind. A.-G. Fr. 788,031, Oct 2, 1935. Salts of fat acids of low mol. wt. and contg. halogen along with compds of metals capable of forming complex compds. with dyes are caused to react with azo dyes capable of forming complex metal compds. Thus, 2-aminobenzoic acid  $\rightarrow$  1-phenyl-3-methyl-5-pyrazolone is heated in water to 135° with  $\text{Cr}_2\text{O}_3$  and  $\text{CH}_3\text{CICOOH}$ . Other examples are given. Cf C. A. 30, 1240.

**Azo dyes and soluble copper compounds thereof** I G Farbenindustrie A.-G. Brit 436,095, Oct 4, 1935. Dyes are obtained by condensation of a mono- or poly-azo dye contg. at least once the grouping  $\text{hal}-\text{NR}'\text{OH}$ , in which R and R' are residues of the  $\text{C}_6\text{H}_5$  or  $\text{C}_6\text{H}_4$  series, the OH is in *o*-position to the azo bridge and the halogen atom, hal, is in *o*- or *para*-position to the azo bridge, with  $\text{NH}_3$ , amines, alcohols, phenolates, sulfites or sulfonates, in presence of a Cu compd. An acid-binding agent and (or) a nitrogenous base may be present. The products are in some cases free from Cu. Cu may be removed from the Cu complex compds. by treatment with acid, alkali sulfides or alkali cyanides. Among examples, (1) the dye 2,4-dichloroaniline-6-sulfonic acid  $\rightarrow$   $\beta$ -naphthol is heated with  $(\text{AcO})_2\text{Cu}$  in EtOH to 78°, KOH is added and the whole boiled 1 hr.; the product on treatment with  $\text{Na}_2\text{S}$  in aq. soln. is converted into the Cu-free dye which gives orange dyes on wool, and (2) the dye 1-chloro-2-naphthylamine-6-sulfonic acid  $\rightarrow$  2,7-naphtholsulfonic acid is heated 1 hr. at 85-90° with an aq. soln. contg.  $\text{CuSO}_4$ ,  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{SO}_3$ ; the product dyes wool red.

**Monoazo dyes.** Imperial Chemical Industries Ltd., Arthur H Knight and Henry A Piggott. Brit. 434,126, Aug. 23, 1935. Dyes for coloring acetate rayon are made by coupling a diazotized *p*-nitro aniline of the  $\text{C}_6\text{H}_4$  or

$\text{C}_6\text{H}_3$  series, free from COOH and  $\text{SO}_3\text{H}$  groups, with a compd. or mixt. of compds. of formula  $\text{ArRNCH}_2\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OH}$ , where Ar is a Ph residue free from COOH and  $\text{SO}_3\text{H}$  groups and has an unsubstituted para coupling position to the N, R is Me, Et,  $\beta$ -hydroxyethyl or  $\text{CH}_2\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OH}$ , n and m being integers greater than 0 such that  $n + m$  does not exceed 10 and preferably does not exceed 4. They may be converted into dispersible dye powders by means of known dispersing agents. They may be used in admixt. with other insol. azo or anthraquinone dyes. In examples, dyes from the components, 2,4-dinitroaniline, 6-chloro- or 6-bromo-2,4-dinitroaniline, 2-chloro-4-nitroaniline, *p*-nitroaniline, 2,4-dinitro-1-naphthylamine, 5-nitro-2-aminidine, 2,6-dichloro- or 2,6-dibromo-4-nitroaniline, and the products obtained by condensing various quantities of ethylene oxide with *N*- $\beta$ -hydroxyethyl-*m*-toluidine, *m*-toluidine, *N*-ethylamine and 3 PhNH<sub>2</sub>, are described. The coupling components may also be obtained by condensing a suitable amine with a mixt. of ethylene chlorohydrin and NaOH.

**Disazo dyes** Arthur H Knight (to Imperial Chemical Industries Ltd.) U S 2,027,178, Jan. 7. See British 395,005 (C A 28, 335).

**Disazo dyes** J R Geigy S. A. Fr. 788,119, Oct. 4, 1935. See Ger 620,258 (C A 30, 874).

**Mordant disazo dyes** Ernst Hug and Max Müller (to Durand & Huguennin S. A.) U S 2,027,777, Jan. 14. Dyes yielding violet-brown shades of good fastness on cotton by chrome printing and which have the general formula 1,4-(alkyl-O)-2-R<sup>1</sup>N-5-R<sup>2</sup>N NCH<sub>3</sub>, where R<sup>1</sup> stands for a sulfonated benzene nucleus and R<sup>2</sup> stands for a benzene nucleus contg. as substituents a hydroxyl and a carboxyl group in *o*-position to each other, are produced by diazotizing a sulfonated amine of the benzene series (NH<sub>2</sub>R<sup>1</sup>), combining the diazo compd. thus formed in an acid medium with an aminohydroquinone dialkyl ether, then diazotizing the aminoazo compd. thus formed and finally combining it with an *o*-hydroxy carboxylic acid of the benzene series. Several examples are given.

**Trisazo dyes.** Ernst Hug and Max Müller (to Durand & Huguennin S. A.). U. S. 2,024,797, Dec. 17. Trisazo dyes yielding in chrome printing on cotton green to blue-green shades of good fastness (especially to Cl<sub>2</sub>), which can be discharged to a pure white by hyposulfite and are suitable for chrome printing on cotton and correspond with the general formula (naphthalene deriv.) 1-HO-8-H<sub>2</sub>N-2-R<sup>1</sup>N NR<sup>2</sup>N-7-R<sup>3</sup>N N-3,6-C<sub>6</sub>H<sub>3</sub>(SO<sub>3</sub>H)<sub>2</sub>, where R<sup>1</sup> represents a benzene nucleus, substituted by  $\text{SO}_3\text{H}$ , NO<sub>2</sub>, COOH groups or halogen, R<sup>2</sup> means a sulfonated benzene nucleus, R<sup>3</sup> means a benzene nucleus bearing as substituents a hydroxy and a carboxylic group in *o*-position to each other and which may contain as further substituents a CH<sub>3</sub> group or halogen are made by combining a 1-amino-5-hydroxynaphthalene-3,6-disulfonic acid first in an acid medium with a diazotized amino compd. of the type R<sup>1</sup>NH<sub>2</sub> and subsequently in an alk. medium with a diazotized amino azo compd. of the type R<sup>2</sup>N NR<sup>3</sup>NH<sub>2</sub>, where R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> have the meanings defined. Several examples with details of procedure are given.

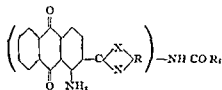
**Tetrakisazo dyes** suitable for dyeing leather brown shades Francis H Smith and Crayton K. Black (to E. I. du Pont de Nemours & Co.) U S 2,027,206, Jan. 7. Numerous examples are given of dyes of the general formula (R<sup>1</sup>N NR<sup>2</sup>N N<sub>2</sub>)(HO)<sub>2</sub>XC<sub>6</sub>H<sub>3</sub>, in which R<sup>1</sup> and R<sup>2</sup> represent aryl groups and X represents groups such as halogen, alkyl, alkoxy, nitro, carbonyl, sulfonic, hydroxyl and H. These compds. are produced by coupling two equivalents of a diazotized mono-acyl-diamino or nitro-amino deriv. of an aromatic compd. with a dihydroxybenzene, treating the resulting compd. with the monoacylamino or nitro groups to amino groups, then diazotizing and coupling with two equivalents of an aromatic coupling component Cf. C. A. 30, 324.

**Polyazo dyes** I. G. Farbenind. A.-G. Fr. 788,238, Oct. 7, 1935. Dyes which give black shades fast to acid on leather are prepd by coupling in mineral acid medium 1 mol. of a tetrazotized *p,p'*-diaminoaryl with 1 mol. of a 1-amino-8-hydroxynaphthalene-3,6-disulfonic acid (I) and

uniting this compd in alk. medium with 1 mol of a mono-diazo compd and 1 mol of a 3,5-diaminophenol in which each of the 2 NH<sub>2</sub> groups contains an aryl group. Thus, tetrazotized benidine is coupled in mineral acid medium with I, then united with diazotized *p*-sulfanilic acid and finally coupled in alk. medium with bis(*p*-chlorophenyl)-3,5-diamino-1-hydroxybenzene.

**Vat dyes** I G Farbenind A-G (Karl Köberle and Christian Steigerwald, inventors) Ger 622,173, Nov. 21, 1935 (Cl 226 3 09). Nitroindibenzanthrones containing more than 4% of N are heated with S or other sulfurizing agent. The reaction may be effected by boiling the materials in a solvent, e. g., naphthalene in the presence or absence of a catalyst, e. g., Cu or Mo or a compd thereof. Black or olive dyes are obtained. Sp. processes are described.

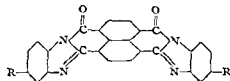
**Vat dyes** I G Farbenind A-G Ir 785,275, Aug. 6, 1935. Dyes of the anthraquinone series are made by process, by processes known in themselves, aminoanthraquinone-oxazoles, -thiazoles or -imidazoles of the formula



in which X is O, S or NH, R is an anthraquinone radical, R<sub>1</sub> a cyclic radical and which may also contain one or more other amino or acylamino groups. Several examples are given. Thus, 1-amino-4-nitro-2-anthraquinonyl 2',3'-anthraquinone-oxazole (by condensing 1-amino-4-nitro-anthraquinone-2-carbonyl chloride with 2-amino-3-hydroxy-anthraquinone and treating with *p* toluenesulfonic acid) is reduced to the 1,4-diamino compd which dyes cotton greenish blue from the vat. Several examples are given.

**Vat dyes** I G Farbenind A-G Fr 785,168, Aug. 28, 1935. Asymmetric indigo dyes are prepared by condensing 7-alkoxy-3-hydroxythionaphthene, containing either in the 4- or 5 position a halogen or an alkyl group or a halogen and an alkyl group, according to the usual method, with 3-hydroxythionaphthene or isatins of the C<sub>10</sub>H<sub>7</sub> series, to form bis(hionaphthene-2,2'-indigo or thionaphthene-2-indole-2'-indigo), and, if necessary, subsequently halogenating the products obtained. Thus, 4-methyl-5-chloro-7-methoxy-3-hydroxythionaphthene is heated with 4-methyl-5,7-dichloro-2,1-diketodihydrothionaphthene 2-(*p*-dimethylamino)anil in AcOH giving a product which dyes cotton violet shades from the vat. Other examples are given. Cl C A 30, 875.

**Vat dyes** I G Farbenind A-G Fr 784,782, Sept. 9, 1935. Dyes are prepared by treating with acid-condensing agents, such as AlCl<sub>3</sub>, bis(anthraquinonylamino)-1,4,5,8-naphthylenebis(arylimidazoles), prepared by the reaction of aminoanthraquinones on naphthylenebis(arylimidazoles) containing an atom of halogen. Examples are given of the prepn of compds of the formula



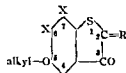
R is 1-anthraquinonylamino group, its 2-Me deriv or its 4-benzamido deriv. Cl C A 30, 623.

**Vat dyes of the anthracene series** Wilhelm Eckert and Otto Brunsdorf (to General Aniline Works) U S 2,028,384, Jan. 21. See Ger 612,930 (C A 29, 6433).

**Vat dyes of the anthraquinonecarbazole series** Donald P. Graham (to I. du Pont de Nemours & Co.) U S 2,028,103, Jan. 14. Anthranthide is fused with AlCl<sub>3</sub> at temps not materially above 250° and the product is

subjected to oxidation (suitably by use of Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in dil. H<sub>2</sub>SO<sub>4</sub> soln.). A dye of brighter and greener shade is obtained than results when higher fusion temps are used. NaCl and Na<sub>2</sub>CO<sub>3</sub> are also preferably included in the mix subjected to fusion.

**Indigoid vat dyes** Norbert Steiger and Eduard Albrecht (to General Aniline Works) U S 2,021,367, Nov. 19. Dyes of various colors and having the general formula



where one of the X's means methyl and the other halogen and R means the radical of a component capable of forming indigoid dyes, have good affinity for vegetable fibers. In some cases their tinctorial properties may be further enhanced by after-halogenation. Alkoxychloromethylhydroxythionaphthene, starting materials for making these dyes, may be obtained by converting the corresponding alkoxychloromethylbenzenesulfonic acids into the sulfonyl chlorides, mercaptans, thioglycolic acids and ring-closing the latter or also by introducing a S-contg. radical into the ortho position to the amino group of a corresponding alkoxychloromethylaminobenzene. These hydroxythionaphthene series, are themselves or after the transformation into their reactive 2-derivs condensed with isatins or hydroxythionaphthene of the benzene or naphthalene series or with the reactive 2-derivs thereof. Several examples with details of procedure are given. Cl C A 30, 875.

**Reducing vat dyes** Louis S. Bale (to F. I. du Pont de Nemours & Co.) U. S. 2,027,141, Jan. 7. An aq. suspension of indigo is treated with HCl, H<sub>2</sub>SO<sub>4</sub>, NH<sub>4</sub>Cl or ZnCl<sub>2</sub> and then with SO<sub>2</sub> and Zn to produce Zn hyposulfite and reduce the dye to its leuco compd. Other vat dyes may be similarly reduced.

**Brominated derivatives of vat dyes of the anthraquinone series** Karl Krauer (to Soc. pour l'ind. chim. A. Bale) U S 2,027,914, Jan. 14. Bromination of a fat dye such as an isodibenzanthrone is conducted in an inert solvent such as PhNO<sub>2</sub> and in the presence of ZnCl<sub>2</sub>.

**Indigoid dyes** Soc. pour l'ind. chim. A. Bale Fr 788,724, Oct. 15, 1935. This corresponds to Swiss 176,362 (C A 30, 297) with the addn that 5,7-dihaloisatin derivs may be used. Fr 788,724. See Swiss 174,270 (C A 30, 297).

**Indigoid dyes** Ernst Stocklin (to Soc. pour l'ind. chim. A. Bale) U. S. 2,027,889, Jan. 14; Brit 475,242, Sept. 19, 1935. Dyes of various colors suitable for dyeing cotton, wool and silk are produced from thionaphthene derivatives

the general formula,  $\text{CY CX CY CX C C CO C (Z) S}$ ,

where X represents halogen, Y alkyl, and Z a reactive group, such as O, aml, 2H, NOH, halogen or H and COOH, by condensing the thionaphthene with an indigoid component, the indigoid dye produced may be subsequently halogenated, e. g., 4,6-dimethyl-5-chloro-7-bromo-3-thionaphthene, 4,6-dimethyl-5,7-dichloro-3-thionaphthene, 4,6-dimethyl-5-bromo-7-chloro-3-thionaphthene, 4,6-dimethyl-5,7-dibromo-3-thionaphthene, the 2-amis, such as for example 2-*p*-dimethylaminoanis and 2-*p*-hydroxyanis, 2-carboxylic acids, 2-oximes, 2-dihals of these compds, as well as 4,6-dimethyl-5,7-dihaloanthionaphthenequinones obtainable from the above 2-amis by saponification with aq. acids may be used, with thionaphthene of the benzene, naphthalene, anthracene and anthraquinone series, indoxyls and isatins of the benzene, naphthalene and anthracene series, phenols, naphthols, hydroxyanthracenes and hydroxycarbazoles, and also  $\alpha$ -diketones of the isocyclic series and the products of these hydroxythionaphthene, phenols, hydroxycarbazoles and  $\alpha$ -diketones substituted in the aryl nuclei by halogen

groups, such as, for instance, Cl or Br, alkyl groups, alkoxy groups, such as, for example, methoxy or ethoxy groups, wherein only one of several substituents, which may be similar or different, can be present. Numerous examples, details and modifications are described.

**Anthraquinone dyes** I. G. Farbenind. A.-G. (Berthold Biebert, inventor) Ger. 622,311, Nov. 23, 1935 (Cl. 22b 3 02). Blue acid wool dyes are prepared by the following process: 1-Chloro-2-methyl-1 (I) denotes anthraquinone is chlorinated to yield 1-chloro-2-chloromethyl-I. The latter, heated with aq.  $\text{Na}_2\text{SO}_3$  soln., yields 1-chloro-1,2-methanesulfonic acid, which is then heated with  $\text{NH}_3$  or an aliphatic or hydroaromatic amine to yield a 1-amino-1,2-methanesulfonic acid. This is halogenated to yield, e.g., a 1-amino-4-bromo-1,2-methanesulfonic acid and finally this is heated with a nitrogenous base, e.g.,  $\text{PhNH}_2$ ,  $p$ -aminoacetanilide or cyclohexylamine, whereby the halogen is replaced by a substituted amino group. Suitable conditions for each reaction are described.

**Azine dyes** Wolfram Vogt and Eugen Huber (to I. G. Farbenindustrie A.-G.) Brit. 435,479, Sept. 23, 1935, addn. to 417,388 (C. A. 29, 12614). Dyes of the azine series contg. in the mol. at least once the group  $\text{alk-SO}_2\text{H}$  attached to N, where alk is an alkylene group of 3 C atoms of which 2 form a bridge between the N and the  $\text{SO}_2\text{H}$ , are synthesized by standard methods by use at least once of an aromatic sulfonic acid of formula  $\text{aryl-N(R)-alk-SO}_2\text{H}$ , where R is H, alkyl, hydroxyalkyl, sulfoalkyl or aralkyl and alk is an alkylene group as described above. In examples, (1) 1,3-bis(phenylamino)naphthalene-8-sulfonic acid is oxidized in the usual manner with an equimol. proportion of 4-aminobutylphenylaminopropanesulfonic acid, the product dyes wool greenish blue shades, and (2) 3-diethylsulfosindoline-1,6-disulfonic acid is condensed with 4-aminophenylbutylaminopropanesulfonic acid, wool is dyed blue shades.

**Dibenzanthrone-azole dyes** Alexander J. Wuertz and Myron S. Whelan (to E. I. du Pont de Nemours & Co.) U. S. 2,028,115, Jan. 14. Vat dyes of the dibenzanthrone-azole series, yielding upon cotton gray to dark blue and black dyestuffs, are obtained by alkali fusion of a benzanthrone-1,2-thiazole or a benzanthrone-1,2-selenazole. Several examples with details of procedure are given and general claim is made to new dyes consisting chiefly of a dibenzanthrone- $\alpha\beta,\alpha'\beta'$ -dithiazole or -diselenazole. U. S. 2,028,116 relates to the production of compds. of the

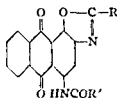
general formula  $\text{R-S-CX-N}$ , where R stands for a bivalent benzanthrone radical to which the S and N are connected in the  $\alpha$ - and  $\beta$ -positions, resp., while X stands for H, alkyl or aryl, such as  $\alpha\beta$ -benzanthrone-C-phenylselenazole or  $\beta\beta'$ -1-halogen- $\alpha\beta$ -benzanthrone-C-phenylselenazole, by reducing an anthraquinone-1,2-selenazole which contains no substituent in the remaining  $\alpha$ -positions and causing the same to react with glycerol in the presence of concd  $\text{H}_2\text{SO}_4$ . Several examples with details of procedure are given.

**Cyanine dyes** Kodak Ltd. and Bernard Beilenson Brit. 435,542, Sept. 19, 1935. In mfg. dyes of the cyanine type by the use of an acid-binding agent, the latter consists of an alkali metal salt of a weak org. acid, preferably in substantially anhyd and finely-divided form. Thus 1-methylbenzothiazole is condensed with diphenylformamidine in presence of anhyd  $\text{Na}_2\text{PO}_4$  and 2-iodoquinoline-1 (I) is condensed with quinaldine-EtI in presence of  $\text{K}_2\text{CO}_3$ .

**Gallocyanine dyes** I. G. Farbenindustrie A.-G. Brit. 436,090, Oct. 4, 1935. Addn. to 417,975 (C. A. 29, 16547). Dyes are prepared by condensing gallic acid or a deriv. thereof, e.g., gallamide, gallic acid arylamides or esters, pyrogallallic acid, with nitroso compds. of secondary or tertiary aromatic amines of formula  $\text{aryl-N(R)-alk-SO}_2\text{H}$ , where R is H, alkyl or substituted alkyl and alk is an alkylene group of at least 3 C atoms. For examples, cf. C. A. 29, 6433<sup>9</sup>.

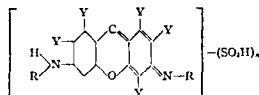
**Mordant dyes** Durand & Huguennin A.-G. Brit. 437,513, Sept. 23, 1935. This corresponds to Ir. 743,107 (C. A. 29, 70919).

**Oxazole dyes** I. G. Farbenind. A.-G. Ir. 787,918, Sept. 30, 1915. Oxazole dyes of the anthraquinone series are prepared by heating 1-hydroxy-2,4-diaminoanthraquinone (I) with aromatic carboxylic acid chlorides, advantageously in the presence of a diluent of high b. p. and advantageously also in the presence of an acid condensation agent and exchanging, in the condensation products obtained, the acylated groups joined to the N with other acylated groups. The products have the formula



R and R' are aryl groups which are different if they both belong to the  $\text{C}_{6}\text{H}_5$  series. Examples are given of the prepn. of dyes from I with benzoyl,  $p$ -chlorobenzoyl and 2,4-dichlorobenzoyl chloride,  $p$ -toluyl chloride, 1-chloroanthraquinone 2(II) and (II)-carbonyl chloride, biphenyl-4-carbonyl chloride and  $\beta$ -naphthoyl chloride.

**Pyrone dyes** I. G. Farbenind. A.-G. Ir. 786,405, Sept. 3, 1935. Dyes of the formula

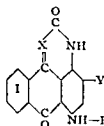


(R is a cyclic radical contg. more than 2 rings, X is alkyl or aryl contg. a COOH group, 1 or 2 of the Y's are halogens, the others H, n is a whole no. not greater than 4) are prepared by condensing fluorescein chloride or bromide or their substitution products with the following amines and subsequently sulfonating: aminodiphenylazenes, amino-hydroxydiphenylbenzenes, aminophenyl-naphthalenes, aminobiphenyls, aminofluorenes, aminofluoranthenes, aminochrysenes, aminopyrenes, aminocyanaphthalenes, 1- or 2-aminocarbazole, 3-amino-N-ethylcarbazole or their substitution products. They may also be prepared by condensing phthalic anhydride or its derivs. with aryl-m-aminophenols and sulfonating or by condensing 4-aryl-amino-m-hydroxy- $\alpha$ -benzoylbenzoic acids with aryl-m-aminophenols and sulfonating. Examples are given.

**Sulfur dyes** Paul Schetelig (to Soc. pour l'ind. chim. & Bâle) U. S. 2,027,323, Jan. 7. Aromatic compds. such as pyrene, fluoranthene, chrysene or aminochrysene are reacted with S at 200-300° to produce dyes which generally dye brown shades.

**Sulfur dyes** I. G. Farbenind. A.-G. (Werner Zierweck, inventor) Ger. 622,274, Nov. 25, 1935 (Cl. 22d. 3). See Brit. 111,431 (C. A. 28, 70373).

**Red to violet acid wool dyes** Klaus Weinand (to General Aniline Works) U. S. 2,025,921, Dec. 31. Anthrapyridone- and anthrapyrimidone sulfonic acids are prepared by reacting with a neutral sulfite upon a compd. of the probable formula



where Y means halogen, such as Cl or Br, X stands for

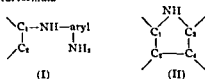


nitrogen, the group  $\text{—CH=}$ ,  $\text{—C=CN}$ ,  $\text{—C=COOR}$ ,

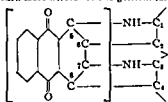
$\text{R}_1 = \text{alkyl}$ , such as Me, Et, Pr, iso-Pr, Bu, etc., R means an unsubstituted or substituted alkyl, Ph, benzyl, or hydroaryl group and wherein, furthermore, the nucleus marked I may be substituted by univalent substituents, such as alkyl groups (Me or Et, for example), halogen atoms (Cl or Br), hydroxy, alkoxy or sulfonic acid groups, etc. Suitable neutral sulfites for performing the reaction are, for example,  $\text{Na}_2\text{SO}_3$ , methylamine sulfite, ethylamine sulfite, etc. Several examples with details of procedure are given.

**Colored reserves.** Durand & Huguenin S. A. Fr 788,336, Oct 8, 1935. Colored reserves are obtained by adding to the reserve, in addition to the quaternary  $\text{NH}_4^+$  compd and the usual ingredients such as thickener,  $\text{ZnO}$ , etc., an azo dye of alk. nature prep'd with a stable diazo compd (e.g., diazoamino compd or nitrosoamine) and  $\beta$ -naphthol or an arylidene of 2,3-dihydroxy-naphthoic acid or acetylacetic acid, the azo dye being developed by an acid treatment. The printed cloth is dried, steamed in a steamer into which a volatile org. acid is caused to pass to develop the colored reserves. Afterward the cloth is steeped in a soln. of an ester of a leuco deriv. of a vat dye and the basic color is developed in the usual manner. Examples are given.

**Carbazole derivatives.** Hermann Hauser and Max Bommer (to Soc. pour l'ind. chim. & Râle). U. S. 2,027,908, Jan 14. Various examples are given of the production of dyes and dye intermediates by treating compds of the general formula



where the carbon pair  $\text{C}_1\text{C}_2$  belongs to a polynuclear radical capable of being vatted in which at least one C atom in *o*-position to the imino group is unsubstituted, and in which the amino group is in *o*-position to the imino group, with diazotizing agents, and heating the azimides thus obtained. The carbon pair  $\text{C}_3\text{C}_4$  may belong for example to an anthranthrene, dibenzopyrenequinone, dibenzanthrone, indigo or thionindigo radical, particularly to an anthraquinone radical whose 1- or 2 position is connected with the imino group, and which is substituted by halogens, alkyl, nitro, alkoxy or by aroylamino groups, such as benzoylamino groups which may be in 4-, 5- or 8-position. The aryl radical may for instance be a benzene, naphthalene or anthracene radical, and these radicals may contain various substituents, such as, for example, halogens, or alkoxy groups, nitro or acylamino groups or alkyl groups. The compds having the above general formula, and which serve as parent materials, may be made by reducing the corresponding nitro compds. Products of the general formula II, where the carbon pair  $\text{C}_1\text{C}_2$  belongs to an anthraquinone radical substituted by a benzoylamino group and the carbon pair  $\text{C}_3\text{C}_4$  belongs to an aryl radical whose carbon atom standing in para position to the imino group is unsubstituted, constitute olive to brown to red to yellow colored powders which dye cotton from the vat olive to brown to red to yellow tints of very good fastness. U. S. 2,027,909 relates to the production of carbazole dyes of the general formula



where the carbon pair  $\text{C}_1\text{C}_2$  belongs to a phenyl radical, the

carbon pair  $\text{C}_3\text{C}_4$  to an anthraquinone radical, and where the carbon pair  $\text{C}_1\text{C}_2$  is linked with the carbon pair  $\text{C}_3\text{C}_4$  and the carbon pair  $\text{C}_3\text{C}_4$  is linked with the carbon pair  $\text{C}_1\text{C}_2$ , which dye cotton from gray to brown to Bordeaux vats brown to Bordeaux to gray to green tints of very good fastness. Numerous examples with details of procedure are given.

**Tetrahydro-3-hydroxyquinoline derivatives.** I. G. Farbenind. A.-G. (Hans Lange, inventor). Ger. 619,825, Oct 7, 1935 (Cl. 12p. 110). Alkylated and arylated amines of the  $\text{C}_{10}\text{H}_9$  series are treated with epichlorohydrin at temps. above  $130^\circ$  with or without pressure and in the presence or absence of solvents. The free bases are obtained from the reaction products by usual means. Thus, butylaniline is heated with epichlorohydrin and the product heated with NaOH to give 1-butyl-3-hydroxytetrahydroquinoline, b.  $177-9^\circ$ . The prepn of 1-ethyl-3-hydroxy-7-methyltetrahydroquinoline, b.  $171-2^\circ$ , 1-benzyl-3-hydroxytetrahydroquinoline, b.  $227^\circ$ , 1-butyl-3-hydroxy-8-methyltetrahydroquinoline, b.  $175-6^\circ$  and 1-methyl-3-hydroxytetrahydroquinoline, b.  $165-6^\circ$ , is also described. The derivs. are used for making dyes.

**N-(Nitrophenyl)- $\alpha$ -tetrahydroanthrylamines.** Henry C. Olpin (to Celanese Corp. of America). U. S. 2,026,744, Jan 7. Compds. such as  $\alpha$ -tetrahydro- $\alpha$ - and  $\beta$ -naphthylamines and their monoalkyl and monoacetyl nuclear substitution derivs are condensed with a nitrated compd of the benzene series contg. at least one labile H atom, such as 2,4-dinitrochlorobenzene (suitably by refluxing in the presence of NaOAc) to form products which are suitable for dyeing cellulose acetate.

**Amino halo anthraquinonesulfonic acids.** Fritz Baumann (to General Aniline Works). U. S. 2,025,370, Dec 24. 1-Aminoanthraquinonesulfonic acids having halogen in the 2 position, H or halogen in the 3 position and H or  $\text{H}_2\text{N}$  in the 4-position are produced by heating initial materials such as 1,4-diamino-2,3-dichloroanthraquinone and like compds with fuming sulfuric acid of 12-30% strength at temps. of about  $110-160^\circ$ . Several examples with details of procedure are given, and the products dye wool from an acid bath blue-violet shades of good fastness.

**Benzanthronethiazoles (dyes and dye intermediates).** Alexander J. Wuerz and Myron S. Whelen (to E. I. du Pont de Nemours & Co.). U. S. 2,024,114, Jan 14. Compds. such as  $\alpha\beta$ -benzanthronethiazole and its methyl and phenyl substitution derivs are obtained from an anthraquinone-1,2-thiazole by reduction (suitably with Fe, glycerol and  $\text{H}_2\text{SO}_4$ ) and reaction with glycerol to form a benzanthronone compd. Numerous examples with details of procedure are given.

**Azabenanthrone derivatives.** I. G. Farbenind. A. G. (Max A. Kunz, Gerd Koehnle, and Karl Koller, inventors). Ger. 622,461, Nov 28, 1935 (Cl. 22b. 3.09). Products useful as dyes or intermediates are obtained by introducing substituents into an azabenanthrone (I) by standard processes. The substituents may then be converted into other substituents in known manner. Numerous methods of procedure are indicated, and examples are given of the prepn of *inter alia*, a bromo-8-I, m.  $214-5^\circ$ , a dibromo-8-I, m. above  $300^\circ$ , a nitro-8-I, m.  $245-6^\circ$ , an amino-8-I, m.  $267-8^\circ$ , a hydroxy-8-I, and its Me ether, a sulfo-8-I, m. above  $300^\circ$ , a cyano-8-I, m.  $203-7^\circ$  and  $\beta$ -1- $\alpha$ -anthraquinonylamino-8-I.

**Dye intermediates.** Joseph F. Turski. U. S. 2,022,579, Nov 26. Amino compds of the hydroxynaphth-anilide type having the general formula  $2,3\text{-C}_{10}\text{H}_6(\text{OH})(\text{CONHR}^1\text{NH}_2)$ , where  $\text{R}^1$  is a group of the benzene series selected from the class having the following formula  $[(p)-\text{C}_6\text{H}_4\text{R}^2]_n$ , where  $\text{R}^2$  represents H, an alkyl or alkoxy group and  $n$  is 1 or 2, are obtained by the reaction of an amino nitro, amino hydroxy or diamino aromatic compd with carboxylic acids of aromatic compds and treating the condensation product to form the dye intermediates which can be converted into azo dyes by diazotization and self-coupling and are suitable for use in dyeing animal, vegetable and artificial fibers. Numerous examples with details and modifications of procedure are given, for production and use of dyes of different colors.



(C. A. 23, 2197). Textiles dyed with azoic colors are stripped by a modification of the process of 400,239 by including in the stripping bath a small proportion, e. g., 0.05%, of anthraquinone or anthraquinone carrying as substituents OH, halogen, amino and (or) alkyl groups. Among examples, cotton yarn dyed with 4-chloro-2-aminotoluene — (on the fiber) diacetoacetic toluidine is treated 30 min at 85–90° in 40 times its wt. of an aq. soln. contg. NaOH, Na hyposulfite and trimethylcetylaminium bromide and having dispersed therein powd. anthraquinone.

**Printing textiles.** Imperial Chemical Industries Ltd. and Allec Wormald. Brit. 435,111, Sept. 16, 1935; Fr. 746,445, Sept. 3, 1935. White resist effects are obtained in dyeing cellulosic material with such sulfuric esters of leuco vat dyes as have affinity for cotton by applying locally to the material at least 1 of the salts, K alum,  $\text{Al}(\text{SO}_3)_3$ ,  $\text{Al}(\text{CNS})_3$ , Cr alum,  $\text{FeNH}_4$  alum,  $\text{K}_2\text{Fe}(\text{CN})_6$ ,  $\text{SnCl}_4$  and stannic oxalate, drying, overprinting or padding with the sulfuric ester of the leuco vat dye and developing the color. Among examples, (1) cotton is printed with an aq. paste contg. British gum and  $\text{SnCl}_4$ , dried, overprinted with an aq. paste contg. Soledon brilliant purple 2R, diethylene glycol, neutral starch tragacanth thickening,  $\text{NH}_4\text{CNS}$ ,  $\text{NaClO}_2$  and  $(\text{NH}_4)_2\text{VO}_4$ , dried, steamed 5–10 min, washed and soaped at the boil.

**Printing textiles.** Durand & Huguennin A.-G. Brit. 435,701, Sept. 20, 1935. Vegetable or animal fibers or fibers of regenerated cellulose, or mixed fabrics thereof, are printed with Cr mordant dyes, other than alizarin dyes, with incorporation in the printing paste of lactic acid, or a salt or ester thereof, and development of the color by steaming. Among examples, a viscose or viscose-cotton fabric is printed with an aq. paste contg. Mordene violet, AcOH, gum arabic thickening and Cr lactate, dried, steamed for 45 min, washed and, if necessary, soaped.

**Printing textiles.** Walter Hugenlocher, Jacob Bänziger, E. Bänziger and Victor Zollikofer. Ger. 622,227, Nov. 23, 1935 (Cl. 8m 1.01). Use is made of anhyd printing pastes contg. all the reagents necessary to fix the dye on the fiber, as well as an oily or fatty thickening agent and glycerol or like compd., e. g., glycol, the purpose of which is to facilitate the penetration of the dye. Sp. compns. are described. The thickening agent may be removed after the printing, e. g., by extr. with a solvent. For reserving parts of the textile material, use is made of compns. contg. Canada balsam or like resin, one suitable compn. being specified.

**Printing textiles.** Soc. pour l'ind. chim. à Bâle. Ger. 622,355, Nov. 26, 1935 (Cl. 8m 1.02). See Swiss 177,231 (C. A. 30, 3019).

**Apparatus for printing materials such as pile fabrics or floor coverings by impregnation with dye compositions, etc.** Melvin R. Greiser (to Carthage Mills Inc.). U. S. 2,027,820, Jan. 14. App. is described for applying liquid mordant, printing and steaming, in timed sequence.

**Dye baths and printing colors for naphthol dyeing.** Heinrich Bertsch (to H. Th. Böhm, A.-G.). U. S. 2,026,817, Jan. 7. A naphthol dye component is mixed with a dispersing agent comprising an aliphatic higher-mol. alc. ester of an inorg. O-contg. acid such as the octadecanoic ester of  $\text{H}_2\text{SO}_4$  or  $\text{H}_3\text{PO}_4$  (the ester used contg. at least one free OH group).

**Dyeing apparatus with circulating liquid.** Clermont-Bonte & Fils (S. & R. I.). Fr. 788,712, Oct. 15, 1935.

**Supports for use in dyeing hank yarns.** Ernst Littke. Ger. 622,354, Nov. 26, 1935 (Cl. 8a 36.01).

**Treating textiles.** Fritz Drechsel. Brit. 435,642, Sept. 25, 1935. See Fr. 777,097 (C. A. 29, 38560).

**Treating textiles.** Soc. pour l'ind. chim. à Bâle. Brit. 435,871, Oct. 1, 1935. Acid-free solns. of  $\text{H}_2\text{O}$ -sol urea- $\text{CH}_2\text{O}$  reaction products of noncolloidal character and consisting substantially of methylol compds. are mixed with a nonvolatile, neutral and easily  $\text{H}_2\text{O}$ -sol substance of noncolloidal character to yield stable solns. which may be used for treating textiles. The solns. may be dried or the ingredients may be mixed dry to yield an easily sol. powder. Among examples, (1) a soln. of dimethylolurea

(I) produced by condensing urea and  $\text{CH}_2\text{O}$  in the presence of active C is mixed with urea to yield a stable soln. that may be dried, (2) a soln. of I is dried, the product ground with a soln. of dimethylolthiourea (II),  $\text{NaCl}$  and  $\text{Na}_2\text{SO}_4$  and the product dried, and (3) I is ground with a soln. of II and the product, after drying and grinding, dissolved in  $\text{H}_2\text{O}$  contg. cane sugar to give a stable soln. for use for producing antireflecting effects on viscose.

**Treating textiles.** I. G. Farbennud A.-G. Fr. 758,128, Oct. 4, 1935. Threads and textiles made from artificial and vegetable fibrous material are made uncrushable by impregnating them with compds., e. g., urea or derivs. thereof, which are condensable with  $\text{CH}_2\text{O}$  to form a resin, and treating them after drying, in a closed vessel, with vapors of  $\text{CH}_2\text{O}$ , if necessary in the presence of a solvent for  $\text{CH}_2\text{O}$ , at a temp. above 100°. In a modification a sol. compd. sepg.  $\text{CH}_2\text{O}$  on heating is added at the same time as the urea, and the whole is then heated without further addn. of  $\text{CH}_2\text{O}$ . Cf. C. A. 29, 8358\*74.

**Treating textiles, paper, etc.** Lilly Pollak (see Hissberger). Fr. 787,950, Oct. 1, 1935. Impregnation by hardenable synthetic resins, e. g., to render cloth uncrushable, is improved by adding hydrotrope wetting agents, such as those known as "Leonol S," "Nekal" and "Tekal" acid, to the solns. of resins used.

**Finishing textiles.** La Cellulose (Soc. anon.) Fr. 788,020, Oct. 2, 1935. A permanent finish resistant to washing and wrinkling is obtained on textiles by impregnating the fiber with an aq. or aq.-alc. soln. contg. a tannin and an org. compd. capable of combining with tannin (if necessary in the presence of acids or substances yielding an acid) to give, on simple drying or heating, an elastic water-insol. compd. The org. compd. may be  $\text{CH}_2\text{O}$ , an amine or a polyvinyl alc.

**Surfaces of textiles.** Robert Mermoz. Fr. 787,902, Oct. 1, 1935. Brilliant or glazed effects are obtained on textiles by stretching the textile on a polished surface and applying one or more printing pastes which traverse the textile and form a glaze in contact with the polished surface. Examples of pastes contain gum lac, alc., olive oil with or without  $\text{TiO}_2$ , Au powder or a dye. The surface may be only partly polished to give mixed effects and a thin layer of paraffin may be applied to it to facilitate removal of the textile therefrom.

**Steeping bast fiber plants.** Ivan A. Makrnov. Ger. 619,770, Oct. 7, 1935 (Cl. 29b. 2). The plants are steeped in a liquor contg. anaerobic bacteria which destroy the stalks to form mainly  $\text{CO}_2$  and  $\text{H}_2$ . The liquor contains the pectinobacteria *amylphilum* as the anaerobic bacteria.

**Oil composition for treating textile fibers such as wool.** Augustus H. Gull (to Gull Corp.). U. S. 2,026,735, Jan. 7. A fatty acid oil such as olive oil or cottonseed oil associated with linoleic acid is hydrogenated to a stage where substantially only the linoleic acid has been largely transformed into oleic acid, thus substantially preserving the fluidity of the oil for fiber treatment while largely eliminating a constituent that tends to form gum on the fiber.

**Artificial wool.** I. G. Farbennud A.-G. Brit. 435,968, Oct. 2, 1935. Addn. to 424,229 (C. A. 29, 4603\*). The process for the manu. of wool-like fibers described in 424,229 is applied to spun material obtained by using a viscose having a ripeness corresponding with a salt point of 0.1–0.2 or lower. In an example, viscose is ripened for 3 days at 20° and, having attained a salt point of 0.1, is spun, treated in thread or skein form to remove acid and S and washed. Excess  $\text{H}_2\text{O}$  is removed by squeezing or centrifuging and the material is treated in countercurrent with aq. MeOH of 35–92% strength. Adhering alc. is removed by pressing or centrifuging.

**Wooly threads of rayon.** Wolfgang Gruber (to A. Wacker Ges. für elektrochemische Industrie G. m. b. H.). U. S. 2,026,736, Jan. 7. See Ger. 594,378 (C. A. 25, 3598\*).

**Artificial filaments, films, etc.** British Celanese Ltd., Henry Dreyfus, Robert W. Moncreff and Frank B. Hill. Brit. 435,806, Sept. 27, 1935. Threads, fabrics, ribbons, etc., having a basis of a cellulose ester, are treated at greater than atm. pressure with a sapon. medium con-

prising a non-metallic nitrogenous base, e. g., an aq. alc. or other soln. of  $\text{NH}_3$ ,  $\text{MeNH}_2\text{OH}$ ,  $\text{MeNH}_2$ ,  $\text{Et}_3\text{N}$ , allylamine, piperidine, diammoniohydroxypropane, ethylenediamine. In 435,947, Sept. 27, 1935, divided on 435,800, the treatment at superatm. pressure is with a medium contg. a mineral spongy agent, e. g., an aq. or alc. soln. of caustic alkali,  $\text{NaOH}$  contg.  $\text{AcONa}$ ,  $\text{NaOH}$  contg. gelatin, egg albumin, casein, leucine, aspartic acid, proline, potato starch, dextrin, etc.;  $\text{Ba(OH)}_2$ ,  $\text{Na}_2\text{HPO}_4$ ,  $\text{Na}_2\text{SiO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{O}$ .

**Conditioning artificial threads** Hugo Hofmann (to American Remberg Corp.) U. S. 2,027,911, Jan. 14, 1935. Threads such as those formed from cuprammonium cellulose soln. are subjected to a fixing soln., stretching and shrinking, and then treated with a fixing soln. App. is described.

**Acetate rayon** Karl Beck Ger. 619,978, Oct. 10, 1935 (Cl. 8k 1). The rayon is improved by treatment with an aq. soln. of  $\text{CH}_3\text{CO}$  and  $\text{MeOH}$  or other substances forming acetal-like substances with  $\text{CH}_3\text{CO}$ , e. g.,  $\text{BuOH}$ . The treatment gives the rayon a mat silky gloss and a soft touch.

**Ornamenting cloth containing cellulose esters or ethers** Rhodiacta Ir. 788,592, Oct. 12, 1935. Designs are obtained by local destruction of the cellulose esters or ethers by treatment with org. peroxides, e. g., benzoyl peroxide, at a high temp.

**Washing dyed textiles** I. G. Farbenind. A.-G. Fr. 786,390, Sept. 2, 1935. Quaternary  $\text{NH}_4$  salts obtained by alkylating the products formed from ethylenediamine polymers, either by elimination (by distn.) of the parts of low b. p., or by condensation with substances contg. at least 2 acylating groups, are added to the washing bath.

**Washing and other processing of artificial threads** I. G. Farbenindustrie A.-G. Brit. 436,003, Oct. 3, 1935. Self-supporting hollow windings or packs of the thread are washed and treated by sucking  $\text{H}_2\text{O}$  or a treating liquid through a no. of packs threaded in superimposed relationship on a vertical perforated tube (a) that lies closely against the inner surface of the packs and is open at its upper end, a closure being maintained at the upper end of the column by means of an impermeable tube that is closed at its upper end and fits closely over a and is held with its lower end pressed against the topmost pack owing to the reduced pressure within a, so that it follows the contraction of the column. After treatment, the packs may be dried while threaded on a.

**Bleaching and washing preparations** Henkel & Cie. G. m. b. H. Brit. 436,235, Oct. 8, 1935. Preps. comprising a per compd. together with a salt of a phosphoric acid contg. less  $\text{H}_2\text{O}$  than  $\text{H}_3\text{PO}_4$  are stabilized in an alk. medium by the addn. of  $\text{Al(OH)}_3$  or of Al salts that yield  $\text{Al(OH)}_3$  under prevailing extl. conditions. Among examples, soap,  $\text{Na}_3\text{PO}_4$ ,  $\text{NaAlO}_2$ ,  $\text{Na}_2\text{CO}_3$  and  $\text{Al(OH)}_3$  are mixed in an edge-runner mill and used for washing textiles.

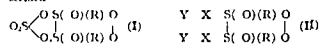
**Starch for finishing linen.** Chemische Fabrik Joh. A. Benckiser G. m. b. H. Fr. 788,602, Oct. 14, 1935.

Alkali metaphosphates, e. g., Graham salt, are added to the starch, whereby a more regular brilliance is obtained.

**Apparatus for the wet treatment of stockings and like textile goods on cores of appropriate shape.** Fritz Schuster Ger. 622,028, Nov. 10, 1935 and 622,029, Nov. 18, 1935 (Cl. 8b 9 04).

**Felt hats** Julien van Puymbroek Brit. 435,076, Oct. 2, 1935. Hat contg. nonsecreted fibers, e. g., wool, and secreted fibers, e. g., rabbit's hair, are made by oiling the material with a product contg. mucilaginous substance, forming a lap from the material in a carding machine, winding the lap upon a forming cone and filling in an acid bath.

**Organic sulfur compounds** Charles A. Pfeifferberger Ir. 784,285, Oct. 7, 1935. Comps., of the formula I which are used to protect cloth from moths, etc., and for the manuf. and application of dyes, are made by causing dichlorobenzene to react with a sulfonating agent contg.  $\text{SO}_2$ , preferably at below  $100^\circ$ . The products may be submitted to a further condensation with amines, imines, amides, alcs., phenols, mercaptans, carboxylic, sulfonic, sulfine and sulfamic acids, giving products of the formula II, in which X is  $\text{NH}$ ,  $\text{SO}$  or  $\text{O}$  and Y is a hydrocarbon radical, substituted or not. The prep. of bis(2,5-dichlorobenzene-4-sulfonic acid anhydride) sulfone and of condensation products of this with  $\text{PhNH}_2$ ,  $\text{BuOH}$ , benzene-sulfonic acid, 1,8-ammonaphthol, 3,6-disulfonic acid, arsenous acid, derivs. of P, Sb, N, Bi and CN,  $\beta$ -chloroaniline and 1,2-dichlorobenzene-4-sulfonic acid is described.



**Apparatus for resizing and drying garments after dry cleaning** Walter Alcorn and Lloyd Hamiter U. S. 2,027,491, Jan. 14. Various structural and operative details.

**Dry cleaning** Warren T. Reddish Brit. 435,576, Sept. 21, 1935. Fabrics, etc., are cleaned by treating with a solvent, withdrawing the solvent, adding  $\text{H}_2\text{O}$  thereto and then returning the mixt. to the washing machine. Homogenizing and other app. are described.

**Dry cleaning, etc.** Compagnie de produits chimiques et électrometallurgiques Alais, Iroges & Camargue. Ir. 788,415, Oct. 10, 1935. Tetrachloroethylene is used in place of trichloroethylene in all chem. or phys. operations where the medium is alk. or may become alk. in one of the steps. This is particularly so in dry cleaning where  $\text{NaOH}$  is used to regenerate the cleaning agent. The formation of spontaneously inflammable compds. is thereby avoided.

**Composite fabric suitable for foot coverings** Charles F. McLean (to Patchogue-Plymouth Mills Corp.) U. S. 2,026,693, Jan. 7. Various structural details of a fabric comprising stuffing warp threads made of paper and holding warp threads of twisted textile fibers such as cotton or wool and filling threads such as a yarn of mixed fibers.

## 26—PAINTS, VARNISHES AND NATURAL RESINS

A. H. SAHN AND CARLTON H. ROSE

**Health hazards among painters with reference to industrial lead poisoning.** L. Holstein. Med. Welt 9, 183-5, 221-6, 302-4 (1935). U. S. Pub. Health Eng. Abstracts 15, 1185, 7 (Sept. 28, 1935).—A valuable paper on lead poisoning and dangers arising from use of solvents and lacquers.

**Are there paints which have a deadening effect on sound?** C. R. Fellers. C. Hornemann. Farbe u. Lack 1935, 557.—Paints which produce a rough uneven surface sometimes lead in deadening sounds and improving acoustics.

**Paints—their disinfectant properties—steps in the production of a germ-proof film.** Samuel Wetchler, Anna Lewis and Frederick Battline. Paint & Varnish Production Mfr. 14, 12, 14, 16, 18 (Jan. 1, 1936).—Exptl. data is given for the development of a permanently bactericidal

paint. A standardized bacteriol. technic using typhoid bacillus and *Staphylococcus aureus* has been developed, which has led to the following conclusions: (1) The nature of pigments, oils and natural and synthetic resin varnishes has no bearing on the germicidal activity; (2) ordinary com. types of paints are only temporarily germicidal; (3) the addn. of the common disinfectants does not render a paint permanently germicidal; (4) the use of a halogenated oil base products a nontoxic paint which will kill bacteria applied to the dried paint film, even after 3-4 yrs. of drying; (5) the germicidal properties are maintained against the washing of the film with soap and water. Ten references.

**Naphthenate driers** M. D. Curwen. Oil Colour Trade J. 88, 1711-14 (1935).—A discussion of naph-

W. H. Roynon

thetic acids, the manuf. of naphthenic driers and the qualities of a good drier. General properties of the naphthenates are discussed. W. H. Boynton

Rust protection. A. V. Blom. *Korrosion u. Metallschutz* 11, 245-9 (1935).—Some of the phys. phenomena affecting the life of paint films are discussed. L. P.

The behavior of pigment suspensions in the electric high-tension field. Erich A. Becker. *Angew. Chem.* 48, 691-6 (1935).—Nine pigments and 6 binders were investigated in an app. which permitted a max. potential difference of 20,000 v., amounting to 6700 v. per cm. The exptl. app., procedure and results are described in detail. The explanation of sediment formation in oil paints, lacquers and enamels, by pptn. due to oppositely charged pigment particles and colloidal particles of the binder could not be substantiated. Three of the lacquer binders did not show any electrically charged particles, while in two lacquers both the disperse phase of the binder and the pigment particles had a positive charge. The proper sedimentation problem is det'd. by three factors. (1) gravitation, which in the first approximation can be expressed by Stokes' law, (2) the elec. charge, which acts as a deterrent factor, (the smaller the pigment particles the greater the effect of the charge and the smaller that of the mass; the pigments are polydisperse throughout), (3) adsorption; it is possible that the pigment particles do not float freely, but are surrounded by adsorptive layers which det. if the particles clng together on collision or sep. freely, and in case of adsorption of reousous substances by the pigment particles a cumulative mass effect and increased sedimentation takes place. K. K.

Hempseed oil. Harry Friedman. *Am. Paint J.* 20, 48, 50 (Jan 13, 1936).—Raw hempseed oil makes an excellent vehicle for grinding colors in oil. Its color is not a serious defect, since its staining power is weak. Its acid no. is neither very high nor very low, and it has the property, unique among the thorough-drying oils, of dispersing hard-grinding pigments and keeping them in smooth paste form over long periods of time. It has a marked absence of after-ellowing tendencies.

Experiences with chlorinated rubber paints. G. Schulze. *Akutschuk* 11, 231-2 (1935).—A discussion, dealing with the various essentials of technic for obtaining successful results. C. C. Davis

Polymerization of hydrocarbons of synthetic rubber manufacture into a paint vehicle, on the plant scale. L. V. Lopatin and B. Ya. Soldatov. *Sintet. Kaschuk* 1935, No. 6, 22-9.—The hydrocarbons of different fractions (25-45°, 45-60° and 60-100°), obtained during the process of synthetic rubber manuf., were washed with water to remove the water-sol. substances and then polymerized in an autoclave (the charge was up to 850 kg.) in the presence of 20% of catalyst (Glukhovskaya clay, that had been heated at 420°) for 30 hrs. at 100°. The products were dist'd. *in vacuo* to remove unchanged gases and bring the viscosity to 79-87% of that of glycerol det'd. in the Ostwald viscometer. Paint prep'd. from 100 parts of the product per 100 parts of ZnO, dried after 12-22 hrs. and adhered well to the surface. The paint is waterproof. A. Pestoff

Flax studies. W. F. Geddes. Can. Dept. Trade & Commerce, Dominion Grain Research Lab., *6th Ann. Rept.* 1934, 31-4.—Practically the same values for the I nos. of the Et<sub>2</sub>O ext. of finely ground flaxseed meal were obtained by drying the meal in (1) a Freas or DeKhotinsky vacuum oven for 15 hrs. at 100°, (2) a Freas air oven for 10 hrs. at 100° in a current of CO<sub>2</sub>, and (3) an Abderhalden app. (with P<sub>2</sub>O<sub>5</sub>) at the temp. of boiling water. There was no appreciable difference between the I nos. of the Et<sub>2</sub>O exts. when the exts. were either (1) dried to const. wt. at 100° in either a Freas or a DeKhotinsky vacuum oven, (2) dist'd. and dried at 100° in a current of inert gas or (3) dist'd. in CO<sub>2</sub> and dried in a DeKhotinsky vacuum oven at 100° for 3 hrs. A study of the refractometric method for detg. the oil content of flaxseed with a mixt. of bromonaphthalene and chloronaphthalene indicated a high correlation between the  $\eta$  and the oil content as det'd.

by the Et<sub>2</sub>O-extn. method. Satisfactory extn. of flaxseed pigments was obtained by using 4 g. very finely divided flaxseed (ground in a high-speed burr mill), adding 100 cc. solvent (a mixt. of varnish makers' naphtha 93 and EtOH 7% by vol.) and shaking for 2 hrs. in a mech. shaker. The novelty variety of flaxseed was definitely higher in pigment than the Bison, Linota and Premost varieties. The concn. of pigment was considerably reduced by aging or storing. The carotene content amounted to about 22-30% of the total carotenoids. The northern areas of Canada yielded flax of somewhat higher oil content and I value than did the central and southern districts. K. D. Jacob

The influence of the structure of alcohol radicals on the film-forming properties of the esters of the acids of linseed oil. A. Ya. Drinberg and A. A. Blagomiravova. *J. Gen. Chem.* (U. S. S. R.) 5, 1229-32 (1935).—Thurman and Crandall (C. A. 23, 5335) and other investigators showed that Me and Et esters of unsat'd. high-mol. acids (linoleic and linoleic acids) do not give films. Only the esters of trihyd. acids form hard films. Mixed linseed-oil acids were esterified at various temps., with and without the aid of HCl, with iso-Am. alc., a mixt. of ceryl and myristyl alc. (obtained from beeswax), ethylene glycol, propylene glycol, glycerol, pentaerythritol and mannitol. The tabulated results show that with the increasing no. of OH groups in an alc. the film-forming properties of the ester of unsat'd. acid improve, while the rate of esterification rapidly declines. The film-forming ability of the esters of dihydric acids is directly related to the position of the 2 OH groups in the alc. The esters of mono- and dihydric acids (the OH groups in the position 1,3) do not tend to autopolymerize and, therefore, do not form films. Esters entirely incapable of forming hard films show the min. value of O nos. The work is being continued. About 20 references. Chas. Blanc

Polymerization: some applications in the organic industry. H. I. Waterman and C. van Vlodrop. *Chimie & Industrie* 34, 1036-44 (1935); cf. C. A. 29, 64437.—The polymerization of linseed oil was studied by applying the diagram of the sp. refraction ( $n_D^{20} - 1$ )/( $n_D^{20} + 2$ ) as a function of unsatn. The graphic theoretical representations based on the const's of Auwers and Eisenlohr were corrected for deviations in the values of the sp. refraction due to unsat'd. glycerides. Two sets of expts. were carried out on a semimicro scale and the results were interpreted by means of the above-mentioned diagram. Supposing that, in the polymerization all the ethylene linkings have been sat'd. and the nature of the O linkings remained unchanged, a theoretical line can be drawn representing the polymerization of linseed oil. As a matter of fact at the beginning of the reaction the sp. refraction decreases relatively slowly, but subsequently the decrease is more rapid so that toward the end the curve reaches the theoretical line. The relatively high specific refraction obtained at the beginning is in agreement with the results of van der Hulst who, by means of the absorption spectrum, showed that at the beginning the polymerization product contains conjugated double bonds. Formation of a conjugated system by isomerization is related to an increase in the sp. refraction. Application of distn. in cathodic vacuum with internal condensation showed that polymerized linseed oils frequently have a higher I no. than the corresponding distillates and residues; this is explained by the presence in the polymerized oil of volatile unsat'd. decompn. products. If polymerization is pushed further, the I nos. of the distillate and residue become approx. equal; simultaneously the percentage of high-mol.-wt. oil increases, or in other words, continuously increasing polymerization is not the only phenomenon involved in the reaction. A. Papineau-Couture

Softening of linseed-oil films. Carlton H. Rose and Don S. Bolley. *Ind. Eng. Chem.* 28, 115-18 (1936).—Unpigmented films of linseed oil, with and without drier, dried and hardened when exposed under const. temp. and humidity to ultraviolet light, then softened again on continued exposure. The percentage of the film mol. in MeAc reached a max. at about the point of max.

hardness and decreased as the film softened again. The less said linseed oils gave dried films which softened less and were less sol in MeAc. A method is given for the detn. of dryness semi-quantitatively on a numerical scale. Drier (Pb, Mn) delayed and reduced the amt. of after-softening. When pigmented with white lead, the films became progressively harder and less sol (no after-softening). The I no., which was about the same for the sol and insol portions of the film, decreased during exposure in all cases and was not related to the softening or soly of the film.

Arnold M. Collins  
Nitrocellulose-ester lacquers and combination lacquers, their production and development. Fritz Zimmer. *Chem.-Ztg.* 59, 941 5(1935), cf. C. A. 30, 307. E. H.

Study of the manufacture of nitrocellulose and acetocellulose lacquers. R. Tersand. *Lauchouche & Gutierrez* 32, 17347 8, 17342 3(1935), cf. C. A. 29, 8465. The properties of various diluents are described.

C. C. Davis  
Comparative water-resistance tests on nitrocellulose lacquers. Bodenbender. *Nitrocellulose* 6, 85 6(1935). L. M. S.

Special applications of nitrocellulose lacquers. A. Kraus. *Nitrocellulose* 6, 139 41, 159 62(1935)—A review. E. M. Symmes.

Modern varnishes. H. W. Talen. *Chem. Weekblad* 32, 606-11(1935)—An address summarizing present knowledge of oil, nitrocellulose and synthetic varnishes.

O. W. Wilcox  
Tung oil as a raw material for varnishes and synthetic resins. I. Ewald. *Farnrobert. Paint Manuf.* 5, 364 7(1935)—A paper dealing with the cultivation, yield, quality and chem. compn. of tung oil. The quality of a tung-oil stand oil as a varnish ingredient increases proportionally as its tendency to frost decreases. "Curing" of tung oil requires heating the oil to a temp. of 270-280°, and this temp. should be attained as rapidly as possible.

The  $\alpha$ -eleo-stearic acid triglyceride is the essential constituent of tung oil on which its desirable varnish properties depend, as in contrast to the  $\beta$ -glyceride it shows less tendency to crystallize or "throw out". This  $\alpha$ -form changes over readily and rapidly into the  $\beta$ -form on heating or under chem. influence. For varnish prepn. the tung oil should receive a treatment to minimize this action. Rapid heating causes the rate of polymerization to exceed that of transformation. II. *Ibid.* 6, 4-7(1936).—An outline is given of the prepn. of tung-oil stand oils, of mixed tung-linseed stand oils, and the Am. method of heating the raw materials required for the varnish in one operation. The Albertol tung-oil varnishes prepnd. by the European method are paler in color, have less tendency to skin and yield somewhat better-flowing and slightly harder films. The varnishes cooked by the Am. method are more gas proof, show less tendency to wrinkle on stoving and when combined with the harder Albertols are a trifle more alkali-resistant. Alkylphenol and alkyl resins are briefly discussed. The frosting tendency of tung oil is minimized by adding an amt. of 1-2% of the tung oil in solid form, after having dissolved it previously in a varnish solvent.

W. H. Boynton  
Future of natural resins. T. Hedley Barry. *Paint Manuf.* 5, 374-6(1935); cf. C. A. 30, 631.—A review indicating that the use of natural resins is bound to increase.

W. H. Boynton  
Direct liquid extraction process for pure lac resin. Lal C. Verman and R. Bhattacharya. *London Shellac Research Bur. Tech. Paper* No. 5, 30 pp(1935); cf. C. A. 29, 27631.—Of the methods previously developed for the prepn. of "pure lac, resin" (I) from whole lac, that of direct liquid extn. is considered the most suitable for a large-scale production. Extns. with various solvents proved that the partition-coeff. theory is untenable in this case. Trichloroethylene (II) as extn. agent gave promising results except that, because of a slight acid decompn., it polymerized the lac strongly. This was, however, conveniently overcome by the addn. of 0.1% triethylamine (III) as a stabilizer. III is recovered together with II, the b. ps. being 89° and 80-8°, resp.

Extn. equal curves in all cases where lac wax is absent follow a simple power law, i. e., they satisfy the equation  $y = ax^n$ , where  $y$  represents the concn. of "ether-sol. resin" (IV) in the extg. solvent and  $x$  the concn. of IV in the product,  $a$  and  $n$  being consts. characteristic for the particular process. The curves representing the extns. with II follow the same law also in cases where lac wax is present. Fit to extn. curves follow an exponential law, i. e.,  $y = be^{mx}$ , where  $y$  and  $x$  have the same significance as above and  $b$  and  $m$  are characteristic consts. for this process. The utility of the curves in relation to the design and development of efficient extn. processes is demonstrated. Certain secondary factors render extn. calcs. approx., viz. (1) the dispersion of I in the solvent, (2) the retention of solvent by the product and (3) the loss of solvent by evapn. The film-forming qualities of I improve with the progressive improvement in its purity.

Obtained by successive extns. up to about five. App. are described and illustrated for the quant. extn. of lac by solvents lighter and heavier than the extd. material and for the production of 1 lb. batches of I. D. Thuesen  
Air-drying asphaltum finishes. E. Assheton. *Paint Manuf.* 5, 371 3(1935). A brief review of various pitches in common use and a few representative formulas of air-drying asphaltum blacks. The blackness of a sample of asphaltum can be evaluated by the following method. I use 10 g. of the pitch with 40 g. rosin. Cool to 149° and add turpentine till the bulk is 100 cc. Place 10 cc. of this soln. in a 1000-cc. cylinder and make up to 1000 cc. with turpentine. Compare with a standard made in the same way.

W. H. Boynton  
The yellowing of paints. Chas. S. Farmer. *Paint Manuf.* 5, 282-3(1935). A brief consideration of chem. yellowing, the choice of oil media, acid values, after-treatment of stand oils, driers, resins pigments and yellowing of phenolic media. W. H. Boynton

Some notes on wood preservation. J. Stewart Remington. *Paint Manuf.* 6, 8-10(1936).—Forms of rot and decay are briefly discussed, also preservatives such as creosote, cuprinol and synthetic-resin varnishes. Several metallic naphthenates are widely used as driers. Diatomite may be used in priming paints. It carries more oil than barytes. A priming coat that limits fungoid attacks contains 35% leaded ZnO 73 kg., bleached diatomite 9.1 kg., raw linseed oil 18 0 l. and white spirit contg. 3% Cu naphthenate 3-8-7 l. W. H. B.

Complex metal compds. of dyes [product for printing-varnishes] (Brit. pat. 435,108) 25. Azo dyes [for coloring lacquers] (Ir. pat. 788,217) 25. Refining resins (Ir. pat. 788,239) 27. Disazo dyes [for coloring lacquers] (Fr. pat. 788,119) 25. Pigment dyes (Brit. pat. 435,817) 25. Varnishes (Brit. pat. 435,762) 13. Preventing corrosion (Brit. pat. 435,420) 9. Cellulose triacetate solns. [for making varnishes] (Ir. pat. 785,030) 23.

Paints and printing inks. August Chwala. Tr. 788-001, Oct. 2, 1935. Basic or amphoteric pigments in paints or inks, are protected against attack by the oily or resinous binders used and consequential thickening by a protective coating incapable of dissolving or swelling in the binder. The coating may be formed by salt compds. on the surface of the particles or by solns. of org. compds., preferably colloidal. Coatings may be formed with oxalic acid,  $H_2PO_4$ , phosphates, Ca glycerophosphate, tannin,  $Al_2(SO_4)_3$  or aluminum.

Dissolving rubber to form a paint, varnish or insulation, etc. John P. Henharen. U. S. 2,028,008, Jan. 14, 1936. Brit. 432,405 (C. A. 30, 6329).

Hand-operated paint mixing device. Frank Trampusch. U. S. 2,027,297, Jan. 7, 1936.

White pigments. I. G. Farbenund. A.-G. Fr. 787-384, Sept. 21, 1935. At least 0.0 mol. of ZnO with 1 mol. of  $TiO_2$  or the corresponding amt. of a Ti compd., is submitted to calcination at 500-1000° until practically all the  $TiO_2$  is combined with the ZnO as titanate in the form of spinel and (or) corundum. Part of the Zn may be

replaced by Mg, and the pigment obtained is reheated in the presence of ZnO and (or) MgO until the addn oxides are wholly or partly absorbed by the titanate.

**Titanium pigment.** Joseph Blumenfeld and Max Mayer (to Krebs Pigment & Color Corp.). U. S. 2,026,862, Jan. 7. A pigment which has good weather resistance is prep'd by suspending  $TiO_2$  in a soln. of a comp'd of Ce, Th or Zr and treating the suspension to ppt. an oxide from the soln in the presence of the  $TiO_2$ , and calcining the resulting mixt.

**Apparatus for mixing pigments and other powders.** Eugen Fässen. Ger. 622,247, Nov. 23, 1935 (Cl. 50/2 01).

**Printing color.** I. G. Farbenund, A.-G. (Walther Lützendorf, inventor). Ger. 619,501, Oct. 2, 1935 (Cl. 15/7 01). See Fr. 774,851 (C. A. 29, 2265). The manuf. of bronze printing ink for use with rubber type is rendered possible by using highly viscous polymerization products of aliphatic vinyl compds, sol in alc, as the binding agent for the bronze powder. Thus, the ink may contain powd Cu bronze, polyvinyl methyl ether, EtOH and the acetate of glycol ethyl ether.

**Printing ink.** P. Chouanard & Fils & Co (Soc. veuve). Fr. 788,400, Oct. 10, 1935. A white or clear-tone ink for printing on colored paper is composed of  $TiO_2$  dispersed in a soln. of casein, e. g.  $TiO_2$  100 is added to a soln. of casein 100 in water 1000 contg.  $NH_3$  13 g.

**Inks.** Encrea Antoine. Fr. 788,383, Oct. 9, 1935. The d. of inks is decreased by incorporating therein 3-10% of aliphatic compds of d. below 1 and sol in water, e. g. acetone, MeOH, AcH, etc.

**Sulfonated drying oils.** Chem. Fab. Stockhausen & Cie. Ger. 622,202, Nov. 25, 1935 (Cl. 12/23 02). Linseed oil and like drying oils are converted into sulfonic esters by treatment with  $H_2SO_4$  of a concn. not above 90%

at a temp. not above 30°. Sp. processes are described. The products resemble Turkey-red oil.

**Synthetic varnish bases.** Adolf Heck (to Cook Paint & Varnish Co.). U. S. 2,027,338, Jan. 7. A condensation product of a phenol 2 mols. with an anhydride of an aromatic dicarboxylic acid 1 mol., formed in the presence of a chloride of Sn, Al, Fe or Zn, is fused with a fatty acid such as that of linseed oil and sufficient glycerol is added to the molten mixt., to produce esterification, with heating to at least 200° until esterification and resinification are effected, so that a resinified material is obtained on cooling, suitable for use in varnishes or plastic compns. U. S. 2,027,339 relates to the manuf. of varnishes by condensing a phenol 2 mols. with 1 mol. of a "polybasic" aromatic carboxylic acid such as phthalic anhydride in the presence of a chloride of Al, Fe, Sn or Zn, heating the condensation product to partially resinify it, fusing it with about 4 times its wt. of rosin and heating with a polyhydric alc. such as glycerol to effect esterification and further resinification, so that on cooling a product is obtained which is sol in coal-tar hydrocarbons, gasoline and drying oils.

**Drying varnished surfaces** under the action of ultra-violet radiation. John J. Brophy (to Turner Tanning Machinery Co.). U. S. 2,020,296, Nov. 12. Various details of app. and operation are described suitable for treating varnished leather, etc.

**Rosin sizing.** Wm. H. Harding and Albert W. Montgomery (to American Cyanamid & Chemical Corp.) U. S. 2,027,166, Jan. 7. For making a low free rosin size contg. over 50% of solids, a soln. of an alkali metal compd such as  $Na_2CO_3$  and rosin mixt. is subjected to a drying heat above 93° but not above 260°, relatively suddenly, so that the mixt. is carried rapidly through and beyond gelling and foaming stages and saponification is accelerated. App. is described.

## 27—FATS, FATTY OILS, WAXES AND SOAPS

E. SCHERUBEL

**The titer of fat mixtures.** C. Bergell *Seifen- und Ztg.* 62, 839-40 (1935).—Tabulated data on the titer of mixts of varying proportions of either (1) rosin, (2) coconut-oil fat acids or (3) soybean-oil fat acids with either (a) tallow fat acids or (b) palm-oil fat acids show that the titer of such mixts cannot be predicted from their compn and the titer of their components.

**Notes on the coconut-fat treatment in the confection industry.** Holscher. *Allgem. Oel- u. Fett-Ztg.* 32, 496-7 (1935).—Notes on treatment and storage to prevent spoilage.

**Jaboty fat.** Alph. Steger and J. Van Loon *Chemistry and Industry* 1935, 1095-7.—There are 2 species of trees whose fruits yield Jaboty fat, *Erisma uncinatum* and *E. calcaratum*. The extd. fat of the former had the following constns: d<sub>4</sub><sup>20</sup> 0.8760,  $n_D^{20}$  1.4360, I no. (Wiss) 5.4, sapon no. 233.0, acid no. 20.0, Ac no. 10, R.-M. no. 0.93, Polenske no. 3.1, m. p. 41.5°, Hehner no. 93.0, unsaponifiable 0.7%. The constns of the fat acids were  $n_D^{20}$  1.4260, I no. 4.9, neutralization no. 246, mean mol. wt. 228, m. p. 41°. All of the constns practically agree with those of the com. fat. The compn. of the fat acids showed 78.1% palmitic and 21.7% oleic acid. The fat could be used for edible purposes after refining. E. S.

**Regenerating fatty substances from waste waters of the wool industry.** V. N. Sokolov *Sherstyanos Delo* 1935, No. 4-5, 24-6.—A description of a simplified  $H_2SO_4$  method of regenerating fats from waste waters and of soap-making by boiling.

**Fatty acids and soaps in 1935.** T. P. Hilditch *Chem. Age* 33, 599-600 (1935).

**Standardization of testing methods.** Anon. *Seifen- und Ztg.* 62, 751 (1935).—A proposed standard method of the Deutscher Verband für die Materialprüfungen der Technik for detg. the pour point of oils is described in detail.

**Modified apparatus for determining the hexabromide number of oils and fats in tropical countries.** P. S. Varma, N. N. Godbole and G. M. Garde, *Allgem. Oel- u. Fett-Ztg.* 32, 456-7 (1935).—The filtering app. for hexabromide ppts. proposed for use where room temps. are high consists of a regular suction filtering app. that has an attachment contg. a cooling mixt. by means of which the ppt. and Gooch crucible can be maintained at low temps.

**The Procter and Gamble oil colorimeter.** H. B. Stevenson *Oil and Soap* 13, No. 1, 18-20 (1936).—The colorimeter is a modification of the Bailey instrument and consists of a light box and a color glass magazine. The box is made of cast Al and one side is formed by a sliding panel which completely exposes the interior. The color magazine is also made of Al and is bolted on the end of the light box. It consists of 4 Al disks or plates which are operated by means of hand wheels attached to concentric hollow shafts extending through the top of the case. The disks are slotted along the periphery. The color glasses are held in the slots by means of spring-steel clips. The disks are supported on ball bearings, and ball bearings are used as spacers between the disks. The concentric hollow shafts are of steel and are fastened to the disks with machine screws. The operating hand wheels are keyed to their resp. shafts and are held in position by lock nuts. The edges of the wheels are notched. A simple ball and spring brake system is mounted on top of the case, the balls engaging the notches in the hand wheels. This device enables the disks to be manipulated one at a time. The disks contain 15 slots for glasses and the values of the glasses in each disk are stamped on the surface of the hand wheels in corresponding positions. In reading a color, the disks are moved until the proper match is obtained and the values of the glasses used are read from the hand wheels. The use of this instrument obviates the

continual breakage of the glasses inherent in any instrument where the glasses are placed by hand. The glasses are also always clean.

The determination of the oil content of seeds with a new extraction apparatus II. P. Jaichenko. *Mashbolno-Zhivore Delo* 1934, No. 5, 18-19; cf. C. A. 29, (1935).—The extr. tube is attached by means of a tinned Cu wire to the condenser and is provided at its flat bottom with a hole which is covered with a disk of filter paper. The seeds are thus exposed to the hot vapors of the solvent.

E. Bielous  
The determination of the oil content [of seeds] by the refractometer. A. Rasteryazev. *Mashbolno-Zhivore Delo* 1934, No. 3, 10-11.—Cover 2 g. of the ground seeds with 15 cc.  $\text{CHCl}_3$  and allow to stand for 12 hrs. at room temp. Calc. the percentage oil content (P) from the difference (D) of the  $n_D$  of  $\text{CHCl}_3$  and the  $\text{CHCl}_3$  soln. by the formula  $10 \times D = 55P \times 10 \times 15 \times 2$ . The method is exact within 0.5%.

I. Bielous  
The flash points of vegetable oils A. S. Slaschev. *Mashbolno-Zhivore Delo* 1934, No. 3, 18-19.—Benzene (0.01-0.03%) lowers the flash point of vegetable oils by 40°. The flash point is also affected by the conditions of the crop and storage.

E. Bielous  
Oils from *Althaea officinalis* and *Malva arborea*. H. Ya. Tropp. *Farm. Zhur.* 1934, No. 4, 134-8.—Complete analyses of the oils are given, showing high percentages of unsat. acids of the linoleic type. The oils (especially that from *Althaea officinalis*) show good drying qualities and can be substituted for linseed oil.

L. Nasarevich  
The Barbados nut in the Cape Verde Islands. Jose Cunha da Silveira. *Anais inst. super. agron. Univ. tech. Lisboa* 6, No. 1, 116-26 (1935).—The  $\text{H}_2\text{O}$ -free shelled nuts contain 56.34-66.04% oil suitable for making soap.

O. W. Willcox  
The effect of drying castor beans on the oil. M. Zhdan-Pushkin and M. Sokolova. *Mashbolno-Zhivore Delo* 1934, No. 6, 15-17.—The drying of the castor beans at temps. of 150-175° for 1-10 min. decreases the acid no. (owing to volatilization of the free acids) and causes polymerization of both the free acids and oil.

E. B.  
Oil of Tennessee red cedar. H. B. Huddle. *Ind. Eng. Chem.* 28, 18-22 (1936).—The history of the production of red-cedar oil is reviewed and the method of production described. The consts. of 3 samples of cedar oil distd. in 1932, 1933 and 1935 are, resp.:  $d_4^{20}$  0.9331, 0.9361, 0.9441;  $n_D^{20}$  1.4534, 1.4554, 1.4574;  $n_D^{25}$  1.5042, 1.5053, 1.5063; sapon. no. 8.71, 5.93, 6.76; acid no. 2.63, 2.50, 0.80. Ac. no. 43.65, —, 27.66; ester no. (calcd.) 6.08, 3.43, 5.06; I no. 60.96, 64.30, 61.79;  $\text{H}_2\text{O}$  0.35%, trace, trace. The uses of the oil in perfumes, as immersion oil for microscopes, in insecticides and in medicine are discussed.

E. Scherubel  
Elm-seed oil. H. A. Schuette and Carl M. Lunde. *Oil and Soap* 13, No. 1, 12-13 (1936).—The chem. and phys. characteristics of Am. elm-seed oil from Wisconsin elms are:  $d_4^{20}$  0.9288,  $n_D^{20}$  1.4534, coeff. of viscosity at 20° (0.338), surface tension at 20° (dynes/cm.) 30.72, uter 14°, I no. (Wys) 24.1, sapon. no. 273, thiocyanogen no. 16.18, hydroxy no. 13.45, R-M. no. 2.1, Polenske no. 33.9, sol. acids as butyric 0.8, Hehner no. 82.23, I no. of fatty acids 23.08, thiocyanogen no. of fatty acids 15.51, sapon. no. of fatty acids 288.7, unsaponifiable matter 1.50%. In its major aspects, elm-seed oil appears to be the counterpart of coconut oil growing in the temperate zone. Capric acid is present to the extent of 50%.

E. Scherubel  
Essang oil. Alph. Steger and J. van Leen. *Rec. trav. chim.* 54, 988-94 (1935).—Essang oil, also known as masana and sanga-sanga oil, comes from a West African tree belonging to the family Euphorbiaceae. The seeds contain 48.8% oil having the following consts.:  $n_D^{20}$  1.4841,  $n_D^{25}$  1.4810,  $n_D^{30}$  1.4780,  $n_D^{35}$  1.4750, Reichert-Meissl no. 0.4, I no. 19.2, thiocyanogen no. 8.6, sapon. no. 193.1, acetyl sapon. no. 194. The percentage compn. of the oil as detd. is as follows: unsaponifiable matter 0.5, satd. acids 9.7, 9-oleic acid 16.0, 9,12-linoleic acid 11.0, linoleic acid 10.0, eleostearic acid 46.0, glycerol 4.4, volatile matter 1.2. The oil is related to Chinese wood oil but is

not poisonous and can be used for edible purposes.

E. Scherubel  
Alcoholysis of olive oil. Yves Volmar and Björge Hanssen. *Compt. rend.* 201, 968-70 (1935).—The fat acids of olive oil were converted to the Me esters and fractionated at a pressure lower than 1 mm. Olive oils from various sources contained 0.19-0.23% arachidin and 1% linolein.

W. Gordon Rose  
Composition of citric acid. R. S. McKinney and G. S. Jamieson. *Oil and Soap* 13, No. 1, 10-11 (1936).—Until recently it was believed that the chief unsat. constituent of citric acid known as couepic acid was an isomer of eleostearic acid, but it has recently been shown to be a 4-keto-3,4,4,4-octadecatenoic acid ( $\text{C}_{18}\text{H}_{31}\text{O}_2$ ) which m. 74-5° (cf. Brown and Farmer, C. A. 29, 8249). The name suggested for it is *heamic acid*. The consts. and compn. as found by the authors are:  $n_D^{20}$  1.5145, sapon. no. 192.6, I no. (calcd.) 218.0, thiocyanogen no. 76.2, unsaponifiable 0.57%, I no. of unsaponifiable 110, satd. acids (Bertram method) 10.7%, oleic acid 5.9, heamic acid 76.2, glycerides of satd. acids 11.2, of oleic acid 6.2, and of heamic acid 81.2%.

E. Scherubel  
Oil from *Peganum harmala*. H. Ya. Tropp. *Farm. Zhur.* 1935, No. 2-3, 72-4.—The oil has  $d_4^{20}$  0.924,  $n_D^{20}$  1.4787, acid no. 6.6, sapon. no. 177.85, Hehner value 93.85, I value 131, ester no. before acetylation 171.25, after acetylation 146.8, Norman's HO value 25.55, Reichert-Meissl value 1.5, Polenske value 2.9, unsaponifiable matter 3.15, thiocyanate value 79.6, hexabromide value 0. The oil contains about 55% of linoleic acid and is best suited for soapmaking.

L. Nasarevich  
Composition of rape-seed oil. Ruchiro Yamauchi and Kentaro Ichihara. *J. Chem. Soc. Japan* 56, 1332-4 (1935).—Fat acids of the oil consisted of behenic 0.8, erucic 55, oleic 14, linoleic 24, linolenic 2 and palmitic 3.5%. It contained also myristic, palmitoleic and stearic acids but the amt. was small. The presence of rape-seed oil in the other oils can be identified by the detection of erucic acid, and the presence of the other oils in rape-seed oil by estg. stearic acid.

K. Kutsuta  
Detection of rape oil in edible fat. J. Grossfeld. *Chem. Ztg.* 59, 935-6 (1935).—The method for detg. the amt. of rape oil in other oils depends on the estn. of the content of erucic acid by detg. the I absorption of the insol. fat acid Pb salts obtained from the sample. The method is similar to the Grossfeld and Peter procedure for estg. isoleic acid in oils. The sensitivity is increased by adding palmitic acid to the test sample so that the insol. Pb salt of the erucic acid is absorbed by the Pb palmitate. As little as a 2% admixt. of rape oil to linseed oil can be detected.

M. M. Piskur  
The adsorption of oil by the hulls of sunflower seeds I. Krasutskii. *Mashbolno-Zhivore Delo* 1934, No. 3, 9-10.—The hulls of sunflower seeds can adsorb as much as 24.51% of oil at a pressure of 400 atm.

E. Bielous  
The determination of moisture in whole ground sunflower seeds, press cake and hulls in Trunkler's apparatus. G. V. Vugovskii and M. L. Ginzburg. *Mashbolno-Zhivore Delo* 1934, No. 3, 21-2.—Concordant results in the detn. of moisture are obtained with Trunkler's app. at 130° by drying the whole seeds, 40, the ground 25, the press cake 25-30, and the hulls 25 min., resp.

E. B.  
Wheat-germ oil. Walter Cusack. *Ann. chim. applicata* 25, 417-23 (1935).—An oil has been obtained by expressing wheat germ which is purer and more stable than the oil obtained by extr. with solvents. (The method is to be published elsewhere.)

A. W. Contern  
Orientation of molecules of beeswax, and its effect on solidity of the comb. Paul Woog and N. Yannaquis. *Compt. rend.* 202, 78-80 (1936); cf. C. A. 30, 1250.—Of 2 portions of honeycomb one was kept at 15° and the other at 38° for 5 months, the latter was much stronger; of 2 portions of beeswax made into ribbons and similarly treated the latter was 58% stronger than the former. The heated wax was more translucent and darker colored. The bearing of these facts on the construction of the comb is discussed.

C. A. Silberrad  
Triethanolamine and other alkylolamines in the oil,



fat, wax and other industries Emil J. Fischer. *Allgem. Oel- u. Fett-Zig.* 32, 489-95(1935)—An extensive review of patents and literature M. M. Fiskur

Progress of the soap industry in year 1935 A. van der Werth. *Allgem. Oel- u. Fett-Zig.* 32, 503-6(1935).—A review of patents M. M. Fiskur

From the notebook of the soap manufacture. Karl Pfaff. *Rieschstoff-Ind.* 10, 223-4(1935), cf. C. A. 30, 8031.—Haas' colloid soap, Spanish hardened olive-oil, mangrove-peel and antiseptic soaps are discussed H. M. Burlage

Soap, soap fillers and washing powder. Ernst Jaschke. *Seifensieder-Zig.* 62, 859-62, 879-81, 901-3(1935)—Methods for filling soaps are illustrated with a no. of practical examples J. W. Perry

Determination of volatile hydrocarbons in soaps Procter and Gamble Laboratories. *Oil and Soap* 13, No. 1, 9-10(1936)—The method requires a source of dry oil-free steam which is passed through the sample, treated with sufficient acid to liberate the fat acids from the soap. The steam is next passed through strong caustic soda to scrub out any volatile fat acids, while the volatile hydrocarbons are condensed with the steam in a suitable arrangement which allows the excess water to flow away leaving the volatile hydrocarbon in the measuring buret. The method can be applied to samples containing substances immiscible with water and volatile with steam. For solvents heavier than water a Budek-Stirling tube should be used. The app. and its arrangement are shown E. S.

Water glass in soap, soap powder and self-acting detergents Fritz Ohl. *Allgem. Oel- u. Fett-Zig.* 32, 453-6(1935)—A review giving the amt. usually used and advantages of using Na silicates in soaps M. M. Fiskur

The development of soap rancidity in the manufacture of fine soap Oculus. *Seifensieder-Zig.* 62, 820-1(1935)—A discussion of how to distinguish between soap rancidity due to (1) the catalytic action of metals, (2) the use of fats bleached with oxidizing agents and (3) the use of unsuitable fat material J. W. Perry

Soap from coriander-seed oil F. Nevelin and A. Kol'yu. *Maslobolno-Zhirmoe Delo* 1934, No. 3, 26—Coriander seeds contain after distn of the volatile oil 11-20% of fatty oil which gives soft Na and K soaps of pleasant odor E. Biellous

The effect of fat solvents and heavy hydrocarbons on the detergent properties of soap M. Nikiforov and K. Chernyak. *Maslobolno-Zhirmoe Delo* 1934, No. 6, 23-5—The light and medium solvents (b 200-25°) improve the detergency of soap, especially the chemically uniform solvents, such as turpentine, tetralin, decalin. Heavy hydrocarbons (b 250-75°) considerably lower the detergency. The addn of 10% of solvent to a hard soap softens the latter to an extent equiv. to the lowering of the titer by 2-3 E. Biellous

The use of alginic acid for soaps Anon. *Maslobolno-Zhirmoe Delo* 1934, No. 3, 28—Expts. showed that Na alginate does not possess any detergent properties E. Biellous

Starch as a component of soap Th. Ruemele. *Allgem. Oel- u. Fett-Zig.* 32, 502(1935)—The use of starch in cosmetic and shaving soaps is recommended M. M. P.

Dry-cleaning soaps and solvents S. W. Putnam. *Soap* 12, No. 1, 25-8, 63(1936).—Chlorinated solvents are replacing petroleum solvents in dry cleaning. The desirable characteristics for a soap for use with any chlorinated solvent are discussed E. Scherubel

Soaps or emulsions for silk degumming? J. I. Springer. *Soap* 12, No. 1, 29-32(1936)—A degumming oil is best in those cases where the softness of the water cannot be relied upon. Soap to which a small quantity of mild alkali has been added is recommended for hosiery except when rayon is mixed with the silk. E. Scherubel

Textile oils (Meyer) 25 Flax investigations (Hutton, Strickland) 15 Is soap coming into its own again? (Volz) 25.

Oils, etc Isser Davidsohn and Robert Strauss, Ger. 619,928, Oct. 9, 1935 (Cl. 23a 3). Oils, fats, waxes or their mixts and products such as soaps and cosmetics are prevented from becoming rancid by addn of up to 3% of aliphatic high-mol. halogenated hydrocarbons, such as chlorinated or brominated paraffins.

Refining oils, waxes and resins Planktoll Chemische Fabrik G. m. b. H. and Johannes B. Carpow. *Fr.* 788,239, Oct. 7, 1935. The material in the liquid, liquefied or dissolved state is weakly acidified and treated with such an amt. of a dry pulverized slime, from sea or fresh water, containing chemically active compds. of Si (i. e., poor in O) that the refined product, sepd. from the purifier by pressing or distn., is not fully neutralized.

Apparatus for cooking materials such as fish, scraps, etc., to release oils and fats Charles B. Upton. U. S. 2,027,131, Jan. 7. Various structural, mech. and operative details.

Lecithin preparations Noblee und Thorl G. m. b. H. *Fr.* 788,632, Oct. 14, 1935. Stable aq. emulsions of vegetable lecithin and oil are obtained by heating fresh soybean residues, bleached, e. g., by H<sub>2</sub>O<sub>2</sub>, and adding while still hot a soln. of water glass. The product is useful as an addn in making soap and in the leather and textile industry. *Fr.* 788,633. Powd. mixts. containing lecithin are prepd. by intimately mixing, at a slightly raised temp., fresh soybean residues (bleached if desired) with one or more appropriate substances such as calcined Na<sub>2</sub>CO<sub>3</sub>, water glass, sugar, cacao, chocolate powder or starch, reducing to powder and drying under vacuum at about 60°. Oil may be removed from the residues by means of acetone or AcOEt before the mixing.

Soap from sperm oil. Walther Schrauth (to "Umchem"). *Chemikalien Handels, A.-G.* U. S. 2,027,936, Jan. 14. Sperm oil is saponified, and the alcs. formed by treating the material with concd. alkali such as NaOH at a temp. of 200-280° are oxidized by use of about 1-2% more of the alkali than the stoichiometrically calcd. proportion.

Soap composition suitable for use with hard water Heinrich Bertsch (to American Hyalcol Corp.). U. S. 2,026,810, Jan. 7. A water-sol. soap is used in combination with a water-sol. salt such as the Na salt of an acid sulfuric acid ester of lauryl alc. in sufficient proportion (suitably about 25%) to prevent pptn. of the soap when used in hard water.

## 28—SUGAR, STARCH AND GUMS

J. K. DALE

Notes on cane milling and mill train design. Leandro S. Fibro, Jr. *Sugar News* 16, 515-17(1935)—The standard equipment in the Philippine Islands consists of 11 rolls, 34" X 78". High grinding rates prevail with sucrose extns. of 92-4%. Knives, Maxwell shredders and Nobel hot maceration have been introduced lately. L. C.

Diffusion battery work H. Claassen. *Deut. Zucker-Ind.* 60, 751-4(1935).—A restatement of the known principles of correct diffusion work aimed at recent tendencies working counter to these principles. Reply H. Gaertner. *Ibid.* 792. F. R. Bachler

Diffusion battery losses W. Paar. *Deut. Zucker-Ind.* 60, 809(1935)—Sugar losses are often hidden because of faulty methods of analysis. Sugar losses in pulp should be detd. only by hot aq. digestion and never in a superficially prepd. press juice. F. R. Bachler

Morgan disintegrator at Aguirre Norman Kay. *Facts About Sugar* 30, 471(1935).—The disintegrator has made it possible to increase the cane tonnage ground by 15%, and also the sucrose extn. Tramp iron was completely removed in front of the disintegrator by means of the detector coil of the General Elec. Co. F. W. Z.

Temperatures in vacuum pans. Alfred L. Webre. *Facts About Sugar* 31, 13-14 (1936); cf. *C. A.* 29, 8385<sup>1</sup>.—The temp. of a boiling massecuite is affected not only by the vacuum, but also by the hydrostatic head of the massecuite, and increases downward to such an extent that the lower strata are below the satn point when the top stratum is supersatd. This is the principal cause of unsatisfactory pan operation, especially with high strikes. Mech. circulation greatly improves conditions by creating a more uniform temp. throughout the pan. After the grain has set, supersatn becomes less important as a criterion for boiling, and fluidity serves as a much better guide. F. W. Zerban

The influence of the presence of inorganic nonsugars on the rotation of sucrose solutions. E. Landt. *Deut. Zuckerrnd.* 60, 002-4 (1935).—An attempt is made to form the basis for a working hypothesis for an explanation of the cause of rotational changes in sucrose solns by taking into consideration the ionic properties possessed by the inorg. nonsugars that are present. Proof for this is still lacking because existing older measurements cannot be evaluated in the light of modern phys.-chem. viewpoints. F. R. Bachler

Viscosity measurements in sugar solutions by measurements of power used during stirring. F. Horn and W. Tagener. *Deut. Zuckerrnd.* 60, 939-40 (1935).—Molasses solns of various concns were stirred at 20° with an electric stirrer. The power consumed was found to be in direct relation to the viscosity. F. R. Bachler

Contraction occurring on dilution of sugar solutions. W. Paar. *Deut. Zuckerrnd.* 60, 937-8 (1935).—Plato's earlier data are reviewed. A table has been calculated showing contraction occurring at 20° when solns contg. 10-80% of pure sucrose are dil. to 5-75%. In impure sugar solns, contraction is appreciably greater explaining the fact why the 1 l. diln. method of analysis gives too high an apparent dry substance value. F. R. Bachler

New method for the determination of true dry substance in solid and liquid products. P. Morizot. *Bull. assoc. chim.* 52, 830-2 (1935).—The material is dried in glass bottles of special design, which are inserted in a double-wall drying oven, the necks of the bottles protruding through the cover. The drying process is hastened by drawing dry air through the bottles by means of an aspirator. The results on sirups, massecuites, molasses and flours agreed well with those obtained by the usual drying method. F. W. Zerban

Determination of dry substance in sugar solutions, especially in sugar factory products. P. Morizot. *Bull. assoc. chim.* 52, 833-9 (1935).—The usual method of drying on sand or pumice stone requires 5-6 hrs., and great attention to details. In the new method (cf. preceding abstr.) less diln. is necessary (1 + 2 for molasses or massecuites). Six to 7 g. absorbent cotton is placed in the drying bottle, the soln contg. about 2 g. of dry substance is poured over it and dried at 105-6°, while a current of air is drawn through the bottle. Const. wt. is obtained in 1 1/2 to 2 1/2 hrs., and the result checks with the usual method within  $\pm 0.24\%$ . If desired, the  $H_2O$  given off may be collected and weighed. F. W. Z.

Does optimum flocculation correspond to an effective maximum defecation? M. Garino. *Ind. sacchar. ital.* 28, 550-2 (1935).—So far the practical results have been excellent for the physicochem. conditions of the juice. No information has been obtained, however, concerning the elimination of the N compounds, which are considered undesirable. The search for supplementary criteria of maximum defecation. *Ibid.* 552-3.—The ultramicroscope has been an important tool. More satisfactory methods for the detection and detn. of melassigenic compds. are required. Final elimination of colloids. *Ibid.* 553-6.—The plant practice has advanced beyond the scientific knowledge of the phenomena involved. L. Cusachs

Variations in the  $pH$  of lime solutions. Renato Salami. *Ind. sacchar. ital.* 28, 539-41 (1935).—There is a decrease in  $pH$  with increase in temp. of  $Ca(OH)_2$  solns and suspensions. Sucrose does not disturb the  $pH$  of the soln

in the presence of the original solute in the solid phase; otherwise the soln. is less basic. L. Cusachs

The application of physicochemical theories to the purification of crude sugar-beet juices. E. J. Hugel. *Bull. assoc. chim.* 53, 3-10 (1936); cf. *C. A.* 28, 2936<sup>1</sup>.—The opinion generally held that beet juice has 2 isoelec. points, 1 in the acid and 1 in the alk. range, is erroneous. Flocculation by electrolytes may occur over a wide range of  $pH$ , depending on the nature and relative concn. of the electrolytes added. However, flocculation by oppositely charged colloids always occurs at a definite  $pH$  provided that the degree of dispersion is the same. *Salfosol*, recommended for the purification of beet juices, is a mixt. of inorg. colloids, of high flocculating power. Its use makes it possible to cut the lime in half, and greatly to reduce filter press work and sugar losses. F. W. Z.

Effects of different treatments at harvest upon the yield of sugar cane. Valeriano C. Calma. *Sugar News* 16, 558-9 (1935).—Topped cane should be harvested immediately, except if the milling of unburned cane is delayed. It is better to cut the burned cane than to allow it to stand in the field. The covering of sound cut cane with trash in the field is a waste of labor and is favorable to deterioration. L. Cusachs

Bacteriological standards for refined cane sugars. Wm. L. Owen and Roy L. Mobley. *Facts About Sugar* 30, 451-2 (1935).—Refined sugars have for some time been controlled as to their content of thermophilic bacteria. They are frequently infected to an even greater extent with other bacteria, yeasts or molds, which may prove detrimental to users of these sugars and also affect their keeping quality. The following tentative standards are suggested: (1) that the percentage mold infection in any lot should not exceed 20, (2) that the no. of mold spores per g. should not exceed 20, (3) that not more than 20% should contain viable yeast cells, (4) that the no. of yeasts per g. should not exceed 50, (5) that the percentage of infection with bacteria other than thermophiles should not be over 50, certainly not over 100. F. W. Z.

Cane sirup. J. O. Carrero. *Puerto Rico Agr. Expt. Sta., Rept.* 1934, 9-12 (1935). cf. *C. A.* 28, 7576<sup>1</sup>.—The juices from 10 cane varieties were studied with respect to their value for sirup manuf. Grinding began when the canes were 6 months old and continued at 15-day intervals. Analyses were made for sucrose, reducing sugars, P, K and coloring matter. S. C. 12/4, P. R. 803 and M. 42 yielded juices of lightest color. Filtration was slightly faster when lime was supplemented with  $H_2PO_4$  than with lime alone. The lime darkened the juices but the color was restored by the addn. of acid. Juices filtered with Filter-cel only yielded sirups of the best flavor. The care exercised in the boiling and concn. of the juice affected the color and quality. The longer the concn. period, the darker became the final sirup. If the temp. was raised unduly in the last stages of concn. caramelization with resultant darkening occurred. When sirups from different varieties were compared, those from Mayaguez 42 ranked 1st, with those from F. C. 999 and B. H. 10 as very close seconds. Sirups which had been fermented were lightest in color and were very attractive.

Decided reductions in color were obtained by the use of activated C or by slightly increasing the acidity of the juice with  $H_2C_2H_3O_2$ . The use of activated C resulted in a loss of flavor. By means of controlled fermentation, the sugar was partly inverted which prevented crystn. of sucrose in the sirup. Natural fermentations were objectionable. C. R. Fellers

Effect of freezing temperatures on cane in the Florida Everglades. B. A. Bourne. *Facts About Sugar* 30, 453-5 (1935).—See *C. A.* 30, 638<sup>1</sup>. F. W. Zerban

Cane-juice purification. H. C. Prinsen Geerligs. *Bull. assoc. chim.* 52, 825-30 (1935).—The principal aim of clarification is the removal of colloids. This is effected in practice by addn. of lime to the isoelec. point, by adsorption on finely divided ppts. of carbonate, sulfite, or phosphate of Ca, and by mutual flocculation.

F. W. Zerban  
Physicochemical studies on sugar-cane jaggery. A

preliminary communication. T. Varahalu *Madras Agr. J.* 23, 389-93 (1935).—The optimum relative humidity for the storage of all types of jaggeries is 50-60%. In good jaggeries the particles usually consist of a hard core of crust sucrose surrounded by a thin envelope of a more deeply colored material with more of the matrix. In bad jaggeries the sucrose is deposited in rhythmic bands or veins of varying widths and sometimes in widely sep patches of varying sizes bounded by thicker layers of the damp matrix material. Independently of the purity (high percentage of sucrose), the most potent of the constituents in the jaggery, affecting the crystn. of sucrose, and thus conditioning the phys. structure and other related properties, is the nonsugar org. matter fraction. In bad jaggeries this fraction contains substances which cause high viscosities, retard the progress of boiling, cause the tenacious retention of moisture and offer resistance to the formation of nuclei and the growth of sucrose crystals. The corresponding fraction in good jaggeries does not seem to have these adverse effects. Juice from cane fertilized with castor cake contained the smallest and least harmful amt. of nonsugar org. matter; the reverse was true in cane fertilized with  $(\text{NH}_4)_2\text{SO}_4$  alone. When cane was grown under dry conditions or on saline soils or when it was irrigated with brackish water, the nonsugar org. matter fraction was present in comparatively larger amts or in a more harmful condition, than when the cane was grown under normal conditions. K. D. J.

The crystallization of sucrose from cane juices. Considered theoretically and practically. L. S. Birkett. *Intern. Sugar J.* 38, 12-15 (1936), cf. C. A. 29, 4198\*.—The theory of crystn. of sucrose from cane juices involves (1) the soly. relationships of those juices, (2) the formation of the crystal nuclei, and (3) the growth of the nuclei into larger crystals. The soly. relationships are described by reference to the phase-rule principles, a ternary system, sucrose-nonsucrose-water, being taken. By reference to this phase-rule diagram the degree of supersat. necessary to produce crystal nuclei at 70° has been detd. for purities from 30° to 100°. The following table is given.

Purity	100	90	80	70	60	50	40	30
Supersatn	1 20	1 25	1 30	1 35	1 40	1 44	1 49	1 54

It is also possible to det. in advance, by use of the diagram, the conditions necessary to obtain a given purity mother liquor (molasses).

D. P. Langlois

A neglected industrial plant the sweet sorghum. *J. Pariss. Ind. sacchar. stal* 28, 497-508 (1935).—A review with 45 citations. L. Cusachs

1 The behavior of sorghum in a diffusion battery. Cesare Grossi *Ind. sacchar. stal* 28, 542-4 (1935).—Preliminary expts suggest that a diffusion battery may be superior to mills because of the better quality juice and higher extn. The battery was run at 100°, with 150% draft. There was a purity rise, and the juice was clear and altogether suitable for the distillery. L. Cusachs

A new method for purifying starch milk and for the preparation of potato starch. Waldemar Kröner and Gerhard Knoblich *Chem. Fabrik* 1935, 510-12.—On the under side of the lower end of an inclined pipe of suitable length and diam is an outlet for the washed starch, and on the upper side of this end is a  $\text{H}_2\text{O}$  inlet. About half way up on the top side is the starch milk inlet, and between this and the wash- $\text{H}_2\text{O}$  discharge at the upper end of the pipe are a no. of metal sheets parallel to the length of the pipe but with 1 side lower than the other. The washed starch settles on the sheets and slides off on to the lower side of the pipe and down to the outlet. Economical and complete purification are claimed. J. H. Moore

Power alec problem and the sugar beet (Munerati) 16  
Starch for finishing linen (Tr. pat. 783,602) 25

4 Clarifying sugar cane juices. Francis R. Forter (one-fifth to Frank G. Campbell). U. S. 2,027,423, Jan. 14. Primary juices from sugar-cane mills are collected in one app; secondary mill juices are collected in a secondary app and subjected to a treatment to effect quick pptn and which involves heating to a relatively high temp and, without decantation, the temp of the secondary juices is quickly reduced and they are delivered with their pptd. and consolidated impurities back to and upon the bagasse mat and are collected for further clarification after they have been strained by passing through the mat (all in a continuous operation). An arrangement of app is described.

Starch. Stärkelfabrik Naugard, eingetr. Genossenschaft in b H (Alfred Parlow, inventor). Ger. 619,984 Oct. 11, 1935 (Cl. 53k 2 01). Food starch is produced by air-drying potato starch and heating it to 60-100°. The starch may be treated with 0.2% of acid before heating.

6 Sugar-cane mills. Charles McNeil Brit. 434,967, Sept. 12, 1935

Feeding apparatus for starch tables. Samuel Shorback (to International Patents Development Co.) Can. 353,958, Nov. 6, 1935. Structural details

## 29—LEATHER AND GLUE

ALLEN ROGERS

Processing New Zealand [dewooled sheep] pelts. D. Jordan Lloyd and P. White. *J. Federation Carriers & Light Leather Tanners* 16, 234 (1935). *J. Intern. Soc. Leather Trades Chem.* 20, 37 (1936).—Recommendations for eliminating "mottled" and "leopard grain": (1) Use the min. concn of  $\text{Na}_2\text{S}$  in dewooling, (2) after pulling, wash with old lime liquor instead of  $\text{H}_2\text{O}$ , (3) lime for 24 hrs. with intermittent stirring, using not more than 0.26 g.  $\text{Na}_2\text{S}$  and not less than 4 g.  $\text{CaO}$  per 100 cc. soln. and employing a mellow lime, (4) stack the limed pelts not longer than 5 days and preferably not over 2, (5) delime with  $\text{NH}_4\text{Cl}$ , pickle with  $\text{H}_2\text{SO}_4$  +  $\text{NaCl}$ .

H. B. Merrill

Tungsten tannage. V. Casaburi and E. Simoncini. *J. Intern. Soc. Leather Trades Chem.* 20, 2-9 (1936).—"Very satisfactory" leather can be made by tannage with metatungstic acid, phospho-, boro- or silico-tungstic acid. About 40%  $\text{WO}_3$  is fixed. The tannage more nearly resembles vegetable than Cr tannage. Combination tannages with Cr, Al or vegetable tannin are feasible. Analyses of various W leathers are given without description of the tanning methods. H. B. Merrill

Study of grease stains on leather. VII. A comparison of solvents for removing kidney grease stains. I. K. Moore. *J. Am. Leather Chem. Assoc.* 31, 4-32 (1936). cf. C. A. 29, 1676\*.—Surface stains can be removed by scrubbing with solvents or detergents, but the improvement is not lasting because interior grease diffuses to the surface. Stained leather can be degreased and the stains removed by immersing in any one of a large no. of solvents. This process is economical only when dealing with a large no. of badly stained skins. The lower alcs., ethers, ketones and pyridine darken the leather and make it very hard. Ability to remove stains was detd. by immersing pieces of naturally stained leathers in solvents for 24 hrs. Rate of removal was judged by immersing artificially stained pieces of known fat content in the solvent (16 ml. per g.) for 2 hrs. and detg. the percentage of total grease removed. Time for disappearance of residual odors, at room temp. and at 28-30°, was noted. Cost, toxicity and fire hazards are discussed. Most rapid grease removal was obtained with low boiling solvents such as petr. ether,  $\text{CHCl}_3$  and various ethers. These are all impractical for reasons of cost, fire hazard

or toxicity. The other solvents examd. differed little in rate of stain removal. "Stoddard solvent" is recommended because of its cheapness, low toxicity and relatively low fire hazard, although its action is rather slow.  $\text{CCl}_4$  in a closed system is also suggested. The solvent may be reused until its grease content reaches about 7%.

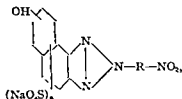
H. B. Merrill  
A method for obtaining casein films of high water resistance on chrome calf leather. M. W. Maslovsky, *Curr. tech.* 24, 382-90 (1935).—Untreated casein films possess little resistance to  $\text{H}_2\text{O}$  regardless of the kind of alkali used for dissolving the casein and regardless of whether the leather surface is previously treated with  $\text{NH}_4\text{OH}$ ,  $\text{AcOH}$  or  $\text{EtOH}$ . The findings of Haupt (*C* 26, 4136) on the  $\text{HCHO}$ -albumin reaction are confirmed. The  $\text{HCHO}$ -casein reaction is different, because the compd is not broken up by steam distn. Most water-resistant coatings are obtained by using a finish high in casein and treating with 10%  $\text{HCHO}$  soln after the coating has dried.

H. B. Merrill

Sturrr for the manuf. of glues and adhesives (Wollenberg) 1. Lecithin preps. [used in the leather industry] (Fr. pat. 788,632) 27. Cleansing agents [for leather] (Brit. pat. 435,465) 13. Drying varnished [leather] under the action of ultraviolet radiation (U. S. pat. 2,020,296) 26

Leather J. R. Geigy A.-G. Ger. 622,213, Nov. 22, 1935 (Cl. 28a 2). For the manuf. of very soft and flexible leather, the deplated hides are treated, before tanning, with an aq. bath contg. small proportions of a neutral pyrophosphate (or neutral pyrophosphate mixt.) and an animal or vegetable albumin, e. g., casein. An example is given.

Azo dyes suitable for dyeing leather in an acid bath. Werner Lange (to General Aniline Works) U. S. 2,022,243, Nov. 26. Dyes dyeing leather red to black tints of good fastness are produced by coupling any diazo compd. with a triazole (pseudoazimino compd.) of the general formula

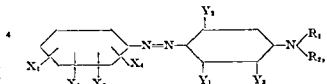


where R is a phenyl or naphthyl radical substituted by at least one nitro group and contg., if desired, further substituents such as sulfonic acid or carboxylic acid

1 radical, halogen, a substituted or unsubstituted amino group (acylamino, alkyl or phenylamino group), an alkyl, hydroxy or alkoxy group, and where  $\pi$  means 1 or 2. These triazoles are obtainable, for instance, by combining in an acid medium an aminohydroxynaphthalenesulfonic acid with a diazotized nitrophenylamine or nitronaphthylamine which may be substituted as indicated above, and by oxidizing the intermediate product thus obtained. Various examples with details of procedure are given.

2 Leather. Georges A. Favre. Fr. 788,387, Oct. 9, 1935. Leather obtained by tanning skins is stabilized by immersing it in an aq. soln. of hydrolyzable and reducing salts, e. g., oxychlorides of Ti and Sb, and "ous" salts of Sn, Sb, Si, Th and Ce and double salts in which one of the elements is one of the above metals.

Dyeing leather. F. M. Senn (to J. R. Geigy A.-G.). U. S. 2,025,618, Dec. 24. Leathers are dyed (usually yellow or orange in the case of numerous examples given), with deep penetration even of chrome leathers, by dyes from diazotized 3-aminobenzenesulfonic acid and bis(hydroxyethyl)aminobenzene or bis( $\beta$ , $\gamma$ -dihydroxypropyl)aminobenzene or other aminoazobenzenesulfonic acid dye of the general formula



where  $X_1$  represents H or  $\text{SO}_3\text{H}$ ,  $X_2$  represents H,  $\text{NO}_2$ , Cl, Me or OR (R being Me, Et or hydroxyethyl),  $X_3$  represents H,  $\text{NO}_2$ , Cl or Me,  $Y_1$  represents H or  $\text{NO}_2$ , with the restriction that at least one of said  $X_1$ ,  $X_2$ ,  $X_3$  and  $Y_1$  must be other than H,  $Y_2$  represents H or  $\text{SO}_3\text{H}$ ,  $Y_3$  represents H,  $\text{CH}_3$ , Cl or OH,  $R_1$  represents H, Me or OMe;  $R_2$  represents H, ethyl, hydroxyethyl or dihydroxypropyl, and  $R_3$  represents hydroxyethyl or dihydroxypropyl.

Dyeing leather. E. I. du Pont de Nemours & Co. Brit. 435,477, Sept. 23, 1935. Chrome- or vegetable-tanned leather is dyed by applying thereto a diazo dye obtained by coupling with *res-cetol* (I) either diazotized aminosalicylic acid (II) or diazotized picramic acid (III) and coupling the monoazo product with either diazotized II or diazotized III. In an example, chrome calf leather is tumbled in a dyeing drum at about 55° with aq.  $\text{NaHCO}_3$  until neutral to litmus, an aq. soln. of the dye II (2 mols)  $\Rightarrow$  I is added, the tumbling is continued 20 min., an aq. soln. of com.  $\text{HCOOH}$  is added and tumbling continued a further 30 min.; the leather, thus dyed a full level yellowish brown, is fat-liquored and dried. The dye may be replaced by II  $\Rightarrow$  I  $\Rightarrow$  III or by III  $\Rightarrow$  I.

## 30—RUBBER AND ALLIED SUBSTANCES

C. C. DAVIS

Semi-ebonite II. P. A. Gibbons and F. H. Cotton *Trans. Inst. Rubber Ind.* 11, 354-76 (1935); of *C* A 29, 646S.—By a special app. and technique, which are described and illustrated, the volatile products formed during the aging of semi-ebonites in air at 70° were detd. Water,  $\text{SO}_2$ ,  $\text{CO}_2$  and  $\text{H}_2\text{S}$  were evolved, and at the same time there were gains in wt. as a result of the formation of non-volatile oxidation products. Practical sole-wearing tests of semi-ebonites showed that they have greater resistance than ordinary leather, but have a greater tendency to premature cracking. A special app. to judge the resistance to bending and to compare pliability, and another app. to measure resistances to flexing are described and illustrated. The pliability diminished with increase in S content and was increased by an antioxidant (Nonox). The resistance to flexing increased with decrease in the S content and in the toughness, so that the lowest forms of

semi-ebonite showed the highest resistances. Aging in air at 70° slowly destroyed the resistance to flexing, and an antioxidant did not retard this deterioration. On the other hand, semi-ebonites vulcanized with an accelerator deteriorated less rapidly. Exposure to ultraviolet light also reduced rapidly the resistance to flexing, and this deterioration was not influenced by an antioxidant. After 6 months of natural aging, semi-ebonites vulcanized with an accelerator lost relatively little resistance to flexing, whereas unaccelerated products deteriorated tremendously. An antioxidant had little effect. The tensile (stress-strain) properties of the various products are also given. With increase in S, the tensile strength increased, and the elongation increased to a max. and then diminished. An optimum combination was reached with 22.5-25% S (based on the rubber). The lowest proportion of S which yielded a semi-ebonite was 17.5-20% for un-

accelerated mixts. and less for those contg. an accelerator. The latter also increased the toughness and ultimate elongation, whereas an antioxidant decreased the toughness. The best of the semi-ebonites compared favorably with a black soling contg. a high proportion of C black. In aging naturally, semi-ebonites toughened and became harder and less elastic, with formation of an inert oxidized film. An antioxidant did not retard these changes. The permanent set after elongation of semi-ebonites was very low, but the recovery was sluggish. C. C. Davis

The internal heating of ebonite during vulcanization. H. A. Daynes. *Trans. Inst. Rubber Ind.* 11, 336-42 (1935).—Math. Theoretical equations for the heat produced and for the rate of loss of heat in the vulcanization of ebonite are derived, and the effects of various factors (reactivity, thermal cond., thickness, temp.) are discussed. There are complicating factors which prevent the quant. application of any such simple theory to practical cases, and for a complete math. solution of the problem, it would be necessary to express in simple algebraic form the relations between temp., degree of combination of rubber with S, and rate of heat production, so that the temp. could be calcd. as a function of time. C. C. Davis

Accelerators of vulcanization. F. Jacobs. *Caoutchouc & gutta-percha* 32, 17336-8 (1935). cf. C. A. 29, 8400.—The properties and uses of a few com. accelerators (S P D., Pipolol X, Pipolene and R-2) are described. C. C. D.

Combined use of two different accelerators. VI. Diphenylguanidine and di-*o*-tolylguanidine. Ichiro Aoe. *J. Soc. Rubber Ind. Japan* 8, 593-602 (1935); cf. C. A. 29, 955.—Ftn of the softening and m. ps. of mixts of various proportions of diphenylguanidine (I) and di-*o*-tolylguanidine (II) indicated that I and II do not form a eutectic mixt. Scorching at 100° was the highest around equal parts of I and II, but it was not bad enough to cause danger in practice. Vulcanization tests showed that acceleration was first manifest around equal parts of I and II, but the differences were not very striking. That stearic acid in the rubber neutralized the accelerators and retarded vulcanization was confirmed by adding stearic acid to the mixts. K. Katsuta

A new method of estimation of mineral substances in vulcanized rubbers by adding an organic accelerator to the solvent. Kiyoshi Maruyama. *J. Soc. Rubber Ind. Japan* 8, 525-33 (1935).—Mercaptobenzothiazole (I) or its K salt (II) in the petroleum solvents used in deig the mineral ingredients in rubber increases the soly. of the rubber. Mix 1 g. of sample with 0.04 g. of II, add 10 g. of paraffin and heat in a weighing flask in an oil bath at 180°. When the sample is completely melted add 0.02 g. of I and shake well. When the mineral ingredients have settled dil with petr. ether, centrifuge, decant the clear soln twice, wash 3 times with a soln of equal parts of acetone and CHCl<sub>3</sub>, dry at 100°. K. K.

Determination of total and free selenium in vulcanizates. E. Kheraskova and L. Vaisbrut. *Ind. Eng. Chem. Anal. Ed.* 7, 407-8 (1935).—Total Se.—Destroy 2 g. of rubber sample with hot HNO<sub>3</sub> (d 1.4), evap. with water to dryness, dil with 100 cc. of hot water, cool, filter, add excess (250 cc.) aq. HCl (d 1.19), then excess cryst. Na<sub>2</sub>SO<sub>4</sub> in small portions, heat overnight at 40-50°, filter, wash the Se residue exhaustively, dry and weigh. As a more expensive alternative, but still better otherwise, treat 2 g. of sample with HNO<sub>3</sub>, dil with 150-200 cc. of water, filter (from nitrosites), neutralize the filtrate with 10% NH<sub>4</sub>OH, acidify with HCl, heat with excess (75-100 cc.) said aq. H<sub>2</sub>NNH<sub>2</sub>·H<sub>2</sub>SO<sub>4</sub>, let stand overnight, filter, wash the residue with hot water, EtOH and Et<sub>2</sub>O, dry and weigh. Free Se.—The method depends on the fact that the aq. Na<sub>2</sub>SO<sub>4</sub> exts. S and Se, and HCHO ppt. only Se from the resultant soln. Heat 1 g. of sample with 200 cc. of 10% aq. Na<sub>2</sub>SO<sub>4</sub> for 30 hrs., repeat, filter, wash, boil the filtrate with 75 cc. of formalin for 1 hr. (cf. Bolotnikov and Gurova, C. A. 29, 2026), filter (free S can be

detd. in the filtrate), wash the residue with HCl, hot water, EtOH and Et<sub>2</sub>O, dry and weigh. C. C. Davis

Chlorinated rubber paints (Schultz). 26. Polymerization of hydrocarbons of synthetic rubber manuf. into a paint vehicle (Lopatun, Soldatov). 26. Heat capacity, entropy and free energy of rubber hydrocarbon (Bekkedahl, Matheson). 2. Plastic materials (resembling rubber) (I. r. pat. 788,645). 13. C black (U. S. pat. 2,027,732). 4. Active C (U. S. pat. 2,027,695 and Brit. pat. 435,345). 18. App. for extruding plastic material such as in making molded rubber articles (U. S. pat. 2,028,044). 13. Amides of carboxylic acids contg. S [as softening agents for rubber] (Ger. pat. 619,299) 10

Machine for spreading rubber latex, etc., on moving sheet material. International Latex Processes, Ltd. Ger. 622,277, Nov. 25, 1935 (Cl. 39a. 10.06). See Brit. 429,235 (C. A. 29, 6806)

Rubber. Egon Meier. Ger. 619,944, Oct. 9, 1935 (Cl. 39b. 5). Conc'd rubber solns. are made by treating a paste of rubber and org. solvent with 61.9% HNO<sub>3</sub>, interrupting the oxidation by adding BaCO<sub>3</sub>, drying by anhyd. Na<sub>2</sub>SO<sub>4</sub> and treating the product with an agent which robs rubber of its stickiness such as water free ZnCl<sub>2</sub> or SnCl<sub>4</sub>, NH<sub>4</sub>CNS, (NH<sub>4</sub>)<sub>2</sub>S, Al stearate, PhNO<sub>2</sub>, BzH, quinine, etc. Examples are given.

Halogenating rubber and like materials. Metallges. A.-G. (Otto Schweitzer, inventor). Ger. 622,471, Nov. 29, 1935 (Cl. 39b. 3). See Fr. 785,257 (C. A. 30, 647)

Preserving rubber. Winfield Scott and Horace G. Byers (to Rubber Service Laboratories Co.). U. S. 2,027,001, Jan. 7. About 3% of a condensation product of an aliphatic aldehyde such as acetaldehyde with a diamine comp. two amino groups such as a diamino diphenylamine is added to a rubber compn.

Thermoplastic rubber. Russell J. Reaney. Fr. 788,241, Oct. 7, 1935. A product which may be used for joining rubber to metal, for making a waterproofing lacquer or varnish and as a substitute for gum lac, is made by combining with rubber chem. compds. which do not separately react with it but which together react with it, at normal or raised temp. Thus, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, 18H<sub>2</sub>O, 17.6 PCl<sub>3</sub> and 12 and rubber 100 parts are mixed together and heated to 160° for 5-15 hrs. and afterward homogenized and purified.

Rubber threads. Thomas L. Shepherd. Fr. 788,456, Oct. 10, 1935. A stream of liquid rubber is brought into contact with a coagulating agent and is drawn while in the state of partial coagulation. App. is described.

Machines for making rubber threads. Caoutchouc Laboratories, Inc. Fr. 788,112, Oct. 4, 1935

Microporous rubber sheets. I. G. Farbenind. A.-G. Fr. 788,075, Oct. 3, 1935. Very thin sheets suitable for diaphragms, etc., are planed or cut from thick sheets of rolls by machines such as are used in the cellulose and wood industries.

Rubber articles. International Latex Processes Ltd. (Giuseppe Venosta, inventor). Fr. 788,499, Oct. 10, 1935. See Brit. 433,441 (C. A. 30, 8024)

Molding hollow rubber articles such as balls. Lewis J. Clayton (to Viceroy Mfg. Co. Ltd.). U. S. 2,027,634 Jan. 14 App. and various operative details are described.

Molding hollow articles such as pipes from materials such as rubber or fiber. Harry Raffovich. U. S. 2,026,906, Jan. 7 App. and various operative details are described, involving heating assembled materials to harden a binder such as by vulcanizing a rubber compn. which is used.

Water bottles of rubber and fabric. Milton B. Reach. U. S. 2,027,290, Jan. 7. Various mfg. details.

Tire tube. Walter E. Shively (to Wingfoot Corp.). Can. 354,002, Nov. 5, 1935. A rubber inner tube is covered on one side with a gas-imperious polyglycol-contg. coating which is made water-resistant by a covering of rubber.

# CHEMICAL ABSTRACTS

Vol. 30

MARCH 20, 1936

No. 6

## 1—APPARATUS, PLANT EQUIPMENT AND UNIT OPERATIONS

W. L. RADGER

Modern developments in chemical balances J. L. Buchan, *J. Sci. Instruments* 13, 1-4 (1936) E. H.

A simple oil micromanometer Otto Beeck, *Rev. Sci. Instruments* 6, 399-400 (1935) —An oil-Hg combination app. is described, with 1 cut. It is sensitive to about 0.0003 mm Hg J. H. Moore

A new method for the registration of density O. Spengler, F. Todt and St. Böttger, *Z. Wirtschaftsprüfung* 85, 680-8 (1935) —A new registering densimeter, Speziometer, registers d to within 0.1° Brix regardless of temp. and purity changes F. R. B.

Improvement of the micro-Kjeldahl method Béla Grodó, *Biochem. Z.* 283, 59-61 (1935) —Description of an all-glass app. in which all the stages of the process (except the digestion) are carried out, whereby greater accuracy in parallel detns. is secured S. Morgulis

Glass vessel for water distillation Bernhard Siede, *Chem.-Ztg.* 59, 925 (1935) —H<sub>2</sub>O-resistant glass is recommended over Sn for cheapness. An automatic small-scale still is described. P. S. Rollet

Some new calorimetric apparatus W. A. Roth, *Chem. Fabrik* 1936, 10-12 —An elec. microbomb with evacuable mantle, a transparent app. for precision measurements with thermoelements and a twin-scale calorimeter are described Six references, 3 cuts J. H. M.

Sedimentometer of new construction Jar Mithauer, *Z. anal. Chem.* 103, 419-22 (1935) —For detg. in tech. analyses the rate at which floating particles sep. out in fluids, the photoelec. cell can be used to advantage. The light rays from the elec. lamp, whose intensity is kept const. by an automatic regulator, pass through a thermo filter, a slit and then into the vessel in which sedimentation is taking place. After this the light passes through another slit and an achromatic condenser before coming in contact with a very sensitive photoelec. cell. The resulting p. d. is measured with a very fine, aperiodic microvoltmeter. Although the principle is very simple, a no. of difficulties had to be overcome W. T. H.

A simple type of humidity chamber F. C. Mead, *J. Sci. Instruments* 12, 394-5 (1935) —An app. designed for making elec. tests on small specimens of material under controlled conditions of temp. and relative humidity. One cut. J. H. Moore

Practical milk-of-lime scale for laboratory and factory H. V. Raven, *Centr. Zuckerrind.* 43, 830 (1935) —A one-l. vessel is filled with milk of lime and weighed on a balance, the pointer of which shows on a quadrant g. of CaO per l. F. R. Bachler

New apparatus for the quantitative recovery of dialyzates F. Loch, *Z. anal. Chem.* 101, 385-8 (1935) —It is sometimes desired to make a complete sepa. of a dissolved crystalline from a dissolved colloid. The usual method of using a semipermeable membrane and running water is out of the question because of the large vol. used in removing all the crystalline. The device here described is based upon the principle of the Soxhlet extra. app. The original soln. is placed in a tube of parchment paper, or Cellophane, and the tube is placed in a rectangular vessel which is about 1/4 filled with water. A siphon tube is placed in the water so that when the level rises above a certain point, the excess is siphoned off, as in the Soxhlet app. The siphoned water, or aq. soln., flows by gravity into a neighboring flask, which is provided with a siphon tube and is connected with a condenser. The liquid in

this flask is boiled gently, the distillate passes into the condenser and runs into the vessel in which the dialysis is taking place. There fresh water is, therefore, constantly being added. The vol. of the aq. soln. at the end of the extra. is merely the vol. of water originally placed in the flask and this can be made as small as desirable. Whenever desired, the soln. in the flask can be siphoned off. W. T. H.

New process for controlling surfaces in the chemical and related industries Alfred Salmony-Karsten, *Industrie chimique* 22, 737-9 (1935) —A brief discussion of the merits of the microcomparator which is essentially a low-power microscope constructed so that it can be removed from its base and applied directly on the surface to be examd., and so that the latter can be observed simultaneously (and in the same field) with a standard surface A. Papineau-Couture

Returnable drums for handling chemicals R. W. Lahey, *Chem. & Met. Eng.* 43, 28-31 (1936), cf. C. A. 29, 4475 E. H.

Protective coating for wood in sodium sulfate Kenneth A. Kobe and Frank A. Morey, *Chem. & Met. Eng.* 42, 682-4 (1935) —Test panels of seasoned Douglas fir coated with a Bakelite-base varnish stood up for 24 days in a boiling satd. soln. of Na<sub>2</sub>SO<sub>4</sub> at 102°, but were darkened E. R. Rushton

Machines for the production of packing and similar products Max Pollmann, *Gummi-Ztg.* 50, 9-10, 34-5 (1936) C. C. Davis

Separation of gases from mixtures by E. Mazza's patented processes. Judes Rabu, *Industrie chimique* 22, 890-900 (1935) —Mazza's centrifugal gas separator (C. A. 29, 4218) is described and numerous possible applications are pointed out A. Papineau-Couture

Tentative standard system of nomenclature for unit operations proposed by A. I. of Ch. E. A. B. Newman, et al. *Ind. Eng. Chem., News Ed.* 14, 59 (1936) E. J. C.

Extraction of acetic acid with isopropyl ether in a spray column J. C. Elgin and F. M. Browning, *Trans. Am. Inst. Chem. Engrs.* 31, No. 4, 639-70 (1935) —Exptl. data are presented for the rate of mass transfer of AcOH between com. iso-Pr<sub>2</sub>O and water in a 2.03-in. diam. Pyrex section spray extn. column whose effective height is 48.75 in. and effective vol. 0.0013 cu. ft. Relations applicable to rate of extn. are discussed briefly and capacity coeffs. calcd. from the data by the equation  $K_{a\omega} = (N/\theta) / V \log \text{mean } \Delta C$ , in which V is the effective column vol. and N/lb. mols. per hr. transferred.  $K_{a\omega}$  depends on the rates of flow of both dispersed and non-dispersed phases, on whether the water or ether phase is dispersed, on the direction of acid transfer, on the inlet acid concn. of the phases and on the diam. of the entrance nozzle for the dispersed phase. Diam. and velocity of transport of the dispersed drops vary greatly with the phase dispersed and acid concns. Data for phys. properties of the 2 solvents as a function of acid concn. are reported C. L. Mantel

Fractional distillation in ring-filled columns Voland Mayor, *Industrie chimique* 22, 730-2 (1935) —Math. A method is given for calcg. the dimensions of a ring-filled fractionating column. The advantages of ring-filled columns are pointed out. A. Papineau-Couture

Heat-transfer coefficients during drop-wise condensa-

tion of steam W. M. Nagle, G. S. Dais, L. M. Blendenman and T. B. Drew, *Trans Am Inst Chem Engrs* 31, No. 4, 593-601 (1935).—A detailed discussion of expts which gave steam side coeff. for drop-wise condensation on Cr-plated pipe with temp. differences from 5.2° to 13.9°F having values between 11,000 and 17,000 B.t.u./hr./sq ft/°F C. L. Mantell

The conditions for drop-wise condensation of steam T. B. Drew, W. M. Nagle and W. Q. Smith *Trans Am Inst Chem Engrs* 31, No. 4, 605 21 (1935); cf C. A. 28, 545<sup>2</sup>.—Clean steam, whether or not it contains noncondensable gas, always condenses in a film on clean surfaces, rough or polished. Drop-wise condensation of steam does not occur unless the cooling surface is in some way contaminated. Although numerous substances, while actually on the surface, make it nonwetttable, only those that are strongly adsorbed or otherwise firmly held are significant as drop-promoters in a condenser. Some contaminants seem to depend for their activity as promoters on the amt. of noncondensable gas present. Some contaminants are specifically effective on certain metals (e.g., mercaptans on Cu alloys), others are quite generally effective (e.g., fatty acids). Boiler steam at the Mass. Inst. of Tech. naturally contains drop-promoters which are effective on some metals but not on others. Drop-wise condensation is induced and maintained more easily on smooth surfaces than on rough C. L. Mantell

Efficient truck refrigeration H. W. Krotzer *Ice Cream Trade J.* 31, No. 11, 33 7 (1935).—Insulators, refrigerants, construction and operation of refrigerating systems are discussed in relation to their use for truck refrigeration A. H. Johnson

A method of obtaining and controlling high humidities at high temperatures W. Lethersich, *J. Sci. Instruments* 12, 348-91 (1935).—Addnl. and sep. heating is supplied by an immersion heater to a tray of H<sub>2</sub>O in the oven, and the addn. of a thermostat in the H<sub>2</sub>O gives close control. In an oven 18 × 20 × 30 in. high a relative humidity of 90% was held const. to 0.1% at 75° for a day. J. H. Moore

Heat transmission in evaporators H. Claassen *Centr. Zuckerrind* 43, 852-3 (1935).—The applicability of the results of researches by the polytechnical school in Karlsruhe for sugar evaporation problems is discussed. Heat transmission in vertical evaporator tubes H. Claassen *Ibid.* 968-9.—An analysis of the results obtained in evaporator tests by the polytechnical school in Karlsruhe with special reference to their applicability in vertical evaporator tubes F. R. Bachler

Heat savings through insulation of flanges Forschungs Heim für Wärmeschutz *Centr. Zuckerrind* 43, 950 (1935).—Heat losses suffered by a pair of unprotected flanges in a pipe line 100 mm in diam. and with temp. differences between flange and air of 50-400°, measured in still air, ranged between 0.4 and 1355 cal. per hr. Suitable insulation reduced these to 7.6-151 cal. With smaller or larger diam. the saving is in proportion. In open air heat losses can be twice as high F. R. Bachler

Modern materials for high-pressure vessels (McAllister)

British Standard Specifications No. 604—1935 Graduated Measuring Cylinders No. 605—1935 Crow Receivers No. 611—1935 Petri Dishes No. 612—1935 Nessler Cylinders London Brit Standards Inst. 2s 2d each

Laboratory fume cupboard V. I. Karmazin and A. T. Cherniul *Russ.* 36,060, Apr. 30, 1934. The fume cupboard is provided with a rotatable floor.

Piezoelectric crystal device René Lucas (to Compagnie générale de télégraphie sans fil). U. S. 2,029,729, Feb. 4, 1934. Structural details.

Compensation colorimeter. P. M. Nemzer. *Russ.* 34,184, Jan. 31, 1934. Construction details. Viscometer Harry T. Booth (to Lubrication Control

Corp.). U. S. 2,028,188, Jan. 21. Structural and operative details of a device with a spring-opposed piston.

Self-luminous devices John E. Paul (to United States Radium Corp.). U. S. 2,028,241, Jan. 21. An elongated glass tube is filled with self-luminous material such as radioactive material (the tube being closed at one end) and surplus air is expelled from the tube by passing the tube through a flame from the closed end to the open end, and the open end is sealed by fusion in the flame without withdrawing the tube from the flame after the air-expelling operation.

Electric timing apparatus suitable for the control of various apparatus Frederick G. Kelly, Jr. (to Engineering and Research Corp.). U. S. 2,029,622, Feb. 4. Various details.

Thermostatic control for electric circuits Carlton W. Bondurant (to Bishop & Babcock Mfg. Co.). U. S. 2,028,893, Jan. 28. Mech. and operative details.

Apparatus for generating heat by catalytic combustion Fr. Weber & Co. Fr. 789,362, Oct. 28, 1935.

Thermostatic valve suitable for steam-heated water heaters, etc. Sten F. B. Soderberg (to Leslie Co.). U. S. 2,029,203, Jan. 28. Various structural and operative details.

Portable thermometer suitable for use with molten solder, etc. William C. Dillon. U. S. 2,028,969, Jan. 28. Various structural details.

Thermometer and associated apparatus for determining the temperature of oils in tanks, etc. Philip S. Williams and Oliver W. Johnson. U. S. 2,028,887, Jan. 28. Structural and operative details.

Thermometer and associated apparatus for determining the temperature of oils in tanks, etc. Philip S. Williams U. S. 2,028,888, Jan. 28. Structural and operative details.

Pyrometric device suitable for exposure to kln gases Herbert B. Henderson and Edward O. Henderson U. S. 2,028,507, Jan. 21. Structural details.

Seamless tanks such as those of copper and nickel layers suitable for holding chemicals Blasius Bart U. S. 2,029,011, Jan. 28. Various mfg. details are described.

Closure for containers such as cans for light oils, etc. Rollin L. Drake (to Texas Co.). U. S. 2,029,259, Jan. 28. Structural details.

Apparatus for dissolving salts Thomas F. Courthope (to Retsof Mining Co.). Can. 351,444, Nov. 26, 1935. A vat has an open top and double bottom in spaced parallel relation to enclose a liquid chamber and inclined at an angle of 40° to insure removal of solid materials. The upper bottom has an opening in its lowest part and acts to support solid materials as a filtering medium. Liquid is conducted into the top of the vat and discharged from the upper part of the chamber between the 2 bottoms.

Apparatus for gelatin and glue extraction, etc. Wm. V. Knoll U. S. 2,028,935, Jan. 28. Various structural, mech. and operative details.

Apparatus for treating and cleaning articles such as those of metal with solvents such as carbon tetrachloride Clarence F. Dunley (to James H. Bell). U. S. 2,028,739, Jan. 28. Various details of app. and operation.

Fractional-distillation apparatus Gerrit Baars (to Shell Development Co.). Can. 354,566, Dec. 3, 1935. Bubble plates in a series consisting of stepwise progressively lowering sections are alternately inclined in opposite directions. Overflow partitions are provided between the plates, and bubble caps for passing vapors through the plates. An overflow pipe is provided at the lower side of the plate for discharging the liquid, and means for supplying the liquid at the upper side.

Distilling apparatus Le Pyrex. Fr. 789,493, Oct. 29, 1935. Means is provided to maintain the level of liquid in a distg. flask constant, the liquid being heated by heat-exchange with the condensing vapors before entering the flask.

Apparatus for distilling wash oils, etc. Percy D. Walmsley and William B. Kendall. Brit. 435,867, Oct. 1, 1935. Volatile constituents are distd. from wash oils,

etc., by flowing the oil down a vertical, packed still provided with a central steam-heated tube around which the packing of Raschig rings, etc., is arranged in annular form and onto which the oil is directed by notched conical rings.

**Water-column displacement weight** Alfred L. Aicher (to Varnal-Waring Co.) U S 2,028,858, Jan 28. An iron shell with a filling of Al is used for gage floats, etc.

**Rectification of air** Mark Benson Can 355,114, Jan 7, 1936. In sep. O and N from air, the air is cooled and passed through a rectifying column the top of which is at approx.  $-195.8^{\circ}$  and the bottom at approx.  $-183^{\circ}$ . The N condensed from the air flows downward in the column, and gaseous O flows upward.

**Rectification of gases** Mark Benson Can 355,115, Jan 7, 1936. Process air is rectified by cooling a primary working fluid composed of air enriched with O. A secondary working fluid mixt. of gases which do not react with one another is cooled by the first fluid, and then further cooled by compression and expansion, and brought into heat exchange with N at atm. pressure from the process air in condensers arranged in series. Liquid N and O are withdrawn from the bottom of the 1st condenser, and liquid N from the bottom of the 2nd condenser, and helium and neon from the top of the 2nd condenser.

**Apparatus for the wet purification of gases** Atehrs J. Hanez, Soc. anon. Belg 407,264, Feb 28, 1935.

**Apparatus for distilling gas solutions** Gerald J. Horvitz Brit 436,003, Oct 3, 1935. In app. for regenerating a soln. used for scrubbing acid gases, e.g., CO<sub>2</sub>, H<sub>2</sub>S, by flowing down a tower heated at its lower portion and having a portion for gradually heating the soln. by recirculating regenerated hot soln. and by the evolved gases to cause its dissociation, a portion for heating the incoming soln. by evolved gases alone is superimposed on and connected with a portion for heating by recirculated hot soln., the lower heated portion completing the dissociation.

**Cups for bubble-tower plates** B. N. Gubrichevskii Russ 34,531, Feb 28, 1934. Construction details.

**Fractional distillation of materials such as petroleum** Earle W. Gard (to Union Oil Co. of Calif.) U S 2,029,528, Feb 4. A vaporizer is connected with a main fractionating column to condense and fractionate the vapors from the vaporizer, and a portion of the reflux condensate from the main fractionating column is withdrawn and transferred to an auxiliary vaporizer whence vapors pass to an auxiliary fractionating column. Overhead fractions are withdrawn from the auxiliary fractionating column and reflux condensate is transferred from it to a point in the main fractionating column lower than that where the reflux condensate is withdrawn from it. Various features of app. are described.

**Heating fluids such as hydrocarbon oils to be cracked in banks of conduits connected in series.** Joseph C. Alther (to Universal Oil Products Co.) U S 2,029,291, Feb. 4. App. and operative details are described. U S 2,029,292-3-4 also describe app. and operative details for heating oils for cracking.

**Filtering materials** Antigas Werk für Luftschutzgeräte G. m. b. H. Fr. 789,336, Oct 26, 1935. Materials, particularly for gas masks, are made by impregnating animal, vegetable, mineral or artificial fibrous material with solns. of org. substances of high mol. wt., such as resins, and then drying or pptg. the substances on the fibers and then drying.

**Continuous separation of partially miscible liquids** A. I. Krasul'shchikov, Russ 37,696, July 31, 1934. The mixt. of liquids is sep'd. in a settling vessel into 2 layers, each of which is led into a sep. fractionating tower. The vapor from the top of the towers is returned to the settling vessel.

**Circulatory evaporator.** Emil Kirschbaum. Ger. 620,907, Oct. 31, 1935 (Cl. 12a, 2).

**Evaporators and condensers in structural combination** John R. Courtney and Harry Ward. Brit. 436,586, Oct 14, 1935.

**Bulk evaporators comprising a vessel housing an in-**

**lined-tube calandria** Charles McNeil Brit 436,592, Oct. 14, 1935.

**Evaporating aqueous solutions such as cyanide solutions** Alexander D. Macallum (to E. I. du Pont de Nemours & Co.) U S 2,029,826, Feb 4. In evap. an aq. soln. of a substance such as NaCN which tends to hydrolyze at elevated temps., the soln. is contacted with a heating device such as a steel cylinder maintained at a temp. above the b. p. of the soln., at such a rate that the soln. is substantially instantaneously evap'd to dryness before substantial decomposition occurs, the pressure during the evapn. being such that the temp. of the evapg. soln. is maintained above the temp. at which substantial hydrolysis would normally occur. App. is described.

**Drying apparatus for fluids** William E. More and George L. Simpson (to Pittsburgh Research Corp.). Can 355,221, Jan 7, 1936. A fluid is passed through a mass of adsorbent material to remove H<sub>2</sub>O from the fluid. The material is heated to free the H<sub>2</sub>O. A dry wash gas is heated and passed through the heated material to remove the H<sub>2</sub>O. The process is repeated with the reactivated adsorbent material.

**Hot air or vapor drier for flat bands, etc.** G. Siempelkamp & Cie. Ger 620,974, Oct 31, 1935 (Cl. 82a, 25.06).

**Device for drying moist suspended materials.** Techno-Chemical Laboratories Ltd. Ger 620,973, Oct 31, 1935 (Cl. 82a, 1.02).

**Filter** P. D. Silin Russ 37,690, July 31, 1934. The filter is composed of two perforated plates so arranged that the openings of one plate are opposite the solid parts of the other plate.

**Filters.** Siemens-Planwerke A.-G. für Kohlefabrikate (Oswald von Wartburg, inventor) Ger 620,692, Oct 25, 1935 (Cl. 12e, 3.02). Addn. to 618,563 (C. A. 30, 9099). The filter formed from C or graphite embedded in a plastic mass, as described in 618,563, is fired as a whole.

**Filters, purifying water, etc.** Victor Ricardo Torcal Ratta and Pedro I. Ondevilla Urizar. Brit 435,543, Sep. 18, 1935. Bacterial bodies for the sterilization of H<sub>2</sub>O and other liquids are made in 3 different ways. (1) materials, e.g., sand, kaolin, usually used for making filtering bodies are mixed with a colloidal sol of an oligodynamically active comp'd. of such a metal and the mixt. is formed into rigid filtering bodies of the desired shape, (2) rigid porous filtering-bodies are impregnated with a sol of such active colloids and coagulation of the colloidal impregnating material is produced by addn. of an electrolyte, and (3) rigid, porous filtering-bodies are introduced as a diaphragm into an electrolytic cell in which the anions of the cathodic compartment and the cations of the anodic compartment ppt. in the interior of said bodies an oligodynamically active gel.

**Porous filters.** Jenae Glaswerk Schott & Gen. Fr. 789,307, Oct. 28, 1935. A layer of large-grained particles of glass are agglomerated by fusion and this layer is afterward coated with a paste of finer particles of glass which is then dried and the particles agglomerated by heating. Cf. C. A. 29, 24017.

**Apparatus containing porous parts** I. G. Farbenind. A.-G. Fr. 789,737, Oct 15, 1935. The porous plates, etc., of filtering or diffusion app. are made of a refractory material, such as Si or its alloys or carbides, porcelain, Zr, graphite, SiO<sub>2</sub>, or of metals or their oxides, e.g., Fe, Ni, Cu, Ag, MgO and Al<sub>2</sub>O<sub>3</sub>. The nonporous part of the app. may be of glass or nonvitreous refractory material, but particularly glass known as "Suprax" and "Vitrosil." Breakage between the porous and the nonporous parts does not take place as is the case when both parts are of glass.

**Rotating suction filter** Maschinenfabrik Buckau R. Wolf A.-G. Ger 620,691, Oct 25, 1935 (Cl. 12d, 16.02).

**Rotary suction filter** William Rausch and John H. Fedeler, Jr. (to Underpinning & Foundation Co.) U S 2,028,949, Jan 28. Various structural, mech. and operative details.

**Centrifugal filtering apparatus** Seitz-Werke G. m. b. H. Fr. 789,356, Oct 28, 1935.



Air or gas filter. Ifans E. Burkholz (to American Air Filter Co.) U. S. 2,029,406, Feb. 4. Structural details.

Filters, adapted for use in drawing off liquor from barrels. Charles D. S. Appleton. Brit. 435,510, Sept. 23, 1935.

Machine for straining or filtering liquids. Seitz-Werke G. m. b. H. and Robert Adams. Brit. 436,350, Oct. 9, 1935. The app. has a no. of sieve drums to the outside of which the liquid is supplied from a passage, the filtrate being obtained from collecting vessels inside the drums. A rotary shaft carries a plate supporting the drums, each of which has an external layer of filter material. The shaft and plate are rotated rapidly and each drum is rotated slowly on its own axis.

Oil filter. Scintilla (Soc. anon.) (Ernst Schaeren, inventor). Fr. 789,682, Nov. 4, 1935.

Filter (with a rotary stack of disks and spacers) suitable for use with oils. Walter L. Chewing (to United Engineers & Constructors Inc.). U. S. 2,029,611, Feb. 4. Structural details.

Filtering device suitable for use with engine oiling systems. James A. Matney. U. S. 2,029,078, Jan. 28. Structural details.

Filter leaf construction for use in filter tanks. Matthew F. Moran. U. S. 2,028,466, Jan. 21. Various structural details.

Stand for welding glass filter plates into the funnel. V. A. Shurenkov. Russ. 37,692, July 31, 1934. Construction details.

Centrifugal separators. Herbert Schulz. Brit. 436,895, Oct. 21, 1935. A centrifuge for sepg. solids from liquids has an inner drum on a spindle arranged at right angles to the axis of the outer rotary casing.

Centrifugal separators. National Acme Co. Brit. 436,550, Oct. 14, 1935. A separator and its driving motor are arranged vertically in sep. housings and a fractional floating connection comprising a pair of horizontal surfaces is arranged between the 2 shafts. The machine is primarily intended to sep.  $H_2SO_4$  from distillate in the refining of crude oil.

Centrifugal separator for classifying solid particles in gases. International Precipitation Co. Brit. 436,491, Oct. 11, 1935.

Centrifugal machines. Gutehoffnungshütte Oberhausen A.-G. Brit. 436,496, Oct. 11, 1935. The hollow bodies or nozzles for the discharge of liquids from sludgy material are situated at parts of the drum wall that are at a greater distance from the axis of rotation than the other parts of the wall.

Centrifugal machines. Vickers Ltd. and Charles J. Salisbury. Brit. 436,915, Oct. 21, 1935. In a centrifuge of the kind used for the treatment of paper pulp and which discharges material from an intermediate layer in the bowl, a blade not moving with the bowl extends through the free surface of the material adjacent the outlet.

Pneumatic separators of the type in which the materials are distributed in a rising air current by a rotating plate. Ernst C. Loesche and Max Berz. Brit. 436,566, Oct. 14, 1935.

Magnetic separators. Herbert H. Thompson and Alfred E. Davies. Brit. 436,138, Sept. 26, 1935. Divided on 432,431 (C. A. 30, 331).

Gravity-separation apparatus. Frederick A. F. Crawford, Wm. A. P. Challenger and Imperial Chemical Industries Ltd. Brit. 435,615, Sept. 19, 1935. Liquid nitric esters, e. g., nitroglycerine, are sepd. from acids used in their manuf. or washing liquids used in their purification by passage through a vessel fitted with a continuous baffle in the form of a strip wound edgewise and with a radial inclination around a vertical axis so that the outer edges of the turns touch or are close to the wall of the vessel.

Strainer suitable for use with aqueous or oily liquids. Howard D. Phillips. U. S. 2,028,520, Jan. 21. Structural details.

Filtering gases. Società italiana Pirelli. Brit. 435,108, Sept. 16, 1935. Auxiliary neutral granular substance is introduced into a previously formed felt of fibrous material, e. g., by depositing a layer of material on the felt and shal-

ing, or by immersing the felt in a soln. or suspension of the neutral substance. Cf. C. A. 30, 909.

Separating materials of different specific gravities. Henry M. Chance. Can. 354,170, Nov. 19, 1935. Materials of different sp. grs. are suspended in comminuted form in a liquid. The heavier materials sink to the bottom and the lighter materials continually accumulate on top. The upper portion of the light materials is forced into the atm. and discharged. Cf. C. A. 29, 1285.

Separating acetylene from gaseous mixtures. Heinrich Schilling and Robert Stadler (to I. G. Farben A.-G.). U. S. 2,029,120, Jan. 24. For sepg.  $C_2H_2$  from a gas mix. such as one contg. a large proportion of  $H_2$ , the  $C_2H_2$  is absorbed in a liquefied substance such as liquefied  $NH_3$  or  $SO_2$ , which under atm. pressure is gaseous at  $15^\circ$  and condensable at a temp. above  $-100^\circ$ .

Humidifiers for bakers' ovens or other apparatus. The Gas Light and Coke Co., Wm. Dieterichs and Peter Lloyd. Brit. 436,599, Oct. 16, 1935.

Apparatus for heating and humidifying air. Kinky C. Wright. U. S. 2,029,208, Jan. 28. Structural and operative details.

Air-conditioning apparatus. George R. Goldthwaite (to B. F. Sturtevant Co.). U. S. 2,029,368, Feb. 4. Various structural and operative details.

Air-conditioning and water-heating system. Charles O. Knudsen. U. S. 2,029,574, Feb. 4. Various structural and operative details.

Air-conditioning and water-cooling apparatus. John C. Wichmann. U. S. 2,028,814, Jan. 28. Structural, mech. and operative details.

Air-conditioning and refrigerating system. Robert E. Keyes (to Cooling & Air Conditioning Corp.). U. S. 2,030,032, Feb. 4. App. and operative details are described.

Air-cooled condensers suitable for refrigerating apparatus. Delbert F. Newman (to General Elec. Co.). U. S. 2,029,890-1, Feb. 4. Structural features.

Heat exchanger suitable for use of furnace waste gases for heating water or air. Heinrich C. Trudsen (to Liesen & Co.). U. S. 2,029,450, Feb. 4. Structural details.

Tubular heat-exchange apparatus suitable for heating water, etc. Thomas L. Murray (to Metropolitan Engineering Co.). U. S. 2,029,437, Feb. 4. Structural details.

Tube and header heat-exchange apparatus suitable for reclaiming heat from waste waters. Lmule J. Parent and John L. Krieg (to Alco Products Inc.). U. S. 2,028,471, Jan. 21. Structural details.

Conveyer for heat treating furnaces. Clarence W. Spicer. U. S. 2,028,479, Jan. 21. Structural and mech. details.

Tube and header heat-exchange apparatus. Wilbur H. Armacost (to Superheater Co.). U. S. 2,029,234, Feb. 4. Structural details.

Tube and header device for cooling engine lubricants. Edwin A. Chambers. U. S. 2,029,057, Jan. 28. Structural and operative details.

Tube and header heat exchanger or cooler suitable for use with volatile liquid refrigerants. Arthur R. Hemphill. U. S. 2,028,213, Jan. 21. Structural details.

Apparatus for exchanging heat between solid and liquid or gaseous substances. Lepol Internationale Patent-verwertungs-G. m. b. H., Ger. 620,620, Oct. 24, 1935 (Cl. 80c 14-10).

Carrying out exothermic catalytic gas reactions such as oxidation of sulfur dioxide. Joseph Bayer, Alfred Halmteier and Karl Hencky (to I. G. Farben A.-G.). U. S. 2,029,604, Feb. 4. Various operative and structural details of a tube and header app. are described.

Temperature regulation of catalytic reaction chambers or converters. Walter Pohl (to Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler). U. S. 2,028,684, Jan. 21. A fluid stream such as one of  $C_2H_2$  and steam is continuously circulated in a closed path, a minor portion of the stream being fed to the catalyst while kept in indirect heat-exchange relation with the circulating stream, and fresh reactants are supplied to the stream to compen-

state for the portion of the stream passed to the catalyst. App. is described.

Furnace suitable for heating materials for drawing or tempering. LeRoy A. Lindberg (to Lindberg Engineering Co.) U. S. 2,029,176, Jan. 28 Various structural and operative details

Condenser with pipes and headers Herbert C. Guild. U. S. 2,029,422, Feb. 4 Structural features.

Roasting furnace with alternate fixed and movable hearths X. de Spirel Belg 407,808, March 30, 1935 Mech features

Furnace and associated apparatus for melting material for "mineral wool" manufacture, etc Lee R. Campbell U. S. 2,029,307, Feb. 4 Structural and operative details

Oil-burning apparatus suitable for furnaces Arthur E. Paige U. S. 2,029,184, Jan. 28 Structural and operative details

Apparatus for supply of fluids under pressure as in supply of compressed air under a desired pressure Max H. Sussin (one-half to Frank L. Barchard) U. S. 2,029,085, Jan. 28 Various structural, mech, elec and operative details

Pump for compressed liquefied gases such as propane or butane Claude F. Tears (to Petroleum Processes Corp.) U. S. 2,028,531, Jan. 21 Structural and operative details

Pump for viscous liquids A. S. Svistunov Russ 33,922, Jan. 31, 1934. An arrangement used in the transfer of viscous liquids, such as for example heavy fuel oil transported in railroad tanks, consists of a steam pump (a portable jacketed unit of steam engine and pump), and a steam coil for heating the liquid

Hand pump for viscous fluids such as oils Rudolph D. Maulis U. S. 2,028,912, Jan. 28 Structural details

Pump and associated apparatus for taking samples of petroleum or other liquids from tanks Augustus W. Gleason and Ernest L. Moyer. U. S. 2,029,231, Jan. 28 Structural and operative details.

Pipe suitable for conveying liquids Charles A. Hill (to Mueller Brass Co.) U. S. 2,029,424, Feb. 4 The smooth inner surface of a pipe which may be formed of Cu or brass produced by piercing, drawing or extrusion is treated with an abrasive to produce irregularities throughout the entire inner surface of the pipe, for retaining a film of liquid on the interior surface of the pipe, so that the coeff. of friction between the wall of the pipe and the mass of moving liquid in the pipe is reduced. App is described

Apparatus for dispensing liquids R. K. Galadzh Russ 34,332, Jan. 31, 1934 Construction details

Apparatus for charging containers with inflammable liquids to a given level. A. G. Lukatzki Russ 34,331, Jan. 31, 1934. Construction details of an enclosed electric mercury switch

Apparatus for drawing samples of liquids from reservoirs L. I. Fundator Russ. 34,198, Jan. 31, 1934 Construction details.

Apparatus for drawing samples of liquids F. U. Baloushev. Russ. 34,199, Jan. 31, 1934. Construction details

Dispensing gases in liquids. Allgemeine Elektrizitäts-Ges. Fr. 789,369, Oct. 28, 1935 The gas is highly diff. with the vapor of the dispersing liquid or the vapor of a liquid miscible with the dispersing liquid.

Apparatus for bringing two liquids into intimate contact. N. V. de Bataafsche Petroleum Maatschappij. Fr. 789,632, Nov. 4, 1935. The 2 liquids flow in countercurrent in a vertical column and are beaten up by a gas to produce turbulence without preventing the counter flow. Cf. C. A. 29, 81.

Bringing different liquids into contact as in treating lubricating-oil stock with selective solvents. Robert E.

Wilson (to Standard Oil Co. of Ind.) U. S. 2,029,690, Feb. 4. Various structural and operative details are described relating to the use of a horizontal inclined app. with partitions and agitating devices, with countercurrent flow of the contacting materials and having zones for vigorous agitation and zones of relative quiescence.

Apparatus for countercurrent contact of materials such as oil stocks and selective solvents Jack Robinson (to Standard Oil Co. of Ind.) U. S. 2,029,691, Feb. 4. Various details of an inclined app. with partitioning baffles and agitators.

Countercurrent contact of materials such as oils and selective solvents such as bis(chloroethyl) ether Robert E. Wilson (to Standard Oil Co. of Ind.) U. S. 2,029,687-8, Feb. 4 Various structural and operative details of a horizontal inclined app. with internal partitions and agitating devices

Distributing and treating liquids The Sharples Specialty Co. Fr. 789,450, Oct. 29, 1935. In feeding an oil mixed with a chem. reagent, means is provided to prevent obstruction of the pipes by the viscous mud formed.

Apparatus (with a rotatable drum) for pulverizing materials such as coal in an air-swept ball mill Martin Frisch (to Foster Wheeler Corp.) U. S. 2,029,917, Feb. 4 Various structural, mech and operative details.

High-pressure joints for pressure vessels Kurt Bredtschneider U. S. 2,029,606, Feb. 4 A thin elastic ferrous metal packing ring is used having a modulus of elasticity of the order of 30,000,000, various structural details being described Various alloy steels, etc., may be used

Safety device for gas lines I. N. Maizel and N. E. Zhuravle-Prokof'ev Russ 34,249, Jan. 31, 1934. Construction details of a pressure release valve

Apparatus for superheating steam. Heinrich Peperhorn (to Superheater Co.) U. S. 2,029,384, Feb. 4 Structural details

Vacuumizing apparatus Thomas M. Rector (to General Foods Corp.) Can 355,195, Jan. 7, 1936 Structural details are described

Apparatus for cooling effervescent liquids Leo Samel. Can 354,498, Dec. 3, 1935 Structural details

Apparatus for solidifying carbon dioxide. Maschinenfabrik Surth Zweigniederlassung der Gesellschaft für Linde's Eismaschinen A.-G. Brit. 434,999, Sept. 12, 1935 Addn. to 404,833 (C. A. 28, 5153).

Apparatus for testing the hardness of materials. Fay H. Willey. Brit. 436,565, Oct. 14, 1935.

Impact device for testing surface finishes. Robert Burns (to Bell Telephone Laboratories, Inc.) U. S. 2,028,190, Jan. 21. Various mech and operative details.

Devices for breaking up foam in washing machines and in machines for the distillation of tar or for brewing fermented liquors. Richard Burslem. Brit. 435,489, Sept. 23, 1935.

Apparatus for cleaning pipe lines Ralph W. Howe, Robert E. Love and Joseph H. Wood, Jr. (to Atlantic Pipe Line Co.) U. S. 2,028,779, Jan. 28 Mech. and operative details.

Apparatus for use of abrasives and liquids in cleaning conduits such as beer coils Elmer O. Fessler. U. S. 2,028,972, Jan. 28. Various structural and operative details

Apparatus for washing tanks such as those used for transporting oil. William Ostling (to Pyrate Corp. of Nevada). U. S. 2,029,788, Feb. 4. Various structural, mech. and operative details

Apparatus for cleaning tanks such as those used for transporting oil. Clare Richard (to Pyrate Corp. of Nevada). U. S. 2,029,795, Feb. 4. Various structural, mech. and operative details, of an app. for cleaning tanks by jets of detergent fluid.

## 2-GENERAL AND PHYSICAL CHEMISTRY

FREDERICK L. BROWNE

Bobuslav Brauner (1885-1935) His life and works  
A Dorabialka *Roczniki Chem* 15, 415-21 (1935)

A Hantzsch *Fr Hein Z. Elektrochem.* 42, 1-4  
(1936).—Obituary with portrait

Bibliography for general chemistry from several periodicals  
I Chemical and Metallurgical Engineering  
Hubert N Alyea *J. Chem. Education* 13, 70-81 (1936)

Laboratory preparation of hydrofluoric acid. Arnold  
Bookhenn *J. Chem. Education* 13, 93 (1936)

Early zinc works in the Lehigh Valley R. D Billinger  
*J. Chem. Education* 13, (10-2) (1936).

Measuring the ability to interpret experimental data  
F P Frutchey, R W Tyler and B. Clifford Hendricks  
*J. Chem. Education* 13, 62-4 (1936)

Proficiency of first-year students in quantitative experiments  
Bruce S Iarquhar and Francis Earl Ray. *J. Chem. Education* 13, 74-5 (1936).

Applicability of the lecture demonstration method to certain groups of students Albert L Elder *J. Chem. Education* 13, 65-8 (1936).

A chapter in teaching acids, bases and salts P M Glasco  
*J. Chem. Education* 13, 69-71 (1936).

Analysis of the dust that fell on April 26, 1928 Al St Cocosinchi  
*Ann ses univ Jassy* 20, 116-17 (1935).—  
A criticism and discussion of an analysis by Cernatescu  
(*C A* 26, 4511). R Cernatescu *Ibid* 173-4.—Cernatescu defends his analysis

Construction of platinum thermometers and determination of their basic points W H. Keesom and B G Dammers  
*Physica* 2, 1071-8 (1935).—The construction and calibration of 5 Pt resistance thermometers for use in the range -190° to 600° are described The optimum annealing temp was 840° to 850°. For each of the thermometers, the value of the resistance was detd at the normal b p. of O (taken as -182.98°, in conformity with a new detn.), the ice point, the steam point and the b p. of S Each of the thermometers fulfilled the following conditions  $R_{100}/R_0 \geq 1.39$ ,  $R_{100}/R_0 \geq 2.645$ ,  $R_{100}/R_0 \geq 1.02$

Comparison of some platinum thermometers with the helium thermometer between 0° and -183° W H Keesom and B G Dammers  
*Physica* 2, 1080-90 (1935).—Five Pt resistance thermometers (cf preceding abstr.) (I and II, wire from Hereaus, III, wire from the National Bureau of Standards, IV, wire from Johnson and Matthey, V, wire from Hereaus), satisfying the conditions stipulated in the definition of the international temp scale, were compared with the He gas thermometer between 0° and -183°. A cryostat precise to  $\pm 0.002^\circ$  is described I, II and III agree within  $\pm 0.003^\circ$ , IV was not compared directly with the others but agrees with them within the reading errors of the He gas thermometer, V differs from I, II and III by  $\pm 0.03^\circ$  at -183°. Between 0° and -100°, the international temp scale lies below the thermodynamic temp scale, the max difference being about  $0.04^\circ$  at -80°. Between -120° and -183°, the international temp scale lies above the thermodynamic scale, the max difference being about  $0.01^\circ$  to  $0.02^\circ$  at -130° to -140°

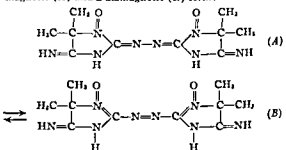
A macro method for melting-point determination Martin Mezeuer and Siegfried Wehrli  
*Helv Chim Acta* 18, 1281-3 (1935).—Because the ordinary microscopic m.p. detn is inadequate for certain substances that sublime, a sealed tube is recommended With a diam of 0.1-0.5 mm. only a few g is required The sample is observed by a low-powered microscope and is heated in a Thiele tube Values agree within  $1^\circ$  with those by the macro method

Rotatory dispersion of aliphatic aldehydes P A Levene and Alexandre Rothen  
*J. Chem. Physics* 4, 48-52 (1936); cf *C A* 27, 5598, 28, 7397, 7094, 30, 4307.—Rotatory-dispersion curves of configurationally related

aldehydes of the type  $\text{HMeEtC}(\text{CH}_3)_n \text{CHO}$  ( $n = 0, 1, 2$  or 3) were detd in the visible and ultraviolet regions of the spectrum Their analysis shows (1) that the band of the aldehydic group at  $\lambda 2930$  is rotatory active and (2) that the sign of its contribution in the first member ( $n = 0$ ) is opposite that of the higher members and (3) that the magnitude of that contribution varies periodically with  $n$ , the no. of C atoms between the aldehydic group and the asym. C atom.

Magnetochemical investigations XVIII  $\text{K}_2(\text{B}_2\text{H}_4)$  and  $\text{K}_2(\text{B}_2\text{H}_4)(\text{OH})_2$  Li Klemm and Wilhelm Klemm  
*Z anorg allgem Chem* 225, 258-61 (1935), cf *C A* 30, 3397.—To decide whether the salt-like product formed by addn of an alkali metal to  $\text{B}_2\text{H}_4$ , having the empirical formula  $\text{KBH}_4$ , gives  $(\text{BH}_4)^-$  or  $(\text{B}_2\text{H}_4)^{2-}$  ions, magnetic measurements were made at temps from 90° to 353°K These show that the compd is diamagnetic Hence, according to the theoretical considerations outlined, the ions are overwhelmingly  $(\text{BH}_4)^-$ . Measurements at room temp indicate that the second compd is diamagnetic and hence has the formula  $\text{K}_2(\text{B}_2\text{O}_2\text{H}_4)$  XIX The magnetic properties of potassium polyoxide and polysulfide Wilhelm Klemm and Heinrich Sodomann  
*Ibid* 273-80.—The susceptibilities of 3 preps of  $\text{K}_2\text{O}$  were measured at 20°, -78° and -183°. Analysis of the results indicates that it is not possible to decide from magnetic measurements whether the highest oxide of K has the formula  $\text{KO}_2$  or  $\text{K}_2\text{O}_4$ . Measurements were made on  $\text{K}_2\text{S}$ ,  $\text{K}_2\text{S}_2$ ,  $\text{K}_2\text{S}_3$ ,  $\text{K}_2\text{S}_4$  and  $\text{K}_2\text{S}_5$  at room temp and at -183°. These compds were all diamagnetic at room temp At low temps  $\text{K}_2\text{S}_5$  showed certain peculiar effects and appeared to be ferromagnetic below -50°

Magnetochemical investigations of organic substances IV. The magnetic behavior of free radicals Eugen Muller, Ilse Muller-Rodloff and Wilhelm Bunge  
*Ann* 520, 235-55 (1935), cf *C A* 30, 3367.—The extent and the heat of dissociation of free radicals can be calcd from their measured magnetic susceptibility The compd of Kenyon and Banfield (*C A* 20, 2837) follows Currie's law, as do di- $\beta$ -amylnitrogen oxide ( $\text{MeOC}_2\text{H}_4\text{N}=\text{O}$  and  $\alpha,\alpha$ -diphenyl- $\beta$ -trimetaphenylhydraeryl,  $\text{Ph}_2\text{N}-\text{N}-\text{C}(\text{H})_2(\text{NO}_2)$  Tributylmethyl (I) forms an addn product (II) with 1 mol  $\text{PhI}$ , which decomposes at 160°. I and II show a magnetic susceptibility corresponding to complete dissociation into free radicals V The magnetic behavior of porphyrins and porphyrindine Eugen Muller and Ilse Muller-Rodloff *Ibid* 521, 81-9.—The paramagnetic susceptibility of porphyrindine (III) was detd, from 90° to 294°K, leading to a value of  $\Delta = -6 \pm 5$ , as against  $\Delta = -12^\circ$  reported by Kuhn, Katz and Franke (*C A* 29, 2173). The magneton factor is constant, showing that III behaves normally Measurements on porphyrindine from 90° to 348°K agree with the above authors' from 273° to 343°, but show at low temps a sharp deviation from Weiss' law This deviation can be accounted for quantitatively by the assumption of an equal between a paramagnetic (A) and a diamagnetic (B) form.



with heat of transformation of 0.56 cal VI A new

method of measurement of the dissociation of hexaarylethanes *Ibid.* 89-94—The degree of disocion and the disocion const of hexaphenylethane are calcd from measurement of the magnetic susceptibility of its solns. The degree of disocion is slightly lower than that found by optical methods, while the heat of disocion is the same.

George Calinaert

The effect of silicic acid on the magnetic properties of dissociated mixtures with iron oxide. W. Lettkebel and K. Bockemuhl *Z. anorg. allgem. Chem.* 225, 209-12 (1935)—Eleven mixts of  $\text{FeO}$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{SiO}_2$  in varying proportions were heated to different temps (from 1000° to 1600°) and the ratio  $\text{Fe}_2\text{O}_3/\text{FeO}$  in each of the end products was detd analytically. The temp at which the magnetism disappeared was also detd. To a first approximation the addn of  $\text{SiO}_2$  produced little effect on the magnetic properties. The results are presented in both tabular and graphic form and are correlated with the work of White, Graham and Hay (*C. A.* 29, 4712\*) on the disocion of the  $\text{Fe}_2\text{O}_3\text{-FeO}$  system, and the magnetic investigations of Kleffner and Kohlmeier (*C. A.* 26, 4005).

W. W. Stiller

Surface magnetization and block structure of ferrite. W. C. Flomere and L. W. Keehan *Am. Inst. Mining Met. Engrs., Inst. Metals Div., Tech. Pub. No. 656*, 15 pp. (1935). cf. *C. A.* 28, 7090\*.—Surface magnetization of polished ferrite crystals with or without  $\text{Si}$  was investigated, the magnetic-powder method was used. The use of a true colloid of  $\gamma\text{-Fe}_2\text{O}_3$  in water improved the powder technic. The maze-like patterns, and the changes in these patterns brought about by suitably changing the magnetizing field, led to the proposal of a block model of cubes spontaneously magnetized along  $\langle 100 \rangle$  or  $\langle 110 \rangle$  axes. A study of patterns on various cuts of the same crystal gave addnl evidence for this structure. The size of the cubes varied from  $0.5 \mu$  to  $3 \mu$ , depending on the particular crystal. The stability of the secondary magnetic structure with respect to the polishing procedure adopted and to magnetic history was shown. It was suggested that the segregation of foreign atoms may account for the observed stability of the block boundaries.

C. L. Mantell

Magnetic rotatory power and dispersion of alkali mercuritetratiodides. René Lucas and Fernand Gallais *Compt. rend.* 202, 129-31 (1936); cf. *C. A.* 29, 2874\*.—The exceptionally high values of Verdet's const. and of the dispersion for  $\text{K}_2\text{HgI}_4$  are due to a very strong absorption band starting from  $\lambda$  2900.

C. A. Silberrad

The magnetic susceptibility of organic liquids. Application to the additivity law. Constantin Silceanu and Dumitru Gheorghiu, *Bull. soc. roumaine phys.* 36, 77-81 (1934), cf. *C. A.* 29, 1693\*.—Mixts of  $\text{Me}_2\text{CO}$  with  $\text{PhNO}_2$  and with  $\alpha$ -methyl-naphthalene obey the additivity law. Deviations in other cases may be accounted for by a chem. change.

R. L. DeRight

Magnetic susceptibility of alkali and alkaline earth halides. Ulrich Viel *Ann. Physik* 24, 697-713 (1935).—The susceptibilities of the alkali and alkaline earth halides, and of  $\text{HCl}$ ,  $\text{HBr}$  and  $\text{HI}$ , in aq. solns were measured for various concns. at room temp and reduced to 20°. For most of the solns,  $\chi$  concns up to 25% by weight were studied, and for  $\text{KI}$  the max. concn was 57%. In all cases the susceptibility is a linear function of the concn as required by the Wiedemann additive law. From the results on solns the susceptibilities of the salts and of the ions are calcd. and the results are compared with those of other investigators. The variation of the susceptibilities of  $\text{NaCl}$  and  $\text{KI}$  solns with temp. was investigated in the region 0° to 50°. Various points of theoretical interest, such as the dependence of the susceptibility of an ion on its nuclear charge no., are discussed.

W. W. Stiller

Ferromagnetic conversion and catalytic activity. IV. Hydrogenation of carbon monoxide and ethylene over nickel and carbon dioxide formation from carbon monoxide over the Heuser alloy,  $\text{MnAlCu}$ , J. Arvid Hedvall and Rune Hedlin, *Z. physik. Chem.* B30, 280-8 (1935), cf. *C. A.* 30, 360\*.—The rates of the reactions change discontinuously at the Curie point when the ferromagnetic prop-

erties of the catalyst disappear. A com. thermal cond. app (cf. *C. A.* 26, 5272) was used successfully for analysis of the  $\text{CO-CO}_2$  mixt.

G. M. Murphy

Behavior of an ion cloud about a dipolar molecule under the influence of an alternating field. Shōten Ōka, *Proc. Phys.-Math. Soc. Japan* 17, 454-66 (1935) (in German).—Math. The ionic cloud about a dipolar mol under the influence of an external alternating elec. field (of frequency  $\omega$ ) was studied from the standpoint of the Debye, 2 interionic theory. Because of the final relaxation time  $\theta$  the ion cloud has a moment of rotation, which the central dipole decreases in its rotary path, entirely analogous to the decrease of the relaxation energy of the central ion in its path. The moment acts in the sense of a diminution of the dielec. const. of solns. of electrolytes and vanishes for high values of  $\omega$ .

G. M. Petty

Dielectric loss characteristics of a chlorinated biphenyl. Willis Jackson, *Proc. Roy. Soc. (London)* A153, 158-66 (1935), cf. *C. A.* 29, 4945\*.—Dielec. power factor-temp. curves are given for a chlorinated biphenyl (about 4 Cl per mol) for 8 frequencies between 50 and  $10^7$  cycles per sec at temps from  $-15^\circ$  to  $+80^\circ$ . The temp of the max. power factor (about 21.5% in each case) increases from  $-5^\circ$  for 50 cycles to  $+40^\circ$  for  $10^7$  cycles and the curves are so sharp that a  $20^\circ$  change in temp. reduces the power factor to less than 1% so that max. dielec. const. and low dielec. loss can be attained by suitable choice of temp. and frequency. The results are discussed in terms of the Debye theory. There can no longer be doubt that the necessary conditions for the appearance of a Debye power factor max. at frequencies as low as 50 cycles can arise at normal temps.

Janet F. Austin

Electrical investigation of solutions of stearamide in paraffin wax. W. Jackson and F. C. Frank, *Trans. Faraday Soc.* 31, 1700-6 (1935), cf. *C. A.* 29, 4985\*.—A series of power factor measurements was made on dil. solns of stearamide in 57-60° paraffin at frequencies  $2.7 \times 10^4$  to  $1.35 \times 10^7$  cycles/sec over the temp. range  $0-80^\circ$ . D. c. cond. measurements were made in this range. The results are interpreted by assuming the presence in soln. of polar groups of about 10 stearamide mols in a group, probably in the form of liquid crystals. The groups appear capable of adsorbing ionized impurities.

C. E. P. Jeffreys

Field strength and frequency dependence of dielectric constants of anisotropic liquids. W. Kast, *Physik Z.* 36, 809-73 (1935).—The results of Jezewski, cf. *C. A.* 23, 25, on *p*-azoxyanisole were analyzed. The decrease of dielec. const. with increasing field strength shows a sat. value well above the optical value. Only a mean moment of the large mol. groups is concerned. The dipoles inside the groups remain sufficiently movable to give only partial orientation on polarization. Increasing frequency as well as field strength must be taken into account in consideration of the decrease of dielec. const.

C. E. P. Jeffreys

Dielectric coefficients of volatile compounds of fluorine and boron. K. L. Ramaswamy, *Proc. Indian Acad. Sci.* 2A, 364-77 (1935).—The dielec. coeffs of  $\text{CF}_4$ ,  $\text{NF}_3$ ,  $\text{CHF}_3$ ,  $(\text{CF}_3)_2\text{N}$ ,  $\text{B}_2\text{H}_6$  and  $\text{B}_2\text{N}_2\text{H}_4$  (in the vapor state) were measured at different temps. to obtain the values for their elec. moments, the values being 0, 0.21, 1.59, 0.46, 0 and  $0.67 \times 10^{-18}$ , resp. Compressibilities at ordinary temp. were measured accurately by refractometric studies and approx. at low temps. by studying variations of dielec. coeff. The significance of the moments is discussed.

L. L. Quill

Electric moments of *p*-quinone and related compounds. D. L. Hammick, G. C. Hampson and G. I. Jenkins, *Nature* 136, 990-1 (1935).—Elec. moments were detd for *p*-benzoquinone in  $\text{C}_6\text{H}_6$ ,  $\text{CCl}_4$  and  $\text{C}_6\text{H}_5$  solns., and for 2,5-dimethyl-1,4-quinone, 2,6-dichloro-1,4-quinone and tetramethylcyclobutane-1,3-dione in  $\text{C}_6\text{H}_6$  soln. The values were all in the range  $0.64-0.73 \times 10^{-18}$  e. s. u. These values are anomalous in view of the sym. structure usually assigned to these compds. and may be due to solvent effect or to an abnormally large atom polarization.

G. M. Petty

The conductivity of calcium, strontium and barium

chlorides in anhydrous glycerol. J. Szper and Z. Gajewski. *J. chim. phys.* 32, 705-14 (1935).—See C. A. 29, 6127.

The electric conductivity of metallic vapors under pressure. Werner Braunbek. *Z. Physik* 97, 482-95 (1935).—On the basis of Bloch's theory of elec. cond. the cond. of a metallic vapor and its dependence upon  $p$  can be estd approx. Vapors of Hg and K were measured. Values found for K vapor are at  $1200^\circ$   $10^{-10} \Omega^{-1} \text{cm}^{-1}$  at a pressure of 1 atm and  $10^{-11} \Omega^{-1} \text{cm}^{-1}$  at 10 atm. Possible existence of metallic cond. at higher pressure is not completely excluded by the expts. The evidence obtained from Hg up to 35 atm and on K up to 10 atm was, however, neg. In Hg the metallic cond. amounted to less than  $10^{-11} \Omega^{-1} \text{cm}^{-1}$  and in K to less than  $10^{-12} \Omega^{-1} \text{cm}^{-1}$ . The lower limit of measurement was detd. by thermal emission and ionization effects. S. Tolansky

Electric conductivity of alkali metal flames. Willy Kisselmann and A. Becker. *Ann. Physik* 25, 49-73 (1936).—The cond. of illuminating gas-air- $N_2$  flames alone, and contg. chlorides of Li, Na, K, Rb and Cs injected in soln. in known amts., was measured with precision in the range 1250-1950°K. The temp. variation of cond. of the pure flame corresponds to an av. of 2.5 v. for the formation of current carriers. For injected flames the cond. is proportional to the square root of the total metal concn. of the flame. The temp. variation of cond. does not change with concn. of metal in the range studied ( $10^{-4}$ - $10^{-2}$  normal). A complete theoretical explanation of the variation of cond. with the nature of the alkali metal is not possible, however, the approximation of expt. to theory is better, the higher the temp. and thus the more complete the dissociation into free metal atoms.

Comparative measurements of the conductivity of the Bunsen flame with direct and alternating currents. H. Ullmann. *Z. Physik* 97, 496-510 (1935).—The resistance of a condenser in the Bunsen flame is measured for an oscillation frequency  $1.50 \times 10^6 \text{ sec}^{-1}$  and the cond. calcd. This is compared with the value given by d-c methods. The a-c method attains the same degree of accuracy as the d-c method. Conclusions are drawn regarding how far the disturbances produced by the electrodes reach into the condenser. Values are given for the flame cond. with a pure flame and with one contg.  $Na_2CO_3$ .

Superconductivity in the light of accepted principles. H. Grayson Smith. *Trans. Roy. Can. Inst.* 20, 305-33 (1935). cf. C. A. 30, 666.

The Joule-Thompson effect of methane, nitrogen and mixtures of these gases. John H. Perry and Carl V. Hermann. *J. Phys. Chem.* 39, 1189-95 (1935).—The Joule-Thompson coeffs. of  $CH_4$  and  $N_2$  are calcd. for the temps., 200°, 250°, 300°, 350° and 400°K. and the pressures, 1, 5, 25, 50 and 100 atms., with the use of the Beattie-Bridgeman equation of state. A method is indicated for obtaining the coeffs. for mixts. of gases from those of the pure gases. Data are tabulated for 3 mixts. of  $CH_4$  and  $N_2$ .

P-V-T relations of gaseous mixtures. E. R. Gilliland. *Ind. Eng. Chem.* 28, 212-15 (1936).—The slope of the isometric of a mixt. can be calcd. by the molal av. of the slopes of the isometrics of the pure constituent gases, all measured at the same molal concn. This rule, together with a method for estg. the internal pressure, furnishes a means of predicting the P-V-T properties of the mixt. For the 3 gaseous mixts. given, the predicted values agree better with exptl. data than do values calcd. from Amagat's or Dalton's law, and is probably within the exptl. error of the data. The 3 mixts. were  $A-C_2H_6$ ,  $H_2$ -CO and  $CH_4$ - $N_2$ .

Isotherms of carbon dioxide between 0° and 150° and pressures from 16 to 250 atmospheres (Amagat densities 18-206). A. Michels and Mrs. C. Michels. *Proc. Roy. Soc. (London)* A153, 201-14 (1935).—The Amagat  $d_s$  and the  $p_0$  values for the isotherms of  $CO_2$  between 0° and 150°, at pressures from 16 to 250 atms. are tabulated and ex-

pressed mathematically. In this range the sp. heat,  $c_p$ , increases with  $d$ .

Isotherms of carbon dioxide between 70 and 3000 atmospheres (Amagat densities between 200 and 600). A. Michels, Mrs. C. Michels and H. Wouters. *Proc. Roy. Soc. (London)* A153, 214-24 (1935); cf. preceding abstr.—Tables in the previous paper are extended to include the values for pressures up to 3000 atm. The sp. heat,  $c_p$ , of  $CO_2$  increases with  $d$ . to a max. at  $d = 226.7$  Amagat units, then decreases and passes through a min. at  $d$  between 390 and 460 Amagat units.

Theory of the equation of state. Satyendra Ray Kollond-Z. 73, 269-72 (1935); cf. C. A. 26, 1172.—A modified Dieterici equation  $y = k + ax$ , where  $y = \log(p_0/dT)$ ,  $k = \log_e(R/M)$  and  $x = 1/p_0T$  ( $d = d$ ,  $R = \text{mol. gas const.}$  and  $M = \text{mol. wt.}$ ) was applied to  $H_2O$  and  $HCl$  in the vapor phase only and to  $NH_3$ ,  $EtOH$  and bromobenzene in the liquid and vapor phases. At conditions far removed from the crit. point, the  $y-x$  curve is a straight line for both liquid and vapor; near the crit. point it is a hyperbola, showing that  $d$  is not const. in this region. The  $y-x$  curve for water vapor shows a marked discontinuity in the region of 2.5 atm. and 115°.

The theory of liquids. V. T. S. Wheeler. *Proc. Indian Acad. Sci.* 2A, 466-76, cf. C. A. 29, 6810.—The derivation and application of the principal equations of the theory are outlined together with the method of calcn. of the properties of a liquid if the temp. and the fundamental properties of the mol. are known. J. E. Austin

Vapor pressure of certain glycols. O. J. Scherholts and M. L. Staples. *J. Am. Chem. Soc.* 57, 2709-11 (1935).—The vapor-pressure curves over the range 10-760 mm. were detd. for ethylene, 1,2-propylene, 1,3-propylene, 1,3-butylene and 2,3-butylene glycols. With the exception of  $(CH_2(OH))_2$ , all data are reported for the 1st time. The  $d_s$  and  $w_p$  are given for all compds., and in addn. the methods for the prepn. of 1,2-butylene and 2,3-butylene glycols are described.

Vapor pressures of some alkyl sulfides. H. W. Thompson and J. W. Linnett. *Trans. Faraday Soc.* 31, 1743-7 (1935).—Vapor-pressure curves for  $EtHS$ ,  $Me_2S$ ,  $Et_2S$  and  $Me_3S$  were detd. The log  $p$ - $1/T$  curves were satisfactory straight lines. The  $b$  ps (760 mm) and heats of vaporization were found to be  $Me_3S$ , 37°, 6910 cal.;  $Me_2S$ , 65°, 7250 cal.;  $Et_2S$ , 91.4°, 8000 cal.;  $EtHS$ , 33°, 6860. Estn. of the Trouton const. indicates that tho. ethers and thio alcs. are normal unassoc. liquids.

The accuracy of viscosity measurements by the Engler method. F. H. Garner and W. E. J. Broom. *Rev. Petroliere No.* 647, 1204-5 (1935); *J. Inst. Petroleum Tech.* 22, 27-31 (1936).—Engler viscometers are less accurate than those by Redwood and Saybolt. This is due to the unsatisfactory official German standardization methods, whereby viscometers having deviations amounting to  $\pm 2\%$  are passed.

The viscosity of deuterium oxide and its mixtures with water at 25°. Grinnell Jones and Holmes J. Fornwalt. *Phys. Rev.* 44, 30-3 (1938).—The viscosity and  $d$  of  $H_2O$ - $D_2O$  mixts. are related by the equation  $\phi = 1/\eta = 1 - 2.00855(\Delta s) + 3.1122(\Delta s)^2$ ; ( $\Delta s = d_{11}^2 - 1$ ). Bingham's equation for additive fluidities has the form  $\phi = 111.94 + 15.611b - 36.538b^2$ , and  $b$  are the vol. and wt. fractions of  $D_2O$ , resp. The 2 equations agree with the data to 0.006 and 0.01%, resp. The La Mer-Baker formula relating mole fraction to sp. gr. (C. A. 29, 900°) is corrected for the  $D_2O$  in ordinary water. If it is assumed  $w_{D_2O} = 0.8337$  centipoises, at 25°  $\eta_{D_2O} = 1.0092$ ,  $\phi_{D_2O} = 90.957$  rebs.

Determination of the ultrasonic velocity in fifty-two organic liquids. S. Parthasarathy. *Proc. Indian Acad. Sci.* 2A, 497-511 (1935).—The ultrasonic velocities at  $7.32 \times 10^6$  cycles per sec. were detd. for 52 org. liquids and water at 23-4° by the method of diffraction of light by ultrasonic waves. The velocity of sound in aromatic compds. is higher in general than in the corresponding al-

phatic compounds although the  $d_s$  of the aromatic compounds are higher. Cyclohexane and its derivatives come midway between the two classes. The velocity of sound in liquids of high  $d_s$  is not as low as the increased  $d_s$  would suggest. The effect of increased  $d_s$  is offset by a diminution of compressibility. Liquids with mols having high elec moment (e.g., alcs, ketones and water) have higher velocities of sound. Lengthening of the mol increases the velocity. The iso alcs show lower velocity than the normal. Adiabatic compressibilities for all the liquids are calcd.

Janet E. Austin  
The stabilization of the color of fufural. N. Chetverikov and M. Lifshitz. *Maslobolno-Zhivoe Delo* 1934, No. 6, 39-40.—The addn of pyrogallol (1:1000 and 1:5000) or satd. solns of NaCl and  $\text{Na}_2\text{CO}_3$  stabilizes the color of fufural for 6-12 months. E. Bielous

Liquid crystals of some cholesterol compounds, and their crystalline superfusion. Paul Gaubert. *Compt. rend.* 202, 141-3 (1936), cf. *C. A.* 30, 992.—Resorcinol, pyrogallol,  $p$ -nitrophenol (but not  $m$ - or  $o$ -), salicin, erythritol, phlorizin, saccharin, isosaccharin, rhamnose and atropthalic and acetic acids also form liquid crystals when melted with cholesterol. Ordinarily on cooling such liquid crystals pass into the solid crystal form at a definite temp, but in some cases, and especially if one component is in large excess and cooling is rapid, they form a solid, sometimes as hard as gypsum, which retains the optical properties of the liquid crystal. On reheating this may either pass into the crystal form, or simply reform the liquid crystal. C. A. Silberry

Plasticity of bismuth. W. F. Berg and L. Sandler. *Nature* 136, 915 (1935).—By use of the method of Andrade and Roscoe (*Proc. Intern. Conference on Physics* 2, 176 (1934)) crystals of Bi were grown *in vacuo*, in N, in H and in  $\text{CO}_2$ . Under all conditions some crystals were soft and so the hypothesis of Berg (*C. A.* 29, 4933) that gas was responsible for the softness is withdrawn. The hardness of Bi crystals obtained by the Bridgman method is tentatively explained as strain hardening. The soft crystals are therefore believed to exhibit the true behavior of Bi (including plasticity). Oden E. Sheppard

The change of electric resistance and reflecting power of metal mirrors condensed at low temperatures. R. Suhrmann and G. Barth. *Physik Z.* 36, 843-5 (1935).—The resistance of Cu, Ag, Cd, Ti and Pb mirrors prep'd by vaporization of the metals on plates kept at 20° or 80° abs decreased on warming to room temp and the light-reflecting power increased. Bi mirrors showed an increase in resistance on warming. This change in resistance indicates an increase in ordered arrangement into lattices.

C. E. P. Jefferys  
The coefficient of expansion of silver iodide and of the halides of thallium. Grinnell Jones and Frederick C. Jelen. *J. Am. Chem. Soc.* 57, 2532-6 (1935).—By use of a special dilatometer the coeffs. of expansion of AgI, TlCl, TlBr and TlI were det'd at 20°, 30°, 40°, 50° and 60°. The value for AgI is  $-0.016 \times 10^{-4}$  and is independent of temp. throughout this range. The thallous salts have pos coeffs. increasing as temp. increases. Coeffs. of Pyrex glass and toluene of  $9.85 \times 10^{-6}$  and  $1.111 \times 10^{-4}$ , resp., were also obtained. The assembly also functions as a thermostat regulator, thus simplifying temp. control.

R. H. L.  
Structural changes taking place upon aging of freshly prepared crystalline precipitates. V. The thermal aging of freshly prepared lead sulfate. I. M. Kolthoff and Charles Rosenblum. *J. Am. Chem. Soc.* 57, 2573-7 (1935); cf. *C. A.* 29, 4987.4.—A sepn. of microscopic aging (sintering) from internal aging was made by following the former by adsorption of wool violet and the latter by the speed of distribution of Th B through freshly ppt'd  $\text{PbSO}_4$ . Between 100° and 200° and for short periods at 305° there was indication of a small but definite increase of external surface. Sintering does not occur below 250°; it becomes pronounced only at 400°. Internal aging occurs at much lower temps with an incubation period. A discussion of thermal aging follows. Aging was greater at 250° than at 300° or even 400°. This was shown to be

due to  $\text{H}_2\text{O}$ . VI. Perfection and aging of lead sulfate precipitated under various conditions. *Ibid.* 2577-9 (1935).—The effect of varying the conditions of pptn. of  $\text{PbSO}_4$  upon perfection and speed of aging of the resulting crystals was studied with the wool violet and Th B treatment. The greater the concn. of reactants the larger is the surface of the ppt. Greater perfection of crystals occurs at lower concns. of reactants. Concn. effects are greater with regard to perfection than those of temp.

R. H. Lambert  
Experiments on piezoelectricity. Gilbert Greenwood. *Z. Krist.* 91, 235-42 (1935) (in English).—To test cases of doubtful piezoelectricity, especially where pyroelectricity has supposedly been found, a more delicate app. was devised, based on the method of Giebe and Scheibe (*C. A.* 19, 3207). The results were: picric acid,  $\text{Na citrate}$ ,  $\text{C}_2\text{H}_4\text{O}_2$ , topaz,  $\text{Li}_2\text{BeF}_4 \cdot \text{H}_2\text{O}$ ,  $\text{ZnBeF}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{C}_2\text{H}_4(\text{NH}_2)_2$ ,  $\text{H}_2\text{SO}_4$ ,  $m$ -bromocyananilide, wulfenite,  $\text{AgNO}_3$ , iodoform and  $\text{KAg}(\text{CN})_2$ , all were neg;  $\text{Li}_2\text{SO}_4$ ,  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ , 1-chlor-2,4-dinitrobenzene, santonine, brucine,  $m$ -chlorocyananilide and methylurea were pos. Of 2 scapolites from Burma, a colorless crystal was pos, and a pink one neg.

L. S. Ramsdell  
The optical anisotropy of molecular crystals as illustrated by some oxalates. Sterling B. Hendricks and W. Edwards Deming. *Z. Krist.* 91, 290-301 (1935) (in English), cf. *C. A.* 29, 7735.—The optical anisotropies of some oxalate crystals are accounted for by the anisotropy and orientation of the oxalate group in the various lattices. Mol. interaction is serious only in the presence of highly polarizable ions. L. S. Ramsdell

Two formation of crystals with irrational surfaces. D. B. Cogoboridze. *Physik Z. Sowjetunion* 8, 208-11 (1935).—Two formation in crystals occurs as commonly with irrational as with rational surfaces. It is thought that the mech. deformation of the crystal governs its type of growth. Helen S. Hopfield

Oxidation of a crystal surface studied by means of cathode-ray reflection. Tasaburo Yamaguchi. *Proc. Phys.-Math. Soc. Japan* 17, 443-53 (1935) (in English); cf. *C. A.* 29, 2842.—The effects of oxidation and of etching of cleaved surfaces of zinc blende on the reflection of cathode rays were investigated. Analysis of Laue diagrams shows that the oxidized product ( $\text{ZnO}$ ) is a single crystal, so oriented that its (103) plane rests on the cleaved (110) surface of  $\text{ZnS}$ , the [010]-axis of  $\text{ZnO}$  is parallel to the [110]-axis of  $\text{ZnS}$ . By taking into consideration the temp. effect in Waller's sense (Theoretische Studien zur Interferenz- und Dispersions-theorie der Röntgenstrahlen, *Dres., Uppsala*, 1925) a new interpretation is given of the formation of the N-pattern. G. M. Pettv

Law of corresponding states of lattice recovery. J. A. M. van Liempt. *Chem. Weekblad* 32, 546-50 (1935).—The hypothesis of lattice recovery by nucleus formation (*C. A.* 21, 3333) has been confirmed repeatedly (van Arkel, *C. A.* 24, 5007). In the recovery of a deformed crystal by heating, time as well as temp. is involved. If the increased energy of a deformed metal is taken proportional to the increased elec. resistance,  $R_d$  (the normal resistance is  $R_0$ ), the degree of deformation will be  $\beta = (R_d - R_0)/R_0$  at 0°. By taking a characteristic temp.  $t$  from  $R_d = R_0(1 + \alpha t)$  it follows that  $t = \beta/\alpha$  and the excess energy of the deformed state  $\Delta E = Mc\beta/\alpha$  with  $c$  the sp. heat. Using  $Mc = 8$ ,  $\alpha = 0.005$  (Tammann value) gives  $E = 1200 \beta$ . The mean time for exchange of place of atoms is smaller for deformed metals than for normal ones by a factor  $e^{4.33/\beta}$ ; for Cu with  $\beta = 6$  the ratio of mean times is 1/400. This decreased time of place exchange causes the tendency to nucleus formation (Cohen, *C. A.* 29, 4714). By heating a deformed metal for a period longer than the mean time mentioned as depending on the local degree of deformation  $\beta$ , it will recover in the corresponding locations. For abs. temp.  $T$ , time  $\theta$  secs. a relation  $T \log \theta = \text{const.} = T(13.5 + 10 \log \theta)$  holds, in which  $\nu$  is the period of vibration of an atom and indicates the relation between  $T$  and  $\theta$  required for the recovery of the metal. Per 1% increase in abs. temp. the rate of place exchange increases 36%.

This law holds also for recrystn. if  $\beta$  is taken as a mean degree of deformation for the entire lattice. The equation is well borne out by data of Mathewson and Phillips (*Trans Am Inst Min Met Eng* 54, 608 (1917); cf *C A* 10, 1157), of Moore and Beckinsale (*C A* 16, 324) and of Karnop (*C A* 24, 3157). In all cases the  $T - \theta$  data gave a const  $T$  ( $\log_{10} \theta + 13.43$ ), the const. decreases with increase of  $\beta$ . At room temp for deformed Cu the recovery requires 80,000 yrs., at 200° 1,000 secs., for deformed Al 23 yrs. at 27°. Such values are of importance for structural use of alloys. B. J. C. v. d. H.

The structure and entropy of ice and of other crystals with some randomness of atomic arrangement Linus Pauling *J Am Chem Soc* 57, 2680-4 (1935).—It is assumed that each mol. of ice is surrounded by 4 others, oriented so as to direct its H atoms toward 2 of the neighboring mols, forming H bonds, and that only one H atom lies near the O-O axis. There are  $(\frac{1}{2})^N$  N configurations for N mols., giving a residual entropy of 0.805 e. u., agreeing well with the exptl. value 0.87 e. u. The structures and entropies of the following crystals are discussed: diaspore, lepidocrocite, pyrrhotite, muscovite, formic acid,  $\gamma\text{Al}_2\text{O}_3$ ,  $\gamma\text{Fe}_2\text{O}_3$ , AgI, NaBr, CaBr<sub>2</sub> and the cubic tungsten bronzes. Twenty-two references. Victor Hicks

The crystal structure of ice at low temperatures F. I. Burton and W. I. Oliver *Proc Roy Soc (London)* A153, 166-72 (1935), cf *C A* 29, 3572<sup>1</sup>.—X-ray diffraction patterns were prepd by the Debye-Scherrer method. When ice is formed at temps  $T_1 > -80^\circ$ , the usual hexagonal structure crystallizes; for  $-80^\circ > T_1 > -110^\circ$ , crystals grow only along certain planes, and for  $T_1 < -110^\circ$ , a glassy state occurs. Glassy ice formed at  $T_1$  changes rapidly to senocryst ice when heated to  $T_2$ . Structures formed at  $T_2$  are maintained when cooled to  $T_3$ . Victor Hicks

The structure of ice II Ronald L. McFarlan *J. Chem. Physics* 4, 60-4 (1936).—The high pressure forms of ice discovered by Tammann and Bridgman (*C A* 29, 2040<sup>1</sup>) have a brief period of stability at very low temps and at atm pressure. This makes it possible to obtain x-ray powder diffraction photographs of these forms. Ice II has a side-centered orthorhombic cell with  $a = 7.80 \text{ \AA}$ ,  $b = 4.50 \text{ \AA}$  and  $c = 5.56 \text{ \AA}$ , it contains 8 mols., has the symmetry of space group  $V_6^2C_{22h}$ , and  $d = 1.21$ . The transition I  $\rightarrow$  II breaks up the  $\text{H}_2\text{O}$  mol., ice II is an ionic crystal. The pressure of 2100 atm (used to form ice II) causes (1) a 3% decrease in the packing vol. of the O ion, (2) a breakdown of the ice I structure by a slipping of the O ion layers relative to each other, (3) a 4-coordinated structure where each O is surrounded by a badly distorted tetrahedron of O ions. G. M. Petty

Glass XII Some new heat capacity data for organic glasses The entropy and free energy of di-lactic acid Geo S. Parks, S. Benson Thomas and Donald W. Light *J. Chem. Physics* 4, 64-9 (1936); cf *C A* 28, 6618<sup>1</sup>.—Heat capacity was measured by the Nernst method upon secondary BuOH (from 102.8 to 281.7°K.) and 3 methylhexane (71.1-289.2°K.), in the glassy and liquid conditions, and upon di-lactic acid (1) in an incompletely crystd state (90-300°K.), (2) in the form of an acid glass and liquid (103.5-303.0°K.) and (3) in the form of a mixed glass and liquid (95.8-267.4°K.). The sp. heats and heat of fusion of pure cryst di-lactic acid were called. The glasses and undercooled liquids show a rapid rise from the heat capacity characteristic of a cryst. solid to that for the liquid state in a transition region of about 10°, the nature of this region is discussed. The molal entropy of liquid di-lactic acid at 298.1°K. is 45.9  $\pm$  1.0 e. u., the free energy of formation is -124,300  $\pm$  2300 cal. XIII Glass formation by a hydrocarbon polymer John D. Ferry and George S. Parks *Ind Eng Chem*—Polyisobutylene, with an av. mol. wt. of 4900, is a highly viscous liquid at room temp. When cooled, it forms a glass with the transitions in thermal properties characteristic of the vitrification of substances of low mol. wt. The heat capacity was detd. (from 118° to 295°K.); it increases 32% between 192° and 202°K. The thermal coeff. of expansion was detd. (160-300°K.), it increases 200%

between 185° and 205°K. The mean transition temp corresponds to a viscosity of  $10^{11}$  poises, agreeing with a general rule for glass-forming materials. The factors on which the transition temp depends are discussed.

G. M. Petty  
Application of plane groups to Weissenberg photographs M. J. Burger, *Z. Krist* 91, 255-80 (1935) (in English).—Each Weissenberg photograph is a projection of a single plane of the reciprocal lattice, which embodies both point group and translation properties. All possible Weissenberg patterns can be predicted from a study of plane point-groups and translations. The method is purely geometric and avoids analytic procedure and indexing of reflections. A few appropriately chosen photographs permit an unequivocal detn. of the centrosymmetrical class, and the unit-cell type and dimensions.

L. S. Ramsdell  
X-ray measurements of diamonds M. Renninger *Physik Z* 36, 834-7 (1935).—Intensity measurements of the forbidden (222) reflections from the octahedral faces of several diamonds were made. The  $F_{222}$  structure factor was estd. to lie between 2.1 and 2.6. C. E. P. Jeffreys

Formation of diamond from geologic and crystallographic points of view Elemér Veres *Bulletin Kórisz Lapok* 63, 302-9, 327-30 (1935).—The different theories are presented. No definite conclusion can be drawn at present. S. S. de Finily

Graphite from diamond Paul Corrie *Compt rend* 202, 59-61 (1936); cf *C A* 30, 926<sup>1</sup>.—X-ray examn of graphite prepd by heating diamond at 1900° indicates much better developed crystallites than those in sugar C heated to 2000°. The resistivity, though less than that of the sugar C, is still much greater than that of Madagascar graphite, (relative) values averaging 0.169, 0.193 and 0.126, resp. The magnetic susceptibility approximated that of the sugar C. C. A. Silberrad

Net density and x ray investigation of carbon Yoshiaki Matsunaga *Mem Coll Sci Kyoto Imp Univ* A18, 215-20 (1935).—Charcoal prepd by carbonizing pure sugar 3 hrs at 250° or 300° contains no sugar, gives an amorphous x-ray pattern and has net  $d$  1.475. On heating above 400° the  $d$  increases and graphite is formed as shown by the x-ray pattern, the limit is pure graphite ( $d = 2.268$ ). Ordinary C is a mixt. of amorphous C and graphite, the proportion depends chiefly on the temp to which it has been heated, and can be calcd. from the observed  $d$ . C. D. West

The crystal structure of CdSb. Arne Ölander, *Z. Krist* 91, 243-7 (1935); cf *C A* 29, 7815<sup>1</sup>.—Powder photograph data indicate an orthorhombic unit cell for CdSb, with  $a = 8.492 \text{ \AA}$ ,  $b = 8.320 \text{ \AA}$ ,  $c = 6.390 \text{ \AA}$ , contg 8 Cd and 8 Sb. The symmetry may be either  $V$  or  $C$ . It is suggested that the arrangement may be a greatly deformed NaCl structure. L. S. Ramsdell

The crystal structure of gallium A. J. Bradley, *Z. Krist* 91, 302-16 (1935) (in English).—A redtn. of the structure of metallic Ga by the powder method conforms Laves' orthorhombic structure (*C A* 27, 1558<sup>1</sup>) but photographs with  $\text{Ni K}_\alpha$  radiation show all 3 axes to be different:  $a = 4.5167 \pm 0.0001 \text{ \AA}$ ,  $b = 4.5107 \pm 0.0001 \text{ \AA}$ ,  $c = 7.6448 \pm 0.0002 \text{ \AA}$ . The space group is  $V_2^2$ , and more accurate values of the parameters are  $m = 0.0785$  and  $p = 0.1525$  (both  $\pm 0.0005$ ). These were obtained from photometer measurements of the highly dispersed lines at the end of the film. This method is described and illustrated. L. S. Ramsdell

The unit of structure of telluric acid,  $\text{Te}(\text{OH})_6$  Linus Pauling *Z. Krist* 91, 367-8 (1935).—A rotation photograph of cubic  $\text{Te}(\text{OH})_6$  shows the unit cell to contain 32 mols., as found by Kirkpatrick and Pauling (*C A* 29, 359<sup>1</sup>) and not 4 mols., as stated by Gossner and Kraus (*C A* 28, 6604<sup>1</sup>). L. S. Ramsdell

Crystallization of antimony anhydride Aurelio Serra *Z. Krist* 91, 371-2 (1935).—Below 100° a cubic form, and above 100° an optically biaxial modification of  $\text{Sb}_2\text{O}_3$ , are obtained by crystn. from  $\text{H}_2\text{O}$  or  $\text{H}_2\text{SO}_4$ . L. S. Ramsdell  
Variation in the lattice constant of zinc oxide V. E. Cosslett *Nature* 136, 958-9 (1935).—The lattice const.

of ZnO films prep'd by withdrawing a washer from the surface of molten Zn varied with age. The values for new films, and for films 12 and 18 months old, resp., were:  $\alpha$ , 3.234, 3.262, 3.279 Å;  $c$ , 1.615, 1.628, 1.637 Å. The probable error of each measurement is 0.005 Å. The lattice consists of Au films remained constant during the same time interval. The use of such ZnO films for high-voltage calibration is inadvisable.

**The structure of the electrolytic oxide layer on aluminum.** I. J. W. Verwey. *Z. Krist.* 91, 317-20 (1935), cf. C A 30, 1310<sup>1</sup>.  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, formed by electrolytic oxidation of Al, is closely related to  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, but does not give an identical x-ray pattern. The structure is described as an averaged structure of the cations. The unit cell contains 4 O-ions in face-centered positions, 2/3 Al ions are distributed statistically over all the interstices between the O ions in such a way that 70% of the Al have a coordination no. 6, and 30% a no. 4, with regard to the O.

**Crystal chemical investigation of aluminum compounds with the spinel type of structure and of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>.** I. Rost Kordes. *Z. Krist.* 91, 193-228 (1935).—LiAlO<sub>2</sub> can be derived from Mg<sub>2</sub>AlO<sub>4</sub> by the substitution of LiAl for 2 Mg. The 2 have similar hardness,  $d$ ,  $n$ , and crystal structures and form solid solutions for the Li-Al spinel,  $d_{100} = 7.903$  Å (or 15.806 Å). Well-crystd  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> is obtained from LiPO<sub>4</sub> melts, and closely resembles the Li-Al spinel,  $d_{100} = 7.895$  Å (or 15.790 Å). These 2 can be referred to a face-centered cube only if the cell dimensions are doubled. Traces of Li-O or H<sub>2</sub>O make  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> more stable. Ferromagnetic  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> is a solid soln of 1 Fe-Fe<sub>2</sub>O<sub>3</sub> and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>. The use of fluorides in the synthesis of oxides and silicates is discussed.

**The crystal system of julenite, Na<sub>2</sub>Co(CSN)<sub>2</sub>·8H<sub>2</sub>O.** A. Schoep and V. Billiet. *Z. Krist.* 91, 229-31 (1935).—X-ray photographs show synthetic Na<sub>2</sub>Co(CSN)<sub>2</sub>·5H<sub>2</sub>O to be identical with julenite. The system is tetragonal, with  $a = 9.22$  Å, and  $c = 5.56$  Å. The cell contains 1 mol., and the measured  $d$  is 1.648.

**The crystallography of ergine.** H. Brasseur. *Z. Krist.* 91, 369-70 (1935).—Crystals of ergine, C<sub>18</sub>H<sub>17</sub>ON<sub>3</sub>·ClH<sub>2</sub>O, are orthorhombic. Oscillation photographs about the 3 axes give  $a = 11.915$  Å,  $b = 16.090$  Å,  $c = 8.140$  Å (all  $\pm 0.27$ ).  $Z = 4$  mols. Space group  $V_4$ .

**An x-ray study of symmetrical trimethylolamine and cyclo-trimethylenetriamine.** Ralph Hultgren. *J. Chem. Physics* 4, 84 (1936).— $\gamma$ -m-C<sub>3</sub>H<sub>7</sub>(NO<sub>2</sub>)<sub>3</sub>Me is orthorhombic;  $a = 14.85$  Å,  $b = 39.5$  Å,  $c = 5.96$  Å, there are 16 mols in the unit cell, giving a calcd.  $d$  of 1.710 (the value usually given is 1.634). Trimethylenetriamine is orthorhombic;  $a = 11.5$  Å,  $b = 13.2$  Å,  $c = 10.6$  Å, there are 8 mols in the unit cell, and the calcd.  $d$  is 1.82. The most probable space group is  $I_4^1$ .

**The crystal structure of  $p$ -dinitrobenzene.** R. W. James, G. King and H. Horrocks. *Proc. Roy. Soc. (London)* A153, 225-46 (1935).— $p$ -Dinitrobenzene crystallizes from Me<sub>2</sub>CO in the monoclinic holohedral class. The crystallographic data of Bodewig (*Ann. Physik* 158, 239 (1876)),  $\beta = 90^\circ 15'$ ,  $a, b, c = 2.038, 1.1043$ , are confirmed. The  $n_D$ 's detd. are:  $n_a, 1.614$ ;  $n_b, 1.64$ ;  $n_c, 1.72$ , with  $\beta = 87^\circ$  to  $c$ . Two centrosym. mols occur in an elementary cell of the space group  $C_{2h}^2$  ( $P2_1/n$ ) with the dimensions  $a_u, 11.03$ ,  $b_u, 5.42$ , and  $c_u, 6.56$  Å. Electron  $d$  projections were made by double Fourier series on the axial planes, and the 18 parameters evaluated. The C<sub>6</sub>H<sub>4</sub> ring is distorted from a regular hexagon of 1.40 Å on a side by the shortening of one of each pair of sides adjacent to C-N link to 1.32 Å, the distortion being ascribed to the displacement of the C atoms linked to the NO<sub>2</sub> groups. Other interat. distances are: N-O, 1.10 and 1.25, and O-O, 2.14 Å. The C-N link is nearly in the plane of the ring, but lies at  $25^\circ$  to the plane of the NO<sub>2</sub> group. The mols. lie in sheets parallel to (101), and are slightly inclined to that plane. The O-H distances between adjacent mols. vary from 3.20 to 3.5 Å, and such links are probably responsible for part of the crystal binding forces. This structure does not agree with that of Hertel

and Schneider (C. A. 24, 2924) or with Banerjee's conclusions (C. A. 29, 1303<sup>1</sup>).

**X-ray analysis of the structure of lidescent shells.** H. Halotidae. S. Ramaswamy. *Proc. Indian Acad. Sci.* 2A, 345-51 (1935), cf. *Ibid.* 1A, 871-9 (1934).—By use of monochromatic x-rays, the x-ray diffraction patterns of the shells of some species of *Halotis* were studied. The patterns obtained with normally incident x rays differ somewhat from those previously obtained with *Turbo* and *Trochus* in which shells there is a random orientation of the  $a$  and  $b$  axes of aragonite. In *Halotis* shells there is a preferred orientation of these axes, with a large error in orientation. The  $c$  axis is normal to the laminations as in other aragonite shells. Patterns obtained with *Nautilus pompilius* and *Margaritifer vulgaris* were measured. The type of orientation of twinned strata of aragonite previously suggested for *Nautilus* is confirmed. The nacreous layer of *M. vulgaris* consists of single aragonite crystals with 110 planes parallel to lines of growth and  $c$  axis normal to the shell surface.

**Adsorption problems.** I. Traube. *Trans. Faraday Soc.* 31, 1730-9 (1935).—Many detns. of drop nos. were made and a relation between drop nos. and concn. of the solns was graphed. The results enable detns. of the capacity of adsorption of surface-active and interface-active substances by various org. substances slightly sol. or insol. in H<sub>2</sub>O. Such substances as anthracene, naphthalene or diphenylamine added to solns of acetyl alc. or caprylic acid absorb such small quantities of adsorbate that the Langmuir theory can be applied. In some cases the adsorption is practically zero. If these substances, insol. in H<sub>2</sub>O, are added in an emulsified state they are adsorbed to a high degree, so that there are several thousand layers on the adsorbent. The adsorbate is held so tightly that washing with H<sub>2</sub>O removes only mol. particles if they are sol. in H<sub>2</sub>O. The Gibbs principle is not applicable. A relation between adsorption and flocculation was found. The 2 components of adsorption, viz., the capacity and intensity factors, were discussed.

**Heats of wetting and the physical significance of the constants in the equation of Rodewald.** I. Adrianov. *Kolloid-Z.* 73, 328-34 (1935), cf. Rodewald, *Z. Physik.* 68, 333, 593-604 (1900).—Rodewald's equation is  $c(w_1 - w_2) = \log(r_2 + 1)/(r_1 + 1)$ , where  $r_2$  and  $r_1$  are the calorimetric heats of wetting of the solid contg.  $w_2$  and  $w_1$  % moisture, resp.,  $s$  measures the extent to which the cohesive forces of the swelling solid are overcome and is opposite in sign to  $r$ , and  $c$  is a proportionality const. A. regards  $s$  as the absorption of energy required to disperse the water over the surface of the adsorbing solid (cf. Patzelt and Grimm, C. A. 16, 518<sup>1</sup>). The const.  $c$  is inversely proportional to the active surface of adsorbent.

**Comparison of efficiencies of activated charcoal.** B. P. Denisovich. *Khim. Farm. Prom.* 1935, No. 1, 47-9.—The charcoal is kept in a buret, the standard I soln. is run through it (1 l. per hr.) and the I is detd. in the filtrate.

**The diffusion of helium through fused silica.** R. O. Braaten and G. F. Clark. *J. Am. Chem. Soc.* 57, 2714-17 (1935).—The rate of flow of He through SiO<sub>2</sub> is a linear function of pressure and thickness of SiO<sub>2</sub>. The energy of activation depends on the temp. and the silicate content. The temp. range from 42.9° to 562° was covered.

**The adsorption of hydrogen on tungsten.** J. K. Roberts. *Proc. Roy. Soc. (London)* A152, 445-63 (1935); cf. C. A. 26, 2357, 3970.—A method is described for studying the adsorption of H<sub>2</sub> on a bare W surface which depends on the fact that the accommodation coeff. for Ne is considerably greater for a surface covered with a film of adsorbed gas than for pure W. It is shown that on a clean W surface chemisorption of H<sub>2</sub> occurs very rapidly at temps. as low as 79°K., satn. of the surface taking place at a partial pressure of 10<sup>-4</sup> mm. or less. The heat of adsorption as measured on a single fine wire varies from 45,000 cal. per mol. for the first H<sub>2</sub> mols. to 18,000 for the



last portion becoming adsorbed. The amt of  $H_2$  required to form a unimol. layer is consistent with the adsorption of  $H_2$  as atoms. The film is stable for heating times of about 1 min. up to about 700°K. Calens show the dipole moment per adsorbed atom to be  $1.94 \times 10^{-18}$  e s u, and the change in work function resulting from covering the surface with a complete film to be 5.2 v. The rapidly occurring chemisorption of  $H_2$  on the W surface differs from the more slowly occurring "activated adsorption." The accommodation coeff. of Ne at 79°K. is 0.32 on a W surface covered with a layer of chemisorbed  $H_2$  compared to about 0.1 for bare W; at 295°K. the coeffs. are, resp., 0.17 and about 0.08. Some properties of adsorbed films of oxygen on tungsten. *Ibid.* 464-77.—The accommodation coeff. of Ne on a W film sat'd with O at room temp. is about 0.36. When the film is evacuated at temps. up to about 1300°K. the accommodation coeff. drops to 0.24, whereas on pure W the value is 0.08. The tightly adhering O film stable above 1300°K. is identified with the one already studied by thermionic methods (cf. C. A. 25, 1731). Its heat of adsorption is about 139,000 cal. per mol. of  $O_2$ . It appears to be a layer of O adsorbed as atoms. The second film is new. It is interpreted as a partial layer of  $O_2$  mols in the gaps that necessarily occur in the first film. The heat of adsorption of the second layer is about 48,000 cal. per mol. of  $O_2$ . It is stable to about 360°K. only. Composite films of oxygen and hydrogen on tungsten. *Ibid.* 477-80.—When a film of W covered with a layer of adsorbed  $H_2$  is exposed to a low pressure of O at room temp. the  $H_2$  is displaced by the O, the total heat of the process being about 115,000 cal. per mol. of  $O_2$ . The  $H_2$  is apparently thrown off as mols. Indications are obtained of a slow disappearance of  $H_2$  when the latter is admitted to a bulb contg. a W film partially covered with O. This observation suggests that the slow "activated adsorption" of the  $H_2$  is connected with the presence of surface O.

P. H. Emmett  
The sorption of hydrogen and deuterium by copper and palladium. I. The behavior of copper and copper oxides. H. W. Melville and Eric K. Rideal. *Proc. Roy. Soc. (London)* A153, 77-88(1935).—At. H and at. D at 20° reduce CuO with the same velocity. Mol. H reduces CuO more rapidly at 156-260° than does mol. D, the difference in energy of activation being 0.4 kcal. Mol. H and D are sorbed by and desorbed from Cu at the same velocities from 71° to 200°, except that  $H_2$  is sorbed slightly more quickly at the higher temps. II. The sorption by palladium and diffusion through copper. *Ibid.* 89-103.—H diffuses more quickly than D through Pd, Cu and Ni at temps. from 150° to 350°, and at pressures from 1 to 200 mm., the differences in energy of activation for diffusion being 0.8 kcal. for Pd and Cu and 0.6 for Ni. In Cu, the rate-detg. step is not due to any process on the gas-Cu interface, because use of the composite films, Cu-Pd-Cu, and Pd-Cu-Pd, did not affect the results. H is more sol. than D in Pd, and the difference in velocity of diffusion is due partly to the greater sol. as well as to the greater mobility of H. A potential barrier exists for the penetration of H from the surface to the interior; the barrier has the same height for D as for H.

L. E. Steiner  
Graphic representation of wetting angles for the method of maximum bubble pressure. L. Lange and K. Nagel. *Kolloid-Z.* 73, 268-9(1935); cf. Hohn and Lange, C. A. 30, 1292.—Wetting angles are sketched and described for wetting and nonwetting liquids at various stages in the formation of the bubble.

Oscar T. Quimby  
Measurements of the surface tension of solutions of potassium and lithium salts of higher aliphatic acids by the ring method with exclusion of carbon dioxide and with special consideration of other disturbing effects. II. A. Lottermoser and Ernst Giese. *Kolloid-Z.* 73, 276-83(1935); cf. *Kolloid-Z.* 73, 155(1935); cf. C. A. 30, 6694.—With the precautions described in the previous paper, surface tension,  $S$ , was measured at temps. of 20-70° on 0.001-1.0% solns. of K and Li salts of the following acids: caproic, caprylic, capric, lauric, myristic, palmitic, stearic, oleic and abietic. The results are reproducible to

$\pm 1$  dyne/cm. When adsorption effects are avoided  $S$  decreases with increasing temp. in many cases linearly, especially at the lower concns. c. In general the temp. coeff. of  $S$  decreases with increasing c as was previously found with Na salts, some exceptions were observed due to changes such as soln. of a ppt. with rising temp. The  $S$ -c curves show the same shape as found for Na salts, the steep portion often being linear if  $S$  is plotted against  $\log c$ . The surface activity is unaffected by the cation with 2 exceptions: Li oleate is more active than the Na or K salt and Na palmitate is more active than the Li or K salt. The Li salts are less sol. than the Na salts. At 0.001 to 0.01% concns. of K soaps Traube's rule is qualitatively verified.

Oscar T. Quimby  
The vapor pressure of drops of S. V. Gorbachev. *Kolloid-Z.* 73, 203-7(1935); cf. C. A. 30, 1411, 6657.—Avoiding the assumption that Poisson's const. (product of the radius of meniscus curvature by the rise of liquid in a capillary) is independent of drop size, G. derives a math. relation based on van der Waals' equation for the vapor pressure of very small drops. For relatively large drops the new relation reduces to the Kelvin equation. The effect of dissolved substances on the vapor pressure of drops also is considered.

Oscar T. Quimby  
Mechanism of the coagulation of sols by electrolytes. V. Sulfur sol. Harry B. Weiser and George R. Gray. *J. Phys. Chem.* 39, 1163-75(1935); cf. C. A. 28, 4328.—The adsorption of Th, Nd, Al, Ba, Sr and Ca during the coagulation of Raffo's S sol was detd. at and above the coagulation concn. Contrary to the observations of Bassett and Durrant (C. A. 26, 2102) the adsorption values are not equiv. at the resp. pptn. concns. The order of adsorption above the pptn. value is the same as that of the pptg. power. Th > Nd > Al > Ba > Sr > Ca. The H-ion displacement by the several cations is in the same order as the coagulating power. Equiv. amts. of H are not displaced at the pptn. values of the several cations (cf. Bolam and Bowden, C. A. 27, 649; Bolam and Mur, C. A. 28, 1910<sup>7</sup>, 5311<sup>4</sup>). The displacement approaches equivalence only when the pptn. values are of the same order of magnitude. With cations of the same valence the order of H-ion displacement as well as coagulating power is related to the size of the ions. A mechanism is proposed to account for the above mentioned phenomena of adsorption and H-ion displacement which accompany the processes of potential reduction and coagulation.

H. B. W.  
Electrolytic coagulation of weakly solvated sols and electrolyte activity. Wolfgang Ostwald. *Kolloid-Z.* 73, 301-28(1935); cf. C. A. 14, 1775; 16, 2053.—The inadequacies of previous theories are discussed to emphasize the importance of a new guiding principle, namely, "not the micelles, but their dispersion medium should be in a corresponding (in the simplest case, identical) physico-chem. state during peptization or coagulation." Thus coagulation should take place at the same activity coeff. of the dominant ion ( $f_+$  or  $f_-$ ) regardless of the salt dissolved in the medium bathing the micelles. Numerous tables of data are presented to illustrate this principle, these are based upon data in the literature for the neutral-salt coagulation of the following hydrophobic sols: (1) negatively charged  $As_2S_3$ ,  $Sb_2S_3$ , Au, Ag, Pt, S,  $MoO_3$ , mastic, Congo red, (2) positively charged  $Fe(OH)_3$ ,  $Al(OH)_3$ , Au. For  $As_2S_3$ ,  $f_+$  is const. within a few % for a given investigator and varies only from 0.68 to 0.83 for different investigators, if one excludes, besides a few inaccurate results, the data for salts like  $MgSO_4$  which are suspected of a tendency to form complex ions with  $As_2S_3$ . The agreement between theory and expt. is no better in other cases (sometimes worse). The constancy of  $f_+$  (or  $f_-$ ) is best for weakly hydrated sols. Based on the above principle a new valence rule is formulated, which requires that the reciprocal of the coagulating molarities for the valence types 1-1, 1-2, 1-3, 1-4, 2-1, 2-2, 3-1, 3-2, 3-3, 4-1, 6-1, stand in the ratio 1:5:2:5:48:64:486:608:729:2560:27,200. That this is not in agreement with the data may be due to some neglected factor or to inaccuracy of the data. The averages for the many investigations on

As<sub>2</sub>S<sub>3</sub> agree in order of magnitude with this set of ratios. In certain special cases the above valence rule reduces to the Ostwald or to the Freundlich rule. The curves obtained by Loeb for charged albumin sols suggest that the decreases in (1) swelling, (2) osmotic pressure, (3) relative viscosity and (4) membrane potential are approx. proportional to the activity coeff. of the dominant ion of the added salt.

Oscar T. Quimby  
A new method of investigating unimolecular films Irving Langmuir and Katherine B. Blodgett *Kolloid-Z.* 73, 258-63 (1935).—A lecture (cf. Langmuir, *C. A.* 28, 6606\* and Blodgett, *C. A.* 29, 4649\*, 5329\*). O T Q

Investigation of the structures of extremely thin fatty films by means of electron diffraction H Motz and J J Trillat *Z. Krist.* 91, 248-54 (1935).—Films 2 mols thick were formed by evap. a drop of Et<sub>2</sub>O or C<sub>18</sub>H<sub>36</sub> soln. of the substance on metal foil or cellulose. With Au, Pt, Ag and Al foil there were obtained the diagrams of the metal plus characteristic ring or point diagrams from the film. Diagrams for paraffin, stearic acid, tristearin, cetyl alc. and beeswax were identical except for small intensity differences. With cellulose mounts, only the diagrams of the samples were obtained. The structures are analogous to those found for paraffin by Muller (*C. A.* 23, 30). All show the same lateral chain distances. Differences lie in the angle that the planes of the zigzag chains make with the *a* and *b* axes. The orthorhombic form of stearic acid was confirmed. L S R

Alkali films of atomic thickness on platinum Herbert Mayer. *Physik. Z.* 36, 845-8 (1935), cf. *C. A.* 29, 7811\*.—A method and app. are described for deposition of alkali metal films on Pt and accurate measurement of their thickness. The change of photoelec. sensitivity of K films on Pt as a function of film thickness was followed, for both outgassed and gas-contg. films. In the former case no max. was reached, but in the latter a max. sensitivity occurs with a unimol. layer. C. E. P. Jeffreys

Some aspects of the solid-liquid interface Wm Clayton. *J. Oil & Colour Chem. Assoc.* 18, 412-34 (1935).—A review of recent progress with 74 references. J W P

The preparation of colloids with ultrasonic waves Neda Marinescu. *Bull. soc. roumaine phys.* 36, 181-9 (1934).—Waves with frequencies of the order of 10<sup>6</sup> are generated by piezoelec. quartz in an oscillating circuit. Such waves accelerate various chem. reactions, lower *b* p. as much as 10° and have profound biophys. effects. Emulsions of Hg in H<sub>2</sub>O, kerosene and various aces were prepd. The particles were uniform spheres 0.5  $\mu$  in diam. The size depends on the amplitude and not on the frequency. Emulsions of Wood's and Rose's metals were prepd. by similar treatment at the m. p., upon cooling, these gave solid particles of the same diam. Some of these colloids have therapeutic effects. Colloids of alkali metals and their alloys in kerosene so prepd. burn spontaneously on contact with air. R. E. DeRicht

The diffusion of electrolytes through a membrane I Michimasa Kōno and Eyo Utsunomiya. *J. Chem. Soc. Japan* 56, 1475-82 (1935).—The velocities of permeability of 0.1 N alkali salts through Cellophane were measured at 25°. They are in the order KBr > KI > KCl > NH<sub>4</sub>Cl > NaCl > LiCl and KNO<sub>3</sub> > NH<sub>4</sub>NO<sub>3</sub> > NaNO<sub>3</sub> > LiNO<sub>3</sub>. T. Katsuragi

Variations of the electrogenic property of a membrane with respect to the concentrations of the solutions that it separates M. Chanoz. *Arch. phys. biol.* 12, 251-4 (1935).—Discussion. L. E. Gilson

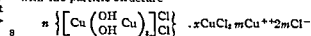
Dimension and cataphoretic velocity of disperse particles. J. Prosz. *Roy. Hung. Palatin-Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met.* 7, 26-32 (1935).—Detns. of velocity and estimates of diam. of very many spherical particles in a paraffin oil emulsion proved that velocity depends on particle size. In the range from 1 to 10  $\mu$  velocity increases directly with diam. of particle. Theoretical explanation by means of the formulas of Debye and Huckel, and of Eucken is attempted. S. S. de Finily

Electrokinetics XVII Surface charge and ion antagonism. Laurence S. Moyer and Henry B. Bull. *J.*

*Gen. Physiol.* 19, 239-47 (1935).—The surface charge (neg. in sign) for cellulose in contact with solns. of 4 K salts follows the order: Cl < SO<sub>4</sub> < CO<sub>3</sub> < PO<sub>4</sub>. For ThCl<sub>4</sub>, the surface charge is neg. at low concns, becoming pos. with higher concns. For NaCl soln. ( $2 \times 10^{-4}$  N) the charges plotted as logs against 1/temp. give a curve with a break at 39°. Since the sp. heat of water shows a min. at 37.5°, and the adsorption of ions probably involves the desorption of water, the break in the heat of adsorption of the charge may result from the change in desorption of water at this point. The explanation probably rests in the smaller dielec. const. at the higher temps., resulting in an increased attractive force between the ions and the charged wall. A marked ion antagonism was found between mixts. of the following pairs of compds. when surface charge was calcd. as a function of concn: NaCl-KCl, KCl-MgCl<sub>2</sub>, NaCl-MgCl<sub>2</sub>, NaCl-CaCl<sub>2</sub>, KCl-CaCl<sub>2</sub>, CaCl<sub>2</sub>-MgCl<sub>2</sub>. This is the first time ion antagonism has been directly traced to elec. effects. The cause of the lower charge for salt mixts. than for single salts probably lies in the tendency toward independent adsorption of the cations of the mixt. while the adsorption of the Cl ions remains much the same for both salt mixts. and single salts. This results in a smaller net neg. charge. Probably ion antagonism in biol. systems is of the same nature.

C. H. Richardson  
Light scattering in gold sols in relation to particle size and shape D. S. Subbaramaya. *Proc. Indian Acad. Sci.* 2A, 358-63 (1935), cf. *C. A.* 29, 5328\*.—Results of measurements of the depolarization of Tyndall scattering corresponding to the incident light *s* being (a) unpolarized, (b) plane polarized with its elec. vector perpendicular to the plane of observation, (c) plane polarized with its elec. vector parallel to the plane of observation, are reported. The results indicate that the particles in Faraday's sols are much smaller than those of other red sols prepd. from nuclear sols with H<sub>2</sub>O<sub>2</sub> as the reducing agent, while those in the blue sols are the largest. In all sols studied, the shapes of the particles were far from spherical. Increased rate of growth caused by enhancing the temp. during prepn. of the sols appeared to increase the size and anisotropy of the particles. L. L. Quill

The structure of the colloidal particles of artificial atacamite A. A. Morozov. *Kolloid-Z.* 73, 288-99 (1935); cf. *C. A.* 27, 884\*.—The atacamite sol (pptd. by reaction of Cu(OAc)<sub>2</sub> and NaCl in aq. soln., filtered and peptized with H<sub>2</sub>O) could not be freed from chloride by dialysis without pptn. Upon diln. of a dialyzed sol with doubly distd. water, the equiv. cond. *L*, of the sol increased as did that of the ultrafiltrate from the dld. sol. Diln. of the ultrafiltrate from the original sol scarcely increased *L* at all. Mixts. of a dialyzed sol with either Cu(OAc)<sub>2</sub> or NaCl or both gave less than the additive values of *L*. A sol contg. 14.56 g. dispersed phase per l. was reversibly coagulated by NaCl in concns. of 0.08 N or more, but coagulated irreversibly by Na<sub>2</sub>SO<sub>4</sub> in concns. of 0.0125 N or more. All the known facts are in harmony with the particle structure



Oscar T. Quimby  
The formation and constitution of crystals of lead salts containing water-soluble colloid. F. D. Miles. *Trans. Roy. Soc. (London)* A235, 125-64 (1935).—The absorption of dextrin, gum arabic or gelatin in growing crystals of PbCl<sub>2</sub>, PbBr<sub>2</sub>, PbI<sub>2</sub>, PbN<sub>3</sub> and PbSO<sub>4</sub> was studied. More than 10% may be firmly bound and 5% may be present in a crystal without confusing its structure. The changes in crystal habit, and alterations in stability of various phys. forms and hydrates, induced by absorption of colloid during both slow and rapid pptn., are described. The relation between amt. absorbed and colloid soln. concn. was followed. Differences shown by the salts are discussed. Microscopic observations of the ppts. were made. The adsorption of dextrin on preformed crystals of chloride and azide was measured and compared with the estd. available surface. Results were checked with dex-

trun purified by dialysis, the osmotic pressure of which was detd. Maltose was not absorbed. Solns contg chloride and dialyzed dextrin were exam'd in the ultramicroscope and their conductivities were measured. There was no evidence of assocn of Pb ion and dextrin in soln. X-ray examn showed that the lattice spacings of the pure salts are unaffected by the introduction of colloid. The difficulties in applying the known methods of estg particle size are mentioned. An approx correction for finite size of the aperture is worked out. The results show that as colloid is taken up the av. particle width is decreased, but the 2 salts exam'd, azide and sulfate, behave differently. The ratio of coherent to incoherent radiation was measured with the conclusion that in the complexes of azide (but not sulfate) extremely fine (colloidal) Pb compd must be present. It is concluded that the constitution of the complexes is that of a connected skeletal growth of crystallites, all in nearly the same crystallographic orientation, with layers of colloid in between. These layers consist of irregular bundles of long dextrin mols anchored in the crystallites by some of their hydroxyls, but having the rest free. **F. D. Rossini**

Reactions of organic anions with basic thorium chloride hydrosols. Reversal of charge with salts of the hydroxy acids and with nitric acid. **Arthur W. Thomas and Chester B. Kremer**. *J. Am. Chem. Soc.* 57, 2539-41 (1935), cf. *C. A.* 29, 7754<sup>1</sup>.—The relative reactivities of K salts of the carboxylic acids were studied by noting the changes in  $pH$  with concn of salt on basic Th chloride hydrosols. Conclusions: Hydroxy org. anions reverse the cationic charge to anionic thionate micelles. Conc'd  $HNO_3$  reverses the charge. The effects of  $H_2SO_4$ ,  $HCl$  and  $HNO_3$  are compared. **Raymond H. Lambert**

Simple lyotropic effects (viscosity). **E. M. Bruins**. *Rec. trav. chim.* 55, 13-16 (1936).—See *C. A.* 29, 6608<sup>1</sup>.

Structure viscosity of lyophilic sols. **I. Gelatin sols**. **Nic. Peskov and S. Averbuch**. *Kolloid-Z.* 73, 182-91 (1935).—An overflow viscometer is described, it consists of 3 capillaries of equal diam. (0.0568 cm), but various lengths (7.1, 13.9 and 20.2 cm) connected to the same reservoir which communicates with a manostat. The amt. of liquid  $S$  flowing through each of the capillaries in a single overflow time  $Z$  is measured under a definite pressure  $P$ . Theoretically both  $PZ/S$ , which is proportional to the viscosity  $\eta$ , and  $\eta/S$  (subscripts refer to two of the above capillaries) should be independent of  $P$  for liquids obeying the Hagen-Poiseuille law. With water, 20% sugar soln, EtOH and AmOH, these ratios were found to be const. within 1% over a  $P$  range of 60-900 mm  $H_2O$ . With an electrodyalized gelatin whose sols had a  $pH$  of 4.4-5, the effect of  $pH$ ,  $P$  and temp. on  $\eta$  of 1 and 2% gelatin sols was studied. A 2% gelatin sol showed no anomalous effects at 34° or higher regardless of  $pH$  or age. At 30° deviations from the Hagen-Poiseuille law were observed only within the  $pH$  range 3.5-5.5, the effect decreasing with time if the sol was previously aged at 20° but increasing with time if the sol was freshly prepd at 30°. At 25° or lower, deviations were observed over the whole  $pH$  range (2-10). These deviations are caused by 2 factors: (1) the formation of an inner structure and (2) the orientation of the micelles. "The abs. value of  $\eta$  does not seem to be connected unconditionally with structure phenomena because sols with a  $pH$  far from 4.5 have simultaneously a greater viscosity and a less pronounced structure." Some measurements are also given with a Krutviscometer and with an Ostwald-Auerbach overflow viscometer. **Oscar T. Qumby**

Viscosity and plasticity of disperse systems. VIII. Application of the A. I. Bachinski formula to the viscosity of hydrogenated fats. **M. P. Volarovich and G. B. Ravich**. *Kolloid-Z.* 73, 339-42 (1935), cf. *C. A.* 30, 12<sup>1</sup>.—The authors measured the  $d$  (Westphal balance) and viscosity (Ubbelohde capillary viscometer) at 5° temp. intervals for sunflower-seed oil (25-95°), hydrogenated fat A (50-140°) and hydrogenated fat B (55-95°). The fat acids of A had a titer of 34.2° those of B, 52.3°. The product of pressure and time of outflow was const.

within  $\pm 1\%$ , the limit of accuracy of the viscosity data. The viscosity-temp. curves for A and B coincided. Plots of the fluidity  $\eta$  sp. vol. gave straight lines for all 3 substances above a fluidity of about 0.05, corresponding to the Bachinski relation (cf. *C. A.* 7, 3000<sup>1</sup>), the break at a fluidity of 0.05 is associated with polymerization.

**Hydroxide gels and hydrated oxide gels and their amphoteric properties**. **R. Fricke**. *Kolloid-Z.* 73, 300 (1935).—(oleum) with Krause, *C. A.* 29, 7753<sup>1</sup>.

**Mechanism and kinetics of thixotropic solidification**. **Wilhelm Heller**. *Compt. rend.* 202, 61-4 (1936), cf. *C. A.* 24, 1782<sup>1</sup>.—The thixotropic gels formed on adding 0.03-0.06 M NaCl to a 110 micromol sol of  $Fe_2O_3$  (2 hrs) were centrifuged for 10 to 190 min., and the percentages of solid g'd. detd. The results are explained by regarding thixotropic solidification as depending on (1) the rate of formation of germs (centers of gelation), (2) the rate of growth of these germs into elementary gels ("geloids") and (3) the rate at which these geloids associate. Thus the question whether the solid resulting from thixotropy consists of a net of gel enclosing liquid, or a uniform and homogeneous gel reduces to a question of time. **C. A. S.**

Coagulation of ferrocyanide sols containing varying amounts of potassium ferrocyanide. **Nirmala Pada Chatterjee**. *J. Indian Chem. Soc.* 12, 671-85 (1935).—An extension of previous work (cf. *C. A.* 24, 5572<sup>1</sup>) to sols of Prussian blue (I), and of U (II), Al (III), Cu (IV), Zn (V) and Cd (VI) ferrocyanides. Concns of chlorides expressed as miliequival./l. necessary to coagulate (1) follow the series  $K > Ba > Ag(NO_3) > Al > Th$ . The other sols were pptd. in presence of varying amts. (0.05-1000 ml. liq.) of  $K_4FeC_6N_6$ . Amt. of KCl necessary increased generally with increase in amt. of  $K_4FeC_6N_6$ . At high concns the effect was irregular. Sols were prep'd by pptn. and then alternate washing and centrifuging until peptization occurred, (I to III were acidic, IV to VI basic). Coagulating concn was detd. by finding the least concn of salt necessary to produce a clear supernatant liquid upon centrifuging for 8 min., the sol with electrolyte. Sp. cond. and cataphoretic speeds for (IV) were detd. Data and curves are given. **E. R. Schierz**

Dielectric constant and conductivity of gelatin sols and gels. **Arkadiusz Piekara and Bruno Piekara**. *Kolloid-Z.* 73, 273-6 (1935); cf. *C. A.* 28, 2594<sup>1</sup>.—Sols and gels contg 0.5 to 1.25% electrodyalized gelatin per 100 g. soln were studied at various temps. (10-40°). The data (4 tables, 6 graphs) show: (1) the dielec. const. of gelatin dispersions ( $D$ ) is greater than that of pure  $H_2O$  ( $D'$ ) and increases linearly with concn., (2) not only  $D$ , but also the elec. cond. of gelatin sols is greater than that of gels at the same temp. and concn.; (3) although  $D$  for a given concn. increases with temp., the difference  $D-D'$  decreases. Two Au sols and an  $\alpha$ -FeOOH sol had  $D$  values identical with that of  $H_2O$ . **Oscar T. Qumby**

Investigations of the effects of some factors on rhythmic crystallization. **Mayel M. MacMasters, Julia E. Abbott and Charles A. Peters**. *J. Am. Chem. Soc.* 57, 2504-8 (1935).—Several types of rhythm are differentiated, the most important ones being coarse rhythm, and fine rhythm (period 0.005 to 0.01 mm). Differences in temp. and concn. do not affect the rhythmic crystn. of  $K_2Cr_2O_7$  from aq. soln. Crystd.  $(NH_4)_2Cr_2O_7$  and  $Na_2Cr_2O_7$  also show a periodic structure. Foreign anions have a specific effect in inhibiting fine rhythm, the order of decreasing effectiveness being nitrate, sulfate, chloride. It is suggested that Liesegang rings and rhythmic crystn. from soln. or melt have a common cause. **J. B. Austin**

The colloid chemistry of rice starch and cooking of rice. **III. Ichiro Sakurada, Toshio Kitano and Keiroku Fuchino**. *Bull. Inst. Phys.-Chem. Research (Tokyo)* 14, 361-73 (1935), cf. *C. A.* 28, 3431<sup>1</sup>.—The transformation velocity of starch from  $\beta$ - into  $\alpha$ -form, generally, occurring at 40-65°, varies with the temp., 65°-16 hrs., 70°-6 hrs., or 90°-20-30 min. cooking being required for the röntgenographically complete transformation. This is also accelerated by an increase in amt. of water added, and by

dipping the rice grains in water before cooking. Grains swell rapidly with the conversion of  $\beta$ -starch of natural rice into  $\alpha$ -starch of cooked rice, and decompose easily with diastase. K. Konda

**Chemistry of celluloid formation II Heterogeneous transformation in the fibrous state affected by the reaction medium** Motoi Wadano, Kurt Hess and Carl Trogus *Z. physik. Chem. B30*, 159-82 (1935), cf. *C. A.* 26, 3485—The reaction between nitrocellulose and camphor to form celluloid was studied in the solvents MeOH, EtOH, PrOH, iso-PrOH, BuOH, sec-BuOH, dimethylethylcarbinol and iso-AmOH. In addition to the earlier known compd., camphor-nitrocellulose I, 2 new ones II and III were found and identified by x-ray and chem. analysis of the solid phase. The relation of the 3 compds. to each other is discussed. A few expts. were also made in  $\text{CaH}_2$ , toluene, m-xylene and cyclohexene as solvents. **III Catalytic reactions in the fibrous state Heterogeneous catalysis by liquid catalysts in the solid substrate** Motoi Wadano, Carl Trogus and Kurt Hess *Ibid.* 183-231—The formation of camphor nitrocellulose I was studied in  $\text{CaH}_2$  soln. with the catalysts  $\text{Me}_2\text{CO}$ , MeOH, EtOH, BuOH and iso-AmOH. The effect of  $\text{Me}_2\text{CO}$  was studied by means of x-ray diagrams. A reaction mechanism is proposed. **IV Camphorization of nitrocellulose with l-camphor and dl-camphor** K. Hess, C. Trogus and M. Wadano *Ibid.* 232-5—The reactions of d-, l- and dl camphor to form (I) are indistinguishable from x-ray analysis. G. M. Murphy

**The partial molal volumes of ammonia and hydrogen in liquid ammonia hydrogen mixtures under pressure at 100°** R. Wiebe and T. H. Tremearne *J. Am. Chem. Soc.* 57, 2601-3 (1935); cf. *C. A.* 29, 981—The vols. of  $\text{NH}_3$ -H<sub>2</sub> mixts. in the liquid phase from 100 to 800 atm. were measured and the partial molal vols. of both constituents calculated. At relatively high concns. used the partial molal vol. of H in liquid  $\text{NH}_3$  is pos. and increases with the concn. of H except at the lowest pressure. The partial molal vol. of  $\text{NH}_3$  decreases as the concn. of H increases. W. C. Fernelius

**The solubilities of l-proline and l-hydroxyproline in water, the calculated heats of solution, and the partial molal volume of l-hydroxyproline** Tetsuo Tomiyama and Carl L. A. Schmidt *J. Gen. Physiol.* 19, 379-82 (1935)—A curve of sol. of the 2 compds. is given, also a table of coeffs. of the soly equations.  $\Delta H$  for l-proline is 1340, for l-hydroxyproline 1506. Partial molal vols. for l-hydroxyproline are given in a table, the calcd. molal vol. is 85.2. C. H. R.

**The diffusion of ammonium salts with reference to other properties.** Lars W. Ohlman *Finska Kemistisamfundets Medd.* 44, 35-55 (1935)—The diffusion rates of  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{NO}_3$  and  $(\text{NH}_4)_2\text{SO}_4$  were detd. at the temps. 12°, 20° and 23-25° and for normalities ranging from 0.1 to 2.0 N.  $\text{NH}_4\text{Cl}$  gives a min. of  $D = 1.368$  at 0.5 N. at 20° which rises to 1.477 at 4 N. and 1.440 at 0.1 N. For  $\text{NH}_4\text{NO}_3$ ,  $D = 1.403$  at 0.1 N., which gradually drops to 1.228 at 7 N. For  $(\text{NH}_4)_2\text{SO}_4$ ,  $D = 1.067$  at 0.1 N., which drops to 0.787 at 6 N. Relative viscosities and  $\eta$ s also were detd. The viscosity shows a min. of 0.9736 for 2 N.  $\text{NH}_4\text{Cl}$ , and a min. of 0.9738 for 2 N.  $\text{NH}_4\text{NO}_3$ , but for  $(\text{NH}_4)_2\text{SO}_4$  it falls steadily from a value of 1.992 at 6 N. to 1.031 at 0.25 N. H. C. Duus

**The mobility of ions** J. J. Hermans *Z. Physik.* 97, 681-9 (1935)—Born's calcn. (*C. A.* 14, 2113) of the mobility of ions is critically discussed and the exact solution found in the case of infinite diln. This is extended to the case of noninfinite diln. and to the first approximation the mobility is proportional to the concn., not to its square root. B. Swerles

**The mobility of hydrogen and hydroxyl ions in aqueous solutions II** G. Wanner *Ann. Physik.* 24, 569-90 (1935); cf. *C. A.* 30, 1286—The theory is further treated mathematically. The theory agrees fairly well with the mobility of  $\text{D}_2\text{O}^+$  and  $\text{OD}^-$  in  $\text{D}_2\text{O}$ . R. E. DeRicht

**Estimation of transference numbers in dilute solutions from limiting ionic conductances** Benton Brooks Owen

*J. Am. Chem. Soc.* 57, 2441 (1935); cf. *C. A.* 26, 4525—By introducing a semiempirical relation into the equation of Jones and Dole or the Longworth parameter equation the estn. of certain transference nos. from limiting conductivities alone is facilitated. By replacing the parameter  $A$  by the quantity  $-\beta\sqrt{2}(2T\epsilon - 1)/A^2$ , characteristic of the whole group of electrolytes conforming to the Longworth equation, generality is gained and precision impaired only slightly. An accuracy better than one unit in the third decimal place of the transference no. is claimed for this modified equation when applied to dil. solns. of univalent electrolytes in which ionic assocn. is negligible. J. W. Shipley

**Coefficient of activity of ions** Marguerite Quintin *Compt. rend.* 202, 123-5 (1936)—From detns. of  $E_{\text{cell}}$  and  $E_{\text{ref}}$  for varying values of  $m$  in the cells Cd (amalgam 2 phases) |  $\text{CdCl}_2$ ,  $m$  |  $\text{KCl}$ , satd. |  $\text{KCl}$  0.1 N |  $\text{Hg}_2\text{Cl}_2$  |  $\text{Hg}$  and  $\text{Ag}$  |  $\text{AgCl}$  |  $\text{CdCl}_2$ ,  $m$  |  $\text{KCl}$ , satd. |  $\text{KCl}$  0.1 N |  $\text{Hg}_2\text{Cl}_2$  |  $\text{Hg}$  the values of  $(E_{\text{ref}})_1$  and  $(E_{\text{ref}})_2$  are deduced as 0.349 v. and -0.2242 v., resp., and therefrom the values of  $a_{\pm}$  and  $a_{\pm 1}$ , a being the parameter in Debye's theory, as 5.4 and about 2 A., resp. (cf. Gronwall, *et al.*, *C. A.* 25, 5336). This value for  $a_{\pm 1}$  agrees fairly well with that,  $G = 1$  A., deduced from Q's earlier results (*C. A.* 22, 2097). C. A. Silberrad

**Activity coefficients of sulfuric acid in anhydrous ethyl alcohol from electromotive-force data** A. W. Scholl, A. Witt Hutcheson and G. C. Chandlee *J. Am. Chem. Soc.* 57, 2342-4 (1935)—The activity coeffs. of  $\text{H}_2\text{SO}_4$  in EtOH are calcd. from  $e$  m f measurements with the cell  $\text{H}_2$  |  $\text{H}_2\text{SO}_4$  |  $\text{H}_2\text{SO}_4$  (s) |  $\text{Hg}$ . W. B. Keighton

**Transition cases in the distribution of ions** Raymond M. Fuoss *J. Am. Chem. Soc.* 57, 2604-7 (1935); cf. *C. A.* 29, 2824—By using a distribution function for the relation of free ions and ion pairs in an electrolyte a crit. point in the concn. is found above which free and assoc. ions become indistinguishable. This concn.,  $3.2 \times 10^{-4}$  M. at 25° for 1-1 electrolytes, is experimentally the concn. above which the simple laws of dil. solns. of electrolytes are no longer obeyed. At higher concns. higher types of assocn. must be considered for the cases of non-aq. solns. For aq. solns. a numerical significance is attached to the phrase "at low concns." J. W. Shipley

**Calomel electrodes and Gouy's equation for diffusion potentials** M. Chanoz, G. Florence and P. Perrotet *Arch. phys. Biol.* 12, 238-50 (1935), cf. *C. A.* 29, 2825—A crit. discussion. Electrodes using satd.  $\text{KCl}$  soln. are preferred. L. E. Gilson

**Volta effect of electrolytic solutions against water, and characteristics of acidity and basicity** Suzanne Veil *Compt. rend.* 202, 121-3 (1936), cf. *C. A.* 28, 4287; 29, 3896—Two Pt wires inserted in a sheet of gelatin at different points show a p.d. due to heterogeneity of the gel. If one wire is inserted in a drop of pure  $\text{H}_2\text{O}$  on the gel the p.d. remains practically unchanged. If the second wire is inserted in a drop of soln. of some salt (lying on the gel) a p.d. is observed. This is termed the electrometric potential ( $e$  p.) of that soln. It may be considerable, e.g., more than 0.5 v. for satd. solns. of  $\text{Fe}(\text{NO}_3)_3$  or  $\text{Na}_2\text{S}$ . It is pos. if the pos. pole is in the soln. in the cell Pt |  $\text{H}_2\text{O}$  | soln. | Pt, and neg. if the pos. pole is in the  $\text{H}_2\text{O}$ . The  $e$ , p. is pos. for solns. that redden litmus, neg. for those that turn it blue, i.e., acidity and basicity are correlated with the Volta effect of the soln. against  $\text{H}_2\text{O}$ . C. A. S.

**Passage of current in electrolytes without electrolysis** M. Vasilescu-Karpen *Bull. soc. roumaine phys.* 36, 9-16 (1934)—Aq. solns. contg. 50% KI and concns. of 4-30%  $\text{I}_2$  conduct  $c$  with no decompn. Since there is no polarization, solns. contg. 30%  $\text{I}_2$  conduct 150,000 times the current for the same  $e$  m f. as those without  $\text{I}_2$ . The reactions  $\text{I} + e \rightarrow \text{I}^-$  at the cathode and  $\text{I}^- - e \rightarrow \text{I}$  at the anode account for the cond., the net result being the transfer of the electricity. The only resistance is due to the increased concns. at the poles. If the electrodes are horizontal with the anode above, gravity aids the diffusion; hence the current is greater than when in the vertical position. The opposite effect is noted if the horizontal

position is reversed. Dependent on the external resistance, the relative position of the electrodes, etc., an unstable region is reached where the voltage between the plates oscillates regularly. This results in a rhythmic flow of current.

R. E. DeRicht

Determining  $p_H$  with the glass electrode. D. Wolfers *Bull soc chim. bel* 17, 1659-72 (1935).—The Morton-type glass electrode gives excellent results with all concns of HCl and HNO<sub>3</sub> less than 2 N. With undiluted human urine the glass electrode gives values 0.1-0.2  $p_H$  unit lower than those obtained with the quinhydrone electrode. When urine is diluted with 3 vols of redist. water the  $p_H$  increases 0.1-0.3 unit as detd. by the glass electrode or 0.01-0.24 unit as detd. by the quinhydrone electrode.

L. E. Gilson

Exchange equilibrium between deuterium and ammonia. Karl Wirtz *Z. physik. Chem.* B30, 289-97 (1935); cf. C. A. 30, 945<sup>4</sup>.—Equal between NH<sub>3</sub> and D<sub>2</sub> was studied in vessels congt. a Pt filament kept at 300°. The gas pressure was 40-50 mm and equil was reached in 1-5 hr. The uncatalyzed equil was studied in quartz at the same temp. Analysis was by thermal cond. The various equil involved may be reduced to 4 independent ones which were taken as  $H_2 + D_2 \rightleftharpoons 2HD$ ,  $K_4$ ,  $NH_3 + HD \rightleftharpoons NH_2D + H_2$ ,  $K_1$ ,  $NH_3 + NHD \rightleftharpoons 2NH_2D$ ,  $K_5$ ;  $ND_3 + NDH_2 \rightleftharpoons 2NHD_2$ ,  $K_6$ . The analytical precision was not high enough to det. all of these equil consts so  $K_4$ ,  $K_5$  and  $K_6$  were calcd from spectroscopic data and used to det.  $K_1$  from the expts. The latter was 2.0, observed and 1.82, calcd. The distribution quotient,  $V = (D/H \text{ in exchange partners}) / (D/H \text{ in hydrogen})$  is about 1.5.

G. M. Murphy

Exchange reactions with deuterium. I. Deuterium and hydrogen chloride. Paul C. Cross and Philip A. Leighton. *J. Chem. Physics* 4, 28-30 (1936).—An app. for detg. exchange equil and rates of exchange of D<sub>2</sub> with H-contg. compds is described. The gas balance is used to analyze H<sub>2</sub>-D<sub>2</sub> mixts. Results on the equil  $H_2 + 2DCl \rightleftharpoons D_2 + 2HCl$  and  $H_2 + DCl \rightleftharpoons HD + HCl$  agreed with theoretical values.

G. M. Petty

A useful integrated form of the equation for calculating change of equilibrium with temperature. J. B. Austin *J. Am. Chem. Soc.* 57, 2428-34 (1935).—The method is useful chiefly for plotting soly data. If soly, expressed as mol fraction, is plotted against abs temp on double logarithmic coordinates the data fall on a straight line. If the temp ( $T$ ) is expressed as the ratio of  $T$  to some suitable reference temp, such as the m. p. of the solvent, the data for all solvents having the same entropy of melting fall on the same straight line.

J. B. Austin

The thermal equilibrium of the cis-trans isomers of dichloroethylene at high temperatures. William Maroney. *J. Am. Chem. Soc.* 57, 2397-8 (1935).—Earlier detns (C. A. 28, 5400<sup>1</sup>) were extended to 875°. The av. molar heat of transition calcd from the equil consts is 618 cal.

J. B. Austin

Constitution of liquid zinc amalgams. Herman A. Liebafsky *J. Am. Chem. Soc.* 57, 2657-62 (1935).—The e. m. f. data for Zn amalgams were reexam. and interpreted on the assumption that Zn<sub>2</sub> and Zn<sub>3</sub> are in rapid equil with monat Zn. This polymerization hypothesis is supported by the exptl e. m. f. values obtained for the more concd amalgams and fairly well for the more dil.

J. W. Shipley

Concentration maxima of endothermic compounds at high temperatures. Application to ozone and nitric oxide. E. Bruner, B. Suzs and E. Rad. *Helv. Chim. Acta* 18, 1468-78 (1935).—The partial pressures for the decompn of O<sub>3</sub> and NO at high temps were redetd. The equil consts approximate the values of the Nernst theorem, but are lower than those previously reported by B. and S. (C. A. 25, 3570). O<sub>3</sub> has its max. concn at approx. 3500°, with a corresponding partial pressure of  $2.19 \times 10^{-7}$  atm. NO has its max. at about 3750°, with a partial pressure of 0.0987 atm. These differences are due to more exact values for the heat of formation.

J. H. Reedy

The direct carboxylation of carbon compounds. III

The free energy of benzoic acid at 522°K. W. D. Bonner and C. R. Kluney. *J. Am. Chem. Soc.* 57, 2402-3 (1935); cf. C. A. 27, 5068-9.—The free energy of formation of C<sub>6</sub>H<sub>5</sub>CO<sub>2</sub>H from C<sub>6</sub>H<sub>6</sub> and CO<sub>2</sub> at 522°K. was calcd to be 18,172 cal., while the equil. const.,  $K$ , was  $10^{-11}$ .

H. W. Leaby

Carbamate equilibrium. I. Equilibrium of amino acids, carbon dioxide and carbamate in aqueous solution, with a note on the Ferguson-Roughton carbamate method. Wm C. Stadie and Helen O'Brien. *J. Biol. Chem.* 112, 723-58 (1936).—The mass-action law was applied to the equil of amino acids (I) and CO<sub>2</sub> (II) and equations are derived that describe these equilibria. The equilibria were experimentally detd. and the validity of the equations was tested. II and I do not combine to yield carbamates (III) at the isoelec. point of I. HCO<sub>3</sub> or CO<sub>3</sub> ions do not combine with I. Formation of III is inhibited by CH<sub>3</sub>O.

Free II reacts only with the amphoteric I and carbamate compds of I are dibasic salts at all  $p_H$  values greater than 7, hence the equil is expressed by  $K_{Am} = (Am^+)(H^+)/(Z^-)(\alpha_{CO_2}P_{CO_2})$ , where  $Am^+$  is the concn of III, Z<sup>-</sup> the amphoteric concn and  $\alpha_{CO_2}P_{CO_2}$  physically dissolved II. The equil of II and I in the absence of H<sub>2</sub>CO<sub>3</sub> or its ions, termed "non-carbonate equil," was experimentally measured in a special app. and is governed by  $(Am^+)[(N) - (B^+) + (Am^+)] / [(B^+) - 2(Am^+)] = \alpha_{CO_2}P_{CO_2}$ ,  $K_{Am}/K_z$ , where  $N$  is total I, B<sup>+</sup> available base, and  $K_z$  the dissociation const. of I. For glycine (IV), alanine and cysteine acid  $pK_{Am}$  at 20° is 5.54, 5.57 and 5.27, resp.

Equil in the presence of H<sub>2</sub>CO<sub>3</sub> or "total equil," was measured by the usual tonometric method and is governed by a similar equation modified to take into account hydration of II, and for IV  $pK_{Am}$  is 4.81. The Ferguson-Roughton method was applied to apply to systems congt. large amts of free II by addn of sufficient carbonic anhydrase to accelerate hydration of II 200- or 300-fold, thus excluding any mode of combination of I and II peculiar to the  $p_H$  range 5-7.

R. C. Elderfield

Esterification as a gas reaction. C. A. Winkler and C. N. Hinshelwood. *Trans. Faraday Soc.* 31, 1730-43 (1935).—The esterification reaction was chosen as a typical reaction occurring in soln. which it was hoped could be induced to take place in the gas phase. The reaction between HCl and MeOH vapor was studied at 450°. It seemed to be confined to a wall reaction. MeOH and AcOH began to react only at temps at which MeOH began to decompose. With MeOH, AcOH and HCl the results were not reproducible, but certain data were recorded. From 25 to 30% esterification occurred at 300° with the proportions MeOH 100, AcOH 50 and HCl 25 mm. Longer reaction time did not increase the amt. of reaction. The tendency to esterification was increased by increasing the partial pressure of MeOH, but was insensitive to temp. It is thought that reaction takes place in an adsorbed layer of MeOH where ionization of the catalyst can take place, and the formation of this layer depends upon various uncontrollable factors. In CCl<sub>4</sub> soln. the reaction velocity falls off only at very low alc. concn. When MeOH concn is further reduced the rate becomes proportional to it and is proportional to  $[AcOH]^{1/2}$ . As in the gas phase the results could not be reproduced well.

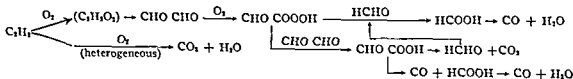
C. E. P. J.

The oxidation of oxalic acid by iodic and accompanied by a change of the stationary adjustment of the intermediate product of the reaction. Emil Abel and Leopold Blumenkranz. *Monatsh.* 66, 181-92 (1935).—Ordinarily the oxidation of (COOH)<sub>2</sub> by HIO<sub>3</sub> yields I ion as an intermediate and I<sub>2</sub>, CO<sub>2</sub> and water as final products. However, when the amt. of I ion in its stationary concn is permanently changed, as by maintaining artificially various concns of the I ion, then a change is brought about in the character of the reaction products. I<sub>2</sub> becomes an intermediate and I ion a final product. The authors attempted an automatic maintenance of the I-ion concn by the addn of AgI + Ag<sub>2</sub>IO<sub>3</sub>, thereby producing a small, const. and calculable I-ion concn,  $(L_{AgI}/L_{Ag_2IO_3})[IO_3^-]$ , where the L's represent the relative soly products. This attempt was not successful because of the slowness in the

establishment of the equilibrium between the solid phase and the substrate. However, without defining it thermodynamically, the authors succeeded in finding a sufficiently constant stationary I-ion concentration from velocity relationships. This was deduced from the reaction-kinetic velocity of homogeneous formation of I-ion and the heterogeneous velocity of its change to the solid phase. The calculation is based on the experimentally determined velocity  $v_s$  of the  $\text{IO}_3^- \rightarrow \text{I}^-$  reaction  $v_s = -d[\text{IO}_3^-]/dt = [\text{IO}_3^-][\text{H}^+][\text{I}^-]^{1/2} + k_p[\text{I}^-]^{1/2} + k_p'[\text{I}^-]$ . The mechanism of this reaction and the kinetics are derived. In the experimental work a concentrated solution of I in benzene was used and kept in constant equilibrium with the aqueous solution by rapid agitation. The details were made at 25° by the method of Abel and Hilferding (C. A. 29, 4247), and the data obtained are given in 4 tables. Experimental results: (1) The oxidation of (COOH)<sub>2</sub> to CO<sub>2</sub> by means of  $\text{HIO}_3$ , in which the  $\text{HIO}_3$  is ordinarily reduced to I, can be varied to yield I-ion by changing the stationary adjustment of the "normally" obtained intermediate reaction product, the I-ion, by artificially increasing the concentration of the latter. (2) This artificial increase to a suitable I-ion concentration is brought about by the addition of  $\text{AgI} + \text{AgIO}_3$  to (COOH)<sub>2</sub> +  $\text{HIO}_3$  solutions. (3) Evolution of CO<sub>2</sub> cannot be observed generally, it accompanies the I reaction under suitable conditions, particularly during the lowering of the I concentration, e.g., by the creation of a suitable distribution equilibrium. (4) Under such conditions a division of the reaction takes place from  $\text{HIO}_3$  to the 2 reaction products CO<sub>2</sub> and I<sub>2</sub>. (5) In agreement with the kinetics resulting from the mechanism developed it was found that an increasing concentration of I favors the production of CO<sub>2</sub>. (6) The ordinary method of calculating the reaction velocity of the I hydrolysis from the distribution of the reaction product between CO<sub>2</sub> and I<sub>2</sub> was shown to be insufficiently checked numerically. Many references. L. S.

The decomposition of propane and n-butane at carbon filaments. Leonard Belchitz and Eric K. Rideal. *J. Am. Chem. Soc.* 57, 2466-9 (1935), cf. C. A. 29, 6128. The decomposition was investigated from the standpoint of radicals and olefin products. The experiments indicate that the initial act is the production of H and the complementary olefin. The latter then dissociates to  $\text{CH}_3$  and the next lower olefin, except in the case of 1,2-butadiene, which undergoes further dehydrogenation to butadiene. The energies of activation for the 2 reactions were determined as 94.2 and 93.2 kJ/mol. H. W. Leachy.

The kinetic treatment of nuclear formation in supersaturated vapors. R. Becker and W. Döring. *Ann. Physik* 24, 710-52 (1935).—Following the work of Farkas (C. A. 21, 1575) and Stranski and Kaishev (C. A. 29, 5324, 6810) a theory is developed for the kinetics of the formation of nuclei in supersaturated vapors. In contrast to the use by these authors of differential equations, series of rigorously constructed algebraic recurrence relations are used, and are solved without involving arbitrary constants. The analysis is carried through for linear, surface and space nuclei. It is applied so as to explain the range of validity of the Ostwald law of progression. The analysis is interpreted in terms of an analogy with the laws of current flow in networks of conducting circuits. M. Muskat.



Formula for the rate of evaporation of adsorbed atoms and molecules. J. K. Roberts. *Trans. Faraday Soc.* 31, 1710-13 (1935).—The formula  $l_p = -2.06 \times 10^{-11} \log_{10} (M/\text{TV}^{1/2})e^{(\phi/RT)}$  was developed to give the rate of evaporation of adsorbed atoms and molecules.  $l_p$  is in sec.  $M$  = mol. wt.,  $\phi$  = mol. heat of desorption in cal. It is consistent with the behavior of adsorbed films of H<sub>2</sub> and O<sub>2</sub> on W. C. E. P. Jeffreys.

Kinetics of the dehydration of gypsum. P. P. Budnikoff and L. A. Schtschukareva. *Kolloid-Z.* 73, 334-9 (1935).—The dehydration of natural gypsum at 107° was approximated by a unimolecular process. The velocity was considerably reduced by 5% NaCl. Addition of 5% KCl stopped the reaction at 107° and the velocity was negligible up to 120°, where the reaction was no longer unimolecular. A sample of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  showed no appreciable rate of dehydration below 130° where the reaction was retarded by 3% NaCl, neither reaction being unimolecular. O. T. Q.

The zero point energy of an activated complex and the reaction  $2\text{NO} + \text{O}_2 \rightarrow 2\text{NO}_2$ . O. K. Rice. *J. Chem. Physics* 4, 53-9 (1936).—It is shown that a reaction whose rate is determined solely by a transitory activated complex in the formation of which classical degrees of freedom must be frozen out into vibrations, must have a positive activation energy at all temperatures, which increases with temperature. A negative activation energy would mean the formation of a stable intermediate compound or "mol. complex". The theory is applied to the reaction  $2\text{NO} + \text{O}_2 \rightarrow 2\text{NO}_2$ . G. M. P.

The dilatometric method for following the hydrolysis of sucrose. David I. Hitchcock and Ruth B. Doogan. *J. Phys. Chem.* 39, 1177-83 (1935).—The velocity constants, calculated from the volume change of sucrose-HCl solutions in dilatometers at 25°, agree with those determined polarimetrically. The rate of volume change in sucrose-invertase solutions is directly proportional to the invertase concentration. The characteristic effects of sucrose concentration and  $p_{\text{H}}$  on the velocity of invertase action are approximately the same in dilatometric as in polarimetric experiments. The total contraction from the complete hydrolysis of 1 mol. of sucrose varies, with concentrations of catalyst and sucrose, from 6.1 to 6.9 cc. Extrapolation to infinite dilution gives 6.92 cc. at 25°. The dilatometric method may be used in place of the polarimetric method. L. E. Steiner.

Thermal decomposition of certain gaseous organic compounds. Morris W. Travers. *Nature* 136, 909-10 (1935).—Further work on the thermal decomposition of gaseous acetylene and ethylene oxide confirms T. C. P.'s earlier opinion (C. A. 29, 1057) that observing the rate of increase of pressure at constant volume as suggested by Hinshelwood (C. A. 28, 7120) does not give a true rate for the chemical reactions or a true idea of the mechanism of the chemical processes involved. The reactions depend upon the surface of the system and involve a "chain" mechanism. O. E. S.

Kinetics of the oxidation of gaseous hydrocarbons. III. The oxidation of acetylene. E. W. R. Steacie and R. D. McDonald. *J. Chem. Physics* 4, 75-81 (1936); cf. C. A. 29, 985, 4246, 7165. The kinetics of the oxidation of gaseous  $\text{C}_2\text{H}_2$  was investigated by the static method. The results show marked irregularities due to variations in the surface. The rate of the reaction is  $-(d/dt)(\text{C}_2\text{H}_2) = K(\text{C}_2\text{H}_2)^{1/2}/(\text{O}_2)^{1/4}$  when  $\text{O}_2$  is in excess. In a packed bed the order is diminished to 1/4. Packing decreases the rate at high pressures but has no effect at low pressures. In an empty vessel the  $\text{CO}/\text{CO}_2$  ratio is independent of the pressure. Packing decreases this ratio, and it further decreases rapidly with diminishing pressure. The apparent heat of activation of the reaction is 35,000 cal.  $\text{HCHO}$  retards the reaction;  $\text{CHO CHO}$  has no effect. The complete course of the reaction is.

G. M. Petty  
Kinetics of the reaction between  $\alpha$ -bromopropionate and silver ions. A heterogeneous reaction taking place on the surface of silver bromide. A. N. Kappanna. *Proc. Indian Acad. Sci.* 2A, 513-24 (1935).—The velocity of the reaction between  $\alpha$ -bromopropionate (I) and Ag ions was studied in very dilute solutions at 27° by detouring the amount of  $\text{H}^+$  formed, by adding KI and  $\text{KIO}_3$  and titrating the  $\text{I}_2$  freed

with 0.01 or 0.003  $N$   $Na_2S_2O_4$ . The hydrolysis of I is extremely slow in pure  $H_2O$  and in solns below 0.001  $N$  even when  $AgNO_3$  is added. The velocity becomes rapid only when  $AgBr$  is present in suspension and decreases if the  $AgBr$  formed in the reaction coagulates. The rate of reaction can be expressed by the equation  $dx/dt = A_1(a-x)(b-x)(c+x)$  in which  $a$ ,  $b$ , and  $c$  are the initial concns. of I, Ag, and  $AgBr$  in suspension. The heterogeneous reaction takes place between I and Ag ions sparsely adsorbed on the surface of the  $AgBr$  particles. Increasing the ratio of the concns of the reactants, other conditions remaining const, causes the velocity const to fall gradually, indicating that the relative orientation of the 2 kinds of ions in the adsorbed layer becomes less and less favorable for reaction. Janet E. Austin

Temperature dependence of the energy of activation in the deacidolization of diacetone alcohol. Victor K. La Mer and Mary L. Miller. *J. Am. Chem. Soc.* 57, 2674-80 (1935).—The velocity const,  $k$ , for the decomposition of diacetone alc. in aq.  $NaOH$  was detd. at 11 temps. from 0° to 50°. At 25°  $k/c$  is independent of the concn. of  $NaOH$  ( $c = 0.02$  to  $0.10 N$ ), but at 50°  $k/c$  decreases slightly as  $c$  increases from 0.008 to 0.06  $N$ .  $E_{act}$  increases from 15,850 cal at 5° to 17,250 cal at 32.5° and then decreases by about 500 cal at 45°. The addn. of 18.5 vol % of  $MeOH$  to the soln increases  $E_{act}$  by 1500 cal at 20°, about as much at lower temps., more at higher temps. Parallel results are obtained for the  $B - T$  curves, the addn. of 18.5%  $MeOH$  producing an increase of one logarithmic unit in  $B$ . A survey of the literature shows that  $dE_{act}/dT$  may be large for reactions involving a halogen compd (3 references), or for reactions which are a hydrolysis or a proton change (7 references). W. B. Keighton

The energies and entropies of activation of the reaction between bromoacetate and thiosulfate ions. Victor K. La Mer and Mildred E. Kammer. *J. Am. Chem. Soc.* 57, 2662-8 (1935).—The effect of ionic attraction on the activation energy,  $E_{act}$ , and on the entropy of activation, or the action const,  $B$ , was studied. For water at 25°,  $E_{act}/2.3RT = E_{act}^0/2.3RT + 0.51Z_AZ_B/\sqrt{\mu}$  and  $B = B^0 + 1.52Z_AZ_B/\sqrt{\mu}$ . At 0 to 37.5° with  $Na_2S_2O_4$  and  $CH_3BrCOONa$  each 0.005  $M$ ,  $dE_{act}/dT$  is about -8.6 cal/deg mole, with 0.18  $M$   $NaCl$  it is about -12.  $B$  also decreases with rising temp. In the presence of low-valence ions ( $Na^+$ )  $\log k$  increases with  $\sqrt{\mu}$  primarily owing to an increase in  $B$ . The addn. of high-valence pos. ions causes a considerable increase in  $E_{act}$ . This rate-depressing effect is more than opposed by the increase in  $B$ . The prevailing theory of catalytic action would predict a lower reaction rate on the addn. of a neutral salt, for  $E_{act}$  is shown to be increased by  $684Z_AZ_B/\sqrt{\mu}$  cal/mole as a result of ionic attraction. The pos. catalytic effect of added neutral salts is due to a larger concomitant increase in  $B$ . This results from an increase in the no. of effective collisions, which in turn arises from the formation of ion clusters. Thus pos. catalysis may result from either (1) a decrease in  $E_{act}$ , which corresponds to providing a lower by-pass in the energy barrier, or (2) an increase in the entropy of activation, which is equiv. to an increase in the no. of systems crossing the energy barrier per unit of time. W. B. Keighton

The influence of nonelectrolytes upon the kinetics of the reaction between bromoacetate and thiosulfate ions. Victor K. La Mer and Mildred E. Kammer. *J. Am. Chem. Soc.* 57, 2669-73 (1935), cf. preceding abstr.—The velocity const. for the reaction between bromoacetate ions and thiosulfate ions in aq. soln at 0° and at 25° is increased by the addn. of nonelectrolytes. Urea raises the dielec. const,  $D$ , but does not affect  $E_{act}$ . Sucrose, glycerol and dioxane each lower  $D$  and decrease  $E_{act}$ . The action const,  $B$ , decreases almost linearly as a function of  $D-1$  from  $D = 88$  to  $D = 55.7$ . This may result from fewer effective collisions as  $D$  decreases because of increased electrostatic repulsion between the reacting ions. When  $D$  is about 50 this repulsive effect is exhausted and ion assocn. between the  $Na^+$  ions and bromoacetate and thiosulfate ions becomes so great that the reaction is sub-

stantially between 2 uncharged ions. In 32% sucrose, as in water, pos. salt catalysis is due to the increase in  $B$  predominating over the simultaneous increase in  $E_{act}$ . W. B. Keighton

Electric conductivity and phase diagram of binary alloys XVII. The system lithium-aluminum G. Grube, L. Mohr and W. Breunung. *Z. Elektrochem.* 41, 880-3 (1935); cf. C. A. 29, 987.—The Li-Al alloys were investigated according to the method of thermal analysis and also by detn. of the temp. coeff. of elec. resistance of the solid alloys as previously described. From these measurements the complete phase diagram was detd. The liquid metals are not sol. in each other in all proportions. The miscibility gap in the liquid state extends at 699° from 44.5 to 60 atoms % Li. Cond. measurements fixed the appearance of a solid soln on the Al side at between 600° and room temp. LiAl forms with excess  $\beta$  solid soln a homogeneous region which extends at 521° from 50 to 56 atoms % Li. The existence of crystal LiAl was detected. It was formed at 521° by peritectic reaction between  $\beta$  solid soln and melt. O. E. S.

Structure of the zinc-cadmium eutectic M. Straumanis and N. Brakss. *Z. physik. Chem. B30*, 117-31 (1935).—The structure of the Zn-Cd eutectic obtained by slow crystn. was compared with that obtained on rapid cooling. Microscopic examn. showed only slight differences. The cryst. fibers are not unit crystals but are composed of alternate layers of the Zn and Cd, growing perpendicular to the cooling surface of the melt. The no. of layers per mm. is 660 in the case of very slow cooling and becomes less with faster cooling. G. M. Murphy

The mutual solubility of aluminum, sodium, potassium and iron nitrates in water in the presence of nitric acid. II. A. I. Zaslavskii, I. L. Fttinger and A. A. Eserova. *Z. anorg. allgem. Chem.* 225, 305-11 (1935); cf. C. A. 29, 6125.—The isotherms at 0° and 20°, for the system  $Al(NO_3)_3$ - $NaNO_3$ - $HNO_3$ - $H_2O$ , are reported. Neither compd. formation nor mixed crystals are found. The solid phases appearing are  $Al(NO_3)_3 \cdot 9H_2O$ ,  $Al(NO_3)_3 \cdot 8H_2O$ ,  $Al(NO_3)_3 \cdot CH_3COOH$  and  $NaNO_3$ . L. E. Steiner

Equilibrium in the system, lithium phthalate-phthalic acid-water. Sterling B. Smith, Wm. A. Sturm and Edward C. Ely. *J. Am. Chem. Soc.* 57, 2406-8 (1935).—Soly. relations in the system  $Li_2C_4H_4O_6$ - $H_2C_4H_4O_6$ - $H_2O$  were detd. and isotherms constructed at 0°, 25°, and 50°. The 3 solid phases present at each of these temps. were  $2Li_2C_4H_4O_6 \cdot 3H_2O$ ,  $LiHC_4H_4O_6 \cdot 2H_2O$  (I) and  $H_2C_4H_4O_6$ . I cannot be purified by crystn. from aq. soln at temps. lower than 60°. W. B. Keighton

Binary systems of  $p$ -dichlorobenzene with biphenyl, naphthalene and triphenylmethane. Ross E. Morris and Walter A. Cook. *J. Am. Chem. Soc.* 57, 2403-6 (1935).—Solubilities, eutectic temps. and eutectic compns. of the 3 binary systems of  $p$ -dichlorobenzene (I) with biphenyl (II), with naphthalene (III) and with triphenylmethane (IV) approach closely those calcd. for ideal solns. The eutectic temp. and eutectic compn. (mole fraction of I) for the 3 systems were: I-II, 26.9°, 0.575, I-III, 39.2°, 0.606; I-IV, 35.9, 0.635. The m. p. of pure IV is 93.2°. W. B. Keighton

Purification and physical properties of organic compounds X. The freezing point diagram for the system acetaldehyde-propionamide. Evald L. Skau and Louis F. Rowe. *J. Am. Chem. Soc.* 57, 2437-8 (1935); cf. C. A. 29, 7770.—The binary  $i$ - $p$  diagram for  $PhNHCOEt$  and  $PhNHCOEt$  was detd. by an accurate static method. The Beckmann method proved unreliable. The system shows compd. formation with incongruent melting. The compd. tends to crystallize in unstable equl. from liquid mixts. near the compd. region. XII. The lower aliphatic bromides Evald L. Skau and Rogers McCullough. *Ibid.* 2439-40.—The b. ps., ds. and i. ps. were detd. for Et, Pr, iso-Pr, Bu, iso-Bu, sec-Bu, and Am bromides. AmBr was found to be dimorphous and monotropic. The unstable form m. -94.6° while the stable form m. -87.9°. H. W. Leach

Catalysis of acetylene polymerization in ultraviolet light by mercury vapor. F. Toul. *Collection Czechoslov. Chem.*

*Communications* 7, 491-2(1935); cf. Kemula, C. A. 29, 7775'.—A reply to tech. objections raised by K. Reply. W. Kemula. *Ibid.* 493-4

**Phase equilibria in hydrocarbon systems XI Thermodynamic properties of mixtures of a crude oil and a natural gas** B. H. Sage and W. N. Lacey. *Ind Eng Chem*, 28, 249-56(1936), cf. C. A. 30, 946.—Sp vols and sp heats were detd. experimentally for several mixts of a crude oil and natural gas from Dominguez Field, Calif. From these data the thermodynamic properties, heat content and entropy were calcd. Detailed tabulations and charts present the behavior of the mixts with changes of temp and pressure. F. L. Browne

**The heat capacity of gadolinium sulfate from 1° to 20.5°K** C. W. Clark and W. H. Keessom. *Physica* 2, 1075-9(1935).—The heat capacity of cryst.  $Gd_2(SO_4)_3 \cdot 8H_2O$  was measured from 1° to 20.5°K and values are tabulated for each 0.5° in this range. The following values of  $C_p$  in cal deg<sup>-1</sup> mol<sup>-1</sup>, taken from the table, illustrate the relation between  $C_p$  and  $T$ : 20.0°K, 4.50; 16.0°K, 2.50; 12.0°K, 1.152; 8.0°K, 0.310; 4.0°K, 0.103; 3.0°K, 0.140; 2.0°K, 0.284; 1.0°K, 0.88. Below 1°K, the heat-capacity curve appears to be rising to the max. theoretically expected for this paramagnetic salt. F. D. Rossini

**Theory of anomalies in specific heat** L. Landau. *Physik Z. Sowjetunion* 8, 113-18(1935).—Math.

Helen S. Hopfield

**Heats of adsorption of gases and vapors upon crystalline adsorbents.** Arthur B. Lamb and Edwin N. Ohl. *J. Am. Chem. Soc.* 57, 2154-61(1935).—The heats of adsorption of  $CH_4$ ,  $C_2H_6$ ,  $N_2O$ ,  $H_2S$ ,  $CO_2$ ,  $CH_3Cl$ ,  $C_2H_5Cl$ ,  $CH_3OH$ ,  $CS_2$ , and  $C_2H_5OH$  on chabasite, of  $CO_2$  and  $CH_3OH$  on brucite, and of  $CO_2$  on dehydrated thomsonite were detd. at 0°. The mol. heats of adsorption of such of these substances as are copiously adsorbed are somewhat larger than those previously observed on charcoal and  $SiO_2$ , etc. The heat of adsorption in general increases with the percentage dehydration of the adsorbent. P. H. Emmett

**Hydrates. heats of solution of hydranion salts at 25°.** H. E. C. Gilbert and V. C. Bushnell. *J. Am. Chem. Soc.* 57, 2611-12(1935); cf. C. A. 29, 2064'.—The heats of soln. in  $H_2O$  at 25° were detd. for hydranion dibromide, anhyd. and hydrated, and of hydranion sulfate, thus completing the series partially reported before. The calorimeter and procedure were as previously described. The extreme slowness with which the sulfate dissolved diminished the accuracy of the results because of the time elapsing while soln. was taking place. The molal heats of soln. of the hydranion salts are compared with those of the alkali and alkaline earth groups. J. W. Shipley

**Sodium hydronide solutions. Heat of dilution at 68°F** John W. Bertetti and Warren L. McCabe. *Ind Eng Chem*, 28, 247-8(1936).—Heats of diln. of solns. const. 13 to 48% by wt. NaOH were measured and corrected to 68°F. The enthalpies at 68°F. relative to infinitely dil. soln. were calcd. F. L. Browne

**Heats of organic reactions III Hydrogenation of some higher olefins** G. B. Kistiakowsky, John R. Ruhoff, Hilton A. Smith and W. E. Vaughan. *J. Am. Chem. Soc.* 58, 137-45(1936), cf. C. A. 29, 4254'.—The reliability of the calorimetric measurements is discussed and some new expts. are presented. It is concluded that the earlier work in these studies is valid. The heats of hydrogenation in cal./mole of the following substances were detd. at 355°K. as: 1-heptene, -30,137 = 37 (60); unsym. methyl ethyl ethylene, -28,491 = 36 (100); 2-pentenes (cis and trans), -27,954 = 28 (100); unsym. methyl isopropyl ethylene, -27,997 = 24 (100); cyclohexene, -28,572 = 10 (100); trimethyl ethylene, -26,920 = 10 (60); tetramethyl ethylene, -26,633 = 19 (100). The deviations represent calorimetric precision, while the bracketed figures are a crude attempt to est. total errors. Some generalizations regarding the effects of substitution on the energetics of an ethylenic double bond are proposed. It appears that with increasing no. of substituent alkyl groups, the heat of hydrogenation is lowered

1 in a progressive manner. Monosubstitution produces a decrease of 27 Cal., disubstitution, 42 (cis) to 52 (trans), trisubstitution, 59, and tetrasubstitution, 62. Thus the differential effect grows smaller with increasing no. of substituents. The effect is independent of the chain length of the normal alkyl radical substituted, but branched groups appear to have greater effect. IV. Hydrogenation of some dienes and of benzene. *Ibid.* 146-53. The following heats of hydrogenation in cal./mole were obtained at 355°K. allene +  $2H_2$ , -71,280 = 103 (200); 1,3-butadiene +  $2H_2$ , -57,007 = 28 (100); 1,4-pentadiene +  $2H_2$ , -60,790 = 64 (150); 1,5-hexadiene +  $2H_2$ , -60,525 = 43 (150); 1,3-cyclohexadiene +  $2H_2$ , -55,367 = 56 (100); benzene +  $3H_2$ , -49,802 = 44 (150); cyclopentadiene +  $2H_2$ , -50,865 = 47 (200). The uncertainties represent calorimetric precision, while the figures in parentheses are the crudely estd. all-inclusive errors. The data are discussed and it is pointed out that 2 double bonds, when placed close together in the mol., exert an unstabilizing effect upon each other. The resonance predictions of Pauling are confirmed but qualitatively. William E. Vaughan

Constitution of complex metallic salts (Mann, Purdie) 6.

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### 3—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

W. ALBERT NOYES, JR.

- Limits of precision in reference to atomic systems. C. G. Bedrag. *Bull. soc. roumaine phys.* 36, 31-3 (1934). Cf. *C. A.* 29, 2798<sup>1</sup>.—A further discussion with reference to the effect of inequalities in the Bohr-Heisenberg theory. R. E. DeRight.
- Elements of the quantum theory. VII. Van der Waals forces. Saul Dushman. *J. Chem. Education* 12, 581-6 (1935). VIII. Perturbation theory. *Ibid.* 13, 84-92 (1936). Cf. *C. A.* 30, 15<sup>1</sup>. E. H.
- Quantized field theory and mass of the proton. Max Born. *Nature* 136, 952-3 (1935); cf. *C. A.* 29, 33<sup>1</sup>, 1317<sup>1</sup>.—Math. Gregg M. Evans.
- Photographic method in the investigation of the atom. Marietta Blau and Ilhertha Wambacher. *Phot. Kor. Suppl.* 70, 31-40 (1934). Cf. Blau, *C. A.* 29, 3653<sup>1</sup>.—A general account of the technique employed in the study of at structure and at processes by the aid of photography. E. R. Bullock.
- The electron configuration of superconducting metals. U. Dehlinger. *Physik Z.* 36, 892-4 (1935). Cf. *C. A.* 30, 924<sup>1</sup>.—A theoretical discussion. C. E. P. Jeffreys.
- Uses and limitations of the spectrograph for industrial control. Thomas A. Wright. *Metal Progress* 29, No. 1, 53-7 (1936). Cf. *C. A.* 29, 7854<sup>1</sup>.—Forty successful uses in different fields are mentioned. W. A. Mudge.
- The dissociation energy of carbon monoxide. R. Schmid. *Roy. Hung. Palatin-Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met.* 7, 171-82 (1935).—Former papers (cf. *C. A.* 29, 779<sup>1</sup>) gave the value of  $D(\text{co})$  as 8.4 v or 194 kg.-cal. Calcs. of various quantities concerning the C atom and CO mol showed that (1) the sublimation heat for solid C equals 108 kg.-cal. and  $\lambda = 205$  kg.-cal. (2) The bond energy C-C is 102 kg.-cal for solid and 99 for aliphatic C compounds. The H-C bond represents 107 kg.-cal. (3) Calcs. of cyanogen yield led to the formula  $\text{CN}(\text{?}) + 9.5 \text{ v} \rightarrow \text{C}(\text{?}) + \text{N}(\text{?})$ , while states  $\text{H}$  and  $\text{?}$  dissociate into  $\text{C}(\text{?})$  and  $\text{N}(\text{?})$ , the latter lying under the former by 1.8 v. (4) The band convergence method led to the calcd. value for ionization potential  $\text{CO} \rightarrow \text{CO}^+ - 13.15 \text{ v}$ . (5) Crit. potentials calcd. are somewhat higher than observed. The value of  $D(\text{co}) = 8.4 \text{ v}$  still needs confirmation. S. S. de Finály.
- Double excitation of helium by electron impact. H. S. W. Massey and C. B. O. Mohr. *Proc. Cambridge Phil. Soc.* 31, 601-8 (1935).—Calcd. values of the probability of excitation of He to doubly excited levels agree qualitatively with the relative intensities and distribution of observed lines. E. O. Wug.
- Collision of K ions with gas particles of inorganic and organic substances. III. Relation between effective cross section and area under mass-spectrographic distribution curve. Otto Schmidt. *Ann. Physik* 25, 92-6 (1936). Cf. *C. A.* 29, 1707<sup>1</sup>.—Irron previous measurements with about 20 mono- and poly-atomic gases it is shown that the ratio of the 2 quantities named (effective cross section by Ramsauer's method) is const. for gases in a related series. C. D. West.
- The quantity of positive ions at the cathode in an electrical discharge. T. I. Câmpian. *Bull. soc. roumaine phys.* 36, 61-8 (1934).—Although the mean energy of the pos. ions is very small, 85% of the current in H and 80% in air is carried by them. These differences are due to the secondary emissions and are diminished by evacuation. The same relative effects in H and the air are found throughout the pressure range 0.1-2.5 mm. R. E. DeRight.
- Photoelectric effect and photoconductance of crystalline semiconductors. C. Athanasu. *Bull. soc. roumaine phys.* 36, 39-50 (1934).—The 2 phenomena are identical, except that in the latter case the liberated electron must travel some distance. Thus, its effect is lost if it is absorbed within the crystal before reaching the electrode. This, in the latter case, is offset by a reduction in temp. which increases the "mean free path". The wave length of max effect on photoconductance is longer than that for the photoelec. effect. Lowering the temp. to  $-145^\circ$  shifts the max of the former toward shorter wave lengths but has no effect on the latter; hence, at this low temp., the 2 points for argentite coincide. As abs. zero is approached the cond. falls off steadily and approaches infinite resistance. R. E. DeRight.
- Effect of pressure on the radiation of the electric discharge in cadmium vapor. V. A. Fabrikant and A. S. Kanel. *Compt. rend. acad. sci. U. R. S. S.* 3, 257-8

(1935); cf. Elenbaas, *C. A.* 29, 361—Variation of the vapor pressure in a discharge in Cd vapor produces changes in radiation as with Hg (cf. Krefl, *C. A.* 29, 1322). Increasing the Cd pressure increases the radiation intensity and yield to a max. at about 0.1 mm., after which they fall to a min. at about 1 mm. and then rise again. A perceptible contraction of the discharge occurs simultaneously with the latter rise. The measurements were made principally on  $\lambda\lambda$  5085, 4800 and 4676 Å. The effect of added inert gases is to flatten the curve, the min. disappearing completely at higher pressures. Å is more effective than Ne.

Temperature distribution and electron density in freely burning arcs. Hans Hörmann *Z. Physik* 97, 539-60 (1935).—A new detn. is made for the lengthwise field strength in freely burning and in stabilized arcs at atm. pressure. The const. of the Ayrton equation is found to be too great by a factor of 2 in the case of the C arc. The ionization in the C arc is not appreciably altered by impurities. From the intensity distribution of the spectral lines in the cross section of the arc the specific radiation density is obtained. Alkali arcs are also examd. The temp. distribution of the arc can be detd. and from this the ionization gradient is obtained. S. Tolansky.

Electron diffusion in the bulb of the mercury rectifier. D. R. Kanaskov *Fizik Z. Soejetunio* 8, 119-35 (1935).—The discharge phenomena in the dark part of the bulb were investigated, an exploring electrode was used. A linear relation was found between the logarithm of the current of electrons of random velocities at a point on the axis of the tube, and the distance of that point from the cathode spot. There is also a linear relation between the logarithm of the electron concn. at a given point on the axis and its distance from the cathode. The change of potential along the axis of the tube is linear. Differential equations for this case are developed on the basis of Schottky's theory, and results obtained from their solution agree with expt. Helen S. Hopfield.

The radial distribution method of interpretation of electron-diffraction photographs of gas molecules. Linus Pauling and L. O. Brockway *J. Am. Chem. Soc.* 57, 2684-92 (1935); cf. *C. A.* 29, 677.—The expression for the coherent scattering is inverted, giving a radial distribution function which is the sum of a Fourier series, the coeffs. of which are the visually estd. intensities of the rings on the electron-diffraction photograph. The method leads directly to the important interat. distances with a probable error of 1 to 2%, and thus eliminates many possible models. The method was tested on Br<sub>2</sub>, Cl<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, CS<sub>2</sub>, COS, CCl<sub>4</sub> and other tetrahedrals. Revised interat. distances and bond angles are given for CF<sub>4</sub>, SiF<sub>4</sub>, CCl<sub>4</sub>, SiCl<sub>4</sub>, GeCl<sub>4</sub>, SnCl<sub>4</sub>, PF<sub>5</sub>, AsF<sub>5</sub>, PCl<sub>5</sub>, AsCl<sub>3</sub>, CH<sub>3</sub>Cl, CHCl<sub>3</sub>, Cl<sub>2</sub>O, (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O, ClO<sub>2</sub> and SO<sub>2</sub>. Victor Hicks.

The electron-diffraction investigation of phosgene, the six chloroethylenes, thiophosgene,  $\alpha$ -methylhydroxylamine and nitromethane. L. O. Brockway, J. Y. Beach and Linus Pauling. *J. Am. Chem. Soc.* 57, 2693-704 (1935).—The at. arrangements in the mols. have been detd. from electron-diffraction photographs by both the radial distribution method (cf. preceding abstr.) and the usual visual method. In phosgene, thiophosgene, and the 6 chloroethylenes, the C-Cl distance varies between 1.67 and 1.73 Å, compared with the normal single bond distance 1.76 Å. This decrease is ascribed to the partial double bond character. The bond angle Cl-C-X varies between 122° and 123°45', compared with the normal tetrahedral angle 125°16'. Other interat. distances detd. are in good agreement with previous data. Victor Hicks.

The dependence of interatomic distance on single bond-double bond resonance. Linus Pauling, L. O. Brockway and J. Y. Beach *J. Am. Chem. Soc.* 57, 2703-9 (1935).—A relation between the bond character of C to C bonds for single bond-double bond resonance and the interat. distance is plotted from certain exptl. data, and tested with other data. The electronic structures of mols. contg. various conjugated double or triple bonds (cf. preceding abstr.) or aromatic nuclei and the dependence of bond angles on single bond-double bond resonance are discussed. Some

C to C distances in polynuclear aromatic hydrocarbons are predicted and found to be in good agreement with exptl. data. Twenty references. Victor Hicks.

Diffraction of electrons by rubber films. K. I. Krulov. *Fizik Z. Soejetunio* 8, 136-52 (1935).—The passage of a beam of electrons through a thin film of natural rubber which is not under strain gives a diffraction picture consisting of rings, showing amorphous structure. Stretching the film produces distinct points in the diffraction pattern, indicating crystal structure. Most (but not all) of these points can be explained by the model of Susich, Mark and Meyer for the unit of crystal structure of this substance. Cooling the stretched film by liquid air gives higher orders of reflection. A film of synthetic chloroprene rubber shows these points even when it is not stretched. The points indicate an orthorhombic crystal as in the case of natural rubber, but with axes of different lengths. H. S. H.

A new optical method for determining the elastic constants of crystals. C. Schaeffer and L. Bergmann. *Atti accad. Lincei* 21, 701-2 (1935).—Quartz crystals are excited to a rapid and elastic oscillation by means of a field varying between  $10^5$  and  $10^6$  oscillations/sec., by utilizing the properties of piezoelectricity. If a ray of monochromatic light is passed through this vibrating crystal, a diffraction pattern is formed which depends on the elastic properties of the crystal, but is independent of the crystal structure, and of the type of excitation. The method can be applied to nonpiezoelectric crystals by cementing the crystal onto a quartz plate of appropriate size. In this way topaz, barite, feldspar and beryl have been investigated. Illustrations of the patterns obtained are shown. A. W. Condon.

Charge potential and secondary emission of electron-irradiated substances. M. Knoll *Fizik Z. Soejetunio* 8, 119-35 (1935).—App. and methods for study of charge potential and electron emission of a wide variety of substances are described. C. E. P. Jeffreys.

The measurement of small light intensities by means of counters. H. Karl H. Kreuchen. *Z. Physik* 97, 625-32 (1935). cf. *C. A.* 29, 5341.—The previously described methods are used. The curves for the photoelec. yields of Zn, Cd and Cu tubes are measured in the region 400 m $\mu$  to 185 m $\mu$  for massive and evapd. counter cathodes. The massive Cd and Zn cathodes when treated with active H<sub>2</sub> are not different from the evapd. cathodes. Treating the evapd. cathodes does not raise the sensitivity. S. Tolansky.

An amplifier for currents of the order of  $10^{-12}$  amperes. Hannes Allén. *Z. Physik* 97, 708-17 (1935).—A thermionic-tube amplifier is described and the theory discussed. The sensitivity is very great and the ionization from individual  $\alpha$ -particles can be measured. S. Tolansky.

Foreign ionization and reduction in sparking potential in gases. W. Rogowski and A. Wallraff. *Z. Physik* 97, 785-91 (1935).—It is shown that the reduction in sparking potential due to foreign ionization obeys the root law of Rogowski and Fucks. With very high foreign ionization the reduction grows slowly and ultimately increases with the fourth root of the foreign gas. S. Tolansky.

Production of cosmic ray showers at a considerable depth below ground level. D. H. Pollett and J. D. Crawshaw. *Nature* 136, 1020 (1935).—Five counters in pentagonal formation at a depth corresponding to 60 m. of H<sub>2</sub>O were used. Three particles were required for simultaneous discharge. Showers were produced in the earth above the app. Shower production was increased by a Pb shield. A thickness of 1 G cm gave the max. effect. A like result is found on the surface. The results indicate that the showers are produced by pos and neg. electrons. R. E. DeRight.

Investigation of ionization by cosmic radiation with a double chamber. W. Messerschmidt. *Fizik Z. Soejetunio* 8, 119-35 (1935).—The absorption of Hoffman "Stöbe" was measured by means of a double chamber. They showed a half-value thickness of 3.5 cm. Pb. The relation between shower groups studied in the Wilson chamber and the "Stöbe" was discussed. C. E. P. Jeffreys.

Theory of the  $\beta$ -disintegration and the allied phenom.

enon Hideki Yukawa and Shiroshi Sakata. *Proc Phys-Math. Soc. Japan* 17, 467-79(1935)(in English).—Based upon Fermi's neutrino theory of  $\beta$ -disintegration (*C. A.* 28, 5329)—It is predicted that, if the difference  $\Delta I$  of proper energies of  $Z$  isotopes with at. nos  $Z$  and  $Z-1$  is larger than  $-mc^2 + \mu c^2$  ( $m$  and  $\mu$  are the masses of the electron and of the neutrino, resp.), the isobar  $Z$  will change into the isobar  $Z-1$  by absorbing one of the orbital electrons. The probability of such a process is calculated, when the electron to be absorbed is initially in one of the  $K$  states, and its bearing on the problems of the mass of the neutrino and the nuclear spin is discussed. The importance of this process in the ordinary disintegration by positron emission is considered for  $\Delta I$  larger than  $mc^2 + \mu c^2$ . G M P

Deviation of electrical charge distribution from spherical symmetry in some atomic nuclei H. Schuler. *Physik Z.* 36, 812-14(1935).—Calen shows a dissymmetry of charge distribution in the nuclei of Cu, Hg and Bi.

C. I. P. Jeffreys  
The asymmetry of the electrical charge distribution of the nucleus of  $^{201}\text{Hg}$  H. Schuler and Th. Schmidt. *Z. Physik* 98, 239-51(1935).—From very exact measurements upon the hyperfine structures of the  $\text{Gr}^{67}\text{P}_1$ ,  $\text{P}_2$ ,  $\text{Gr}^{67}\text{S}_1$  and  $\text{Gr}^{67}\text{P}_1$  terms of the spectrum of Hg I, deviations from the Landé interval rule are found. These are attributed to an asym. distribution in the nuclear electric charge, and from this the quadrupole moment is found to be  $0.5 \times 10^{-24}$ . In Hg there is not a pure Russell-Saunders coupling. The relativity correction for the terms is considered. The fact that the quadrupole moment has a positive value indicates that the nucleus is lengthened in the direction of the nuclear spin axis. A new detn. is made for the fine structure of the green Hg line 5461 Å and an improved fine-structure analysis made. S. Tolansky

Creation of positive and negative electrons by heavy charged particles. Yoshio Nishina, Shin-ichiro Tomonaga and Minoru Kobayashi. *Sci. Papers Inst. Phys. Chem. Research (Tokyo)* 27, 137-78(1935).—Math. E. O. Wig

Production of neutrons by annihilation of protons and electrons according to Fermi's theory F. Bloch and C. Møller. *Nature* 136, 987(1935).—Math-theoretical. G M P

Absorption of residual neutrons Leo Szilard. *Nature* 136, 950-1(1935), cf. *C. A.* 29, 2441<sup>1</sup>, 4257<sup>1</sup>.—Neutrons which have passed a 1.5-mm Cd sheet are only 10% absorbed by another Cd layer of 0.5 mm, yet are  $1/2$  absorbed by a 0.4 mm sheet of In. Discussion of the theory involved follows, showing that this is contrary to current theory. These residual neutrons show strongly selective absorption in certain elements such as I. G M E

Resonance levels for neutron capture Hans v. Halban, Jr. and Pierre Preiswerk. *Compt. rend.* 202, 133-5 (1936), cf. *C. A.* 30, 19<sup>2</sup>.—Results obtained by the authors and others (cf. Bjerge, et al., *C. A.* 29, 6458<sup>7</sup>, Szilard, preceding abstr.) are summarized. It is probable that in the region of energies greater than  $kT$  there are maxima of resonance for the capture of neutrons by nuclei (cf. Perrin and Elsäesser, *C. A.* 29, 2837<sup>4</sup>; Bethe, *C. A.* 29, 5010<sup>9</sup>). C A S

Experiments with slow neutrons P. I. Lukurskil and T. V. Zareva. *Compt. rend. acad. sci. U. R. S. S. (N. S.)*, 3, 393-6(1935).—From the fact that slowing down of neutrons is accompanied by an increase in the intensity of nuclear reactions, it may be concluded that most slowly moving neutrons possessing thermal velocities are most effective in producing reactions. This was proved by the temp. effect found with a Ag target. The neutron source was a tube contg. Rn and powd. Be. The Ag target was subjected to the action of the neutrons and the decay curve then obtained with a Geiger-Müller counter. With a 10-cm. layer of paraffin surrounding the target no increase in activity of neutrons was observed when the wax was cooled from room temp. to that of liquid air. In a series of expts. with the target surrounded by paraffin 1.7, 1.0 and 0.8 cm. thick, the increase in activity on the cooling was 13, 24 and 45%, resp. The thicknesses used were sufficient for the neutrons previously

1 slowed by  $\text{H}_2\text{O}$  and paraffin to acquire the velocity corresponding to the temp. The expts. confirm the temp. effect and show the absorption of neutrons by paraffin. With the 10-cm. layer absorption entirely compensated for the temp. effect. C E P J

Experiments with neutrons slowed down at different temperatures C. H. Westcott and H. Niewodniczański. *Proc. Cambridge Phil. Soc.* 31, 617-24(1935).—Expts. are described with neutrons slowed down by passage through a paraffin block cooled to liquid-N or liquid-H temps. and also with liquid H replacing all or part of the paraffin block. Absorption of neutrons by Cu, Ag, Rh and Cd increases as the temp. of the block is lowered. The transformations produced in these substances also increase but to a smaller extent, the effect appearing to depend on the thickness of the layer of cooled paraffin or liquid hydrogen. E O Wig

Slowing down of neutrons by collision with protons Hans von Halban, Jr. and Peter Preiswerk. *Nature* 136, 951-2(1935).—Spheres with radii from 5 to 15 cm. were filled in turn with  $\text{H}_2\text{O}$ ,  $\text{F}_2\text{O}$ ,  $\text{C}_2\text{H}_6$  and a liquid paraffin. With a source of neutrons at the center and an activated Ag plate in the surface the absorption by the liquids was measured and plotted against the mass of H per cc. of the liquid. Differences are found to be unexplained by differences in H d; so they must be due to differences in the structure of the compounds. Gregg M. Evans

Disintegration by slow neutrons J. Chadwick and M. Goldhaber. *Proc. Cambridge Phil. Soc.* 31, 612-16(1935), cf. *C. A.* 29, 2441<sup>1</sup>.—Bombardment of all the elements up to Al and also of Cl, A, Ca, Ni, Cu, Zn, In and U by slow neutrons results in marked disintegrations with only Li and B. N shows a small effect. The reaction with B is  $\text{B}^{10} + n \rightarrow \text{Li}^7 + \text{He}^4$  rather than that previously suggested (*C. A.* 29, 2441<sup>1</sup>). E O Wig

Production of secondary  $\gamma$  rays by neutrons R. Fleischmann. *Physik Z.* 36, 806-8(1935); cf. *C. A.* 30, 377<sup>4</sup>.—Paraffin, Fe, Cu, Cd and Pb emit  $\gamma$ -rays on the capture of a slow neutron. The yield is about 1 quantum per neutron. C E P Jeffreys

Evaluation of the exactness of Bethe and Peierls' formula, which is derived from the splitting of the deuteron by  $\gamma$  rays. V. I. Mamaschikov. *Physik Z. Sowjetunion* 8, 200-7(1935).—A formula for the cross section of the deuteron is given, with reference to the photoeffect on the nucleus. It is developed as a power series in  $r_0$ , the greatest distance at which forces between the proton and neutron are manifest. The first member of the series agrees with the formula of Bethe and Peierls, which is therefore considered only an approximation. Helen S. Hopfield

Emission of positrons by a Th B + C source Sergio De Benedetti. *Compt. rend.* 202, 50-2(1936), cf. *C. A.* 29, 3707<sup>4</sup>.—The no. of positrons resulting from the action of radiation from Th B + C on screens of Cellophane, Al and Pb, after deduction of those arising in the source and traversing the screens, is in accordance with the theory of materialization of  $\gamma$ -rays. Emission of positrons by radiation other than  $\gamma$ -rays is not detectable after such radiation has traversed a thickness of matter of 0.55 g./sq. cm. C A. Suberrad

Proton spectra of the elements magnesium, silicon and sulfur on bombardment with fast  $\alpha$  rays O. Hazen. *Physik Z.* 36, 804-6(1935).—Mg, Si and S emit protons on  $\alpha$  ray bombardment, and the yields indicate that the emission is associated with the principal isotopes. The energies of the protons allow the energies of the excited states of the nuclei produced, Al, P and Cl, to be estd. These energies are the same within the limits of error. The nuclei differ only by an  $\alpha$ -particle. C E P. Jeffreys

Chemical detection of artificial transmutation of the elements P. A. Paneth and H. Lohr. *Nature* 136, 950(1935).—Froughe has been obtained from bombardment of B with neutrons to be identified spectroscopically and to be measured. 2200 millieuries of radon were used and  $1.3 \times 10^{-7}$  cc of He was obtained. G M E

Recall by  $\beta$ -decay. F. Bloch and C. Møller. *Nature* 136, 911-12(1935).—The probable relation between the

direction of emission of the electron and that of the neutrino in a  $\beta$ -decay is discussed. Oden F. Sheppard

The excitation of K-radiation from nitrogen, oxygen and neon by  $\alpha$  particles Wolfgang Rietzel. *Ann. Physik* 24, 71-18 (1935).—Counter experiments on the excitation function for the intensity of the K-radiation excited in  $N_2$ ,  $O_2$  and Ne  $\alpha$ -particles (13.35 mm range in air) gave curves of the Bragg type for the total  $\alpha$ -particle ionization in air. The  $N_2$  curves show a flat max. at an  $\alpha$  particle range of 30 mm. The max for  $O_2$  lies at 35 mm while that for Ne lies beyond 45 mm. However, the Ne curve indicates an inflection point at 13 mm. Morris Muskat

The magnitude and structure of the absorption coefficients of harder  $\gamma$  rays W. Gentner. *Physik Z.* 36, 810-12 (1935), cf. *C. A.* 29, 2074.—The absorption coeff. of  $\gamma$  rays in Pb was measured as a function of wave length. The absorption coeff. of Th C' rays ( $h\nu = 2.65 \times 10^6$  e.v.) was plotted against  $\lambda$ . The results agree with the theory and show that Compton effect, photoelectric effect, and pair formation are sufficient to account for  $\gamma$  ray absorption. C. I. P. Jeffreys

Recording the ionization curve of a single  $\alpha$ -particle Hannes Alfvén. *Z. Physik* 97, 718-24 (1935). The ion column produced by a single  $\alpha$  particle moves with constant velocity in a uniform field, so that the total ionization can be brought into a discharge chamber wherein it produces a current obeying Bragg's law. The current is of the order of  $10^{-12}$  amp and can be measured and observed with an oscillograph. The method employed may have applications for observing individual nuclear reactions. S. Tolmnsky

The distribution of thorium C' in thallium salt solutions Johannes Zirkler. *Z. Physik* 98, 75-6 (1935), cf. *C. A.* 29, 2910.—It has been shown that the radioactive TI isotopes Th C' does not distribute itself between the TI<sup>+</sup> and TI<sup>3+</sup> ions according to the masses present. If the radioactive isotope is added to a mixt. of TI<sup>+</sup> and TI<sup>3+</sup> of equal concn., then NH<sub>3</sub> pptn. of the TI<sup>3+</sup> shows that the TI<sup>3+</sup> contains about 30% of the total  $\gamma$ -ray activity, and the ppt. formed with the TI<sup>+</sup> and HCl contains 70% of the  $\gamma$ -ray activity. The relative abundances of the TI isotopes 203 and 205 are 29.6 and 70.4%. The valency change thus leads to an isotope sep., the heavier isotope tending more to go to the single-valued ion form. S. T.

Determination of the radium content of rocks Wm. D. Vry. *J. Chem. Physics* 4, 40-8 (1936).—The determination of the age of common rocks by the "He method" (*C. A.* 28, 2088) necessitates accurate detns. of Ra. The method of compensating the background by the use of two opposed ionization chambers is applied to Ra detns., and the app. is an observational limit of  $5.5 \times 10^{-12}$   $\alpha$  per g. Ra for  $n$  hourly readings. A summary of results for a suite of rocks from a single horizon and the present status of a geol. time scale is given. G. M. P.

Radioactivity of oil waters in Czechoslovakia I. Böhmeček, V. Santholzer and I. Ulrich. *Nature* 136, 910-11 (1935).—The Ra contents of 30 samples of water from different oil-well borings at Gibly and one sample from a boring at Jasna, Czechoslovakia, were detd. The highest Ra content was  $7.25 \times 10^{-16}$  g. per l. in water from a depth of 262 m. Only 8 samples had Ra contents as high as the order of  $10^{-16}$ , the others being of the order of  $10^{-17}$  to  $10^{-18}$ . No correlation between depth and Ra content was found. The presence of elements of the Th series could not be proved. The Ra contents of the oil waters of Czechoslovakia are much lower than in oil waters of Russia as pointed out by Khlopina and Verbitskii (*C. A.* 26, 5007).

Electrolytic separation of the oxygen isotopes Geoffrey Ogden. *Nature* 136, 912 (1935).—The electrolytic sep. coeff. for the isotopes of O is found theoretically to be between 0.8 and 1, depending on the width and height of the energy barrier, if account is taken of the possibility of "tunneling" of the barrier by the O<sup>16</sup> or O<sup>18</sup> complex. This sep. coeff. apparently depends on the nature of the electrode and of the electrolyte, but the above range of values is in good agreement with the available exptl. results. This means that while the concn. of D increases

1 from about 1/5500 to 100% by the electrolytic method the O<sup>18</sup> isotope concn. increases from 0.15 to 0.6% when the most favorable coeff. is used. Oden F. Sheppard

Isotopes of nickel J. de Gier and P. Zeeman. *Proc. Acad. Sci. Amsterdam* 38, 810-13 (1935).—As a result of work with the parabolic mass spectrograph none but even isotopes was detected, the 61 isotope reported by Aston (cf. *C. A.* 29, 3910) being absent. The 61 isotope, about which Aston was in doubt, is confirmed. The proportions of isotopes 58, 60, 62 and 64 were 68.1, 27.2, 3.4 and 0.4%, resp. Gregg M. I. vras

Isotopic structure of iridium A. J. Dempster. *Nature* 136, 909 (1935).—Direct mass analysis of the Ir ions formed by a high frequency spark between electrodes made of Pt-Ir alloy has verified the prediction of Venkatesachari and Subram (cf. *C. A.* 30, 199) relative to the existence of Ir isotopes 191 and 193. The heavier isotope is the more abundant. These two isotopes fill the only gaps in the mass no. scale in this region. Oden F. Sheppard

State of electrons in crystal lattices P. Hund and B. Mrowka. *Physik Z.* 36, 888-91 (1935)

C. I. P. Jeffreys  
Artificial crystal lattices for interference with visible light Walter Kraus. *Physik Z.* 36, 841-3 (1935).—A method is described for obtaining artificial crystal lattices by the use of standing light waves. C. I. P. Jeffreys

Effect of Röntgen and  $\gamma$  rays upon piezoelectric crystals I. Seidl and I. Huber. *Z. Physik* 97, 671-80 (1935).—Quartz and Rochelle salt crystals are examined with x-rays and with radioactive radiations. The short waves employed produce a definite increase in the piezoelectric constants. The piezoelectric constants are increased in some Rochelle salt crystals and in others are reduced. Quartz crystals exhibit diverse behavior. The influence upon the piezoelectric constants is attributed to a secondary effect. Radioactive radiations turn Rochelle salt crystals yellow. S. Tolmnsky

X-ray interference by contact of radiation source and crystal II Seemann. *Physik Z.* 36, 837-41 (1935).—With the single-crystal antineutrone with sep. approaching zero of the radiator and lattice, interference lines were clearer and more completely obtained. The exptl. arrangement is described and some diagrams of rock salt are shown. C. I. P. Jeffreys

Quantitative analysis of mine dusts—an x-ray diffraction method George L. Clark and Dexter H. Reynolds. *Ind. Eng. Chem., Anal. Ed.* 8, 36-40 (1936).—A known amt. of a crystal substance not present in the dust is added to it. The ratio of the photographic ds. of adjacent diffraction lines from the added and sought constituents is detd. photometrically. The amt. of the sought constituent is then read from a curve giving the relation of similar ratios to the compn. of the sample; the data for this curve are obtained from synthetic standards. For quartz, the detns. can be duplicated to within 5% of the amt. sought. Victor Hicks

New investigations on lines of helium and hydrogen with crossed electric and magnetic fields W. Stuehling. *Physik Z.* 36, 822-5 (1935), cf. *C. A.* 30, 379.—Further work on the combined Stark and Zeeman effects is described. C. I. P. Jeffreys

The influence of foreign gases upon the higher series lines of sodium Chr. Lächthauer and P. Schulz. *Z. Physik* 97, 699-707 (1935).—The broadenings produced in the lines of the ends of the Na series by He, Ne, Ar, H<sub>2</sub> and N<sub>2</sub> are measured. With all the gases the breadth falls with the series no. of the line, finally attaining a constant limiting value. The final value reached depends only upon the foreign gas used. Apart from He, the final widths and the displacements increase with the order no. The interaction of the gases differs at the beginning and end of the series. There exists an asym. effect to the red. S. Tolmnsky

The spectrum of rubidium K. W. Meissner. *J. Physik* 98, 453-4 (1935).—With reference to the results of Dutta and Bose (cf. *C. A.* 30, 379) it is pointed out that the sep. of the D term of Rb has already been measured with great accuracy by Ramb (*C. A.* 25, 5089), who fur-

ther showed, in contradiction to D. and B. that the 3D term is double

S Tolansky  
Dispersion of air, krypton and xenon in the short-wave ultraviolet. Wilhelm Krongraber. *Z. Physik* 98, 17-22 (1935).—The dispersion curves are measured with a Jamin interferometer of quartz. For air the region examined is 5095-2144 Å, for Xe and Kr it is 6707-2025 Å. Dispersion formulas are obtained which give absorption frequencies of a shorter wave length than those measured by Cuthbertson (*C. A.* 5, 232). According to the quantum theory such a deviation is expected from the correspondence principle.

S. Tolansky  
The spectrum of rotation-vibration of the molecule HD. G. Wick. *Atti accad. Lincei* 21, 709-14 (1935).—HD, which is not rigorously homonuclear, probably shows a faint rotation-vibration spectrum. The vibration spectrum is calculated.

A. W. Conner  
The absorption spectrum of hydrogen. II. The ordering of the D level into the term scheme of hydrogen from exposures of H<sub>2</sub> and D<sub>2</sub>. H. Beutler, A. Deulner and H. O. Jünger. *Z. Physik* 98, 181-97 (1935), cf. *C. A.* 30, 22.—By using the continuum of a He discharge the absorption spectra of H<sub>2</sub> and D<sub>2</sub> are taken in the region 900-600 Å with a 1-m grating. In para-H<sub>2</sub> the (D → A) bands are completely resolved for rotation structure, but in D<sub>2</sub> the resolution of the higher rotation levels is limited. The heads of the bands form the R(O) lines. Analysis of the vibration series is obtained by using the isotope effect to a 1st approximation and the empirical band formulas obtained are  $H_2, \nu_0 = 111,819 + 2315(\nu' + 1/2) - 59.25(\nu' + 1/2)^2$ ,  $\nu'' = 3-10$ ,  $D_2, \nu_0 = 112,428 + 1632(\nu' + 1/2) - 30.5(\nu' + 1/2)^2$ ,  $\nu'' = 4-11$ . The series in H<sub>2</sub> begin at  $\nu' = 3$  and in D<sub>2</sub> at  $\nu' = 4$  as only these lie above the photochem. dissociation limit  $H_2 \rightarrow H + H^+$  and their rotation terms can predissociate. The absorption lines are much broadened, being recognizable only because of the limited resolution of the spectrograph. The D level is classified as  $3p^2\ ^3P_2$  and fits well into the term scheme. The following formulas are proposed:  $H_2, \nu_0 = 111,859 + 2316.0(\nu' + 1/2) - 60.75(\nu' + 1/2)^2 + 0.167(\nu' + 1/2)^3$ ,  $D_2, \nu_0 = 112,485 + 1638.5(\nu' + 1/2) - 30.405(\nu' + 1/2)^2 + 0.0591(\nu' + 1/2)^3$ . Agreement is not exact. Richardson's recently analyzed emission bands fit this scheme badly.

S. Tolansky  
The absorption spectrum of copper hydride. B. Grundstrom. *Z. Physik* 98, 129-32 (1935).—New absorption measurements are made upon CuH bands occurring at 2240 Å, the dispersion used being 2.2 Å/mm. P, Q and R branches are found for the rotation fine structure. The rotation constants for H are  $B_0 = 7.9$  and 7.29 for  $\nu = 0, \nu = 1$ , resp., and  $D_0 = 5.10 \times 10^{-4}$  and  $-5.78 \times 10^{-4}$  for the same levels.

S. Tolansky  
The explanation of the perturbations in the second positive nitrogen bands. D. Coster and F. Brons. *Z. Physik* 97, 570-2 (1935).—The previously examined perturbation in the  $^4D$  level of the N<sub>2</sub> bands is not altered by the analysis of Gerb (*C. A.* 29, 780P). The perturbations have the noteworthy properties that (1) a weakening in the perturbed lines takes place without noticeable displacement, (2) the perturbation extends only over one or very few lines, (3) no so-called superfluous lines occur.

S. Tolansky  
Predissociation of the oxygen molecule. Paul J. Ilory. *J. Chem. Physics* 4, 23.7 (1936).—Data from the emission and fluorescence spectra of O<sub>2</sub> and from photochem. expts. are assembled to show that the O<sub>2</sub> mol. predissociates in the region of band absorption above 1751 Å. On the basis of this interpretation, the initial process in the photochem. reactions of O<sub>2</sub> caused by light in the region of Schumann-Runge band absorption is the formation of normal  $^3P$  atoms as a result of the predissociation, rather than the reaction of an optically excited mol. with a normal mol., as heretofore has been supposed.

G. M. P.  
The absorption spectrum of the iodine molecule in the vacuum ultraviolet. Heinrich Cordes. *Z. Physik* 97, 603-24 (1935).—The spectrum is observed with a fluor spar vacuum spectrograph of large dispersion (2.3 Å/mm at

1700 Å) in the region 1950-1500 Å. The band heads can be arranged into 8 progressions. The individual band series and a continuous absorption band at 1505 Å are described. The absorption systems correspond to transitions in the I<sub>2</sub> mol. from the  $^2\Sigma_g^+$  ground level in various excited mol. states. Two of the levels are classified as components of a  $^3\Pi_u$  multiplet and two others as ( $^3\Pi_u, \nu$ ),  $^3\Pi_u, \nu$  and ( $^3\Pi_u, \nu$ ),  $^3\Pi_u, \nu$ . In the limit the excited mol. dissociates into a  $6s^2P$  and a  $5p^2P$  I atom. The extrapolation of the convergence limit of a further band system is given as  $7.33 \pm 0.10$  eV. The upper level of this system is  $^3\Sigma_u^+$ , the atom splitting into  $^3S_1$  and  $^3P_1$ . The ionization potential of the I atom is given as  $8.9 \pm 0.2$  eV. The ultraviolet fluorescence spectrum is discussed.

S. Tolansky  
Absorption spectra of tellurium dioxide and monoxide. Choong Shin Paw. *Compt. rend.* 202, 137-8 (1935).—The spectrum of TeO<sub>2</sub> consists of 3 regions: (a) from  $\lambda$  2430 into the Schumann region, structureless, (b) from 2910 to 4700 with max. at 3580, and (c) with max. at 4980, b and c consist of bands, a and b appear at about 720°, c at 1250°. Two periodicities are indicated with certainty, 650 and 219 cm<sup>-1</sup>, belonging, resp. to the normal and excited states of the mol. If the d of the vapor is too low the TeO spectrum (cf. *C. A.* 30, 969P) appears, together with 4 new bands on the short λ side, due to predissociation.

C. A. Silbert  
The influence of heavy water of crystallization on the line like absorption spectra of chrome alums. G. Jooz and H. Böhm. *Physik. Z.* 36, 826-7 (1935).—If D<sub>2</sub>O is substituted for H<sub>2</sub>O in the chrome and selenate alums, a shift in the absorption lines is observed. This indicates that the lines are due to lattice and complex vibrations.

C. E. P. Jeffers  
The ultraviolet absorption spectrum of water. Erwin Haas. *Biochem. Z.* 282, 224-9 (1935).—The absorption of freshly distilled water was determined with waves of a definite length. Since saturation of the water with 100% CO<sub>2</sub> has little effect on the light absorption of water, it is argued that the temp. effect is not due to an increase in the dissociation of the water.

S. Morgulis  
Absorption spectrum of acetylene in the ultraviolet. Aurel Ionescu. *Bull. soc. roumaine phys. 36*, 151-67 (1933); cf. *C. A.* 29, 2948.—A review. R. E. DeR.

R. E. DeR.  
The flame spectra of some aromatic compounds. W. M. Vaidya. *Proc. Indian Acad. Sci. ZA*, 352-7 (1933), cf. *C. A.* 29, 1324.—A previous investigation by V. of a band system attributed to HCO as the emitter in the spectrum of the flame of ethylene is reviewed, and arguments for the existence of the HCO band are given. The flame spectra of C<sub>6</sub>H<sub>6</sub> and C<sub>6</sub>H<sub>5</sub> were also found to contain the

HCO band system, but it is less intense than in the ethylene spectrum. A similar investigation of the flames of C<sub>6</sub>H<sub>6</sub>, PhMe, PhOH, resorcinol, pyrogallol, BzH, BzOH, Ph<sub>2</sub>O, PhNH<sub>2</sub>, PhNO<sub>2</sub> and C<sub>6</sub>H<sub>5</sub>N showed the presence of the HCO bands. Their appearance in the aromatic flames was explained as due to the double bond of the benzene ring and the breaking up of the oxygenated mols. formed by the direct incorporation of the O<sub>2</sub> mol. A gradual variation in the spectra of the flames of aliphatic series, and considerable uniformity for those of the aromatic series was observed. A tentative but incomplete scheme for the combustion of C<sub>6</sub>H<sub>6</sub> on the hypothesis of the direct incorporation of the O<sub>2</sub> mol. is given. The scheme is based on spectral studies of C<sub>6</sub>H<sub>5</sub>N, C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub> and C<sub>6</sub>H<sub>5</sub>NO<sub>2</sub>.

L. L. Quill  
Resonance fluorescence of benzene. H. G. R. Cuthbertson and G. B. Kistakowsky. *J. Chem. Physics* 4, 9.15 (1936), cf. *C. A.* 26, 584t.—The analysis of the resonance fluorescence of C<sub>6</sub>H<sub>6</sub> given previously is amplified and corrected. Upon absorption of the 2536 Å Hg line, only the changes of the quantum no. of one ( $\nu_1, \nu_2 = 990$  cm<sup>-1</sup>) vibration of the nonexcited C<sub>6</sub>H<sub>6</sub> mol. are unrestricted in fluorescence. In other vibrations the changes of the quantum nos. are not larger than unity (2 when required by selection rules). Five additional frequencies were tentatively identified. 432, 791, 1542, 2537 and

3174 cm.<sup>-1</sup>. The fluorescence spectrum of C<sub>4</sub>H<sub>4</sub> contains the frequencies 944 (as unrestricted) and 2460. The resonance fluorescence of C<sub>4</sub>H<sub>4</sub> was quenched by He, H<sub>2</sub>, N<sub>2</sub>, CO<sub>2</sub> and cyclohexane; this fluorescence is quenched by being changed into high-pressure fluorescence. There is little or no loss of electronic excitation energy by C<sub>4</sub>H<sub>4</sub> molecules on collisions. The quenching action is unspecific, and appears to increase as the kinetic cross section of the foreign gas mols. New high-pressure fluorescence bands were observed in C<sub>4</sub>H<sub>4</sub> in the presence of foreign gases. These support Henri's analysis (C. A. 23, 1056) of the absorption spectrum as consisting of 2 close electronic levels of the excited mol. Attempts to excite resonance fluorescence in C<sub>4</sub>H<sub>4</sub> derive were generally unsuccessful. PbF and PhMe, even at 0.01 mm pressure, emit nearly continuous spectra. Other derivs tried have too little fluorescence to be studied at low pressures. G. M. P.

**Absorption and fluorescent spectra of certain naphthalenic hydrocarbons** R. Titeica *Bull. soc. roumaine phys.* 36, 69-75(1934).—A study of the spectra of the series C<sub>10</sub>H<sub>8</sub>,  $\alpha$ -CH<sub>3</sub>C<sub>10</sub>H<sub>7</sub>,  $\alpha$ -(C<sub>6</sub>H<sub>5</sub>)C<sub>10</sub>H<sub>7</sub> shows that the substitution of successively heavier radicals shifts the spectra toward the visible with no alteration in their nature. The fluorescent spectra are intense but simple. Evidence is not conclusive whether they can be interpreted by the frequency 481 cm.<sup>-1</sup> or whether one of 1400 cm.<sup>-1</sup> is also necessary. R. E. DeRight.

**The light absorption of porphyrin. III.** A. Stern and Hans Wenderlich *Z. physik. Chem.* A174, 321-34 (1935), cf. C. A. 30, 3824.—Comparisons were made of the absorption curves of 2 chloroporphyrin- $\alpha$  dimethyl esters that are isomers but differ greatly in structure. The presence of a ring between  $\gamma$  C atom and position  $\beta$  in the ester produced with MeOH-HCl could be confirmed by comparing its absorption curve with that of rhodoporphyrin-XV dimethyl ester. The absorption of a porphyrin unsubstituted in the  $\beta$ -position and with an isocyclic ring was measured and only a small blue displacement found as compared with that corresponding to  $\beta$ -position in Et-substituted porphyrin. Various derivs of the chlorophyll- $\beta$  series were studied and regularities found similar to those of the  $\alpha$ -series. The effect of hydration of vinyl group in the 2-position upon light absorption in both  $\alpha$ - and  $\beta$ -series is discussed. R. H. Baechler.

**Infrared photography of chromium compounds** J. Rzymkowski *Naturwissenschaften* 23, 610(1935).—Several Cr compounds (tri- or hexavalent Cr or perchromates) reflect light of 720-880 m $\mu$  regardless of their visible spectrum. Only a few Cr compounds form an exception, viz. CrCl<sub>3</sub>, Cr<sub>2</sub>S<sub>3</sub> and Cr<sub>2</sub>O<sub>3</sub>. This observation is valuable for identification of old paintings. B. J. C. v. d. H.

**Structure and absorption of benzocyclopentadienone oximes** Pauline Ramart-Lucas and Joseph Hoch *Compt. rend.* 201, 1857-90(1935).—Oximes have spectra whose form and band positions are similar to those of ethylenic hydrocarbons, whereas the isoximes have absorption bands which occupy, in the neighborhood of the visible end, the same position as those of analogous saturated hydrocarbons. The formation of the isomers may depend on the mode of splitting out H<sub>2</sub>O from the addn complex of H<sub>2</sub>NOH and the ketone which in turn may depend on the mobility of the O and OH groups influenced not only by the no. and nature of the radicals adjacent to the functional group but by the deformations of the valence angles. Measurement of the ultraviolet absorption of the derivs of indanone (III) and  $\alpha,\alpha$ -dimethylindanone (IV) and comparison of their curves with those of ethylenic,  $\alpha,\alpha$ -dimethylenic and hydrazidene showed them to be true oximes, although they may be regarded as derived by simple cyclization from trialkylacetophenones which have the isoxime structure. As the no. of CH<sub>3</sub> groups in the chain increases, the unsubstituted ketone gives a true oxime, while the disubstituted compd yields an isoxime. Thus tetralone oxime has the unsatd form and  $\alpha,\alpha$ -dimethyltetralone oxime shows the ring structure. Although all solns of the oximes of benzosuberone and dimethylbenzosuberone contain an equim. mixt. of the 2 forms, the main proportion of the former is in the oxime form and of

the latter in the iso modification. Similar results have been previously noted in studies of the analogous open-chain compds, MeC<sub>6</sub>H<sub>4</sub>CO(CH<sub>3</sub>)<sub>2</sub>Me and MeC<sub>6</sub>H<sub>4</sub>COCMe<sub>2</sub>(CH<sub>3</sub>)<sub>2</sub>Me. C. R. Addmell.

**The Raman effect from cholesteric points of view.** Pál Szarvas *Magyar Chem. Folyóirat* 41, 95-114(1935).—A general summary of the theory of the effect based on publications of K. W. F. Kohlrausch, A. Smekal, etc.

**Polarization of Raman scattered light.** W. Hanle and F. Heidenreich *Physik. Z.* 36, 851-3(1935).—The polarization of the edges of Rayleigh lines and the circular polarization of Raman lines from several substances were studied. For a series of Cl compds the assocn. of Raman lines with detd. mol. vibrations are studied. C. E. P. Jeffreys.

**The Raman spectrum and fundamental vibration frequencies of silane (SiH<sub>4</sub>).** Fred B. Stitt and Don M. Yost *J. Chem. Physics* 4, 82(1936).—The Raman spectrum of gaseous SiH<sub>4</sub> (5 atm) contains the lines 2187 and 978  $\pm$  5, of liquid SiH<sub>4</sub> at -120°, 2175 and 907  $\pm$  10. A complete assignment of the Raman and infrared lines of SiH<sub>4</sub> is given. G. M. P.

**Infrared absorption spectra of liquefied gases: methane.** Constant Corin and Jacques Herry *Compt. rend.* 202, 41-4(1936).—The absorption spectrum of liquid CH<sub>4</sub> and of its soln in liquid N<sub>2</sub> between 0.8 and 2.7  $\mu$  consists of 11 bands, for which the harmonics and combinations of the 4 fundamental frequencies of the CH<sub>4</sub> mol, as well as the intensities are given (cf. Kohlrausch, C. A. 29, 4673). Four of the bands (A 0.88, B 1.00, C 1.15 and D 1.38) correspond closely with similar bands of C<sub>2</sub>H<sub>6</sub>, and are due to intra- and not inter-mol. vibrations, indicating that CH<sub>4</sub> groups within a mol, as in C<sub>2</sub>H<sub>6</sub>, absorb more strongly than when in different mols as in CH<sub>4</sub>.

**Some new features in the Raman spectra of carbon and silicon tetrachlorides** R. Ananthakrishnan *Proc. Indian Acad. Sci.* 24, 452-8(1935).—Two new faint lines in the Raman spectrum of CCl<sub>4</sub> are reported. 434 cm.<sup>-1</sup>, interpreted as the harmonic of 217.9 cm.<sup>-1</sup>, its appearance is probably due to its proximity to the total symmetric vibration frequency 459 cm.<sup>-1</sup>, and 145 cm.<sup>-1</sup> apparently a differential tone arising from 459 and 314 cm.<sup>-1</sup>. There is a well-defined wing to the red side of the line 459 cm.<sup>-1</sup>. For SiCl<sub>4</sub>, the new line 440.5 cm.<sup>-1</sup> (near the total symmetric vibration frequency 423 cm.<sup>-1</sup>) is regarded as the harmonic of 221 cm.<sup>-1</sup>. Janet E. Austin.

**The rotation vibration spectrum of the methyl halides in the photographic infrared at 1.11  $\mu$ .** H. Verleger, *Z. Physik* 98, 342-52(1935).—With the use of the new Agfa infrared plates the rotation vibration spectra of CH<sub>3</sub>Cl, CH<sub>3</sub>Br and CH<sub>3</sub>I are taken with large dispersion (3-m. grating) in the region 11,100 Å. The bands show a simple set of approx. equidistant lines which show the characteristic intensity alternations exhibited by the  $\perp$  bands of a mol. with an axis of threefold symmetry. The mean sepn. of the lines for chloride, bromide and iodide, resp., are 8.47, 9.39, 9.61 cm.<sup>-1</sup>. The spectra are examd. in absorption and when the pressure in the absorbing tube is raised a second band system overlaps that occurring at the lower pressure. The intensity of this system grows when the pressure is increased. The bands are classified as belonging to the vibration combination (2 $\nu_1$  +  $\nu_2$ , 2 $\nu_3$ ). S. Tolansky.

**Infrared evidence for the existence of an isomeric form of hydrocyanic acid.** Dudley Williams *J. Chem. Physics* 4, 84(1936).—All org. cyanides have an intense infrared absorption band near 4.5  $\mu$ ; the isomer in which the C is bivalent gives more intense bands with a wave length 0.2  $\mu$  higher. The data of Nef (*J. Am. Chem. Soc.* 26, 1549(1904)) are reanalyzed; the results show that the aq. soln of HCN contains about 2% of the isomer HNC. G. M. P.

**Quantitative absorption measurements on the CH harmonics of simple hydrocarbons. I.** The halogen derivatives of methane, ethane and ethylene. B. Timm and R. Mecke *Z. Physik* 98, 363-81(1935).—The total absorptions of the third CH harmonic ( $\nu = 4$ ) of the follow-

ing 18 halogen derivs of methane, ethane and ethylene are detd by photographic photometry in the region 0.8 to 0.9  $\mu$ . Chloroform, methylene chloride, Me chloride, bromoform, methylene bromide, Me bromide, pentachloroethane, tetrachloroethane, 1,1,2-trichloroethane, 1,2-dichloroethane, Et chloride, Tr chloride, tetrabromomethane, 1,2-dibromomethane, Li bromide, trichloroethylene, *cis*-dichloroethylene and vinyl chloride. A general formula is deduced for the relation between the dipole moment of any bond and the absorption strength of its bands. It is shown how the dipole moment of homopolar bonds can be estd from the measurement of the total absorption in a series of harmonics. The moment of the aliphatic CH valency is estd as  $0.3$  to  $0.4 \times 10^{-18}$  e.s.u. The absorption strength of the CH frequencies is to a first approximation proportional to the no. of CH bonds in the mol. Deviations occur with methane and the dihydrides of ethane. The influence of the halogen is very small. In general a C=C bond reduces the absorption strength. The Br compds have stronger absorptions than the corresponding Cl compds. S. Tolansky

**Raman effect of acetylenes** II Duodoacetylene, liquid acetylene and deuterioacetylenes George Glocker and Charles L. Morrell *J. Chem. Physics* 4, 15-22 (1936), cf *C A* 28, 5335\*, 29, 681\*—An app designed for Raman work consists of 8 discharge tubes arranged concentrically around a Raman tube. Cylindrical filters may readily be used. The vibrational Raman spectra of  $C_2H_2$ , liquid  $C_2H_2$  and gaseous  $C_2H_2$ ,  $C_2HD$  and  $C_2D_2$  were observed and are interpreted in the usual manner on the basis of symmetry considerations.  $C_2H_2$  mols., which are linear in the gaseous state, are seriously distorted in the liquid state. Some lines are accompanied by faint satellites which may be due to rotation effects. The free energies of  $C_2HD$  and  $C_2D_2$ , and the free-energy change and the equl const of the reaction  $C_2H_2 + C_2D_2 = 2C_2HD$  were calcd for the temp range 273-700°K. G. M. P.

**Raman spectra and molecular structure of ethane and its derivatives** San-ichiro Mizushima and Yonezo Morino *J. Chem. Soc. Japan* 56, 1464-70 (1935)—As a continuation of previous work (*C A* 29, 971\*, 2850\*) Raman spectra of  $C_2H_6$  and its derivs were studied in relation to free rotation. The results for  $CH_3X$ ,  $CH_3X$ , together with those obtained in other lines of expts (dipole moment, x-ray scattering, Kerr effect and sp heat), can best be explained by the theory of hindered rotation, but not by the assumption of *cis-trans* isomerism. T. Katsura

**Rotational Raman scattering in benzene** S. Bhagavantam *Proc. Indian Acad. Sci. A*, 342 4 (1935), cf *C A* 29, 2449\*, 2949\*—In a study of the intensity distribution in the rotation wing accompanying the Rayleigh lines in liquid  $C_6H_6$ , the E<sup>1</sup> Hilger spectrograph is used. Results agree perfectly with those obtained earlier with spectrographs of lower dispersion. L. L. Quill

**Raman spectrum of benzene-*d*** R. C. Lord, Jr. *J. Chem. Physics* 4, 82-3 (1936)—The shifts of 3052 and 3108  $cm^{-1}$  reported for  $C_6D_6$  by Wood (*C A* 29, 6505\*) are believed to be due to  $C_6D_5H$ . The shifts of 3573 and 3627  $cm^{-1}$  reported by him for  $C_6H_6$ , which he believed to be excited by Hg-4358, are reassigned to 2 other Hg lines, giving shifts of 990  $cm^{-1}$ . G. M. P.

**Raman spectrum and molecular structure of benzene** San-ichiro Mizushima and Yonezo Morino *J. Chem. Soc. Japan* 56, 1470-4 (1935)—For benzene structure hexagonal symmetry is more probable than trigonal. Calcs of the modes of vibration for  $C_6Cl_6$  and  $C_6(CH_3)_6$  are given. T. Katsura

**The infrared absorption spectra of some anthracenic hydrocarbons** II Applications to the analysis of hydrocarbons present in oils P. Lambert and J. Lecomte *Ann. combustibles liquides* 10, 1077-92 (1935), cf *C A* 29, 1324\*—The infrared absorption curve has been detd from 6 to 20  $\mu$  for 4 substituted 9,10-dihydroanthracenes as follows 9,10-disubstituted, 9,10-di-isomyl, 9,9-disubstituted, and 9,9,10,10-tetra-substituted. The comparison of the curves with those previously obtained on frac-

tions of lubricating oil show the applicability of the method to the identification of hydrocarbons. G. C.

**Raman spectra of amino acids and related compounds** I. Ionization of the carboxyl group John T. Edsall *J. Chem. Physics* 4, 1-8 (1936)—Raman spectra are reported for 4 amino acids, glycine, *dl*-alanine, *dl*- $\alpha$ -aminobutyric acid,  $\alpha$ -amino-isobutyric acid, and for their hydrochlorides, for 3 fatty acids,  $HCOOH$ ,  $HOCH_2COOH$ , and for their Na salts; and for  $CH_3C(=O)OH$ ,  $CH_3C(=O)ONa$ ,  $MeNH_2HCl$ ,  $LiNH_2HCl$ , *dl*-alanine methyl ester, *dl*- $\alpha$ -aminobutyric acid ethyl ester, urea,  $AcOMe$  and  $Me_2CO$ . The influence of the solvent water on the Raman spectra of aq solns is discussed. The "carbonyl" frequency, lying near 1670  $cm^{-1}$  in the pure fatty acids, shifts to 1720  $cm^{-1}$  when they are dissolved in water. No shift is found for the corresponding frequency in  $AcOMe$  or  $Me_2CO$ . The presence of a charged  $NH_3^+$  group on the C atom to the carboxyl increases this frequency by about 20  $cm^{-1}$ . On ionization of the carboxyl group it is found that (1) the "carbonyl" frequency vanishes in all cases investigated. The behavior of the amino acids in this respect is entirely consistent with their structure as amphoterics ions. (2) A group of lines in the region 1200-1420  $cm^{-1}$  undergoes characteristic changes in position and intensity. (3) In most cases there is a powerful line in the region 750-930  $cm^{-1}$  whose frequency increases by 20-40  $cm^{-1}$  on ionization. The frequency of this line is decreased by about 50  $cm^{-1}$  for each addl Me group on the C atom adjoining the carboxyl. (4) Ionization markedly decreases the C-H frequency in  $HCOOH$ , and also certain strong frequencies in  $MeNH_2$  and  $EtNH_2$ . G. M. P.

**Raman spectrum of oxalic acid** W. Røge Angus and Alan H. Leckie *J. Chem. Physics* 4, 83-4 (1936)—The Raman spectrum of oxalic acid is given, and is compared with spectra given by Hibben (*C A* 30, 384\*) and by Rao (*C A* 29, 5350\*). Hibben's results disagree with those of the other investigators, his conclusions that the 2 carboxyl groups behave differently may be invalid. G. M. P.

**Raman spectrum of crystalline selenous acid** C. S. Venkateswaran *Current Sci.* 4, 309-10 (1935)—At an exposure of 4 hrs. transparent crystals of  $H_2SeO_4$  give a very intense spectrum consisting of a large no. of very sharp lines. The frequency nos. of these lines in  $cm^{-1}$  are 910(1), 909(2), 888(10), 862(0), 706(3), 597(8), 524(3), 364(0), 395(2), 287(3), 251(6) and 199(3). The nos. within the parentheses indicate the relative intensities of these lines and a dash above some of them shows that anti-Stokes lines are also present. The lines were obtained with 4046 and 4358 radiations of the Hg arc with equal intensity. The spectrum of the solid is markedly different from that of the aq soln of this acid. W. J. Peterson

**Raman spectra of glasses** Th. G. Kuyumzlis *Z. Physik* 97, 561-9 (1935)—The Raman spectra of a no. of the glasses made by Schott and Genossen are examd. Contrary to previous observations, an individual spectrum is observed for each glass. The spectra consist partly of sharp and partly of broad Raman lines. The frequency  $\sim 800$ , which occurs, is of interest. It is not characteristic of  $SiO_2$  as it is found in Si free glass. A new observation is the existence of continuous bands directly linked to the primary lines. The band structure is different for each glass. The bands exhibit sharp limits on the long wave side. S. Tolansky

**Ultra-luminous spectrograph of the Pic du Midi** Hubert Garrigue *Compt. rend.* 202, 44-5 (1936)—An improved form of Cabannes and Dufay's spectrograph (*C A* 28, 6332\*) with F 0.55, and automatic registration, is described, giving much improved results for the night sky spectrum. The line 5577 is const. from night to night, but diminishes in intensity with increased angle of elevation. C. A. Silberrad

**The sensitized decomposition of hydrogen with electrons of controlled energy** Geo. Glocker and Lloyd B. Thomas *J. Am. Chem. Soc.* 57, 2352-7 (1935)—H<sub>2</sub>

mols have been dissociated, by electrons of known speed with Hg atoms as a sensitizer (the Cario and Franck expt. in which electrons are used in place of light). From the photochem. reaction it is predicted that electrons of 4.9 e. v. should initiate the reaction. This is not confirmed. At 0.02 mm. of H<sub>2</sub> and 0.002 mm. Hg vapor decompn. starts at 7.7 v. J. B. A.

Photoelectric effect and photochemical sensitiveness Cécile Stora. *Compt. rend.* 202, 48-50 (1936), cf. C. A. 29, 39151.—In the course of expts. to det. the difference in the photoelec. effect of the xanthane and quinonoid groups of dyes it was shown that the pos. effect of the former increases in the order uranin, eosin, erythrosin, i. e., the reverse of that of the fluorescence. The photopotential of the xanthane dyes is markedly reduced by hyposulfites, phenol, amines and TiCl<sub>3</sub>, but K<sub>2</sub>I while reducing that of erythrosin increases that of uranin and eosin. The action of gases (O, N and H) in this respect also differs for the 2 groups. These differences are attributed to differences in their chem. constitution.

C. A. Silberrad. Photochemical stability of crotonaldehyde. Francis F. Blacet and Jack G. Root. *J. Am. Chem. Soc.* 58, 73-5 (1936).—Monochromatic Hg vapor radiation from 2300 to 3600 Å. covering at least 3 types of absorption was used on crotonaldehyde vapor and no decompn. or appreciable polymerization occurred. The system of conjugated double bonds seems to contribute greatly to the photochem. stability of the mol. as contrasted with the corresponding satd aldehydes. The data are discussed. A predominating reverse reaction is postulated.

William H. Vaughan. Emission spectra of chemical reactions. René Audubert. *Compt. rend.* 202, 131-3 (1936), cf. C. A. 29, 32385.—With counters with Al and Cu cathodes, of which the max. sensitivities are for 2400 and 2000-2250 Å., resp., the radiation emitted by 14 reactions has been exam'd. In 4 cases (oxidation of EtOH by CrO<sub>3</sub> and action of Br or I on Na<sub>2</sub>CO<sub>3</sub> or K<sub>2</sub>CO<sub>3</sub>) detection was with the Al cathode only; in 3 (oxidation of glucose by KMnO<sub>4</sub> and of Na<sub>2</sub>SO<sub>3</sub> and K<sub>2</sub>SO<sub>3</sub> with O<sub>2</sub>) with that of CuI only, and in 7 (NaOH and HNO<sub>3</sub> or H<sub>2</sub>SO<sub>4</sub> oxidation of alk. pyrogallol or Na<sub>2</sub>SO<sub>3</sub> with O<sub>2</sub> and action of amalgams of Na, Mg or Al on H<sub>2</sub>O) by both.

C. A. Silberrad. Photochemical reaction between sodium formate and iodine and a relation between chemical reactivity and light absorption. N. R. Dhar and P. N. Bhargava. *J. Phys. Chem.* 39, 1231-44 (1935); cf. C. A. 29, 24301.—The reaction between Na formate and I<sub>2</sub> in the aq. soln. is unimol. in the dark and semi-mol. in light. Temp. coeffs. between 15° and 25° are: dark 1.77; 8500 Å. 1.75, 6640 Å. 1.71; 5700 Å. 1.66, 4295 Å. 1.61, 3512 Å. 1.57; 3485 Å. 1.57; 3340 Å. 1.52; 3125 Å. 1.45. The velocity of the reaction is proportional to the 1/2 to 2/3 power of the incident light. The quantum yield varies from 26 to 64 and increases with temp. and frequency of incident radiation. The light absorption of the mixt. is greater than the sum of the absorption of the reacting substances due to the activation of I<sub>2</sub> by the Na formate, and is proportional to the concn. of reacting substances.

Arthur A. Vernon. Hydrolysis of glucosides and other organic compounds by ultraviolet light. A. Guillaume and G. Tanret. *Compt. rend.* 201, 1037-60 (1935).—Two % solns. of glucosides (salicin, amygdalin, etc.) and of some esters were placed in *crystg.* dishes, covered with quartz plates and irradiated with ultraviolet light for 3-24 hr. periods. Ultraviolet light has a marked hydrolyzing action on glucosides and some esters.

E. D. Walter. Reduction of ferric chloride under the action of α-, β- and γ-rays. G. W. Spicer. *Trans. Faraday Soc.* 31, 1706-10 (1935).—FeCl<sub>3</sub> in aq. soln., in anhyd. FeCl<sub>3</sub> soln. and in the form of anhyd. crystals, was reduced by α rays from Rn. Reduction was incomplete in the solid, probably owing to lack of penetration. With γ- and β-rays complete reduction was effected in FeCl<sub>3</sub> soln., but none occurred in aq. soln.

C. E. P. Jeffreys. An instance of negative photocatalysis. Jaroslav

Milbauer. *Chem. Listy* 29, 267-8 (in French 268) (1935); *Phot. Korr.* 71, 94-5.—The decompn. of an active MeOH soln. contg. 28.2 g. Na. N-chloro-*p*-toluenesulfonamide (I) per l. proceeded at a rate directly proportional to the light intensity. In the presence of 0.02, 0.2 and 1.0 mg. of the dyes Nile blue, Brilliant green, Capri blue, phos. safranin, fuchsin, primary phtol green and methylene violet per 10 cc. of I soln., the decompn. was slower than in their absence. Exposures were made to the rays of a Phillips 400-w. or of an Osram Vitalux lamp. Since simultaneous expt. with these dye mixts. made in darkness did not show a loss of the activated Cl, the process is one of negative photocatalysis. With the same range of concn. the sensitizers malachite green, primary phtol yellow and methylene blue did not show negative photocatalysis, for in the darkness they consumed activated Cl and disintegrated themselves. For following the decompn., M. added 2 cc. 2 N HCl to the dye soln., followed by 8 cc. of a 0.2 N KI soln. and then titrated the excess I with 0.05 N Na-SO<sub>3</sub> with starch as the indicator. Frank Marech.

The yellow coloration caused by the action of ultraviolet light on mixtures of pyridine with sugars, monohydric alcohols and acetone. Remo Cantieni. *Helv. Chim. Acta* 18, 1420-7 (1935), cf. C. A. 29, 57487.—The yellow color (photopyridine (I)) developed in solns. of pyridine, H<sub>2</sub>O and fructose is more readily formed and more readily bleached than in solns. contg. glucose, galactose, sucrose or lactose. MeOH and EtOH are not very effective in the formation of I. Acetone inhibits the formation of I.

W. F. Bruce. Absorption and fluorescent spectra of the coloring matter of litmus and red cabbage. I. G. Popescu. *Bull. soc. roumaine phys.* 36, 33-5 (1934).—For the coloring matter of red cabbage solns. in alk. and ether have a slight yellow tint and in H<sub>2</sub>O are violet, in acid are red and in base are green. For the litmus the colors are blue, violet, red and blue, resp. In every case the absorption bands are wide and diffuse, their position being shifted by a change of solvent. The litmus has almost continuous absorption in the ultraviolet. The fluorescent spectra are continuous with maxima between 4500 and 5000 Å. An alc. soln. of litmus has a diffuse band at 5700-6300 Å.

R. E. DeRight. The influence of light on the surface tension of soap solutions. H. L. D. Mahajan. *Z. Physik* 98, 348-95 (1935), cf. C. A. 26, 3164.—The total reduction in surface tension by light increases up to a max. at a concn. of 6.25 X 10<sup>-4</sup> g. of Na oleate per cc. of soln. and diminishes when the soln. is further weakened. This limit varies with age, purity and the type of soap. The violet radiation of the visible spectrum produces a strong reduction, the greatest effect being caused by ultraviolet light. The reduction in surface tension is due to a chem. change in the soap soln. The effect is not due to heat and still occurs when heat and adsorption effects are eliminated.

S. Tolinsky. The fluorescence of ruby, sapphire and emerald. C. S. Venkateswaru. *Proc. Indian Acad. Sci.* 2A, 459-63 (1935).—The photoluminescence spectra of natural crystals of ruby, sapphire and emerald (beryl) at 35° are given. The fluorescence of emerald (exposure 24 hr.) shows 2 intense sharp lines at 6806 and 6827 and weak bands at 6946, 6908, 6736, 6433, 6378 Å. The fluorescence of sapphire (exposure 48 hr.) shows 2 intense and sharp lines at 6927 and 6942 and a series of bands resembling the spectrum of ruby. 7130, 7000, 6902, 6802, 6753, 6600, 6592. A 4-hr. exposure gave a very intense spectrum of ruby showing lines at 7027, 6902, 6945, 6927, 6814, 6791, bands at 7206, 7222, 7104, 7123, 7089, 7050, 6733, 6690, 6650, 6592, 6495, 6430, an unresolved blackening between 6992 and 6945, and continuous spectrum between 6100 and 6900. Since the spectra of sapphire and ruby are similar and the strong lines in that of emerald correspond to the weak doublet 6792, 6814 in ruby with diffuse bands on either side resembling the bands of ruby at 200° it is concluded that the luminescence is due in each case to the same agent, probably Cr. Janet F. Austin. "Optimum" concentration of active foreign materials



in phosphorescent crystals Gerhard Schumann. *Z. Physik* 98, 252-61 (1935).—The crystals examined are  $\text{CaO}$ ,  $\text{CaS}$ ,  $\text{CaSe}$ ,  $\text{SrO}$ ,  $\text{SrS}$ ,  $\text{SrSe}$ ,  $\text{BaO}$ ,  $\text{BaS}$ ,  $\text{BaSe}$ ,  $\text{MgS}$  and  $\text{ZnS}$ . The active foreign materials used are  $\text{Mn}$ ,  $\text{Fe}$ ,  $\text{Ni}$ ,  $\text{Cu}$ ,  $\text{Zn}$ ,  $\text{Ag}$ ,  $\text{Sb}$ ,  $\text{Ir}$ ,  $\text{Nd}$ ,  $\text{Sm}$ ,  $\text{Pb}$  and  $\text{Bi}$ . In connection with the relation between the imperfection in structure and the after-glow of crystal foreign phosphorescents it is shown that the existing "optimum" concepts already held for foreign materials are not sufficiently definite. S T

Cathodic phosphorescence for examination of minerals and thin sections of rocks Pierre Urbain *Compt. rend. soc. géol. France* 1935, 112-13 —A new exptl. method is described. C A Silberrad

Coloration of compounds of different elements by cathode rays Hantaro Nagaoka and Tadao Mishima. *N. J. Papers Inst. Phys. Chem. Research (Tokyo)* 28, 77-94 (1935).—The chlorides of  $\text{Na}$ ,  $\text{K}$ ,  $\text{Pb}$ , the fluorides of  $\text{Ca}$ ,  $\text{Na}$ ,  $\text{Li}$ ,  $\text{Mg}$ ,  $\text{Al}$ ,  $\text{Ti}$ ,  $\text{Mn}$ ,  $\text{Cr}$ ,  $\text{Zn}$ ,  $\text{Ce}$  and  $\text{Pb}$ ,  $\text{PbBr}_2$ ,  $\text{K}_2\text{S}$ ,  $\text{SnOCl}_2$ ,  $\text{PbCO}_3$ ,  $\text{Pb}$  silicate,  $\text{PbO}$ ,  $\text{Sb}_2\text{O}_3$ , and  $\text{Kb}$  alum placed in powder form in a pencil of electrons in a Coolidge tube develop colors as with x-rays, but much more readily. The intensity of light reflected from the colored spots was measured and plotted against the voltage of the cathode rays forming the spot. These curves often show breaks that correspond to x-ray voltages. The theories advanced to explain the colors arising in crystals by electron bombardment are discussed. E O Wug

The nature of the  $U$  centers in alkali halide crystals P Tartakovsky and V Poddubnyi *Z. Physik* 97, 765-73 (1935).—When  $\text{NaCl}$  crystals are heated to 550-600° the absorption of infrared radiation at  $\approx 1.2 \mu$  strongly increases. The transition of the  $U$  into  $F$  bands under various radiations is studied. It is shown that electrons in the  $U$  levels can move to the  $F$  levels in 2 ways, namely directly involving absorption of light quanta  $\approx 4$  v, and indirectly requiring 6.4 v. A photoelectric current can be observed when ultraviolet radiation is used. The  $U$  centers are capable of recombining with introduced  $\text{Cu}$  ions producing a red coloration. It is considered that the  $U$  centers are electrons remaining in the place of  $\text{Cl}^-$  ions which have wandered away. S Tolansky

Examination of some of the optical and electrical properties of didymium glass K Provad, D K Bhat-tacharya and L M Chatterjee *Z. Physik* 98, 324-35 (1935).—The fluorescence bands of didymium glass irradiated by sunlight are identical with the Raman

spectrum, if one considers the incident exciting radiation frequency as that of the strong absorption bands possessed by didymium glass. The infrared frequencies calculated upon this assumption agree very well with the known values for glasses. Three new frequencies are found at 2.5, 3.1, 6.0  $\mu$ . The photoelectric cond. of the glass is examined to try to explain the characteristic differences occurring in the fluorescence bands when filters are employed with the incident sunlight. S Tolansky

The dust effect of Mitra Lj Splat *Z. Physik* 98, 306-8 (1935), cf. Mitra, C. A. 29, 7813.—The light intensity employed by Mitra is not enough, and his exptl. precautions are insufficient. Stray light effects are suspected. The results of Mitra are criticized. S T

Detection of  $\text{O}_2$  (Shlesinger-Konstantinova) 7. Alkal films of at. thickness on Pt (Mayer) 2

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#### 4-ELECTROCHEMISTRY

COLIN G FINK

Electric furnaces to bright anneal high-carbon steel Willard Roth *Metal Progress* 29, No. 1, 35-38 (1936).—Better temp. and atm. control are reflected in quality of product and economy of operation. Practical data are given. W A Mudge

The manufacture of high test cast irons A Portevin and R Lenoire *Bull. assoc. tech. fonderie* 9, 301-4 (1935).—High-test cast iron, made in elec. furnaces and contg. 2.60 to 2.80% total C is about 50% superior to malleable iron. Most elec. furnaces for cast iron are basic-lined to facilitate desulfurization, but the basic slag has a tendency to enter the mold, the fluidity of the iron falls rapidly with the temp., and the cast-iron surface is not as uniform as that of cupola iron, whereas acid-lined furnace cast iron does not have any of these disadvantages. Cupola cast iron contg. 2.70% C and less than 2% Si has better properties than malleable iron. G T M

Development of the electrolytic production of aluminum in France previous to 1925 A v Zeiler *Z. Elektrochem.* 42, 27-31 (1936). E H

Characteristics of anodized aluminum R E Pettit *Product Eng.* 6, 292-4 (1935).—The aluminate process is described with photographs, and also its fields of application. The oxide coating produced by the electrolytic action is 0.001 in. thick. Ann Nicholson Hurd

Peculiarities in formation of metallic deposits Jean

Bulliet *Rev. met.* 32, 518-21 (1935).—With very low c.d.s. electrodeposition occurs only along grain boundaries of the base metal or along scratches present on it. Crystal structure of the base is reproduced in the deposit independently of the space lattice dimensions of the base. Force of attraction have a pronounced influence here and appear to be greater than of mol. order because an easily visible unoriented layer can be seen under a microscope between the base and the oriented deposit. Roughness produced with low densities persists, occasionally, when the rest of deposit is made with high densities. This can be explained by a hypothesis that a film of electrolyte which is impoverished by the current and is located next to the surface of the base is of about the same thickness as the projections originally deposited. The film does not follow the contours of the surface, maintaining the same thickness, but is reduced by the height of these projections so that they are located closer to normal electrolyte, which leads to an increased precipitation in corresponding areas. This hypothesis is supported by an expt. which is described. J D G

Six years of observation of chromium [plate] in a job shop Wm A Vignos *Platers' Guide* 32, 11-14 (Jan. 1936).—The Ni sub-coat should be controlled as regards Cl, acidity and metal content, with temp. and c.d. having an important bearing on the deposits. Arrange

ment of the work in regard to the anodes is important in Cr plating. The coating produced during the first few seconds det. the character of the final Cr plate. The size and shape of anodes, the sulfate content and the effects of trivalent Cr and of Fe are briefly reviewed. W H B

Kinetics of anode-layer formation on metals. II. Surface layers of lead chloride on lead. L Yu Kurtz. *Compt rend acad sci U R S S [N S]* 3, 307-8 (1935), cf. *C A* 29, 7198. —During electrolysis with a Pt cathode and a Pb anode in 0.5 N HCl, satd. with PbCl<sub>2</sub>, with a const. c. d., the potential was const. at first, then increased suddenly, after which there was a linear increase with time. Visual observation showed that the sudden increase takes place when the surface is completely covered with a thin layer of PbCl<sub>2</sub>, and the linear increase is due to the thickening of this layer. The thickness of the layer at this point (1.82 - 0.45 μ) decreases with the c. d. but the porosity and elec. cond. increase with the c. d., as with the Ag halides. Comparison of the porosity of the layer with that of compressed PbCl<sub>2</sub> showed that it is small, and the difference in the polarization curves for Pb and Ag is due to the difference in porosity of the salts. I R Rushton

The decomposition and equilibrium reaction potentials of fused potassium chloride. Roy C Kirk and W I Bradt. *Trans Electrochem Soc* 69, 14 pp. (preprint) (1936). —Existing discrepancies in the literature concerning the decompn. potential of fused KCl at 500°, with graphite electrodes, were reconciled. Neumann's direct method value of 2.8 v. was verified as the decompn. potential. By means of a revised direct method, the p. d. between the anode and cathode was found to increase with increasing anodic c. d., as applied through a second cathode. A max. value of 3.37 v. was obtained with an anodic c. d. of 10 amp./sq. cm. This is quite close to the calc'd. equal reaction potential and in exact agreement with the results obtained by Cambi and Devoto by the commutator method. Neumann's value of 2.8 v. should, therefore, be called the decompn. potential and 3.37 the equal reaction potential. C G I

Energy levels in electrochemistry. Wilder D Bancroft and James E. Magosin. *J Am Chem Soc* 57, 2561-5 (1935). —The reduction reactions which may take place in an electrolyte depend upon the relative energy levels required for the specific reactions. That reaction requiring the lowest energy level will take place in preference to one requiring a higher energy level. It will be evolved electrolytically preferentially to reduction if the reduction requires a higher energy level to be reached than that required for the production of nascent H. In H<sub>2</sub>SO<sub>4</sub>, solns. lower energy levels were required for a sp. reaction where the concn. of H<sub>2</sub>SO<sub>4</sub> increased or the temp. rose or the activating adsorption increased. The energy level for the production of intermediate products is also a detg. factor in the course of electrolytic reactions. Reactions are irreversible if the required energy level for the reversible process is not attained. Irreversible reactions may give a definite e. m. f., e. g., the sulfate-sulfate electrode potential may be expressed as  $nFE = RT \ln P' \times P_{SO_4}$ . No distinction need be made between the processes of hydrogenation and reduction. J W Shipley

Dielectric properties of anodic layers in aluminum electrolytic condensers. J E. Lilenfeld, L. Chandler, Jr., and S. Goldman. *Physica* 6, 416-25 (1935), cf. *C A* 26, 333. —Measurements of the dielec. properties with a new type of especially stable electrolytic capacitor showed that variations of the properties may be divided into 2 classes: lagging and nonlagging. The former are characteristic of anodic layers. Anodic layers are similar to other composite dielectrics in regard to nonlagging variations. An oscillographic study of harmonics verified the lagging nature of the changes of capacity with operating voltages (a. c. and d. c.) and demonstrated for the first time the existence of a nonlagging variation of the a. c. cond. with voltage. To minimize lagging changes most of the expts. were made with layers formed in electrolytes of low OH-ion concn. and operated in extremely viscous electrolytes of high sp. resistance and low active OH-ion concn. Where a large percentage of harmonics was de-

veloped, anodes formed and operated in decidedly amp. electrolytes were used. From the absence of evidence of changes of capacity due to pressure caused by the electrostatic field it was concluded that the layer could not be of a gaseous nature. Allen S. Smith

A study of aluminum mercury-zinc anodes in acid zinc-plating baths. A K. Graham and P. G. Kolupaev. *Metals & Alloys* 7, 14 17 (1936), cf. *C A* 29, 1718.

A study of Al-Hg-Zn anodes in a ZnSO<sub>4</sub>-Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>-NH<sub>4</sub>Cl bath at pH values of 2 to 4 and 3 other acid Zn baths of com. compn. at a pH of 4.0. Current efficiencies, anode polarization, sludging tendency, bath voltage, pH variations and character of deposits are reported. A comparison with com. pure Zn anodes is made. Downs Schaaf

Mercury | mercuric oxide | saturated barium hydroxide and calcium hydroxide electrodes. G. J. Samuelson and D. J. Brown. *J Am Chem Soc* 57, 2711-4 (1935).

The two electrodes Hg|HgO(s), Ba(OH)<sub>2</sub>(s) and Hg|HgO(s), Ca(OH)<sub>2</sub>(s) were prep'd and studied from the standpoint of ease of prep'n, constancy, reproducibility and effect of temp. change. The potential for the Hg|HgO(s), Ba(OH)<sub>2</sub>(s) including a liquid junction was +0.1462 - 0.0004(t - 25°) = 0.0002 t, versus H as zero. With ordinary materials a satisfactory electrode of the same voltage was obtained. The potential of the Hg|HgO(s), Ca(OH)<sub>2</sub>(s) was +0.1923 + 0.00010(t - 25°) = 0.0010 t, and with ordinary materials the voltage was well within ±0.002 v. of the above value. J W Shipley

An unknown portion of the calomel half-cell and the estimation of bromide-chloride mixtures. F. L. Hahn. *J Am Chem Soc* 57, 2537 (1935), cf. *C A* 29, 5379.

Expts. indicated that small amts. of bromide in the KCl of the calomel cell influenced the e. m. f. Even the purest grades of com. KCl contained bromide up to 0.1% and the calomel will contain varying quantities of bromide. It is suggested that it may be the presence of bromides which causes the irregular change in e. m. f. of reference calomel cells with the passage of time. The bromide effect on the e. m. f. of a calomel electrode is suggested as a method of estg. bromide in chloride. J W Shipley

Potential of the Ag(s), Ag<sub>2</sub>CrO<sub>4</sub>(s), CrO<sub>4</sub>--- electrode. Jessie V. Cann and Gretchen B. Mueller. *J Am Chem Soc* 57, 2525-7 (1935). —The potential of the Ag(s), Ag<sub>2</sub>CrO<sub>4</sub>(s), CrO<sub>4</sub>--- electrode was det'd. by means of e. m. f. measurements in an app. similar to that used by Randall and Cann (*C A* 24, 3154). The normal electrode potential was found to be  $E^\circ = -0.4163$  and  $\Delta F^\circ = 20,506$  cal. J W Shipley

Study of the bismuth electrode. D. N. Mehta and S. K. Kulkarni. *Jatkar J Indian Inst Sci* 18A, Pt. 14, 109-13 (1935), cf. *C A* 29, 7824. —Expts. with the Bi electrode with interference colors on the surface due to the oxide film show that it may be used to measure pH within the range 5.0 to 7.4 only. In the relation  $E = E_0 + 0.00611 \text{ pH}$  (30°), the av. values of  $E_0$  in the useful range for the half cell Bi|Bi oxide|soln. with the sat'd. calomel and normal H<sub>2</sub> reference electrodes are -0.2300 and -0.4737 v., resp. The temp. coeff. of the Bi|Bi oxide electrode is -0.0015 v./1°. Energy calcs. indicate the existence of Bi<sub>2</sub>O in the surface film. Allen S. Smith

Polarographic studies with the dropping-mercury cathode. LII. The electroreduction of benzaldehyde. M. Tokunaka. *Collection Czechoslov. Chem. Communications* 7, 392-403 (1935), cf. *C A* 27, 1279. —The electroreduction of BzII at the dropping-Hg cathode was investigated by obtaining current-voltage curves polarographically. The reduction proceeds in two stages, each requiring 1 F per mol. of BzII. The first stage gives probably PhCH(OH)CH(OH)Ph, if not a free radical; the second gives PhCH(OH). The dependence of the cathode potential at which the process starts on the pH of the soln. was studied; for pH = 1, the potential is -0.9 v.; it decreases approx. 0.06 v. for each pH unit; from pH 7 both stages of electroreduction occur and the potential decreases approx. 0.025 v. for each pH unit. In neutral soln. the potential is -1.5 v.; bivalent cations make the potential more pos. In solutions of tetramethylammonium salts the electroreduction of BzOH takes place at -2.3



**Storage batteries** Albert Ricks. Ger 620,587, Oct. 24, 1935 (Cl 21b, 17). An active mass for Pb storage batteries is made by mixing finely powd. PbO and carbonates of Pb obtained by absorbing CO<sub>2</sub> by Pb oxides. The Pb carbonates are not powd. as fine as the PbO.

**Electric capacitor.** Frank M. Clark and Ralph A. Ruscetta (to General Elec. Co.). U S 2,028,590, Jan. 21. A film-forming armature such as one of Al foil is closely spaced to another armature and the space between the armatures contains a coned aq salt soln contg fine particles of salt in suspension.

**Electrolytic condenser** Willem C. van Geel and Hendrik Emmens (to Radio Corp of America) U S 2,029,451, Feb 4. A metallic electrode such as Al carrying an oxide coating is used with an electrolyte contg a soln of an acid phosphate and an alc. such as glycerol or the like for preventing attack of the oxide coating.

**Electrolytic condenser** Preston Robinson and Carleton Shugg (to Sprague Specialties Co) U S 2,028,564, Jan 21. Structural details.

**Electrolytic condensers, electrolysis** Siemens & Halske A-G Brit 435,300, Sept 19, 1935 See Fr 785,674 (C A 30, 3034)

**Film forming electrolytic paste for electrolytic condensers.** Paul Hetenyi (to Solar Mfg Corp) U S 2,028,775, Jan 28. Glycerol 36, NH<sub>4</sub> borate 56, potato starch 7 and agar-agar 1% are used together.

**Electrodeposition of metal** Alexander G Russell (to Electrical Research Products, Inc) Brit 435,694, Sept 25, 1935. In the deposition of separable deposits on a metallized non-conducting surface, e. g., a wax master sound record, metal clips are applied to the edges of the mold to prevent curling away of the deposit.

**Coating metals** James H. Gravell Brit 435,773, Sept 27, 1935 See Fr 783,250 (C A 29, 78331)

**Electrodeposition of metals such as nickel** Paul R. Pine (to Harshaw Chemical Co) U S 2,029,786-7, Feb 4. Brit 436,042, Oct 3, 1935 See Fr 781,542 (C A 29, 61507).

**Electrodeposition of magnesium.** P. F. Antipin Russ. 36,019, Apr 30, 1934. A Mg alloy is obtained by electrolysis of a hot coned aq soln of a Mg salt with a cathode of low-melting alloy such as Wood metal, Rose metal or other metal characterized by a high superpotential in the sepn. of H<sub>2</sub>. The alloy thus obtained is then made the anode in a molten electrolyte such as carnallite.

**Electrodeposition of aluminum.** N. N. Tumanov. Russ. 38,327, Aug 31, 1934. The resolin of the Al deposited from aq soln on the cathode is prevented by a diaphragm over the cathode (on the bottom) of liquids immiscible with water such as aniline or chloroform.

**Bright aluminum surface.** Ralph Bryant Mason and Martin Tosterud (to Aluminum Ltd) Can 354,510, Dec. 3, 1935. An Al surface is buffed to produce a reflection factor of 74.9%. It is then made the anode in an electrolytic cell with a soln contg 10.0% by wt CrO<sub>3</sub> and 1.0% of a 48% H<sub>3</sub>I<sup>+</sup> soln. A d c is pressed for 10 min., the electrolyte being at 40-58°. The reflecting surface then has a reflection factor of 87.2%. The article is then anodically oxidized in a 7% H<sub>2</sub>SO<sub>4</sub> soln for 10 min., and the oxide coated surface treated for 10 min. with boiling H<sub>2</sub>O and polished with soap powder. The reflection factor of the product is 85.3%.

**Bright aluminum surface** Ralph B. Mason and Martin Tosterud (to Aluminum Ltd.) Can 354,511, Dec. 3, 1935. An Al article is buffed to a reflection factor of 75.9% and electrolytically treated at 60° in an electrolyte contg 25% by wt. H<sub>2</sub>SO<sub>4</sub> soln and 2% of 48% HF soln. for 10 min. It is then anodically treated in a 12% H<sub>2</sub>SO<sub>4</sub> soln at 24°. The oxide-coated surface obtained is heated with boiling H<sub>2</sub>O for 10 min. and polished with soap powder; it has a reflection factor of 84.5%.

**Electrolytic surface treatment of aluminum and its alloys** Ralph B. Mason and Martin Tosterud (to Aluminum Co of America). Brit 436,154, Oct. 7, 1935. A bright surface of high reflectivity is produced on Al or Al alloy by anodic treatment in a soln. contg 1-60% of

H<sub>2</sub>SO<sub>4</sub> together with HF. A current d of 10-100 amp. per sq ft. and temp of 30-70° are suitable. D. c. or a c. may be used. The treated surface may be coated with clear lacquer or varnish or it may be subjected to further anodic treatment in a H<sub>2</sub>SO<sub>4</sub> or (COOH)<sub>2</sub> bath to produce a clear, transparent oxide coating which does not appreciably impair the reflectivity. Cf. C. A. 30, 3947, 1935, 481. Oct 7, 1935, the bright surface is produced by anodic treatment of the Al, etc., in an electrolyte contg. CrO<sub>3</sub> and HIF.

**Coating aluminum** Siemens & Halske A-G Fr. 789,699, Nov 4, 1935. Galvanic coatings are obtained on Al by subjecting the Al to a preliminary anodic treatment in an electrolyte contg HF, HI, H<sub>3</sub>PO<sub>4</sub> or H<sub>2</sub>PO<sub>4</sub> and HF.

**Oxide layers on aluminum** Veremigte Aluminium-Werke A-G Ger 620,894, Oct 29, 1935 (Cl 48a 16). The layers are produced on Al or Al alloys by electrolysis in an (CO<sub>2</sub>-H<sub>2</sub>) bath contg also a small amt of oxidizing agents such as peroxides or salts of inorg. acids, e. g., K<sub>2</sub>MnO<sub>4</sub>, K<sub>2</sub>CrO<sub>4</sub> or K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, and HNO<sub>3</sub>, added to (COH)<sub>2</sub>. Cf C A 29, 17253

**Oxidation of aluminum alloys** Carlo Borgo Fr. 789,617, Nov 4, 1935. The surface hardness of Al alloys, particularly those contg Cu, is increased by electrolytic oxidation using as electrolyte an aq soln of oxalic and tartaric acid, in which are dipped the pieces to be oxidized connected to the anode, while the cathode is composed of any conductor.

**Oxide and colored films on aluminum** Pentol Soc. Anon Fr 788,873, Oct 18, 1935. Films of oxide are produced electrolytically on Al and its alloys in a bath contg an aromatic sulfonic acid, e. g., PhSO<sub>3</sub>H, toluene-p-sulfonic acid and mono- and di-sulfonic acids of C<sub>6</sub>H<sub>4</sub>. Fr 788,874. The bath contains a substance having a capillary action, e. g., Na sulfocimates, Na isopropylphenathene-sulfonate and diamyl ester of sulfophthalic acid. Fr 788,875. See Swiss 175,366 (C A 30, 759) Fr 788,876. A protective tube for elec. conductors is made of Al or an Al alloy, the surfaces of which (exterior and (or) interior) are covered with a film of oxide impregnated with insulating materials. Cf C A 30, 757. Fr 788,877. See Swiss 175,365 (C A 30, 757) Fr 788,878. Paper or cardboard is covered with sheets of Al or its alloys, the surface of which has been oxidized. Fr 788,879. The film of oxide formed on Al is colored, and a design is produced by corroding a part with an alk. agent. Fr 788,880. See Swiss 176,415 (C A 30, 759).

**Metallic films by cathodic disintegration** Julius von Bosse and Kurt Richter (to La Dispersion Cathodique) U S 2,028,853, Jan. 29. For accelerating the cathodic disintegration of metallic electrodes such as those of Au or Ag in a vacuum for deposition upon an article to be coated without excessive heating of the article, the size of the electrodes is so adjusted that the greatest cross sectional dimension of the electrodes is from three-tenths to approx. equal to the mean free path of the atoms formed by disintegration of the cathode. Cf C. A. 29, 10211.

**Removing electrolytic deposits** Kelsen Special Sheet Holding Soc. Anon. Brit 436,282, Oct. 9, 1935. Electrolytic deposits of metal are loosened from cathodes by dissolving the metal on particular surfaces or along particular lines as desired, the solvent selected being one that reacts with the deposit but does not attack the cathode. Thus Cr steels and steels which, in addn. to Cr, contain Ni may be used as cathodes and Cu deposited thereon is removed with dil. HNO<sub>3</sub> or H<sub>2</sub>SO<sub>4</sub>.

**Electroplating apparatus** John Kronsbein and Charles F. Neale Brit 435,631, Sept 25, 1935. Articles are carried through the plating bath by a partially submerged rotating wheel or disk constituting an electromagnet on the periphery of which the articles are supported.

**Zinc plating.** Soc. d'Electrochimie, d'Electrometallurgie et des acieres Electriques d'Ugine Fr. 789,076, Oct. 22, 1935. Electrolytic coatings of Zn are obtained by adding carbamide to solns of neutral salts of Zn.

**Chromium plating** N. D. Bryukov. Russ. 37,956, July 31, 1934. To a plating bath of H<sub>2</sub>CrO<sub>4</sub>, Cr<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>,

$ZnCrO_4$  and  $MnCrO_4$  are added hydrates of  $CaCO_3$  or  $CaO$  in such proportion that the ratio by wt of the  $Ca$ ,  $Zn$  and  $Mn$  is 1:1.0:0.3.

Bath for electroplating with lead. A. B. Yakulov and P. E. Borozhin. Russ. 37,957, July 31, 1934. To a bath contg.  $Pb(OAc)_2$ ,  $AcOH$  and a protective colloid is added  $NH_4OH$ .

Electrolysis. *Patent Soc. Anon. Brit.* 436,270, Oct. 8, 1935. See Swiss 176,639 (*C. A.* 30, 319).

Apparatus for the electrolysis of alkali chlorides. Charles Pouyand. Ger. 620,902, Oct. 30, 1935 (*Cl.* 121.9).

Apparatus for the agitation of the electrolyte in cells for the electrolytic etching of zinc or other metals. José G. Walling. Brit. 436,176, Oct. 7, 1935.

Heavy water and hydrogen. Albert F. Knowles. Brit. 435,450, Sept. 23, 1935. In the electrolysis of water for the concn of heavy water, the water vapor carried off by the gases is condensed before washing of the gases and is used as feed to an electrolytic cell in the next stage of concn. The feed water may serve for washing. The washed hydrogen may be brought into intimate contact with the feed water in a coke tower, the  $D$  interacting with the  $H_2O$  to form a certain amt of heavy water. The hydrogen from the later stages of concn is liquefied and the "heavy" and "light" gases are sep'd by fractional distn. The  $D$  so obtained is burnt in air to yield almost pure  $D_2O$ .

Electrolytic generation of hydrogen. Siemens & Halske A.-G. Brit. 435,719, Sept. 26, 1935.  $H_2$  generated under pressure in an electrolytic cell is led by a pipe to a pressure-tight flask filled with  $H_2O$  or other inert and incompressible liquid, the flask being filled with  $H_2$  by displacement and the displaced  $H_2O$  passing by a pipe to fill a fresh flask from which the displaced air escapes by a capillary.

Storage-battery electrodes. The Expanded Metal Co. Ltd. and Harry P. Salmon. Brit. 436,268, Oct. 8, 1935. The electrode in the form of a box filled with active material is made of expanded or partly expanded metal and may be made by bending a sheet of the metal into shape and welding the free edges together. *Cl. C. A.* 29, 408.

Oxide-coated cathode. Irwin F. Lowry (to Canadian Westinghouse Co. Ltd.). Can. 354,260, Nov. 19, 1935. For cathodes to be used in gas-filled tubes, such as rectifiers, for heavy duty a coating of  $BaO$  or  $SrO$  is used as the electron-emissive material.

Rotatable cathode for electrolytic cells such as those used for depositing metals in sheet form. Allen C. Jephson and Ernest B. Custer (to National Radiator Corp.). U. S. 2,029,285, Jan. 21. Various structural and mech. details.

Engine spark plug with a palladium-containing contact. Robert John and Erwin I. Spellmeyer (to Carbex Chemical Co. of Ill.). U. S. 2,028,749, Jan. 23.  $Pd$  or a  $Pd$  alloy is used on one or both electrodes.

Refining metals. Ivar Rennerfelt. Fr. 789,700, Nov. 4, 1935. The air in an induction furnace is replaced by an inert gas such as  $N$  and the metal is strongly beaten by  $CO$ , water gas or  $H_2$ . The metal is protected against any harmful action of the inert gas by a layer of slag.

Refining auriferous material. Deutsche Gold- und Silber-Scheideanstalt vormals Roessler. Brit. 435,731, Sept. 26, 1935. See Fr. 784,928 (*C. A.* 30, 359).

Electric induction furnaces. Heraeus-Vacuum-schmelze A.-G. and Wilhelm Rohn. Brit. 436,490, Oct. 11, 1935. Added to 382,002 (*C. A.* 27, 2888). In a furnace according to 382,002, a coil for high frequency current, preferably wound about a vertical axis, is arranged between the pancake coils and the hearth, and the turns of the latter coils cross the turns of the high frequency coil to a preponderating extent.

Induction furnace for melting metals. A. I. Boldurev and N. N. Barushnikov. Russ. 37,776, July 31, 1934. Construction details.

Electric smelting furnace. Akt. Ges. Brown Boveri & Cie. Ger. 620,778, Oct. 29, 1935 (*Cl.* 31a.2.40).

Automatic electrolytic vulcanizer. Maurice Alléra. Fr. 789,515, Oct. 30, 1935.

Electrically heated coke ovens. Akt.-Ges. Brown, Boveri & Cie. Fr. 789,422, Oct. 29, 1935.

Electrically heated annealing furnace. Deutsche Bekleidungsindustrie G. m. b. H. Ger. 620,820, Oct. 28, 1935 (*Cl.* 18c.8.01).

Charging electric metallurgical furnaces. Paul I. Mignet and Marcel P. Perron. Ger. 620,786, Oct. 30, 1935 (*Cl.* 40c.16.01).

Electric resistance heater suitable for use externally of tanks or conduits. John A. Knight. U. S. 2,029,673, Jan. 28. Structural details.

Electrode steam boilers. Siemens-Schuckertwerke A.-G. (Heinrich Gelius, inventor). Ger. 620,592, Oct. 24, 1935 (*Cl.* 21b.7). A perforated float-supported sleeve for tapping off water with a high salt content is described. Thus keeps the cond. of the water from becoming too high.

Treating air electrically. Constantin P. Yagiou. Brit. 436,467, Oct. 11, 1935. Mixts of pos and neg ions in desired proportions are obtained by supplying to 1 or more sharp electrodes an alternating voltage greater than 800 v., causing an air flow over the electrodes and varying the length of the path from the electrodes to earth, whereby the rate of destruction of the neg. ions relative to that of the less mobile pos. ions can be varied. App. is described. *Cl. C. A.* 29, 3244.

Heating fluids by electricity. Gaston H. C. Roux. Brit. 436,676, Oct. 16, 1935. A fluid, e. g., a gas, is heated by passage through a refractory insulated container provided with electrodes and a resistance filling of magnetite alone or mixed with  $MnO_2$ , wolframite, sulfides of  $Pb$ ,  $Zn$ ,  $Fe$  or  $Cu$ , or non-conducting materials.

Electron-discharge device. Victor O. Allen (to Hygrade Sylvania Corp.). Can. 355,060, Dec. 31, 1935. A radio tube has an electron-emissive cathode coated with alk. earth oxides and an anode consisting substantially of entirely graphitized  $C$ , substantially free from occluded gases and entrapped carbonaceous products, and from loose amorphous  $C$ . *Cl. C. A.* 29, 4634.

Photoelectric tube. George R. Stilwell (to Bell Telephone Laboratories, Inc.). U. S. 2,029,040, Jan. 23. An anode such as a  $Ni$  ring is used with a light-sensitive electrode having a base metallic layer such as  $Na$  carrying a thin film of dielec. material such as  $Na$  sulfide and an overlying thin film of light-sensitive material such as  $Na$  which in turn is covered by another thin film of dielec. material. Various structural details of associated app. are described.

Photoelectric tube. Jan H. de Boer, Johannes Bruyens and Marten C. Teves (to Radio Corp. of America). U. S. 2,029,414, Feb. 4. An anode is used with a photoelec. electrode contg. alkali metal such as  $Cs$ , and a metal such as  $Sn$  capable of alloying with the excess alkali metal of the tube is disposed outside the vicinity of the electrode. Structural details are described.

Thermionic valves, etc. The M.-O. Valve Co. Ltd. and Mark Benjamin. Brit. 436,527, Oct. 14, 1935. In the production of deposits of alkali and alk. earth metals within sealed vessels for photoelec. cathodes, a mixt of an oxide or carbonate of the metal and  $Al$ , the no. of atoms of  $Al$  being equal to or greater than the no. of  $O$  atoms or  $CO_3$  radicals, is heated in  $H_2$  at atm. pressure to about  $1000^\circ$ . The product is introduced into the vessel as pellets, etc., and heated in *vacuo* to  $1000^\circ$  or over. The process is not practically possible for  $Cs$ , and rarely so for  $K$ ,  $Na$ ,  $Ca$ , and oxides of  $Ba$ ,  $Sr$ ,  $Ca$  and  $Li$  are preferable.

Colored liquids in electrical signs. Fuzo Mukasa. U. S. 2,029,183, Jan. 28. App. is described in which differently colored liquids of different sp. grs. such as water and oil are used, layers of which are caused alternately to flow through an illuminated pipe.

Metal-vapor lamp. Harvey C. Rentschler (to Canadian Westinghouse Co. Ltd.). Can. 355,173, Jan. 7, 1936. A high-pressure Hg-vapor lamp has an env. envelope contg. electrodes of the activated type and a charge of starting gas. A sufficiently small amt of  $Hg$  is inside the envelope that it can be completely vaporized in the operation of the lamp with sufficient elec. energy.

Apparatus for salvaging arc-lamp carbons by mechanical turning. Wallace E. Cross. U. S. 2,029,522, Feb. 4. Mech. and operative details.

**Flashlamps.** N. V. Philips' Gloeilampenfabrieken. *Brit. 436,047*, Oct. 3, 1935. In addition to the material producing the actinic light, a complex Co compound the color of which is changed by  $H_2O$ -vapor is also enclosed in the bulb to indicate the leakage of air thereinto. Co or Ni cobaltcyanide and hydroazonomium cobaltcyanide are suitable compounds.

**Flashlamps.** N. V. Philips' Gloeilampenfabrieken. *Brit. 436,694*, Oct. 16, 1935. A lamp comprises a wire or band of Mg or Mg alloy having a periphery less than  $1500\mu$  and obtained by drawing through dies producing a comparatively small reduction of section, preferably a decrease in circumference of 6% at most per die, the zone of deformation being heated to  $250-400^\circ$ .

## 5—PHOTOGRAPHY

R. R. BULLOCK

**Relation between exposure and density for x-ray exposures.** J. E. de Langhe. *Z. wiss. Phot. 34*, 174-80 (1935).—On the basis of several hypotheses, a general equation is derived for the blackening on development ( $d$ ) of an emulsion layer after exposure with x-rays. From the discussion of this equation, the following conclusions are drawn: (1) In its lower part, the  $d-E$  curve is linear. From the equation of this linear part, it appears that the  $d$  is proportional to the expression  $\Sigma a_i n_i$  [where  $a_i$  is the projected area after development, and  $n_i$  is the no. of grains per sq. cm. of the emulsion surface]. Accordingly, for a unidisperse layer, the  $d$  is proportional both to the individual, and to the total, grain surface. The  $d$  is a function of the wave length through the expression  $f(\gamma) \gamma h$  [where  $\gamma$  is the no. of nuclei formed per quantum]. (2) The max. slope of the  $d-\log E$  curve is independent of the wave length of the incident x-rays. It can be calculated from 2 of the equations for a unidisperse single-grain layer.

**Fine-grain chemical development.** R. Namas. *Progresso fot. 41*, 97-102, 139-45 (1934). *Phot. Abstracts 15*, 194 (1935), cf. *C. A. 29*, 3925<sup>7</sup>.—Formulas for  $p$ -phenylenediamine developers are often not given with sufficient detail to det. the form in which the  $p$ -phenylenediamine is used. Many developers stated to give fine grain give very incomplete development of the image and require increased exposure. If their activity is increased by adding alkali, graininess is also increased. Some of these developers do not give better results than those obtained with a metol developer contg. just sufficient alkali to free the base; such a developer is improved by the addn. of a small amt. of safranine.

**Sensitometric testing without a photometer.** Th. Mendelssohn. *Phot. Ind. 32*, 886-7 (1934). *Phot. Abstracts 15*, 224; cf. *C. A. 29*, 3925<sup>7</sup>.—Both in measuring the  $d$  of an absorbing medium and in deriving characteristic curves of photographic emulsions, testing without a photometer involves the detn. of points of equal  $d$  on 2 sensitometric strips. To facilitate this, Hübl has recommended placing the 2 strips in juxtaposition under a mask contg. a narrow slit 1.5 mm. wide, and moving 1 strip with respect to the other until equality of  $d$  is obtained. By this method, a probable error of 1 mm. is claimed, and M. has checked this for 3 contrasts of printing paper. Results are quoted and discussed.

**Sensitometric study of the Artigue process with two sensitive surfaces superposed.** J. B. Turdy. *Photo four tous 10*, 167-70, 185-8 (1934). *Phot. Abstracts 15*, 224.—On account of rapid plates being "backed" and the impossibility of using 2 superposed plates with most plate holders, T. suggests the use of 2 emulsion-coated films placed back to back. Such "tandem-negatives" have been sensitometrically studied by developing the 2 component films separately in different developers, 1 to give a soft negative and 1 a hard negative. Three development times were used, and characteristic curves are reproduced both for the negatives separately and for the various possible combinations of them as composite negatives.

**Progress and applications in infrared photography.** John Eggert. *Veröff. d. wiss. Zentral-Lab. phot. Abt. 4*, 98-118 (1935).—An account is given of the further extension of sensitivity into the infrared as far as  $1360\mu$  rendered possible by the use of the cyanine dyes with 9- or 11-membered polymethenyl chains, which were

obtained in 1933, with special reference to the properties of the Agfa Infrared plates. For the later part of this article see *C. A. 29*, 6203<sup>1</sup>. R. R. Bullock

**Stability of various sensitizing solutions for infrared rays.** Giovanni Semerano. *Ann. chim. applicata 25*, 473-81 (1935).—A photographic film which has been sensitized to the infrared is not very stable. Aq., as well as alc., solns. of pinacyanol, rubrocyanine, neocyanine and alloxyaniline, neutral, as well as with addn. of acid or  $NH_3$ , were used. The neutral solns. are most stable. Those compounds having a methene chain in  $o$ -position with respect to the quinoline N are more stable than those where the chain is in  $p$ -position. Heavy constituents in the methene chain also increase the stability of the mol. A. W. Contieri

**Deviation from Schwarzschild's law in the ultraviolet.** Maurice Lambrey and Jean Corbière. *Compt. rend. 201*, 1351-2 (1935).—When the density,  $d$ , of a plate is plotted against  $\log E_0/E$ ,  $E_0$  being a fixed and  $E$  a variable illumination, for  $\lambda$  4300-3230 Å, with the time of exposure,  $t_0$ , const., the curve obtained differs materially from that obtained by plotting  $d$  against  $\log t/t_0$  with const. illumination  $E_0$ , and time of exposure,  $t$ , variable. The Schwarzschild exponent calculated from these results varies, the variation increasing with increasing rapidly as  $\gamma$  (the factor of development) decreases and  $d$  increases, in approx. the same way as  $\gamma$  was found to do (Fabry and Buisson, *C. A. 18*, 2845). C. A. Silbrad

**Influence of atmospheric oxygen and of the desensitizer concentration on the sensitivity of desensitized photographic layers.** Marietta Blau and Hertha Wambacher. *Sitzber. Akad. Wiss. Wien, Math.-naturw. Klasse, Abt. IIa*, 144, 403-8 (1935); cf. *C. A. 29*, 4685<sup>2</sup>.—For exposures at ordinary pressure, the effect of treating Agfa Contrast plates with pinakrytol yellow soln. is to increase the steepness of the intensity-scale, and to decrease that of the time-scale,  $d$  curve. This effect is greater, the higher the concn. of the dye, but, at all the concns. used (0.001% to 0.2%), decreases with decreasing pressure (to 6 mm.). It is considered that the facts of desensitization are best explained by an oxidation theory, according to which the desensitizing dye acts as intermediary between the oxygen of the air and the development nuclei produced by light. Solarization, which is almost prevented by the highest dye concn. used at atm. pressure, is progressively less affected by lower dye concns. and at lower pressures; and the desensitizing action of KBr and of KI, and the bleaching-out action of the latter, become less pronounced under the lowest pressure. R. R. Bullock

**Modern development papers.** Wandelt. *Phot. Ind. 32*, 1350-2 (1934); *Phot. Abstracts 15*, 218.—Typical characteristic curves for the 7 contrasts of Agfa "Lupeke" glossy gaslight curves are reproduced, together with a table of sensitometric constants in which are quoted the gradation—measured by the length of the projection on the abscissa of the region of the characteristic curve lying between the  $d$ s 0.02 and 1.7—and the  $\gamma$  values. W. points out that neither the gradation nor the  $\gamma$  value alone characterizes a paper, as 2 papers may have similar gradations for very different  $\gamma$  values and vice versa.

**Luz, Antoine.** Les filtres colorés ou écrans compensateurs en photographie. Paris: J. de France. 96 pp. F. 4.50

**Person, Alfred.** Bildmässige Leica-Photos durch Ton-

trennung nach dem Person-Verfahren. 2nd ed. Frankfurt: Bechhold 83 pp. M. 7.50.

Underberg, G.: Le développement au diamidophénol acide. Paris: J. de France 78 pp. F. 4.50.

**Photographic diazotype layers** Leonard R. Harper and Donald W. Powell. Brit. 435,874, Oct. 1, 1935. Pos. diazotype prints are produced by exposing to light under a design a surface sensitized with a diazo compd. that couples in practically neutral or acid soln., e.g., diazo-1-amino-2,5-diethoxy-4-benzoylamino benzene, diazoethyl- $\alpha$ -bromoglyoxylate, 2,6-dichloro-4-amino-phenylhydrazine, diazo- $\alpha$ -chloro- $\alpha$ -ketopropaldehyde-2-chloro-4-amino-phenylhydrazine, and developing the surface with a soln. that is neutral or alk. or slightly acidified with  $H_2BO_3$  and contains a salt of an aromatic org. acid that is readily pptd. in aq. soln. by a stronger acid and has no deleterious effect on the print. Developing solns. named contain (1) phloroglucinol (I) and  $BzONH_2$  (II), (2) I, II and  $H_2BO_3$  (III), (3) I, II, Na K tartrate and III, and (4) Na phenylacetate,  $Na_2WO_4$  and III.

**Photographic diazotype processes** I. G. Farbenindustrie A.-G. Brit. 436,587, Oct. 7, 1935. Light-sensitive material for the production of pictures in 1 or more colors by synthesis of 1 or more azo dyes comprises 1 or more layers, e.g., gelatin, paper, cellulose hydrate, dyed with 1 or more azo dye components substantive to the layer. Sparingly sol. components having acidic or OH groups are used in the form of salts and soly may be promoted by addn. of MeOH. The layer may be dyed with an azo coupling component, a light-sensitive diazo compd. added and a colored image produced by development after exposure. Gelatin treated with both components may be worked up into an Ag halide emulsion and the Ag image bleached in a bath tanning the gelatin at the exposed parts. Dye formation occurs at the unhardened parts on treatment with acidified  $NaNO_2$  soln. Red pictures are obtained from 2,3-hydroxynaphthoic acid  $\alpha$ -naphthalide with ureabus ( $p$ -amino benzoyl  $p$ -phenylenediaminesulfonic acid) (I) and blue with diazotized diaminidine. The gelatin may be dyed with the azo coupling component and the Ag image converted into an anti-diazotized image as described in Brit. 387,197 (C. A. 27, 5016). Yellow pictures are obtained from di- $o$ -sulfo benzylidene-4,4'-diaminodiphenylurea, obtained by reaction of 4,4'-diaminodiphenylurea with 2 mol. proportions of  $o$ -sulfo benzaldehyde, and the anti-diazotized  $\beta$ -naphthylamine. The gelatin may be dyed with the azo-coupling component, the Ag halide caused to absorb  $p$ -nitrobenzenediazonium chloride, and coupling effected. The gelatin may be dyed with both compds., the Ag image converted into an insol. nitrite compd. and diazotization effected by treatment with dil. HCl. A red picture is obtained from the urea produced by the action of  $COCl_2$  on  $p$ -amino benzoyl- $p$ -amino benzoyl J and acid and the I obtained by the process of Brit. 344,791 (C. A. 6, 1858).

**Photographing on superposed films of different sensitivities** Clyde F. Gillette (to United Research Corp.) U. S. 2,028,975, Jan. 28. Various optical and operative details.

**Motion picture photography** Charles B. Dreyer (one-half to Henry F. Boeger). U. S. 2,028,275, Jan. 21. Light from an object to be photographed is passed through a front emulsion sensitive to a range of light wave lengths but insensitive to a certain band of wave lengths within the first mentioned range and thence to a rear emulsion preferentially sensitive to the wave band to which the front emulsion is not sensitive, and those portions of the object being photographed which it is desired to accentuate are illuminated with light contg. wave lengths to which both emulsions are sensitive while the remainder of the

scene is illuminated with light of wave lengths mainly within the range to which one only of the emulsions is preferentially sensitive.

**Colored photographs** Franz Lejeune. U. S. 2,023,077, Jan. 29. A process for the production of colored photographs in natural colors on a single printing surface in which three printing images produced by tanning in a picture like manner and corresponding to a blue print image, a yellow print image and a red print image are soaked with metallic solns. involves embodying in the printing surface a ppt. in the form of a water-insol. Pb compd., such as Pb carbonate, a dimethylglyoxime ppt. and a ferric salt ppt. and afterward soaking the blue print image with a  $K_4FeC_6N_6$  soln., placing the blue print in intimate contact with the printing surface to form a Prussian blue ppt. in it, stripping off the blue print image, further soaking the yellow print image with a chromatic salt soln. and placing the yellow print image in register and intimate contact with the printing surface carrying the blue image to form a yellow ppt., stripping off the yellow print image, soaking the red print image with a Ni salt soln. and placing the red print image in register and intimate contact with the printing surface provided with the blue image and yellow image to form a red ppt., and stripping the red print image.

**Photographic sound records** I. G. Farbenindustrie A.-G. Brit. 435,473, Sept. 23, 1935. In the photographic recording of sound by the variable d. method and in the prepn. of a pos. point from an original negative in such manner that the product of the gammas of the positive and of the negative may be 1, the neg. film is developed to a gamma greater than 1, the exposure having been such that the ds. corresponding to the max. and min. lie on the straight part of the characteristic curve; a print is then made on an undyed film, the exposure being such that the d. lies on the straight part of the curve, and the development of the positive is such that its gamma is the reciprocal of that of the negative. A detailed example is given, with formulas for metal and metal hydroquinone developers. Cf. C. A. 30, 404<sup>1</sup>.

**Photographic developers** I. G. Farbenindustrie A.-G. Fr. 789,616, Nov. 4, 1935. Halogen substitution products of 2-methylaminophenol (I), e.g., 5-chloro-, 4-chloro- and 4,6-dichloro I, are used for developing Ag halide emulsions with fine grain.

**Photographic developers** I. G. Farbenindustrie A.-G. Fr. 789,640, Nov. 4, 1935. 2-Methylaminophenols substituted in the ring by alkyl, OH or MeO groups, e.g., the 4-methyl or 5-methoxy derivs., are used for developing Ag halide emulsions. Cf. C. A. 29, 419<sup>4</sup> and preceding abstr.

**Photographic emulsion** Béla Gáspár. U. S. 2,028-279, Jan. 21. A color-photographic material comprises a Ag halide emulsion having incorporated in it differently colored dyes having varying powers of resistance to the effect of an agent serving locally to discharge the dyes for the production of a color image.

**Photographic emulsions** Max Schmid (to Soc. pour l'ind. chim. à Bâle). U. S. 2,029,940, Feb. 4. See Brit. 428,305 (C. A. 29, 6520<sup>4</sup>).

**Printing papers** J. Halden & Co. Ltd. and John Holden. Brit. 435,936, Sept. 30, 1935. Printing paper and cloths sensitized with ferroproussiate soln. are coated on the sensitized side with an oxidizing agent in the form of a fine, dry powder not easily sol. in  $H_2O$  so that it will exert an intensifying effect late in the development stage. Suitable agents are Sr, Mg and Ca peroxides or hypochlorites and Cl derivs. of aromatic sulfonamides. Cf. C. A. 29, 2106<sup>1</sup>.

**Apparatus for developing blue prints with a gaseous developer** A. F. Prokin. Russ. 34,307, Jan. 31, 1934. Construction details.

## 6—INORGANIC CHEMISTRY

A. R. MIDDLETON

The development of the chemistry of rhenium. Ida Noddack. *1A Congr. intern. quim. pura aplicada, Madrid 3, 334-40* (1934), cf. *C. A.* 28, 723.—A review of Re and its compds. The heats of formation of Re oxides are compared graphically with data for the oxides of neighboring elements. Re is in a middle position, which accounts for the stability of its oxides. There are only small differences in the heats of formation of the oxides, and for this reason reactions in which part of the Re is reduced and part oxidized to a higher valence are possible. L. R. R.

Palladium carbide. Norbert G. Schmahl. *IX Congr. intern. quim. pura aplicada, Madrid 3, 468-74* (1934).—CH<sub>4</sub> or a mixt. of CH<sub>4</sub> with H in contact with Pd powder formed by reducing PdO with H reaches equilibrium in several hrs. The C content of the solid was 4.4%, corresponding closely to Pd<sub>2</sub>C<sub>3</sub> (4.31% C). Photographs of x-ray spectra are given which show that it is a true compound. The compd. shows the same capacity to absorb H as Pd, and this is explained by assuming a double intercalated structure, according to the theory of Hagg (*C. A.* 25, 2615).

E. R. Rushton  
Preparation of carbon suboxide. A. Klemenc, R. Wechsberg and G. Wagner. *Monatsh.* 66, 337-44 (1935), cf. *C. A.* 29, 6170.—A discussion of the prepn of C<sub>3</sub>O, from Cl<sub>2</sub>(CO<sub>2</sub>H) (I) and P<sub>2</sub>O<sub>5</sub> at 140-150° (not over 20% yield) and by sublimation of (AcO)HC CH(OAc) CO O CO

(II) in CO (at 710°, 9.3% C<sub>3</sub>O and 0.97% AcOH) and in CO<sub>2</sub> (21% C<sub>3</sub>O and 60% AcOH at 650° or 810°). An app. is illustrated. The carefully purified C<sub>3</sub>O is very stable and a sample at 120 mm Hg was unchanged after 1 year. The sample from II appears to be more stable than that from I. C. J. West

Chlorous anhydride. Michel Kanter. *Compt. rend.* 202, 209-10 (1936).—By effecting the interaction of KClO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> in presence of undecylic acid the proportion of ClO<sub>2</sub> is much diminished, i. e., the reaction 2ClO<sub>2</sub> = Cl<sub>2</sub>O + Cl<sub>2</sub>O<sub>2</sub> is favored at the expense of 3ClO<sub>2</sub> = Cl<sub>2</sub>O + 4ClO. The spectra of Cl<sub>2</sub>O<sub>2</sub> and ClO<sub>2</sub> have been detd., that of Cl<sub>2</sub>O (OOC<sub>2</sub>Cl<sub>2</sub>) showing distinct analogies to those of SO<sub>2</sub>Cl<sub>2</sub> and CrO<sub>2</sub>Cl<sub>2</sub>. C. A. Silberrad

Arsenates of barium, a sesquiasenate. Henri Guérin. *Compt. rend.* 202, 225-7 (1936).—Ba(AsO<sub>3</sub>)<sub>2</sub> begins to evolve O (and As<sub>2</sub>O<sub>3</sub>) at 500° and, provided it is not heated above 700°, forms the sesquiasenate Ba<sub>2</sub>As<sub>2</sub>O<sub>7</sub>, which at 750-800° passes into Ba<sub>3</sub>As<sub>2</sub>O<sub>7</sub>, and thus above 800° into Ba<sub>4</sub>(AsO<sub>3</sub>)<sub>3</sub>, the only arsenate stable at high temps. C. A. Silberrad

Hydrated barium and strontium nitrites. Jean Bureau. *Compt. rend.* 202, 307-9 (1936).—The nitrites were prepd by the action of the products of catalytic oxidation of NH<sub>3</sub> on solns. of the hydroxides and concn. in vacuum, or pptn. by LiOH in cold. The system Ba(NO<sub>2</sub>)<sub>2</sub>·H<sub>2</sub>O has a eutectic, Ba(NO<sub>2</sub>)<sub>2</sub>·H<sub>2</sub>O (hexagonal)—H<sub>2</sub>O, at -5.8° (20.3% Ba(NO<sub>2</sub>)<sub>2</sub>). The hexagonal form is stable to 40°, c = 52%, above which the monohydrate assumes the rhombic form and the soly is given by c = 36 + 0.400t, the satd. soln. b. 114.8°. At 116° the anhyd. salt is formed, which m. 267° and begins to decompose at 270°. Sr(NO<sub>2</sub>)<sub>2</sub>·H<sub>2</sub>O has a eutectic, Sr(NO<sub>2</sub>)<sub>2</sub>·4H<sub>2</sub>O (cubic) H<sub>2</sub>O, at -8.7° (26.4% Sr(NO<sub>2</sub>)<sub>2</sub>), soly c = 31 + 0.645t, at 15° (c = 39.55%) it changes to Sr(NO<sub>2</sub>)<sub>2</sub>·H<sub>2</sub>O (hexagonal), soly. (at 15-100°) c = 36.75 + 0.1916t. The anhyd. salt decomposes before melting. C. A. S.

Hydrolytic equilibria of calcium aluminate hydrates. Frick Peter von Polheim. *Zement* 24, 643-8, 659-63, 677-83 (1935).—It is difficult to reach equilibria between the various Ca aluminates hydrates in contact with lime solns. at 20-40° because of the slowness of reaction. Another difficulty arises in the variations of cry. strn. tendencies of the several aluminate hydrates, resulting in formation of intermediate products. Three distinct Ca aluminate hydrates of different lime contents are in equilibrium with their solns. at 20°. There are 3 lime water concns. with

each of which 2 solid phases occur, namely Al(OH)<sub>3</sub> and dicalcium aluminate hydrate, di- and tricalcium aluminate hydrate, and tri- and tetra-calcium aluminate hydrate. The concn. of solns. yielding tricalcium aluminate hydrate was found to be 560 mg. CaO and about 80 mg. Al<sub>2</sub>O<sub>3</sub> per l. The values were confirmed for the di- and tetra-calcium aluminate hydrates as 300 mg. CaO with 100 mg. Al<sub>2</sub>O<sub>3</sub> per l. and 1070 mg. CaO with less than 3 mg. Al<sub>2</sub>O<sub>3</sub> per l., resp. All 3 hydrates form hexagonal plates at 20°. The dicalcium salt shows a characteristic glittering, even in the finest state of division. Only the tricalcium aluminate hydrate is found at 37°, with CaO concns. between 360 mg. per l. and satn. H. F. Kriege

Preparation of sulfur and magnesium sulfate from sulfur dioxide and magnesia. I. Hiroshi Hagisawa. *Bull. Inst. Phys.-Chem. Research* (Tokyo) 14, 1081-90 (1935).—S and MgSO<sub>4</sub> are prepd by the thermal decompn. of an aq. soln. of Mg(HSO<sub>3</sub>)<sub>2</sub>. When this soln. is heated at 130-150° in a closed tube with or without addn. of MgSO<sub>4</sub>·6H<sub>2</sub>O with shaking, S and MgSO<sub>4</sub> are formed in accordance with the following reactions: 3Mg(HSO<sub>3</sub>)<sub>2</sub> = 3MgSO<sub>4</sub> + H<sub>2</sub>SO<sub>4</sub> + 2S + 2H<sub>2</sub>O, Mg(HSO<sub>3</sub>)<sub>2</sub> + MgSO<sub>4</sub> = 2MgSO<sub>4</sub> + S + H<sub>2</sub>O. In these reactions S acts as a catalyst, the preliminary addn. of S therefore accelerates the reactions to a great extent. Even then the 1st reaction does not proceed to completion (about 95%), while the latter comes nearly to an end in an hr. at 140° and within 20 min. at 150°. In this case a little Sr<sub>2</sub>O<sub>2</sub> is found in the reaction product, the max. pressure in the reaction vessel is about 20 atm. at 140°. K. Konda

Hyposulfites. II. The reduction of sodium bisulfite by zinc amalgam. 1. Toyosaku Murooka. *Bull. Inst. Phys.-Chem. Research* (Tokyo) 14, 1091-7 (1935).—Reduction of concd. Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> soln. (av. 38.76%) with Zn amalgam in liquid form gives Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> in very good yield; the rate of utilization of Zn is about 95%. The reduction velocity is greatly affected by stirring, this reaction proceeds smoothly, and comes to an end within 70 min. at 10° if the soln. is stirred sufficiently. The end point can be detd. by potentiometric titration or quantitative analysis.

III. The reduction of sodium bisulfite by zinc amalgam. 2. *Ibid.* 1154-60 (Abstracts 67-8).—The method of reduction with Zn amalgam also gives good results in the prepn. of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> by reducing Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> soln. with addn. of H<sub>2</sub>SO<sub>4</sub> and dil. H<sub>2</sub>SO<sub>4</sub>. Slight decompn. of free H<sub>2</sub>S<sub>2</sub>O<sub>4</sub> is, however, inevitable when H<sub>2</sub>SO<sub>4</sub> is added, the yield of H<sub>2</sub>S<sub>2</sub>O<sub>4</sub> is somewhat lowered at temps. over 30°, the amalgam to be used is sufficient in an amt. less than 1.5 times of the theoretical based on the Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. The concn. of the solid substance remaining after the complete reduction is given approx. by 3ZnSO<sub>4</sub>·Na<sub>2</sub>SO<sub>3</sub> + Zn(OH)<sub>2</sub>. K. K.

The action of hydrogen sulfide on chromates. H. B. Dunnichoff, G. S. Kotwani and M. A. Hamid. *J. Phys. Chem.* 39, 1217-29 (1935).—The brown solid formed in the intermediate stages of the reduction of 5% K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> consists of Cr(OH)<sub>3</sub>, Cr(OH)<sub>2</sub>, a coordinated Cr<sub>2</sub>(OH)<sub>4</sub>, Cr<sub>2</sub>S<sub>2</sub>O<sub>7</sub> and free S while the filtrate contains K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, K<sub>2</sub>S<sub>2</sub>O<sub>7</sub> and K<sub>2</sub>SO<sub>4</sub>. The amt. of K<sub>2</sub>S<sub>2</sub>O<sub>7</sub> in soln. decreases with the amt. of chromate present until the 2 disappear together and no sulfide is present as long as any chromate remains. The tetrasulfate is formed from the oxidation of the thiosulfate by the chromate and is then reduced by the alk. sulfide. No sulfate is formed if the hydroxylion concn. is above a certain value (not given). The final products of the reaction are a ppt. composed of a mixt. of Cr(OH)<sub>3</sub>, S and a complex Cr thiosulfate in which the ratio of ionic to coordinated thiosulfate is approx. 2:1 and a soln. contg. K<sub>2</sub>S<sub>2</sub>O<sub>7</sub> and K<sub>2</sub>S at ordinary temps. or K<sub>2</sub>S<sub>2</sub>O<sub>7</sub> and K<sub>2</sub>S at 90°. Sulfite is the precursor of both thiosulfate and sulfate and some sulfate appears if the reaction is carried out rapidly at 90-95°, but no sulfate is formed after the reaction mixt. becomes alk. Arthur A. Vernon

The action of carbonic acid on ferrous sulfide. Y.



Kauko and P. Ilauio.  *Suomen Kemistilehti* 8B, 40-50 (1935) (in German).—A preliminary report.  $\text{H}_2\text{CO}_3$  reacts directly with  $\text{FeS}$ , forming a carbonate which goes into soln as a bicarbonate and ppts as the carbonate on removal of the  $\text{H}_2\text{CO}_3$ .

E. E. Jukkola

The volatilization of silica and silicon as silicon sulfide. A technological study. P. Dolch. *Chem. Fabrik* 1935, 512-14.—Historical Applications to the metallurgy of Fe and to removal of Si from  $\text{Al}_2\text{O}_3$ , etc., are discussed.

J. H. Moore

Fluorination of sulfuryl chloride—sulfuryl chlorofluoride. Harold S. Booth and Carl V. Herrmann. *J. Am. Chem. Soc.* 58, 63-69 (1936).—Sulfuryl chlorofluoride,  $\text{SO}_2\text{ClF}$ ,  $b$  7.1  $\pm$  0.1°,  $m$  -124.7  $\pm$  0.1°, was prepd. by the action of  $\text{SbF}_3$  on  $\text{SO}_2\text{Cl}_2$  in the presence of  $\text{SbCl}_3$  under pressure. The latent heat of vaporization at the b. p. is 4.339 cal. At 0°,  $d$  is 1.623. Surface tension is 17.2 dynes per cm. It is hydrolyzed by  $\text{H}_2\text{O}$  and absorbed by  $\text{NaOH}$ . There is no reaction at room temp. with glass, Hg or brass.

L. R. Rushton

Chlorofluorides of germanium. Harold S. Booth and Wm. C. Morris. *J. Am. Chem. Soc.* 58, 90-93 (1936).—The fluorination of  $\text{GeCl}_4$  by means of  $\text{SbF}_3$  in the presence of  $\text{SbCl}_3$  yields  $\text{GeF}_2\text{Cl}_2$ ,  $m$  49.8°,  $b$  37.5°,  $\text{GeF}_2\text{Cl}_2$ ,  $m$  -51.8°,  $b$  -2.8°;  $\text{GeF}_2\text{Cl}_2$ ,  $m$  -60.2°,  $b$  -20.3°, and  $\text{GeF}_4$ . The chlorofluorides hydrolyze in air more readily than  $\text{GeCl}_4$ . They rearrange to form  $\text{GeF}_4$  and  $\text{GeCl}_4$ , even at -78° and are explosively reduced by Cu to germanous salts.

L. R. Rushton

Double salts of arylidiazonium chlorides and heavy metal chlorides. Preparation of organic tin compounds by means of diazo compounds. A. N. Nesmeyanov, K. A. Kocheshkov, V. A. Klimova and N. K. Gipp. *Ber* 68B, 1877-83 (1935), cf. *C. A.* 23, 5172, 28, 1990a.—Sols of arylidiazonium chlorides were prepd. by diazotizing 1 mol. of arylamine in 300 cc. concd. HCl and 300 g. ice with solid  $\text{NaNO}_2$  and dilg. to 1 l. The double salts were obtained by mixing a soln. of the metal chloride in HCl ( $d$  = 1.19) with a 1 l. soln. of the arylidiazonium chloride in 5 N HCl, both sols. having been cooled in a freezing mixt. The double salt was filtered off, washed with 5% HCl, then  $\text{EtOH}$  and  $\text{Et}_2\text{O}$ , and dried in the air. Double salts of the following arylidiazonium chlorides with  $\text{ZnCl}_2$ ,  $\text{CdCl}_2$ ,  $\text{SnCl}_4$ ,  $\text{PbCl}_2$ ,  $\text{PbCl}_2$ ,  $\text{AuCl}_3$ ,  $\text{TiCl}_4$ ,  $\text{BiCl}_3$  and  $\text{FeCl}_3$  were prepd.  $\text{PhN}_2\text{Cl}$ ,  $o$ -,  $m$ - and  $p$ - $\text{MeC}_6\text{H}_4\text{N}_2\text{Cl}$ ,  $o$ -,  $m$ - and  $p$ - $\text{ClC}_6\text{H}_4\text{N}_2\text{Cl}$ ,  $p$ - $\text{IC}_6\text{H}_4\text{N}_2\text{Cl}$ , and  $o$ - and  $p$ - $\text{O}_2\text{NC}_6\text{H}_4\text{N}_2\text{Cl}$ ,  $p$ - $\text{HOC}_6\text{H}_4\text{N}_2\text{Cl}$ ,  $o$ - $\text{MeOC}_6\text{H}_4\text{N}_2\text{Cl}$  and  $p$ - $\text{BrC}_6\text{H}_4\text{N}_2\text{Cl}$  formed double salts with all of the chlorides except  $\text{FeCl}_3$ ,  $m$ - $\text{O}_2\text{NC}_6\text{H}_4\text{N}_2\text{Cl}$  with all except  $\text{CdCl}_2$ . The compn. and decompn. temp. of each salt are given. The derivs. of  $\text{AuCl}_3$  were golden yellow, those of  $\text{PbCl}_2$  yellow, those of  $\text{FeCl}_3$  bright lemon yellow, those of  $\text{PbCl}_2$  bright orange, all the other salts (except the  $\text{NO}_2$  and  $\text{I}$  compds. which were bright yellow) were colorless. The salts contg.  $\text{Cd}$ ,  $\text{Bi}$ ,  $\text{Sn}$ ,  $\text{Pb}$  and  $\text{Pt}$  were relatively stable, those of  $\text{Zn}$ ,  $\text{Fe}$  and  $\text{Ti}$  were easily decompd. Conversion of the double salts contg.  $\text{Sn}$  into org. Sn compds was carried out as follows: the solvent was brought to the desired temp., the metal powder was added to it with vigorous shaking, and then the double salt was sprinkled in, gradually but yet rapidly enough so that the temp. remained as const. as possible. At the end of the energetic reaction the inorg. ppt. was filtered off, the solvent evapd. from the filtrate, and the viscous residue extd. with petroleum ether. Upon concn. of this soln. the org. Sn halide crystd. In the case of low melting and difficultly crystallizable compds. the liquid obtained after removal of the petroleum ether was dild. with  $\text{EtOH}$  and then mixed with an excess of 5%  $\text{NH}_4\text{OH}$ . The diaryl tin oxide ( $\text{R}_2\text{SnO}$ ) which sep'd. was filtered off, washed and dried. The yield of  $\text{R}_2\text{SnCl}_2$  from ( $\text{R}_2\text{N}_2\text{Cl}$ ),  $\text{SnCl}_4$ , depends upon the conditions of the reaction and the chem. nature of R. The best reaction medium was  $\text{AcOEt}$  and the best temp. was its b. p. With ( $\text{PhN}_2\text{Cl}$ ),  $\text{SnCl}_4$  and Cu powder the yield was only 10% of  $\text{Ph}_2\text{SnCl}_2$  (I), decompn. with Zn dust gave a better yield, with Sn powder the yield of I was 25%. Other salts of the type ( $\text{RN}_2\text{Cl}$ ),  $\text{SnCl}_4$  that were decompd. with metal were those in which  $\text{R}$  =  $o$ - $\text{MeC}_6\text{H}_4$ ,  $p$ - $\text{ClC}_6\text{H}_4$ ,  $p$ - $\text{BrC}_6\text{H}_4$ , and  $o$ -

$\text{MeOC}_6\text{H}_4$ ; all gave only compds. of the type  $\text{R}_2\text{SnCl}_2$ . Decompn. of ( $o$ - $\text{MeO}$ ,  $\text{CC}_6\text{H}_4\text{N}_2\text{Cl}$ ),  $\text{SnCl}_4$ ,  $m$  102-4°, gave ( $o$ - $\text{MeO}$ ,  $\text{CC}_6\text{H}_4\text{N}_2\text{Cl}$ ),  $\text{SnCl}_4$ ,  $m$  164°. ( $\text{PhN}_2\text{Cl}$ ),  $\text{PbCl}_2$ , in  $\text{Et}_2\text{O}$  with Zn dust gave a small quantity of  $\text{Ph}_2\text{PbCl}_2$ ,  $m$  205°.  $\text{PhN}_2\text{Cl}$ ,  $\text{PbCl}_2$  (10 g.) with Cu powder gave  $\text{Ph}_2\text{PbCl}_2$ , which was isolated as  $\text{Ph}_2\text{PbO}$  (0.2 g.), a similar result was obtained with  $\text{PhN}_2\text{Br}$ ,  $\text{PbBr}_2$ , a yellow salt which decompd. at 85°.

Louise Kelley

System cobalt chloride-ammonium chloride-ammonia-water. Marcel Châtelet. *Compt. rend.* 202, 216-17 (1936).—Calorimetric examn. of this system shows that the first reaction is  $\text{CoCl}_2 + 2\text{NH}_4\text{OH} = \text{Co(OH)}_2 + 2\text{NH}_4\text{Cl}$ , but as the concn. of  $\text{NH}_3$  increases there is first no ppt., and then formation first of the ion  $[\text{Co(NH}_3)_4]^{++}$  and then of  $[\text{Co(NH}_3)_5]^{++}$ , the equal concns.  $(\text{Co})^{++} \times (\text{NH}_3)/[\text{Co(NH}_3)_4]^{++}$  and  $[\text{Co(NH}_3)_5]^{++} \times (\text{NH}_3)/[\text{Co(NH}_3)_4]^{++}$  being, resp. 3 and 95. C. A. S.

The constitution of complex metallic salts. III. The parachors of palladium and mercury in simple and complex compounds. Frederick G. Mann and Donald Purdie. *J. Chem. Soc.* 1935, 1549-63, cf. *C. A.* 28, 4330f.—In a homologous series with the formula ( $\text{R}_2\text{S}$ ),  $\text{PdCl}_2$  the parachor for Pd falls regularly from 30 when R is Me to -7 when R is Am. The same effect is noted when homologous phosphines or arsines are substituted for sulfides in the Pd compds. The  $m$  p. of the compds. also falls as the series is ascended. Since the series for  $\text{Pd(SR)}$ , and  $\text{Hg(SR)}$ , also show the drop in parachor for the metal, the effect cannot be due to coordinate links. The results are explained in terms of the strain const. theory of Mumford and Phillips (*C. A.* 24, 83). The parachor values for  $\text{Ti}$ ,  $\text{Be}$  and  $\text{Al}$  given by Sugden ("Parachor and Valency," Chapter 7, *C. A.* 24, 546) do not take account of this effect, and so are incorrect, as are his ideas on single bonds drawn from them. The parachors of compds. of the type  $\text{CR}_3(\text{SEt})_2$  are normal. When  $\text{PhSH}$  is added to a chloropalladate soln.,  $\text{Pd(SPh)}$ , ppts quantitatively and decomposes to Pd when heated. It can be used for the  $d_{\text{rel}}$  of Pd. The following compds. are described:  $\text{AmS}$ ,  $b$  103-9° (sulfonate,  $b$  163-4°,  $m$  60°);  $\text{BuPhS}$ ,  $b$  137-9°,  $m$  103-9°;  $\text{iso-BuPhS}$ ,  $b$  126-7°,  $m$  67°;  $\text{C}_6\text{H}_5(\text{CH}_2\text{SPh})_2$ ,  $b$  264°,  $m$  103-9°;  $\text{CO}(\text{CH}_2\text{SPh})_2$ ,  $b$  256-60°,  $m$  67°;  $\text{CMe}_2(\text{SEt})_2$ ,  $b$  93-100°,  $m$  69°;  $(\text{MeEtS})_2$ ,  $\text{PdCl}_2$ ,  $m$  67°, for the series ( $\text{R}_2\text{S}$ ),  $\text{PdCl}_2$ , when R is Pr,  $m$  59°, Bu,  $m$  32°,  $\text{iso-Bu}$ ,  $m$  163°. Am,  $m$  41°, the heptyl and octyl compds. are oils,  $[(\text{CH}_2)_7\text{CH}_2\text{S}]_2$ ,  $\text{PdCl}_2$  decomposes *in vacuo* at room temp. ( $\text{PhBuS}$ ),  $\text{PdCl}_2$ ,  $m$  118°, ( $\text{iso-BuPhS}$ ),  $\text{PdCl}_2$ ,  $m$  96°, ( $\text{Ph}_2\text{S}$ ),  $\text{PdCl}_2$ ,  $m$  170°,  $[(\text{PhCH}_2)_2\text{S}]_2$ ,  $\text{PdCl}_2$ ,  $m$  154°, ( $\text{Et}_2\text{S}$ ),  $\text{PdCl}_2$ ,  $m$  107-8° (decompn.), for the series ( $\text{R}_2\text{S}$ ),  $\text{Pd(NO}_2)_2$ , when R is Me,  $m$  137-8°, Et,  $m$  161-3°, Pr,  $m$  163-4°, Bu,  $m$  165-6°, Am,  $m$  163°, for the series ( $\text{R}_2\text{P}$ ),  $\text{PdCl}_2$ , when R is Pr,  $b$  183°,  $m$  96°, Bu,  $b$  183°,  $m$  96°,  $m$  60°, Am,  $b$  211°,  $m$  47°, ( $\text{Bu}_2\text{P}$ ),  $\text{PdBr}_2$ ,  $m$  73°; ( $\text{Bu}_2\text{P}$ ),  $\text{PdI}_2$ ,  $m$  64-5°, ( $\text{Pr}_2\text{P}$ ),  $\text{Pd(NO}_2)_2$ ,  $m$  167-8° (decompn.), for the series ( $\text{R}_2\text{As}$ ),  $\text{PdCl}_2$ , when R is Me,  $m$  235°, Pr,  $m$  55°, Bu,  $m$  54°, Am,  $m$  10-11°, ( $\text{PrAs}$ ),  $\text{PdBr}_2$ ,  $m$  49°; ( $\text{EtAs}$ ),  $\text{Pd(NO}_2)_2$ ,  $m$  176-7°; for the series  $\text{Pd(SR)}$ , when R is Pr,  $m$  209-10°, Bu,  $m$  142°, Am,  $m$  83°, for the series  $\text{Hg(SR)}$ , when R is Bu,  $m$  85°, Am,  $m$  66°, heptyl,  $m$  58°, heptyl,  $m$  75°, octyl,  $m$  71°. The correct  $m$  p. of  $\text{Et}_2\text{S}$   $\text{HgCl}_2$  is 77.5-8.5°. It decomposes on standing.

H. M. Leicester

The structure and configuration of certain diamminopalladium compounds. Frederick G. Mann, Dorothy Crowfoot, David C. Gattiker and Nora Wooster. *J. Chem. Soc.* 1935, 1612-52.—Besides the known yellow *trans*-( $\text{NH}_3$ ) $_2$  $\text{PdCl}_2$  (I), a red compd. (II) of this formula results when formation is slow. It differs from the cis isomer I and II give the same oxalate, darkening without melting at 175-85°, which is a cis compd. and with KI gives the cis diiodide, a brown powder. I and II also give a trans dinitrate, decomp. at 231-2°. Thus the change from cis to trans and reverse occurs very easily in these compds. X-ray evidence shows that II is *trans*, but a different cryst. modification from I *cis*-( $\text{NH}_3$ ) $_2$  $\text{Pd(NO}_2)_2$  is prep'd. from I in a large excess of  $\text{NaNO}_2$  and  $\text{NH}_4\text{OH}$ , and darkens without melting at 234°. It easily

changes to the trans form.  $[(\text{NH}_3)_2\text{Pd}][\text{Pd}(\text{NO}_2)_4]$  changes easily into the monomeric trans compd. *trans*-( $\text{NH}_3$ ) $_2$  $\text{PdI}_2$  is prepd. from either the cis or trans dichloride and KI. It is a yellow powder, which dissolves in  $\text{Me}_2\text{CO}$  and immediately reprecipitates in the form of reddish blue octahedra. The I atoms in this modification, which is also trans, are irregularly placed between the regular layers forming the rest of the crystal in a form of polysynthetic twinning which has been predicted but not previously observed. This structure is due to the extreme rapidity of growth of the crystals, which permits an entirely haphazard orientation of the diiodide mols relative to one another. H. M. Leicester

Compounds of cyclic diamines with metallic salts zinc salts R. Cernatescu and Marg. Poni *Ann. soc. chim. Jassy* 21, 394-406 (1935), cf. *C. A.* 29, 2104. Polyethylene diamine with Zn halides forms simple addn compds. contg. 1 mol. base and 1 mol. Zn salt. With naphthyl-1,4-diamine compds. are formed of the type  $2\text{R}(\text{NH}_2)_2 \cdot \text{ZnCl}_2$ . This is compared with the behavior of Cd, which forms compds. of the type  $2\text{Ca}(\text{NH}_2)_2 \cdot \text{Cd}(\text{NO}_3)_2$ . O. W. Wilcox

Complex calcium aluminates—action of organic salts J. I. Goret *IX Congr. intern. chim. pura applicada, Madrid* 3, 352-8 (1931), cf. *C. A.* 28, 577-8, 29, 2834. The prepn. of Ca acetate aluminate,  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{Ca}(\text{AcO})_2$ ,  $3\text{H}_2\text{O}$  and Ca propionate aluminate,  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ , and curves showing the equal behavior of lime and  $\text{Ca}(\text{AcO})_2$  and lime and  $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$  for the univariant systems in which the solid phases are the resp. double salts with (1)  $\text{CaO}$  and  $4\text{CaO} \cdot \text{Al}_2\text{O}_3$ , (2)  $\text{Al}_2\text{O}_3$  and  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$ , and (3)  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$  and  $4\text{CaO} \cdot \text{Al}_2\text{O}_3$ . Data on Ca formate aluminate were given in *XIII<sup>e</sup> Congr. de chimie industrielle*. L. R. Rushton

Alkali bromine salts and bromopyridine derivatives of rhodium. Pierre Poulenc *Ann. chim.* (11), 4, 567-657 (1935).—The action of alkali bromides on Rh bromide was studied and decided differences were found between the Br and Cl derivs. The following new compds. were prepd. and analyzed and described:  $\text{Na}(\text{RhBr}_4) \cdot 12\text{H}_2\text{O}$ ,  $\text{K}(\text{RhBr}_4) \cdot 6\text{H}_2\text{O}$ ,  $\text{K}_2(\text{Rh}_2\text{Br}_8) \cdot 6\text{H}_2\text{O}$ ,  $\text{K}_2(\text{Rh}_2\text{Br}_8) \cdot 6\text{H}_2\text{O}$ ,  $\text{K}_2(\text{Rh}_2\text{Br}_8) \cdot 6\text{H}_2\text{O}$ ,  $(\text{NH}_4)_2(\text{Rh}_2\text{Br}_8) \cdot 6\text{H}_2\text{O}$ ,  $\text{Cs}(\text{Rh}_2\text{Br}_8) \cdot 6\text{H}_2\text{O}$ ,  $\text{K}(\text{RhCl}_4 \cdot \text{Br}) \cdot 4\text{H}_2\text{O}$ ,  $\text{K}_2(\text{Rh}_2\text{Cl}_4 \cdot \text{Br}_2) \cdot 6\text{H}_2\text{O}$ . The action of pyridine on Rh bromide is extremely

complex, a large no. of different salts being formed. Combinations of Rh bromide with pyridine hydrobromide in media more or less acidified with HBr are described:  $\text{H}_2\text{Py}(\text{Rh}_2\text{Br}_8)$ ,  $\text{H}_2\text{Py}(\text{RhBr}_4) \cdot 3\text{H}_2\text{O}$ ,  $\text{H}_2\text{Py}(\text{Rh}_2\text{Br}_8) \cdot 9\text{H}_2\text{O}$ ,  $\text{H}_2\text{Py}(\text{RhBr}_4\text{Py})$ ,  $\text{HCsPy}(\text{RhBr}_4\text{Py})$ ,  $[(\text{NH}_4)_2\text{RhCl}](\text{RhBr}_4\text{Py})$ ,  $\text{H}_2\text{Py}(\text{RhBr}_4)$ ,  $\text{HCsPy}(\text{RhBr}_4)$ ,  $\text{Cs}(\text{RhBr}_4 \cdot \text{H}_2\text{O})$ ,  $[(\text{NH}_4)_2\text{RhCl}](\text{RhBr}_4 \cdot \text{H}_2\text{O})$ . A study was also made of the reaction products of increasing proportions of free pyridine on Rh bromide. The dipyrindine derivs.  $\text{M}_2(\text{Py}_2\text{RhBr}_4)$ , tripyridine derivs.  $(\text{Py}_3\text{RhBr}_4)$  and tetrapyrindine derivs.  $(\text{Py}_4\text{RhBr}_4)\text{Br}$  were prepd. and a study was made of their decompn. in aq. a. alc. and chloroform media as well as of the action of  $\text{AgNO}_3$  on  $(\text{Py}_3\text{RhBr}_4)\text{Br}$ . M. McMahon

A ferric ammonium carbonate Victor Auger and Marie Gallissot *Compt. rend.* 202, 2245 (1936).—By the action of a sol.  $\text{Fe}^{+++}$  salt, e. g.,  $\text{FeCl}_3$ , on a large excess of aq. soln. of  $\text{NH}_4\text{HCO}_3$  in presence of solid  $\text{NH}_4\text{HCO}_3$  and in an atm. of  $\text{CO}_2$ , a ppt. of microscopic yellow prisms is obtained, which can be washed with 1.5% soln. of  $\text{NH}_4\text{HCO}_3$  and dried over  $\text{H}_2\text{SO}_4$  in an atm. of  $\text{CO}_2$ . The compn. corresponds with  $\text{NH}_4\text{Fe}(\text{OH})_2\text{CO}_3 \cdot \text{H}_2\text{O}$ , or more probably  $[\text{Fe}(\text{OH})_2\text{NH}_4(\text{H}_2\text{O})]\text{HCO}_3 \cdot \text{KHCO}_3$  and  $\text{NaHCO}_3$  give clear liquids which soon deposit  $\text{Fe}(\text{OH})_3$ . C. A. Silberrad

The preparation of alkyl or phenylgermanium trihalides of the type  $\text{RGeX}_3$  and of methylenegermanium hexachloride. Arakel Telakirian and Michel Lewinsohn *Compt. rend.* 201, 835-7 (1935).—Alkyl and phenylgermanium trichlorides were prepd. by reaction of  $\text{Cs}(\text{GeCl}_4)$  (I) with alkyl or aryl halides (preferably iodides) in sealed tubes at elevated temp. in 60-80% yield. EtI and I heated 30 hrs at  $110^\circ$  gave  $\text{I}(\text{GeCl}_3)$  (60%), PhI and I heated 30 hrs at  $270^\circ$  formed  $\text{Ph}(\text{GeCl}_3)$  (80%),  $\text{CH}_3\text{I}$  and I gave  $\text{CH}_3(\text{GeCl}_3)$  (II),  $\text{Ph}_3\text{GeI}$  in 80% yield after 30 hrs at  $200^\circ$ . II was hydrolyzed by  $\text{NH}_4\text{OH}$  to germanomalonic acid,  $\text{CH}_2(\text{COOH})_2$ . The other trihalides reacted similarly, forming germanonic acids from which the trihalide could be regenerated by treatment with HCl. L. W. Scott

Straumanis, M. *Isa. revads kompleksu savienojumu teorija* [Theory of complex compounds], Riga. The Author. 62 pp.

7—ANALYTICAL CHEMISTRY

W. T. HALL

Quantitative spectral analysis studies IV R. Breckpot and A. Mevis *Ann. soc. Bruxelles* 55B, 261-92 (1935); cf. *C. A.* 29, 3623. —The most persistent rays and their apparently relative intensities, measured by the logarithmic sector and based upon the spectra of Cu, are given for Ti, V, Cr, Mn, Co, Ni, Mo and In. W. T. H.

Titrimetric colorimetry P. Karsten *Pharm. Weekblad* 72, 1327-39 (1935).—The principle of titrimetric colorimetry is applied to the following detns.:  $\text{Fe}^{+++}$  by  $\text{KSCN}$ ,  $\text{SCN}^-$  by  $\text{FeCl}_3$ , Bi by  $\text{KI} + \text{I}$ , Cu by  $\text{NH}_4\text{OH}$ ,  $\text{Cu}$  by  $\text{K}_2\text{Fe}(\text{CN})_6$ , Cu by  $\text{FeSCNHH}_4$ , Pb by  $\text{Na}_2\text{S}$ , Cu by  $\text{Na}_2\text{S}$ , Cr by  $\text{OC}(\text{NH}_2)\text{NH}_2$ , Mn as  $\text{HMnO}_3$ , phenolphthalein with 0.1 N NaOH, salicylic acid with  $\text{Fe}^{+++}$ , adrenaline with  $(\text{NH}_4)_2\text{MoO}_4$ , HCN as  $\text{HSCN}$  and Mn in powder. A. W. Doty

An analytic procedure for removal of phosphate, oxalate, fluoride, silicate and fluosilicate ions from group III. Selim Augusti. *Ann. chim. applicata* 25, 448-51 (1935). —The HCl soln. from the pptn. of group III is heated, and  $\text{NH}_4\text{OH}$  added until a permanent ppt. is formed. This is redissolved with just enough HCl, and excess  $\text{CO}_2$  is then added together with a slight excess of neutral  $\text{Pb}(\text{OAc})_2$  soln. This ppt. the ions  $\text{PO}_4^{3-}$ ,  $\text{C}_2\text{O}_4^{2-}$ ,  $\text{F}^-$ ,  $\text{SiO}_4^{4-}$  and  $\text{SiF}_6^{2-}$  as insol. lead salts. These are filtered off, the soln. is acidified with excess HCl and the excess  $\text{Pb}(\text{OAc})_2$  pptd. with  $\text{H}_2\text{SO}_4$ . A. W. Contiers

Stability of aqueous solutions of acid potassium phthalate. James I. Hoffman *J. Research Natl. Bur. Stand.*

*ards* 15, 583-4 (1935) (Research Paper No. 594). —Standard aq. solns. of acid K phthalate do not change in strength during 1 yr. under av. lab. conditions. The soln. was also unaffected by direct summer sunlight. L. R. Rushton

Preservation of standard solutions of oxalic acid and sodium oxalate II Saburo Ishimaru *Science Repts. Tohoku Imp. Univ.*, First Ser. 24, 411-25 (1935), cf. *C. A.* 17, 910. —The results of numerous expts. carried out with solns. tested at intervals during a period of 7 months show that the titer of 0.5 N oxalic acid or of 0.2 N  $\text{Na}_2\text{C}_2\text{O}_4$  remains const. provided the storage bottle is wrapped with black paper. With 0.01 N  $\text{H}_2\text{C}_2\text{O}_4$ , or solns. less than 0.2 N in  $\text{Na}_2\text{C}_2\text{O}_4$ , the solns. are stable in the wrapped reagent bottles provided they are 3-6 N in  $\text{H}_2\text{SO}_4$ . W. T. H.

Micro Dumas generation of carbon dioxide. Walter S. Ide *Ind. Eng. Chem., Anal. Ed.* 8, 56 (1936). — $\text{CO}_2$  for the microdetn. of N by an adaptation of the semimicro method of Berli and Burkhardt (*C. A.* 20, 2961) is generated from magnesium contained in the closed end of a microcombustion tube. Allen S. Smith

Volume extraction analysis Adolph Bolliger *Australian Chem. Inst. J. & Proc.* 2, 312-17 (1935). —A review of B's method of volumetric pptn. analysis in which the unpptd. remnants are extd. with an org. solvent immiscible with  $\text{H}_2\text{O}$ . Methylene blue is used for the detn. of certain o-polynitrophenols, especially picric acid and picrolonic acid and their compds., and some inorg.



New colorimetric determination of iron with the aid of pyrogallol. A. P. Palkin. *Zashchita Lab.* 4, 1106 (1935).—Prep the reagent by dissolving 5 g pyrogallol in 100 cc. of satd.  $\text{Na}_2\text{SO}_3$  soln. Acidify slightly a soln of Fe with pure  $\text{H}_2\text{SO}_4$  and dil to a point where a sample of the soln will give with the reagent approx the same intensity of coloration as the standard soln. Withdraw 5 cc of the soln, add 85 cc of  $\text{H}_2\text{O}$  and then the reagent dropwise to a const color, and compare with the standard soln. A standard soln contg 0.0025 g Fe in 1 cc is prepd. from 0.1244 g  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  as above and dil to 1 l. Metals capable of giving colored solns, such as Cu, Ni, Co and Cr, must be sepd. The sensitiveness of the method is 1.7  $\mu\text{g}$ . Chas Blane

Determination of small quantities of lead in tartaric acid. Sándor Ránky. *Magyar Árt. Lektor* 9, 427.9 (1935).—Dissolve 20 g tartaric acid in 50 cc distd water and add ammonia until any ppt formed dissolves. Neutralize to litmus with  $\text{AcOH}$  (litmus paper indicator). Add 2-3 drops of 50%  $\text{Na}_2\text{S}$  soln and mix with 30 cc glycerol. Compare the resulting brown color with standards. S S de Finály

Iodometric microanalysis of lead. M. N. Gapechenko. *Zashchita Lab.* 4, 1014-16 (1935). cf. Ivanov, C. A. 8, 2132—10 to 10 cc of a  $\text{Pb}(\text{NO}_3)_2$  soln add 5 cc of freshly prepd. 10%  $\text{NaHSO}_3$  and centrifuge for 10 min. Wash the  $\text{PbSO}_3$  ppt by decanting and centrifuging with  $\text{H}_2\text{O}$  until all  $\text{NaHSO}_3$  is removed. Dissolve the  $\text{PbSO}_3$  in 0.5-2 cc of 2 N  $\text{NaOH}$ , add 0.001 or 0.01 N I soln (depending on the amt of  $\text{PbSO}_3$ ), transfer the mixt into a flask, acidify with 20%  $\text{H}_2\text{SO}_4$  and titrate back the excess of I with  $\text{Na}_2\text{S}_2\text{O}_3$ . Calc Pb from the actual amt of I consumed in the oxidation of  $\text{SO}_3^{2-}$  to  $\text{SO}_4^{2-}$ . C B

Potentiometric titration of molybdenum with silver nitrate. P. Spacu. *Bul soc stinte Cluj* 8, 317-20 (1935).—Potentiometric titrations of  $\text{MoO}_4^{2-}$  have been described which were based on the pptn of either  $\text{PbMoO}_4$ ,  $\text{Hg}_2\text{MoO}_4$  or  $\text{BaMoO}_4$ . It is now shown that a similar titration can be carried out as an electrode of Ag against a calomel half cell with  $\text{AgNO}_3$  as reagent, provided sufficient I OH is added to reduce the soly of the  $\text{Ag}_2\text{MoO}_4$  formed. In titrating 5-cc portions of 0.4 N  $\text{Na}_2\text{MoO}_4$ , to which 50 cc of 15-50% alc was added, the results obtained were all within 1% of the truth. W T H

New volumetric method for the determination of nickel. G. Spacu and V. Armeanu. *Bul soc stinte Cluj* 8, 206-10 (1935).—The method resembles in principle the KCN method of Moore but the titrating KCN soln contains pyridine (Py). On adding this reagent to a Ni soln a light violet ppt. is formed according to the equation  $\text{Ni}^{++} + \text{Py} + \text{H}_2\text{O} + 2\text{CN}^- \rightarrow [\text{NiPy}_2\text{H}_2\text{O}](\text{CN})_2$  and the ppt dissolves in excess KCN to form  $[\text{Ni}(\text{CN})_4]^{2-}$ . As reagents, an approx. 0.1 N standard soln of  $\text{Ni}(\text{NO}_3)_2$  and a soln. contg 20 g KCN and 10 cc of pyridine per l are used. The titer of the latter changes slowly, it should be kept in a dark bottle and titrated against the Ni soln on the day the analysis is made. The titration can take place directly or indirectly. In the former case, take a measured vol of the KCN soln, add 1 cc. of pyridine and titrate with the neutral Ni soln until the above-mentioned ppt begins to dissolve slowly. Now add 100 cc of hot water, heat over the burner and after the soln has become clear, add more of the Ni soln dropwise until finally a faint ppt. is produced which does not dissolve in 2-3 min. Or, the unknown Ni soln can be titrated dropwise with the KCN soln until the soln becomes clear after 2-3 min. The KCN soln. must be standardized in exactly the same way. In the indirect method, add a few drops of pyridine to the soln Ni salt or to the concd and neutral soln. of the salt, and titrate in the cold, with continuous stirring, until an excess of KCN is present as is shown by the fact that the light violet ppt. has dissolved completely and the soln. has assumed a pale yellow tint. Now titrate back with the standard Ni soln. until a turbidity forms in the stirred soln. which does not dissolve after 2-3 min. The results obtained in titrating 25 portions of Ni soln. corresponding to 0.06 to 0.18 g. of Ni were all within 0.2 mg. of the truth. W. T. H.

Determination of tin in minerals and in metallurgical residues by means of 0.1 normal potassium bromate. Léopold Deutsch. *Ann. chim. anal. chim. appl.* 18, 10 (1936).—For detg Sn in minerals it is recommended to fuse the sample with  $\text{Na}_2\text{O}_2$ ; alloys should be dissolved by heating with  $\text{H}_2\text{SO}_4$  and  $\text{Na}_2\text{SO}_4$ . After reducing with 5 g. of *ferrous reductum* it is recommended to filter and use an aliquot for the Sn detn. The Sn is reduced to metal by adding approx 1 g of Al and the Sn is then dissolved in 60 ml of concd. HCl by heating. After the reduction the soln is cooled out of contact with air, by allowing  $\text{NaHCO}_3$  soln. to flow into the flask as necessary. The cold soln. is titrated with  $\text{KBrO}_3$  in a soln. contg.  $\text{ZnCl}_2$ ,  $\text{ZnI}_2$ , and starch as indicator. In alloy analysis, the Sn and Sb are titrated together in the above manner after the Sb has been detd. by itself with  $\text{KBrO}_3$  in the usual manner. W. T. Hall

Spectrophotometric determination of minute quantities of zinc in organic substances. J. Dabrowski and L. Marchlewski. *Biochem Z* 282, 387-91 (1935); *Bull. intern acad polonaise, Classe sci math nat.* 1935 A, 479-85.—Zn is pptd as the 8-hydroxy quinoline compd. This is decomposed by HCl and the 8-hydroxy quinoline-HCl is detd by its extinction quotient. The org. matter is ashed at 500° in a quartz dish, and the ash is warmed with 1 cc concd. HCl, dil with  $\text{H}_2\text{O}$  and filtered. The filtrate is dil to bring the HCl concn to about N. Bring to a boil and pass  $\text{H}_2\text{S}$  for 2 min to ppt any Cu. Filter again, wash with HCl satd. with  $\text{H}_2\text{S}$ , and evap to 1 cc. Add 0.25 cc. 20% citric acid, neutralize with  $\text{NH}_4\text{OH}$  to methyl orange, then add 0.25 cc of a special mixt. (20 cc.  $\text{HCOOH}$ , 3 cc  $\text{NH}_4\text{OH}$  and 20 g.  $(\text{NH}_4)_2\text{SO}_4$  made up to 100 cc) and 0.2 cc  $\text{HCOOH}$ . Warm to 40° and pass in  $\text{H}_2\text{S}$ , bring to a boil, then cool while passing  $\text{H}_2\text{S}$ . Collect the ppt, wash with  $\text{H}_2\text{O}$ , dissolve in a little 2 N HCl, evap partly and make alk with  $\text{NH}_4\text{OH}$  and remove the excess  $\text{NH}_4\text{OH}$  by warming. Dil the soln to 80 cc. and ppt the Zn as  $\text{Zn}(\text{C}_8\text{H}_7\text{NO})_2$  with 5 cc of the 8-hydroxy quinoline reagent (4 g 8-hydroxy quinoline and 8 cc  $\text{AcOH}$  in 100 cc) after first adding 2 cc 30%  $\text{AcOH}$  and 3 g  $\text{AcONa}$  and warming to 40°. Heat to 90° and leave overnight. Wash the ppt with  $\text{H}_2\text{O}$ , dissolve in 2 N HCl, make up to vol and exam in a spectrophotometer. From the detn. of  $\alpha$  the concn is detd from  $c = a/d$ . S Morgulis

Determination of zirconium. G. A. Ampt. *Australian Chem Inst J & Proc* 2, 321-31 (1935).—A mixt. of equal parts of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$  and  $\text{Na}_2\text{CO}_3$  is a good flux for all Zr materials. B interferes with the detn of Al, but it can be removed by successive evapns with  $\text{MeOH}$  and  $\text{H}_2\text{SO}_4$ . The fusion is dissolved in HCl and  $\text{SiO}_2$  is pptd. by fuming with  $\text{H}_2\text{SO}_4$ . The ppt. is treated with  $\text{HCl}$ , the residue is fused with  $\text{K}_2\text{S}_2\text{O}_8$ , dissolved in  $\text{H}_2\text{SO}_4$  and added to the filtrate. Ti and Fe are detd. colorimetrically in aliquots. Zr can be pptd. as the phosphate from acid soln, but Ti is also pptd. partially and must be detd in the ppt and filtrate. Ti, Fe and Zr can be pptd. with cupferron ignited and weighed and Zr calcd. by subtracting Ti and Fe previously detd. E. R. Rushton

The determination of small amounts of mercury by photometric titration. Shizo Hirano. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 648-7 (1935), cf. C. A. 30, 1326<sup>1</sup>.—A  $\text{Cu}_2\text{O}$  photoelectric cell was used with the previously described app. Gum arabic was added to the sample as protective colloid. Fxptl results are given for titration with 0.01 M and 0.001 M  $\text{Na}_2\text{S}$  soln. and good results were obtained in both cases. The presence of Cu and As had no effect. The determination of small amounts of lead by photometric titration. *Ibid.* 648-50.—The app. and method described above were used. The results reported are accurate. Pb detns in crude  $\text{H}_2\text{SO}_4$  and in flint glass are also reported. Karl Kammermeyer

Determination of mercury in mercuric cyanide. E. Cattelan. *J. pharm. chim.* 22, 454-6 (1935).—Introduce into a 100-cc 1 phenemey flask 10 cc.  $\text{Hg}(\text{CN})_2$  soln, 10 cc. distd.  $\text{H}_2\text{O}$  and 10 drops of 25% HCl. Heat until boiling starts and add, drop by drop, 2 cc. of 50%  $\text{Na}_2\text{S}_2\text{O}_3$  soln. Insert a funnel into the mouth of the flask and maintain a gentle boiling for 15 min. Finally add 2 cc. of 20%

$\text{Na}_2\text{SO}_4$  soln and boil for 15 min, shaking 4 or 5 times. Recover the ppt. in a tared 1 G crucible with a porous bottom of Jena glass, wash with hot distd.  $\text{H}_2\text{O}$ , then dry at  $100^\circ$  for 1 hr. The  $\text{Hg}_2\text{S}$  ppt. is too fine to be recovered on a 1 G crucible. S. W. Goldstein

Determination of bromine. G. E. Batrak *Mid. Exptl. (Ukraine)* No. 2, 109-4 (1935).—The method of Bernhardt and Uco (cf. C. A. 19, 2965) was modified by burning the mixt. on a sand bath, decolorizing with animal charcoal (in acidified medium) and subsequent filtering through an ordinary filter (instead of Hugo's filter), thus obtaining a clear soln and preventing loss of Br by eliminating the repeated burning. The sensitivity of Schiff's reagent was detd. not by the diffuse violet color but by the appearance of typical lilac rings with a pink tinge and a yellow layer on the top after placing carefully along the sides of the test tube a few drops of  $\text{Cl}_2$  water. In the absence of Br the rings are just yellow. After some training B could det. even 10% of Br (the original method was only sensitive up to 5%). S. A. Corson

Determination of active chlorine in chlorinated lime, Labarraque's and Dakin's solutions. Carl Staimer *J. pharm. Belg.* 17, 775-7 (1935).—The active Cl content is not only the Cl present as hypochlorite, but all the Cl liberated when the sample is treated with an acid. If enough chloride is present, the active Cl will be twice the amt. present as hypochlorite. S. W. Goldstein

Determination of iodine with iodides R. L. Ralgorodskaya and E. S. Binova *Farm. Zhur.* 1935, No. 1, 23-5.—Free I is titrated with  $\text{Na}_2\text{S}_2\text{O}_4$ , the iodides are then titrated with  $\text{AgNO}_3$  and the free I equiv. is subtracted from total iodides. L. Nasarevich

Determination of iodine in extracts from brine. I. Orlov and Tz. Kapanova *Khim. Farm. Prom.* 1935, No. 1, 44-6.—Iodine liberated with the nitrite from the acidified brine is extd. with activated charcoal, ground and extd. with  $\text{Na}_2\text{SO}_4$ , filtered, made up to the mark and titrated with the standard  $\text{As}_2\text{O}_3$  in the presence of  $(\text{NH}_4)_2\text{CO}_3$ , eosin as indicator. To det. I in the waste liquors it is oxidized to iodate with  $\text{Br}_2$ , excess Br removed with phenol and the I liberated from the added KI is titrated with 0.001 N  $\text{Na}_2\text{S}_2\text{O}_4$ . To det. the Cl the I is removed by boiling with  $\text{HNO}_3$  and then titrated according to Volhard. The acids in the crude I are extd. with petr. ether, shaken with standard alc.  $\text{NaOH}$ , and the excess is titrated back. L. Nasarevich

Determination of iodine in kelp. J. B. McKean *Analyst* 61, 11-13 (1936).—Dry 25 g. of the crude sample to const. wt. in a water oven. Grind the dry sample to pass through a 20-mesh sieve and dry again. Boil 20 g. with 200 ml. of water for 30 min.; filter and make up to 500 ml. Mix and take 50 ml. for the I detn. Dil. to about 400 ml., add 10 cc. of 20%  $\text{AcOH}$  and 1 ml. of  $\text{Br}_2$ . Boil until only a slight yellow color remains, cool and add about 0.5 g. of phenol dissolved in a little glacial  $\text{AcOH}$ . After 2 min. or more, add an excess of KI soln and titrate with 0.05 N  $\text{Na}_2\text{S}_2\text{O}_4$  soln. W. T. H.

Selenium as the catalyst in Kjeldahl digestions. K. Scharer *Z. Pflanzenenergie, Dungung Boden* 41, 203-7 (1935).—Powd. Se (pure red pptd. Se, Merck) and  $\text{CuO}$  were compared as catalysts for Kjeldahl digestions and the former proved the better. C. J. Schellenberger

New titrimetric method for the determination of nitrogen in ammonium salts and in foodstuffs. N. V. Shirokov and V. Volovinskaya *Z. Unterzuch. Lebensmittel* 70, 249-4 (1935).—The principle for the detn. of  $\text{NH}_3$  in  $\text{NH}_4$  salts depends on the treatment of a soln with  $\text{Na}_2\text{CO}_3$  in the presence of a Ag salt. The  $\text{NH}_3$  set free by the  $\text{Na}_2\text{CO}_3$  combines with the Ag to form the complex cation  $\text{Ag}(\text{NH}_3)_2^+$ , while the excess Ag is filtered from the soln as  $\text{Ag}_2\text{CO}_3$ . The Ag in the complex is detd. by the Volhard titration or alim. The  $\text{NH}_3$  is then calcd. from the formula of the Ag complex. In the estn. of total N in foodstuffs, the Kjeldahl method is first employed, with  $\text{H}_2\text{SO}_4$ ,  $\text{CuSO}_4$ , and  $\text{K}_2\text{SO}_4$ . After the digestion, a soln of  $\text{NaOH}$  is added until a permanent ppt. of  $\text{Cu}(\text{OH})_2$  remains. This soln is then filtered, made up to the mark and an aliquot taken for the detn. of  $\text{NH}_3$  as described above for  $\text{NH}_4$  salts.

The results obtained by these methods show no variations which are of practical significance. P. L. Dunlap

Determination of moisture in sulfidic copper ores. L. M. Tol'son and P. O. Ferstater. *Zarodskaya Lab.* 4, 1031-8 (1935).—The nature of various forms of moisture contents in S-bearing Cu ores and methods of their detn. were studied with a view of developing better methods of lab. control of ore treatment. Sulfidic Cu ores contain 4 forms of moisture: water adsorbed on the surface, mechanically occluded (intercryst.) water, water of crystn. ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , etc.), and water of constitution ( $\text{CuCO}_3$ ,  $\text{Cu}(\text{OH})_2$ ,  $\text{CuCl}_2 \cdot 3\text{Cu}(\text{OH})_2$ ,  $\text{CuSO}_4 \cdot 2\text{Ca}(\text{OH})_2$ , etc.). The detns. are made with 5 g. samples (not less) powdered to 120 mesh. Det. adsorbed water by the difference in wts. of a sample dried in a vacuum desiccator over  $\text{CaCl}_2$  for 24 hrs. and following exposure to air for 2 hrs. Det. the intercryst. water by deducting the value of adsorbed water from the detn. of the sum of the 2 forms of water by the  $\text{CaCl}_2$  method (cf. C. A. 29, 7854). Det. the water of crystn. by the increment in wt. obtained by drying a sample in an elec. oven at  $250^\circ$  for 2 hrs. and following exposure to water vapor in a desiccator for 3 hrs. and drying over  $\text{CaCl}_2$  for 24 hrs. All forms of moisture, except that of intercryst. water, are const. for the ores of the same origin, the detn. of which is required only in the complete analysis of the ore. The results of detn. of intercryst. water by the usual methods of drying are misleading because of the partial loss of water of crystn. Intercryst. water can be detd. with an accuracy to 0.03% for air-dry ore and 0.2% for damp ore by drying a 5-10 g. sample in an elec. oven at  $70^\circ$  for 3 hrs. or in a vacuum desiccator at  $60^\circ$ . The intercryst. water is rapidly evapd. in the air, hence the detns. of moisture contents in prep. lab. samples are not representative of the ore in the bulk. The true contents of intercryst. moisture can only be detd. at the supply base by drying several kg. of crushed (not powd.) sample at  $70^\circ$ . Chas. Blanc

Determination of aluminum oxide in aluminum and its alloys. S. I. Sukkov and B. M. Korotetskaya. *Zarodskaya Lab.* 4, 1104 (1935).—The detn. depends on the soly. of metals and usually of  $\text{Al}_2\text{O}_3$  in  $\text{CuCl}_2 + \text{KCl}$  in  $\text{H}_2\text{O}$  and on the soly. of ignited  $\text{Fe}_2\text{O}_3$  and usually of  $\text{Al}_2\text{O}_3$  in  $\text{HCl} + \text{HNO}_3$ . Treat a 3 g. sample with 100 cc. of the reagent (1 mole of  $\text{CuCl}_2$  and 2 moles of  $\text{KCl}$  in 1 l.  $\text{H}_2\text{O}$ ) first in the cold and then on a hot plate until all metals are dissolved. Dil. the soln with hot water and filter through a triple filter. Wash the ppt. with hot 20%  $\text{HNO}_3$  and then with water, and ignite in a Pt crucible. Evap. the  $\text{SiO}_2$  in the residue with  $\text{HF}$  as usual, digest the residue with concd.  $\text{HCl}$  on a hot plate, transfer the mixt. into a beaker, treat it with aqua regia, dil., filter, ignite and weigh  $\text{Al}_2\text{O}_3$ . The accuracy of the detn. is equal to that obtained by the method of alk. fusion. Chas. Blanc

Determination of gallium in aluminum. J. A. Scherrer. *J. Research Natl. Bur. Standards* 15, 585-9 (1935) (Research Paper No. 833).—Ga is present in most bauxites and is, therefore, in most samples of Al. wares. In analysis, it can be sep'd. from Al by taking advantage of the fact that Ga can be pptd. by cupferron in solns which are 0.6 N in  $\text{H}_2\text{SO}_4$ , provided an excess of 15-20 ml. of 6% cupferron soln is added. In the analysis of a sample of Al weighing 10 g. and contg. 0.01% Ga, a vol. of 450 ml. is sufficient to prevent contamination with Al. The ignited cupferron ppt. should be dissolved in 1.2 N  $\text{HCl}$  and exam'd. for Sn, Cu, V, Ti and Zr which will likewise be pptd. by cupferron. After detg. the wts. of these 5 elements, the Ga can be obtained by difference. If, on the other hand, Ga alone is to be detd., it is better to make use of the fact that  $\text{GaCl}_3$ , like  $\text{FeCl}_3$ , can be extd. by ether from a soln in 6 N  $\text{HCl}$ . The vol. of the acid soln. should be about 400 ml. prior to the extn. and 3 extns. with 100 ml. of ether are necessary and sufficient. From the ether ext. the Ga is obtained eventually after pptn. with cupferron. Analysis of numerous samples of Al showed values ranging from 0.0005 to 0.016% Ga and the 2 methods of analysis gave uniform results. The Ga in the small ppts. was identified spectrochemically and found to be practically pure. W. T. H.

Volumetric determination of nickel in steels. A. L. Vorob'ev. *Zavodskaya Lab* 4, 1105 (1935).— $\text{Na}_2\text{P}_2\text{O}_7$  for the titration of Ni by the method of Gel'band (C A 29, 75<sup>2</sup>) is prep'd by dehydrating  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$  at 200–50° and heating the residue in an elec oven at 600° for 1 hr.

Standard methods of chemical analysis of manganese bronze. *Am Soc Testing Materials, A S T M Designation* B27–35, *Am Standards Assoc, A S A No. K3–1935*, 11 pp.

Testing for sea-water damage. W. M. Deaber. *Analyst* 61, 14–22 (1936).—Samples of galvanized ware, Cu-coated wire, bristles, sugar, corn meal, cacao beans, gum arabic, Gruyère cheese, prunes, dates, walnuts, sheep's wool and carpets have been exam'd. A sample of wool damaged by sea water showed a reduction of the  $\text{K}_2\text{O}$  from 2–3 to 0.16% and an increase of  $\text{Cl}^-$  from 0.02–0.3 to over 1%, showing that K salts had been washed out and NaCl absorbed. In the search for a test which would be more sp than the test for  $\text{Cl}^-$ , the following method for Br was devised. Soak the suspected sample for some time to remove sol halides. To the aq ext add 0.5 g of  $\text{Ca}(\text{OH})_2$  and evap to dryness. Treat the residue with 2 ml of water, add 2 ml of a soln prep'd by dissolving 150 g of  $\text{CrO}_3$  in 80 ml of water and pass a stream of  $\text{CO}_2$  through the soln to expel liberated Br, and cause the vapors to come in contact with filter paper which has been impregnated with 0.2% soln of fluorescein in alc. If Br is present in the vapors, red eosin is formed. The quantity of Br present can be est'd by comparison with standard stains but it is best, after an orienting expt., to base the estn on a stain produced by about 1.7 g of Br. Since sea water contains 3–3.4% of NaCl and the ratio of  $\text{Cl}^-$  to Br is approx 2:1 it is possible to prove contamination with sea water if the aq ext from damaged goods shows about this ratio.

Analyzing the "carbon" deposits in tractor engines. I. G. Semendo and B. B. Kroll. *Neftekhim Kholozhilo* 29, No. 10, 75–76 (1935).—1- $\frac{1}{2}$  lb sample with a suitable solvent for grease, etc., under a reflux condenser, weigh the residue and subtract the wt. of its ash.

Standard methods of routine analysis of dry red lead. *Am Soc Testing Materials, A S T M Designation* D19–35; *Am Standards Assoc, A S A No. K16–1–1935*, 5 pp.

Determination of fumigants. II. An improved vacuum apparatus for the measurement of gas concentrations. A. B. P. Page. *J Soc Chem Ind* 54, 421–427 (1935); cf. C A 27, 792<sup>3</sup>. III. Microdetermination of ethylene oxide and hydrogen cyanide. O. F. Lubatti. *Ind* 421–6T, cf. C A 27, 245<sup>3</sup>.—The methods described are suitable for use with Page's improved vacuum app for measurement of gas concn (cf. above). Absorption of ethylene oxide in a 0.025 to 0.1 N  $\text{H}_2\text{SO}_4$  soln contg. 49–53 g/100 cc.  $\text{MgH}_2$  gives 100% recovery. The excess acid is titrated with 0.025 N NaOH. The cyanide is absorbed in 0.2 N NaOH soln and titrated iodometrically. Details of technic are given. P. S. R.

Rapid detection of ozone. M. Shlesinger-Konstantinova. *Sotzialist Rekonstruktsiya i Nauka* 1935, No. 6, 167.—The intensity of soln luminescence of a specially selected substance after having reacted with  $\text{O}_3$  is proportional to its quantity. Notwithstanding the fact that  $\text{O}_3$  itself does not fluoresce, its quantity is det'd. by the quantity of the fluorescing substance formed from it and the special substance by a photometric method of visual estimation.

Determination of the total oxidizing power, of nitrite, of ozone and of the total chlorine content of fresh and of poisoned air. II. H. Cauver. *Z anal Chem* 103, 385–416 (1935), cf. C A 30, 1324<sup>3</sup>.—The nitrite is det'd in the same way as the total oxidizing power, by its action on KI in the presence of dil.  $\text{H}_2\text{SO}_4$ , after the removal of ozone by heating the air to 200°. The introduction of the necessary furnace causes more complication in the app, with resulting loss in accuracy, so that in biol. and hygienic expts it is usually best to rely upon the value obtained for the total oxidizing power.  $\text{O}_3$  is det'd. by the loss of I

caused by passing the air through a neutral KI soln.; this method is useless in the presence of  $\text{Cl}_2$ . The total  $\text{Cl}_2$  content, or all substances that form water-sol. chlorides, is det'd. by passing a measured vol. of the air through KOH soln. and eventually detg the  $\text{Cl}^-$  content of the KOH soln. by titration with  $\text{AgNO}_3$  according to either Mohr or Volhard. Since about  $\frac{1}{4}$  of the free  $\text{Cl}_2$  forms  $\text{ClO}_2^-$  in the absorbent, this method does not give accurate results in the analysis of  $\text{Cl}_2$  gas.

Colorimetric determination of nitric acid and of nitrates. R. Cernitescu and F. Gheller. *Z anal Chem* 101, 402–6 (1935).—Buznea and Cernitescu (C A 22, 1297<sup>2</sup>) proposed a sensitive colorimetric test for nitrate which depends upon the red coloration produced by the reaction with a soln. of *m*-diaminophenol in conc'd  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$ . This test can be used as a basis for the colorimetric detn of small quantities of nitrate. 1st test to make sure that no interfering substances are present, if they are found, they must be removed by a suitable method, e. g., halide with  $\text{Ag}_2\text{SO}_4$ . Make an orienting expt. to find the approx quantity present and then take a suitable amt of the sample for the colorimetric comparison.

Determination of hydrobromic acid in hydrochloric acid. 1. Chelle. *Bull. soc. pharm. Bordeaux* 73, 188–90 (1935).—0.005 mg HBr can be detected in 10 cc.  $\text{HCl}$  by using the Demégné-Chelle reaction (C A 7, 740, 2231).

Determination of butyric acid in commercial acetic acid. Ladislav Klinc. *Ann. chim. anal. chim. appl.* 18, 6–9 (1936), cf. C A 29, 705<sup>2</sup>, 7574<sup>2</sup>.—Butyric acid is oxidized by  $\text{H}_2\text{O}_2$  and  $\text{AcMe}$  is formed. Scott-Wilson's reagent (10 g.  $\text{HgCN}$  in 600 cc. of water added to an equal vol. of 30% NaOH soln. and the next added slowly to 100 cc. of 0.725%  $\text{AgNO}_3$  soln.) will detect  $\text{AcMe}$  equiv. to as little as 0.01 mg. of butyric acid. By applying this test to numerous samples of  $\text{AcOH}$  the conclusions drawn are: (1) Synthetic  $\text{AcOH}$  contains no butyric acid. (2) Pyroigneous acid can be obtained pure but most samples contain some butyric acid, sometimes as much as 9–10%. (3)  $\text{AcOH}$  obtained by fermentation is likely to contain 0.25–0.28% butyric acid.

Electrometric titration of carbonic acid and of sodium bicarbonate in dilute solutions. Holvard Llander. *Inv* 1935, 75–83.—The  $pH$  values of solns. of  $\text{CO}_2$  and of  $\text{NaHCO}_3$  were det'd. for various dilns by means of a glass electrode against a standard calomel cell and the curves det'd. for the titration of such solns. with NaOH or with  $\text{Na}_2\text{CO}_3$ . The results appear to agree satisfactorily with values expected from the ionization consts. of  $\text{H}_2\text{CO}_3$ .

Field method of determining phosphoric acid in phosphonates. M. I. Zagorskii. *Zavodskaya Lab* 4, 1039–42 (1935).—The colorimetric detn gives accurate results without the use of heat. Add to 5 g. of powd. sample 50 cc. of 5%  $\text{HCl}$  and shake in a Wagner app. for 30 min. Dil. the filtrate to 250 cc., withdraw 5 cc. of the soln. and dil. to 240 cc. To 5 cc. of this soln. add 5 cc. of the soln. (25 g. of  $\text{NH}_4$  molybdate in 100 cc.  $\text{H}_2\text{O}$  mixed with 75 cc.  $\text{H}_2\text{SO}_4$  in 125 cc.  $\text{H}_2\text{O}$ ) and 5 cc. of the soln. (5 g. hydroquinone in 250 cc.  $\text{H}_2\text{O}$  mixed with 0.25 cc. of conc'd.  $\text{H}_2\text{SO}_4$ ). After 5 min. of shaking, add 25 cc. of 20%  $\text{AcONa}$  (cryst.) soln. dil. the soln. to 250 cc., and compare with the standard solns. prep'd by treating  $\text{KH}_2\text{PO}_4$  as above. A portable set for field analysis of phosphonates is illustrated and described.

The determination of sodium chloride in salt. C. W. Foulk and John R. Caldwell. *J. Am. Water Works Assoc.* 27, 1712–16 (1935).—The procedure consists in pptn. of about 94% NaCl by conc'd.  $\text{HCl}$  and then the balance, after excess acid has been removed, with a  $\text{Mg}$  uranyl acetate reagent—as Na  $\text{Mg}$  uranyl acetate. Full details of operation are given and from the results shown the method is quite accurate.

Spectrophotometric determination of potassium chloride in sylvinite. R. Romann and C. Spatel. *Bull. soc. chim.* [5] 2, 2168–70 (1935).—The common ion effect of KCl on the soly. of a less sol. colored salt of K, such as the picrate,

is followed in the instrument. This method agrees within 0.2% with the perchlorate method. Gregg M. Evans

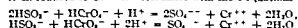
Potentiometric determination of metavanadate with silver nitrate. P. Spacu. *Z. anal. Chem.* 103, 422-4 (1935).—The titration of 0.1 N  $\text{NH}_4\text{VO}_3$  with 0.2 N  $\text{AgNO}_3$  is described. The soln to be titrated contained 20% of LiOH in a total vol of approx 90 ml. As indicator electrode a wire of Ag was used against a normal calomel cell. The ppt formed is orange-yellow  $\text{AgVO}_3$ .

W. T. H

Microalkalimetric studies. III. Microdetermination of ammonium. J. Mika. *Rev. Hung. Pakist. Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met.* 7, 92-110 (1935). cf. C A 29, 5773.—Details for carrying out a micro distn are given. S. S. de la Peña

Determination of chlorate and perchlorate in the presence of considerable nitrate. F. S. Tomula. *Z. anal. Chem.* 103, 427-30 (1935).—Dissolve about 10 g. of conc. alkali nitrate in a little water, add 3-5 ml of 40%  $\text{HCHO}$  soln, 1 ml of 0.5 N  $\text{FeCl}_3$  soln and 2 cc of 2 N  $\text{HNO}_3$ . Allow to stand on the water bath for 0.5 hr., add 20 ml of concd  $\text{HCl}$  and evaporate to dryness. Dissolve the residue in a little water, evaporate again with the same vol of  $\text{HCl}$  and repeat twice more. Finally treat the residue with 50 ml of concd  $\text{HCl}$ , filter through sintered glass and wash with a little concd  $\text{HCl}$ . Evaporate the filtrate to dryness. It will weigh about 0.5 g. in the analysis of  $\text{NaNO}_3$  samples. Dissolve the residue containing chloride and unchanged perchlorate in a little water, make up to exactly 100 ml., mix and determine the  $\text{Cl}^-$  content in an aliquot part of the soln by the argentometric method of Volhard. Then, in the remainder of the soln add a corresponding quantity of  $\text{AgNO}_3$  and filter off the  $\text{AgCl}$  ppt. Evaporate the filtrate to about 40 ml and determine perchlorate by the method of Tomula and Pelkonen. C A 28, 3027.—In another portion, reduce the chlorate with Zn and dil.  $\text{H}_2\text{SO}_4$ , determine the  $\text{Cl}^-$  by the Volhard method and correct for the original  $\text{Cl}^-$  content. W. T. H

Potentiometric titrations of sulfite and sulfoxylate alone and in the presence of hyposulfite. J. Lobering. *Z. anal. Chem.* 101, 392-8 (1935).—Chromic acid in the presence of dil. acid oxidizes sulfite and sulfoxylate to sulfate and is itself reduced to  $\text{Cr}^{++}$  in accordance with these equations:



The end point is best determined potentiometrically with a Pt gauze electrode balanced against a calomel cell. The soln should contain 1-2% of  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$ . It is possible to oxidize both sulfite and sulfoxylate in dil. acid soln without oxidizing any hyposulfite, but the temp must be kept below 40° to prevent decomposition of hyposulfite. Frisch Muller. *Ibid.* 340. A. Mutschkin. *Ibid.* 342. S. Polheim. W. T. H

Detection of ferrocyanide in the presence of ferrocyanide. I. M. Korenman. *Z. anal. Chem.* 101, 417-19 (1935).—Indigo carmine in  $\text{Na}_2\text{CO}_3$  soln is oxidized by ferrocyanide to form pale yellow disulfo-dihydroindigo. The reaction is very sensitive. To 3 cc of the soln to be tested and to 3 cc of pure water in another vessel, add 10 drops of satd.  $\text{Na}_2\text{CO}_3$  soln. Now if 2-3 drops of 0.02% indigo carmine soln is added to the pure water sample, the liquid assumes a blue tint. The same vol of indigo carmine soln added to the sample containing ferrocyanide will soon lose its color. As little as 3-5 of  $\text{K}_4\text{Fe}(\text{CN})_6$  can be detected in 3 cc of soln. W. T. H

Microchemical determination of some cations with the aid of hexamitrophenylamine. O. G. Shelintz. *Zarodskaya Lab.* 4, 1047-52 (1935).—The Na salt of  $(\text{O}_2\text{N})_6\text{C}_6\text{H}_2\text{N}_6$  ( $\text{H}_6\text{NCH}_6(\text{NO}_2)_6$ ) reacts with K, Ca, Sb, Ti, Be, Zr, Pb and  $\text{Hg}^{++}$ , giving characteristically shaped and colored crystals. K can be detected in the presence of Na, Li, Mg, Ba, Sr and Ca.  $\text{NH}_4^+$  gives a similar crystal ppt and therefore must be removed. Al, Fe, Cr, Ni, Co, Cu, Bi, V, Th, and  $\text{Hg}^+$  form amorphous ppts. Other cations do not affect the reaction. Photomicrographs and description of various crystals are given. Chas. Blanc

1. Rapid determination of sulfate and the sum of iron and aluminum ions in commercial hydrochloric acid. V. K. Gontarik. *Zarodskaya Lab.* 4, 1043-6 (1935).—The Ind. son method of detn. of sulfate in conc.  $\text{HCl}$  was modified by evapng.  $\text{HCl}$  to dryness and titrating the residue in  $\text{H}_2\text{O}$  with 0.1 N  $\text{Ba}(\text{OH})_2$  in the presence of phenolphthalein as indicator to a pink and thereafter to a point when a small fraction of the soln after filtration gives no turbidity on the addn of  $\text{BaCl}_2$ . By this procedure all sulfates, except  $\text{Al}_2(\text{SO}_4)_3$  and  $\text{Fe}_2(\text{SO}_4)_3$ , can be detd with accurate results. The latter are detd by the difference between the titration results in the presence of phenolphthalein and methyl orange as indicators. Similar results, with a considerable saving in time in evapn of  $\text{HCl}$ , can be obtained by microanalysis based on the same procedure. Chas. Blanc

Analysis of methylene blue. K. D. Shestrebacher. *Khim. Farm. Prom.* 1935, No. 2, 117-18.—A small sample of the dye is treated with excess of standard  $\text{SnCl}_2$ , heated and titrated in  $\text{CO}_2$  with standard  $\text{K}_2\text{Cr}_2\text{O}_7$ . The appearance of permanent blue indicates the end point. L. N.

Spectrophotometric determination of chlorophyll a chlorophyll b, carotene and xanthophyll. A. Sprecher von Bernegg, E. Heierle and F. Almasi. *Biochem. Z.* 233, 45-52 (1935).—A method is described for detg. all 4 pigments in a small leaf with an accuracy of 5%. S. M.

4. Principle of formaldehyde, alcohol and acetone titations. George M. Richardson. *Proc. Roy. Soc. (London)* B119, 65-6 (1936). cf. C A 28, 5007.—In titrations of this type, the initial point and the end point must be defined clearly in statements of analytical results. Joseph S. Hepburn

Analysis of formaldehyde. I. Determination of formaldehyde. A. Mantegazza. *Materie plastiche* 2, 54-6 (1935). II. Determination of methanol in formaldehyde. *Ibid.* 117-22.—A complete review of the chem. methods of analysis, together with the more rapid gravimetric and phys.-chem. methods (detn. of d or n) for the analysis of mixts of  $\text{MeOH}$  and  $\text{HCHO}$ . G. A. Brown

Acid base titrations in alcohol-water mixtures. III. Titration of alkaloids and alkaloid salts. H. Baeggaard-Rasmussen and F. Reimers. *Dansk. Tidskr. Farm.* 9, 3-8 (1935) (summary in English). cf. C A 28, 5367.—The  $p_n$  values of dil.  $\text{HCl}$  and  $\text{NaOH}$  in alc. solns, detd with a H electrode at 23°, were in 50% alc. 1.32 and 2.20 vs. 0.1 and 0.01 N  $\text{HCl}$ , resp., 13.5 and 13.07 for 0.1 and 0.01 N  $\text{NaOH}$ , resp., and in 75% alc. 1.58 and 2.74 vs. 0.1 and 0.01 N  $\text{HCl}$ , resp., 14.3 and 13.70 for 0.1 and 0.01 N  $\text{NaOH}$ , resp. The end points of the reaction scale, which according to Bjerrum (C A 26, 3716), are the max and the min  $p_n$  obtainable in dil. solns (max.  $p_n$  were approx. 0.3 and 15 in 50% alc. and approx. 0.3 and 15 in 75% alc.)

7. The acidity constants of the alc. solns of the alkali metal investigated were detd, partly colorimetrically and partly electrometrically. In addn to the previously described buffers, mixts of solns of cocaine and cocaine  $\text{HCl}$  were also used, their  $p_n$  values were detd with H and quinhydrone electrodes. The value of  $p_n - (p_n/0.072)$  at 23° of cells of the type Pt | quinhydrone, alc. soln | aq.  $\text{KCl}$  | aq. N  $\text{KCl}$ ,  $\text{H}_2\text{Cl}_2$ ,  $\text{Hg}$ , detd. by measurement against buffer solns of known  $p_n$  was 7.41 ± 0.02 in 50% alc. and 7.58 ± 0.02 in 75% alc. (cf. Conant and Fisher C A 14, 3866; Michaelis and Mizutani, C A 19, 255). 20, 561, 2108). In the majority of cases  $p_n$  was detd in mixts of solns of alkaloid base and alkaloid salt containing sufficient  $\text{KCl}$  to make the salt concn 0.05 N. From the results obtained  $p_n$  is calculated from the equation  $p_n = p_n - \log (\text{Calculated base}/\text{Calculated salt})$ . The variation of  $p_n$  with the alc. concn for various alkaloids is shown in a diagram in comparison with the variations of  $p_n$  for the most important indicators used in the titration of the alkaloids and their salts. From an approx. detn. of the limits of error in the titration of a basic alkaloid (i.e., the distance of the  $p_n$  of the titration end point to the nearest end point of the reaction scale and to the  $p_n$  of the alkali), it can be told which alkaloid can be titrated in alc.-w. or mixts and which indicator should be used (cf. Linderstrom-Lang, C A 22, 4726). Solns. contg. 50% alc. are

preferable because the accuracy of the titration decreases with increasing concns. of alc. The majority of the alkalooids could be titrated satisfactorily in 50% alc with bromophenol blue as indicator. In many cases it is advantageous to dissolve the alkalooid in alc, add methyl red and titrate to the color change which in alc occurs too soon, then the soln may be dild with water without risk of pptn and the titration finished in aq soln where methyl red in most cases is very suitable. The changes in the  $pH$  of the alkalooids and the position of the basic end point of the reaction scale in alc-water mixts as compared with that for water, render the titration of alkalooid salts with a base more accurate in alc-water media than in pure water, the accuracy increasing with the concn of alc. It is shown that most of the alkalooids studied can be titrated in 75% alc with phenolphthalein, or better with Porriar blue (cf. Ol-en and Reimers, *C A* 26, 5848) as indicator. The following alkalooids were used in the investigation: cocaine, morphine, narcotine, papaverine, quinine, cincholine, strychnine, brucine, atropine, procaine, metine, ephedrine and cocaine. D Thuesen

Estimation of acetone in methyl and ethyl alcohols S D. Sumwalt and M C T Katti *J Indian Inst Sci* 18A, 115-22(1935).—Messinger's method for detg AcMe depends upon treating the sample with NaOH and  $I_2$ , which causes the AcMe to be oxidized to AcONa and CHI<sub>3</sub>. After the reaction is over, the soln is made acid and the excess  $I_2$  titrated with  $Na_2S_2O_3$ . This method gives accurate results for detg AcMe in aq solns even when as little as 0.5 mg of AcMe is present in 100 cc. In the presence of MeOH Messinger's method gives results which are erratic and correspond to values which are too high. The reason for this is that MeOH is oxidized by NaIO and the principal product is HCO<sub>2</sub>H. If it is assumed that the rate of oxidation of MeOH is proportional to the concn of NaIO at any time, a logarithmic equation can be derived for the total quantity oxidized and the calcd. results then agree with those obtained experimentally. Other methods for detg AcMe were tested and that of Scott-Wilson, as modified by Marriott is the most suitable. By this method it is possible to detect 0.1 mg of AcMe in 100 cc. of soln even when EtOH and MeOH are present. The method should not be used, however, for pptg. more than 8 mg of AcMe at a time and in the titration, 1 cc. of N NII.CNS corresponds to 0.12 mg of AcMe rather than the theoretical value of 0.80 mg. The ppt. must be allowed to stand at least 20 min., and 8-9 times the theoretical amt. of reagent must be used.

W T H

Grouping of organic solvents and organic compounds with Magdala red. Hermann Eichler *Z anal Chem* 103, 425-7(1935).—Magdala red has a yellowish red color in solns of  $pH$  2 or lower but becomes red at  $pH$  4 and fluorescent. In basic solns there is no fluorescence. With respect to their behavior toward this dyestuff, org compds can be classified as follows: (1) Compds which dissolve Magdala red with fluorescence at room temp and which in their original condition reddens gray Magdala red paper and color it throughout. To this class belong alcs, lower ketones, fatty acids, aldehydes, Turkey-red oil and phenols; nitrobenzene dissolves the dyestuff with very weak fluorescence. By the addn of compds. of group 3, the fluorescence usually disappears. Aq solns of the above substances do not as a rule give the Magdala red reaction. (2) Substances which do not dissolve Magdala red at ordinary or higher temps and do not act upon Magdala red paper. Hydrocarbons of the aliphatic and aromatic series, their halogen derivs, fats, C<sub>6</sub>H<sub>6</sub>, ethers, fatty and ethereal oils belong to this class. Mixts of these substances with sufficient EtOH dissolve Magdala red with red fluorescence, if the substance does not mix with EtOH, only the alc. layer is colored. (3) Compds which dissolve Magdala red without fluorescence and color Magdala red paper red. Upon the addn. of a suitable quantity of AcOH or of concd. HClO<sub>2</sub>, fluorescence results as soon as the  $pH$  is lowered to about 4. Besides these classes, there are solns, in which Magdala red dissolves only at an elevated temp. with fluorescence; these

are water, aq solns. of fatty acids and dil. mineral acids of  $pH$  about 4. W. T. H.

Sulfonation of naphthalene. I Determination of the relative proportions of  $\alpha$ - and  $\beta$ -naphthalenesulfonic acids. Robert Lantz *Bull. soc chim* [5] 2, 1913-32(1935); cf. *C A* 29, 6593<sup>7</sup>.—A procedure for the detn. of the relative amts of  $\alpha$ - and  $\beta$ -naphthalenesulfonic acids (I and II) has been worked out for use in a future publication on the monosulfonation of naphthalene. Under definite conditions, I is desulfonated and pptd quantitatively as an insol polybromonaphthalene leaving II entirely in soln. as a sol polybromonaphthalenesulfonic acid. The amt. of I in a mixt of I and II can be detd either by estg. the H<sub>2</sub>SO<sub>4</sub> resulting from the elimination of the HSO<sub>3</sub> group or better by detg the insol bromonaphthalene by CrO<sub>3</sub> oxidation. I from the results of detailed investigation a method of carrying out the bromination either in the presence or absence of H<sub>2</sub>SO<sub>4</sub> has been formulated. About 0.17 g. of the Na salts of I and II in powder or in H<sub>2</sub>SO<sub>4</sub> soln. is sealed into a bomb tube with stated amts. of 65% HBr and Br. The tube is heated in boiling H<sub>2</sub>O for 3-5-6 hrs. according to the method adopted. The cooled brominated product is taken up in 100 cc H<sub>2</sub>O and, after boiling off the excess Br, is filtered. The washed residue is taken up in 50 cc concd H<sub>2</sub>SO<sub>4</sub> and the detn is completed by oxidation with concd CrO<sub>3</sub> soln (*C A* 26, 3691). The results are calcd on the basis of a bromonaphthalene contg 4 atoms of Br (if H<sub>2</sub>SO<sub>4</sub> is not used) or 4.5 (with H<sub>2</sub>SO<sub>4</sub>). It has been shown that naphthalene brominates more slowly than I under the given conditions and that the above procedure can be used for mixts contg a small amt. of naphthalene which should be calcd as I in the estn.

C R Addinall

Analysis of commercial xanthates C A Scherbakova *Zarodskaya Lab* 4, 1053-61(1935).—The simplified method of analysis of xanthate flotation reagents depends on the difference of the results obtained by iodometric titration of aq soln of xanthate together with the impurities (thiosulfates, sulfides and thiocarbonates) and that of the impurities alone by a preliminary decompn of the xanthate with HCl. The products of xanthate decompn (CS<sub>2</sub> and the alcs), incapable of reacting with  $I_2$ , do not affect the titration of impurities. Considerable advantages of the aq iodometric detn. are claimed over the 2 of the best known methods of acidimetric and alc. iodometric detns. For the analysis of xanthates contg. thiosulfates and no sulfides and thiocarbonates, dissolve 4-5 g. of a xanthate in a little cold water, ppt. carbonates and sulfates with an excess of 20% BaCl<sub>2</sub> (not more than 50 cc), dil the soln to 500 cc., let settle, filter (discarding the first 50 cc of the filtrate), and use the filtrate in the following detns. If the soln. is alk., neutralize 25 cc. of the filtrate with NaOH in the presence of phenolphthalein, add a starch soln and excess of  $I_2$  soln., and titrate back with  $Na_2S_2O_3$ . Treat 25 cc. of the filtrate in a glass-stoppered flask with HCl (25 cc of 0.1 N for Et xanthate, 10 cc of 0.5 N for Pr and Bu xanthates, and 10 cc of 1 N for iso-Am xanthate, allowing the mixts. to stand for 10, 30 and 45 min., resp.). Neutralize the soln. with a small excess of CaCO<sub>3</sub>, and proceed with the iodometric titration as above. In the analysis of xanthates contg. contaminating sulfides and thiocarbonates, any loss of H<sub>2</sub>S, formed in the decompn. of xanthate with HCl, is avoided by following the above procedure in the manner given. Introduce 25 cc of xanthate soln contg 2-3 drops of phenolphthalein soln into a filtering flask fitted at the top with a dropping funnel (1) contg. HCl, and at the side neck with a long rubber tubing connected with a dropping funnel (2) contg NaOH soln. Move the funnel (2) to the lowest possible level and open the cock, decompose the xanthate soln in the flask by introducing HCl from the funnel (1). Pour in the NaOH soln. in the funnel (2). Neutralize the excess of NaOH with a few drops of HCl in the funnel (1). Disconnect the app. and proceed with the iodometric detn. Chas. Blanc

Rapid methods for determining trinitrophenol in commercial picric acid. N. Ya. Ugnaychev and D. A. Rikhter. *Zarodskaya Lab* 4, 1062-6(1935).—The resp.



basis of the 2 methods of detn. of  $\text{HOC}_4\text{H}_7(\text{NO}_2)_2$  (I) in picric acid by titration with  $\text{HgNO}_3$  and by pptn. with ammoniated  $\text{CuSO}_4$  and iodometric back titration of excess  $\text{CuSO}_4$  in the filtrate are:  $2\text{I} + \text{Hg}_2(\text{NO}_3)_2 = 2\text{HNO}_3 + [\text{C}_4\text{H}_7(\text{NO}_2)_2\text{OH}]_2\text{Hg}_2 + \text{H}_2\text{O}$  and  $2\text{I} + [\text{Cu}(\text{NH}_3)_4]\text{SO}_4 + 2\text{NH}_4\text{OH} = [\text{C}_4\text{H}_7(\text{NO}_2)_2\text{O}]_2\text{Cu}(\text{NH}_3)_4 + \text{H}_2\text{O} + (\text{NH}_4)_2\text{SO}_4$ . Any dinitrophenol present is not pptd. Prep a standard soln of  $\text{HgNO}_3$  by digesting 20.5 g  $\text{Hg}_2(\text{NO}_3)_2$  with 300–500 cc  $\text{H}_2\text{O}$  acidified with  $\text{HNO}_3$  (1–2 cc of 10%  $\text{HNO}_3$  for each 100 cc  $\text{H}_2\text{O}$ ) at 50–60°. Decant from the undissolved residue, dissolve it in a little  $\text{HNO}_3$ , mix the 2 solns., dil. to 1000 cc. and standardize the soln. by titrating 0.5% soln. of picric acid until a drop of the supernatant soln. fails to give a dark spot on the test paper. Prep. the test paper by adding dropwise 1%  $\text{AgNO}_3$  to 10%  $\text{Na}_2\text{S}_2\text{O}_3$  to a brown discoloration of the soln., followed by satg. filter paper with this soln. and drying. Prep a standard soln. of ammoniated  $\text{CuSO}_4$  by adding 200 cc of 10%  $\text{NH}_4\text{OH}$  to 6–7 g  $\text{CuSO}_4$  in 300–500 cc  $\text{H}_2\text{O}$  and dilg. to 1000 cc. For the standardization of the soln., introduce an excess of the soln. (25 cc.) into 50 cc. of 0.2–0.3% of picric acid contg. 2 cc. of 10%  $\text{NH}_4\text{OH}$ , shake well, filter and wash the ppt. with 0.5%  $\text{NH}_4\text{OH}$ . Make the filtrate slightly acid with  $\text{H}_2\text{SO}_4$  (10 cc. of 10%  $\text{H}_2\text{SO}_4$ ), add 5–8 cc. of 20%  $\text{KI}$  and titrate with 0.5 N  $\text{Na}_2\text{S}_2\text{O}_3$ . Equally accurate results are obtained by dissolving the ppt. in 5–10 cc. of dil.  $\text{H}_2\text{SO}_4$  and titrating as above. Similar procedures are used in the detn. of I in com. picric acid.

Chas. Blanc

Microcrystalline reaction of glycolcol. Geo. Deniges. *Bull. soc. pharm. Bordeaux* 73, 168–72 (1935).—Place a few particles of the sample on a slide and add a drop of phosphotungstic acid soln. with the aid of a fine rod. If the solid does not become entirely moistened, a very small droplet of the reagent may be added or the mixt. may be stirred gently with a very fine rod. Examine without a cover-slip under a magnification of 130–50×. If the glycolcol sample is a soln., a drop should be carefully evapd. on a slide and the reagent added to the residue.

S W Goldstein

Identification and colorimetric determination of glycolcol by means of alloxanic reagent. Georges Deniges. *Bull. soc. pharm. Bordeaux* 73, 161–8 (1935).—Place a little (less than 0.5 mg.) of glycolcol in a round-bottomed porcelain dish (2–3 cm. diam.) and dissolve with agitation in 1 drop of D.'s alloxanic reagent (*Bull. soc. pharm. Bordeaux* 1901, 161). Allow to stand at room temp. A pink color is apparent after 15 min., and becomes more pronounced on standing, being very strong after 2 hrs. After 4 hrs. the color has changed to an intense reddish violet. Addn. of 1 cc. of water to the residue forms a violet-red soln. One part of the soln. is treated with 1–2 drops of  $\text{NaOH}$  soln. to give a violet color, while the other half of the soln. is treated with 1–2 drops of 5% ( $\text{AcO}$ ) $_2\text{Zn}$  soln. contg. 2 cc. of glacial  $\text{AcOH}$  in 100 cc. of soln. to give a yellow-orange color. A set of standard tubes can be prepd. with known amts. of glycolcol treated as above.

S W Goldstein

A colorimetric method for the determination of glucose and levulose by their osazones. V S Butkevich and M. S. Gaevskaya. *Compt. rend. acad. sci. U. R. S. S. [N. S.]*, 3, 113–16 (1935).—Hertzfeld's method, *C. A.* 27, 1374, of detg. glucose in the presence of other reducing substances gives correct values (precision  $\pm 2.5\%$ ), only when the time of heating for osazone formation is 1.5 hrs. and not 1 hr. as prescribed by H. The formation of glucosazone is about 0.5 complete in 1.5 hrs. Heating a longer or shorter period gives results that are high or low, resp. Formation of levulosazone is complete in 1.5 hrs., hence, it can be detd. by the same method, but the value obtained is twice the true value. Glucose and levulose can be detd. in the same soln., if the time of heating is prolonged to 5 hrs. In this case the precision is somewhat lower, exceeding 10%, if the concn. of glucose is 4 times that of levulose.

W Gordon Rose

Volumetric determination of the sodium salt of glycerophosphoric acid and notes on the behavior of the calcium salt. S. Babich. *Z. anal. Chem.* 101, 398–401 (1935).—

1 Dissolve 0.1–0.2 g. of the salt in 25 cc. of water, add 5–10 cc. of 0.5 N  $\text{Pb}(\text{NO}_3)_2$  and boil 5–10 min. Cool, dil. to 50 cc., stir carefully and filter. To an aliquot part of the filtrate, add phenolphthalein and titrate with 0.1 N  $\text{Na}_2\text{CO}_3$  soln. until a distinct pink color prevails. Heat to boiling to see if the color fades, if it does add a drop or 2 more of the soda soln. From the vol. required of  $\text{Na}_2\text{CO}_3$ , the excess  $\text{Pb}(\text{NO}_3)_2$  is known and when this is subtracted from the original vol. used, the difference is a measure of the quantity of glycerophosphoric acid present. Residue obtained in this way with the Na salt agree well with those obtained by acidimetric titration. The procedure also gives good results when Na cacodylate is present, which is not true of the acidimetric method of analysis. The behavior of the Ca salt of glycerophosphoric acid is interesting. Analysis by the above procedure shows that about 7.3 cc. of  $\text{Pb}(\text{NO}_3)_2$  is required when the theory demands 8.76 cc. Similarly when it is attempted to det. the Ca present, only a corresponding quantity of Ca can be pptd. as  $\text{CaC}_2\text{O}_4$ . In the filtrate from the  $\text{CaC}_2\text{O}_4$  ppt., more Ca can be found after destroying all org. matter with hot  $\text{HNO}_3$ . Possibly part of the Ca in the glycerophosphoric acid has replaced an  $\text{OH}$  group and is present as glycerate.

W. T. H.

Microdetermination of protein nitrogen in the presence of ammonium salts. André Roche and Francis Marquet. *Bull. soc. chim. bel. 17*, 1630–2 (1935).—The protein is pptd. as the tannate by Hédin's reagent (tannin 70,  $\text{NaCl}$  100 and  $\text{AcOH}$  50 g. per l.) and washed free from  $\text{NH}_4$  salts with a mixt. of Hédin's reagent 1, satd.  $\text{K}_2\text{SO}_4$  5 and water 94 parts. The usual Kjeldahl detn. is then made. The method is adapted from that of Kossel. L. E. G.

A sensitive reaction for the detection of nitrogen bases. H. Wachsmuth. *J. pharm. Belg.* 17, 705–8 (1935).—To 10 cc. of a very dil. soln. of the alkaloidal base add 0.5 cc. of 15%  $\text{CuSO}_4$  and a trace of  $\text{KCN}$  (or 1–2 drops of a 1% soln.). The coloration appears immediately without heating. A turbidity may form if the soln. of the base is too concd. An excess of  $\text{KCN}$  is undesirable as it hastens the decoloration. The reaction is not specific for the  $\text{CN}$  ion. Morphine produces a yellow-green, but the result is doubtful with concns. below 1.15,000–1.20,000. Pyramidone produces a violet, which is definite with concns. below 1.700,000. Apomorphine produces a red, which passes through rose to brown to gray and finally to a stable green. The color can be removed by shaking with org. solvents. The coloration was observed with concns. about 1.2,000,000. Adrenaline produces a red, and a rose color is still perceptible with concns. of 1.5,500,000.

S W Goldstein

Color tests for distinguishing between tetrahydronaphthalin and decahydronaphthalin. A. Castiglioni. *Z. anal. Chem.* 101, 414–17 (1935).—Mix 1 cc. of the hydrocarbon, or mixt. of hydrocarbons, with 50 cc. of 50% alc. and of this mixt. take 1 cc. for the test. To it add 1 cc. of  $\text{HCHO}$  in 30% aq. soln. and 10 cc. of concd.  $\text{HCl}$  or 2 cc. of  $\text{H}_2\text{SO}_4$ . If  $\text{HCl}$  is used, heat to boiling and allow to cool but with  $\text{H}_2\text{SO}_4$  do not heat. With the  $\text{HCl}$ , decalin gives an orange color with as little as 0.05 g. of hydrocarbon and tetralin gives no color when as much as 0.2 g. is used. With  $\text{H}_2\text{SO}_4$ , on the other hand, 0.01 g. of tetralin suffices to give a distinct pink coloration and 0.1 g. of decalin is required to give a yellow color. If  $\text{HCHO}$  is replaced by furfural, a more sensitive test is obtained but both hydrocarbons give the same color.

W T H

Forensic chemistry in the service of the state. Guillermo Q. Quibilan. *Rev. filipina med. farm.* 26, 307–16 (1935). Pitfalls in medicolegal toxicology. Pablo Anzuarez and Guillermo Q. Quibilan. *Ibid.* 361–7.

E J C

A new crystalline derivative of blood pigment. M. Wagenaar. *Z. anal. Chem.* 103, 417–18 (1935).—In previous papers, *C. A.* 24, 1402, 3258, a summary of all the known cryst. derivs. suitable for the identification of blood pigment has been given. In this connection, it may be mentioned that the hemin crystals of Teichmann are of particular importance. Some difficulties that occur in applying this important test are not encountered with a

new test which is even more conclusive. If a trace of blood fluid, such as can be obtained from particles of blood or from blood stains on clothing, is covered carefully with a cover glass, so that no small objects such as particles of hair or sand will prevent the direct contact of the cover glass with the object glass, and then a few drops of acetone is allowed to flow upon it, the particles of blood pigment will be surrounded by acetone. The cover glass presents too rapid evaporation. Finally if 1 drop of dilute mineral acid is added, soon crystals of a crystalline compound of acetone and blood pigment develop even in the cold. Photomicrographs of the characteristic crystals are shown.

W T H

Constitution of complex metallic salts [detn of Pd] (Mann, Purdie) 6. Improvement of the micro-Kjeldahl method (Groák) 1. Unknown property of the camelot half-cell and the estn of bromide-chloride mixts (Hahn) 4. Colored compd formed in the Sullivan reaction for guanidine (Sullivan, Hess) 10. Identification of phenols with 2,4-dinitrochlorobenzene (Bost, Nicholson) 10.

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Analysis of brass and other zinc alloys. V. S. Bugakov and V. D. Nesukhaev. Russ. 37, 003, July 31, 1934. The Zn is removed from the disintegrated alloys by sublimation in vacuo.

## 8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND J. F. SCHAIER

A method for the experimental investigation of hydrothermal solutions, with notes on its application to the solubility of silica. C. Stansfield Hitchen. *Bull. Inst. Mining Met.* No. 375, 1-20 (1935), *cf C A* 29, 6181.

Inversion of quartz to tridymite. I. Natural tridymite. Seiji Kondo and Toshiyoshi Yamachi. *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 651 (1935).—Microscopic measurements were made on natural tridymites (I) (Japanese) and x-ray powder photographs were taken to determine the properties of I and natural cristobalite (II).  $n_D$  was 1.477-1.479 for 5 different I, which agrees well with the values reported by Mallard. Interplanar spacings and intensities were calculated for 29 lines on the diffraction pattern given by an anticathode of Fe. II. X-ray distinction between tridymite and cristobalite. *Ibid.* 651-2.—Artificial tridymite (III) was prepared by heating powder quartz with  $\text{Na}_2\text{WO}_4$  or water glass, and artificial cristobalite (IV) by heating powder quartz with  $\text{NH}_4\text{Cl}$  or  $\text{TiO}_2$ .  $n_D$  of III = 1.409-1.474, which agrees well with Fennel's data. III showed lower  $n_D$  and higher lattice constants than I. II contains a small amt. of I and  $n_D$  accordingly = 1.478-1.483. However, the main portion is isotropic, II having  $n_D$  = 1.482. IV was almost pure, having  $n_D$  = 1.484. II showed higher lattice constants than IV. X-ray data are given for tridymite and cristobalite, permitting positive identification.

Ankerite and cosalite from Vasko, László Tokody and Gábor Vavrinec. *Földtani Közlemény* 65, 301-5 (1935).—A yellowish ankerite contained  $\text{CaO}$  29.09,  $\text{MgO}$  10.30,

$\text{FeO}$  15.74,  $\text{MnO}$  0.66,  $\text{CO}_2$  43.81, insol. 0.40%. The cosalite analysis shows Pb 39.55, Cu 2.71, Fe 0.25, Bi 40.21, Sb 0.04, S 17.20, insol. 0.74, sum 100.70%.  $\text{sp. gr. } 2^{\circ} = 6.63$ . Microscopic and crystallographic data are given.

Changes in the luminescence colors on fresh cleavage surfaces of calcite. K. Obenauer. *Zentr. Mineral. Geol.* 1935A, 357-61.—Fresh cleavage surfaces of calcite crystals give differently colored luminescence in ultraviolet light. In drawing mineralogical conclusions it is necessary to use only the colors produced on fresh surfaces (cf. Köhler and Leitmeier, *C A* 28, 2301).

Hydrothermal aragonite from andesite and limestone in Hungary. György Kertai. *Földtani Közlemény* 65, 354-62 (1935).—The probable formation of the crystals is discussed. Crystallographic data are given.

Petrological notes. 1-10. Sertaró Tsuboi. *Japan. J. Geol.* 12, 109-13 (1935) (in English); *cf. C A* 29, 3946.—Chem. and optical data are given for Japanese pyroxene, hornblende, anorthite, cummingtonite and biotite from different sources.

The crystallization of orthorhombic and monoclinic pyroxenes from synthetic silicate melts. D. P. Grigor'ev. *Zentr. Mineral. Geol.* 1935A, 353-7; *cf. C A* 29, 6542.—Mixts corresponding to  $3\text{MgO} \cdot \text{CaO} \cdot 3\text{SiO}_2$ , to which  $\text{CaF}_2$  had been added, were heated at 1400° for 4 1/2 hrs and cooled slowly. The material obtained consisted chiefly of enstatite, often bordered by monoclinic diopside which was apparently formed by the reaction enstatite + liquid

= diopside Enstatite does not crystallize from melts if  $F_2$  is absent (cf. Bowen and Scharrer, *C. A.* 29, 4250), probably because of the fact that the inversion temp enstatite-clinoenstatite is lower than the temp. at which complete crystn occurs in the absence of  $F_2$ . M F

The refraction and birefringence of a zircon K. Chudoba *Zentr. Mineral. Geol.* 1935A, 362-7, cf. Epler, *C. A.* 22, 3823, 4120—A green  $a$ -zircon,  $d_{10} = 4.150$ , had  $n_x$  1.8560, 1.8629, 1.8701 and  $n_z$  1.8671, 1.8739, 1.8809 for Li, Na and Ti light, resp. Another zircon with the same  $d$  was found on optical examn to be largely  $\delta$  zircon (uniaxial, high birefringence) with some  $c$ -zircon (biaxial) and with a border zone of  $a$ -zircon (uniaxial, low birefringence). Michael Fleischer

Minerals of lead slags Petrological methods of investigating slags W. I. Lober *Chem. Erde* 10, 67-115 (1935)—Existing information is summarized (120 references), and in many cases recast, e.g., into diagrams for the systems  $CaO$ - $FeO$ - $SiO_2$  and  $Ca$ - $SiO_2$ - $Na_2SiO_3$ - $Zn$ - $SiO_2$ - $Fe_2SiO_5$ , with their comps., ds., optical properties and miscibility limits. Suitable petrological methods are described, in particular pulverization followed by sepn by  $d$ , optical examn, and chem. analysis of the sepd portions, with full details, and numerous micrographs. In this way the constituents of various slags were detd. In those from "mixed ore" (Lower Harz) were identified fayalite, willemite,  $\alpha$ -celadon, sphalerite, wurtzite and magnetite. In lead slags were found various orthosilicates: fayalite, willemite and monticellite (between the first 2 of which a gap of immiscibility from above 17% fayalite to 36% willemite was found), melilite and justite, several feldspars in small amt., pyroxene (near hedenbergite), magnetite and (often ferruginous) zinc spinel, and various sulfides: galena, chalcocopyrite, chalcocite, bornite, pyrrhotite, with sphalerite and wurtzite, the Zn occurring as sulfide and silicate being in approx. equal amts. C. A. Silberrad

Investigation of minerals containing rarer elements Allantite found in Kanbe village, Mara Prefecture J. I. sutaro Takubo and Heihachiro Ukwai *J. Chem. Soc. Japan* 56, 1523-5 (1935), cf. *C. A.* 28, 3892—The compn. of this mineral is believed to be  $Ca_2(AlOH)(Al, Ce, Fe)_3(SiO_4)_2$ . T. Katsura

Sorption of gas by minerals V. Jitsusaburo Same-shima and Noriyoshi Morita *Bull. Chem. Soc. Japan* 10, 487-90 (1935), cf. *C. A.* 28, 2646—The partially or wholly dehydrated minerals, mordenite, thomsonite, scolecite, epistilbite and phillipsite were tested with  $NH_3$  and  $CO_2$ . Dehydration was carried out by heating 2.5 hrs. at 200° *in vacuo*. Mordenite was heated to 350° for 30 min without effect, while heating thomsonite to 350° for 1 hr. caused complete dehydration. Mordenite sorbed considerable amts. of both gases and is a sorbent of the chabazite type. The other minerals sorbed little  $CO_2$ , but did sorb considerable amts. of  $NH_3$ , with which they appeared to combine chemically. Completely dehydrated thomsonite sorbed but little  $NH_3$ . *VI. Ibid.* 490-4—Dried or dehydrated sepiolite, talc, serpentine, asbestos, acid clay, bentonite and vivianite were tested with  $NH_3$  and  $CO_2$ . Sepiolite sorbed considerable amts. of both gases and is a sorbent of the silica gel type. Acid clay and bentonite sorbed  $NH_3$ , as did asbestos to a slight extent, but no  $CO_2$ . Talc, serpentine and vivianite sorbed neither gas. John E. Mubery

Chemical and physico-optic relations in the brittle mica group G. Koch *Chem. Erde* 9, 453-63 (1935)—Numerous analyses with values of  $d$ ,  $\alpha$ ,  $\beta$ ,  $\gamma$  and  $2E$ , many new, are given of the brittle micas. These lead to the formula  $H_2(Ca, Na)_2Al_2[Al_2Si_2O_{10}]_2$  for margarite, increase in Na being accompanied linearly by decrease in  $\alpha$ ,  $\beta$ ,  $\gamma$  and  $E$ , and increase in  $\gamma$ - $\alpha$ , and  $H_2CaMg_2[Al_2Si_2O_{10}]_2$  for clintonite, with which are included zanthophyllite and brandisite. Ca and Na are interchangeable in the margarites, and Mg and Al between them and the clintonites, all being intermiscible. Chloritoid is  $H_2[Si_2Al_4Al_2Fe_2O_{10}]_2$  and otterite  $H_2[Si_2Al_4Al_2Fe_2O_{10}]_2$  with Mn replacing part of the Fe (with proportionate decrease

in  $d$ ). Analyses of paragonite confirm the formula  $H_2NaAl_2Si_2O_{10}$ . C. A. Silberrad

Synthesis of pyrophyllite Robert Schwarz and Georg Trageser, *Z. anorg. allgem. Chem.* 225, 142-50 (1935), cf. *C. A.* 29, 7873—Pyrophyllite has the compn.  $Al_2O_3 \cdot 4SiO_2 \cdot H_2O$  and does not lose  $H_2O$  below 690°. By heating feldspar, plagioclase or kaolin with 0.5 N HCl at temps. above 320°, pyrophyllite can be obtained, but it is accompanied always by some of the initial substance. The material is heated in a Ta crucible in a steel autoclave. H. Stoertz

Thermal decomposition of talc R. H. Ewell, E. N. Bunting and R. F. Geller *J. Research Natl. Bur. Standards* 15, 551-6 (1935) (Research Paper No. 848)—Water in excess of 1 mol was mostly driven off between 390° and 500°. This water loss was accompanied by a small endothermic heat effect, but not by any change in crystal structure or optical properties. The one mol of combined water was driven off between 800° and 840°. This water loss was accompanied by a large endothermic heat effect and an increase in true sp. gr. from 2.83 to 2.91, and by breakdown of the talc into enstatite and amorphous silica. Inversion of the enstatite to clinoenstatite took place gradually, both phases being observed in material heated at 1200°, and only clinoenstatite in material heated at 1300°. The latter also showed conversion of the amorphous silica to cristobalite. The data support the hypothesis of Foshag and Wherry (*C. A.* 17, 513) that water in talc in excess of 1 mol is not constitutional and may be held electrostatically between basal cleavage planes. P. S. Roller

Constitution of the clay minerals C. E. Marshall *Science Progress* 30, 422-31 (1935); cf. *C. A.* 29, 7850—A review with bibliography. Joseph S. Hebrum

Chemical structure of clay minerals and corresponding silicates of bi- and tri-valent metals Julius Holzer *Chem. Erde* 9, 464-85 (1935)—Of 28 such minerals 132 old analyses have been selected as trustworthy and recalcd. to show the at. content of di-(except Ca) and tri-valent metals,  $Si$ ,  $O$ ,  $OH$  (detd. by amt. of  $H_2O$ ),  $Ca$ ,  $Na^+$ ,  $K$  and  $H_2O^-$ . The ratio  $Si/O$  is very near 2.5 in a large no. of cases. In 45 where the divergence is considerable it has been made exactly 2.5 by a suitable transfer of  $H_2O^+$  to  $H_2O^-$ , in many cases definitely supported by consideration of the dehydration results. On the basis of these revised analyses the structures of the minerals are discussed in the light of recent x-ray results, such as those on chlorite, muscovite, kaolin, pyrophyllite, etc. Eighty-four references. C. A. Silberrad

Composition of halloysite and powdery dolomite from Buda Gabor Vavrinec *Magyar Chem. Folyoirat* 41, 70-7 (1935)—Analysis of halloysite gave  $SiO_2$  37.82,  $Al_2O_3$  31.15,  $FeO$  0.12,  $CaO$  0.45,  $MgO$  0.25,  $P_2O_5$  0.17,  $SO_3$  traces,  $CO_2$  0.26,  $H_2O$  29.93, org. matter traces, sum 100.15%. Drying expts. showed that water content of halloysite behaved similarly to that of hydrogels of pure  $SiO_2$  or  $Al_2O_3$ . Curves showing  $H_2O$  content of halloysite according to lowering tensions would be like the dehydration curves of van Bemmelen. Also soly expts. with concd.  $H_2SO_4$ , dil. HCl and concd. HF were made. A special calorimeter was constructed to det. reaction heat of pure  $SiO_2$ , silica earths,  $Al(OH)_3$ ,  $Al_2O_3$  and various halloysites. The latter gave 602 and 530 cal., resp., the theoretical values calcd. from their analyses were 607.6 and 521.1 cal., resp. The components of halloysite ( $SiO_2$  and  $Al(OH)_3$ ) are probably united not chemically but as a phys., eventually colloidal, mixt. This statement affirms that of Mehmel (*C. A.* 29, 4799) about the crystal structure of halloysite. A new analysis of a dolomite flour from Martonhely, Budapest, gave  $CaO$  39.92,  $MgO$  21.44,  $FeO$  0.13,  $CO_2$  46.60,  $P_2O_5$  0.08,  $SO_3$  0.05,  $SiO_2$  0.22, HCl insol.  $SiO_2$  0.13, HCl insol. 0.15,  $H_2O$  at 150° 0.06, loss on ignition 0.20, sum 100.08%. S. S. de Finily

The rare earth content of European and Japanese clay shales. E. Minami *Nachr. Ges. Wiss., Göttingen, Math. physik. Klasse*, Pachtgrupp IV, 1, 155-70 (1935); cf. *C. A.* 28, 4001, 30, 1335—The rare earth content of

all the clays, detd. gravimetrically, was close to 0.02%. The amts. of each element present were detd by x-ray spectrographic methods. Ce, Y, Nd, La and Th predominated.

**Lias clay from Dobbertin, Mecklenburg-Schwerin** F K Schlunz, *Chem Erde* 10, 116-25 (1935), cf. C. A. 27, 5689, 28, 1960.—This clay was examd by similar methods to those used for clays from the neighboring localities, Papendorf and Mallus. Quartz and feldspar increased, muscovite and kaolin decreased as particle size increased; the only clay mineral found by means of x-rays was halloysite; of heavy minerals the proportion therein of pyrite (78.4%) was very much greater than in either of the other 2 clays above mentioned (28.0 and 34.2%, resp.), while that of hornblende was small. Of electrolytes Ca and Cl ions were low, SO<sub>4</sub> high. C. A. Silberrad.

**Spectro-analytical investigation of tectites** E Preuss, *Chem. Erde* 9, 305-418 (1935).—By use of the Mannkopf and Peters' spectral method (cf. C. A. 25, 5110), analyses of a large number of tectites and analogous materials have been made, with results shown in the annexed table, in which the mol. totals of the oxides of Al, Fe, Mg, Ca, Na and K are made up to 100, and SiO<sub>2</sub> stated in proportion (figures in col. 1 indicate the number of samples of which the analysis is the average)

	d	■	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	FeO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O
Moldavites (14)	2 354	1 4828	515	38	12	7	17	5	3 3 13 5
Australites (2)	2 448	1 5111	378	36	5	18	5	14	6 7 6 4
Indochina (8)			331	37	20	0	14	14	5 6 8 7 7
Bilitonites (5)	2 477	1 5178	380	37	21	5	14	5	0 5 8 5
Average (36)			359	36	18	15	5	0	8 8 5
Norwegian loam			212	13	5	20	18	12	7 9 5
Lithosphere			190	29	18	17	17	5	12 6 5

There were also found Li, Be, B, Sr, Ba, Ti, Zr, V, Cr, Mn, Ni, Cu, Ga, Ge, Sn, Pb and Se in amts comparable with those in the lithosphere, the chief differences being the relatively smaller content of the more volatile elements, e. g., Cu, Ge, Sn and Pb in the tectites, the most variable amts were those of Cr and Ni. Glasses were also analyzed.

	FeO	CaO	NaO	MnO	TiO <sub>2</sub>
Darwin glass	2.3	0.035	0.04	0.04	0.4
SiO <sub>2</sub> glass (Henbury)	6.0	0.007	0.12	-	-
SiO <sub>2</sub> glass (Wabar)	5.0	0.0093	0.13	-	-
SiO <sub>2</sub> glass (Libyan desert)	0.3	0.0006	0	-	-
Moldavites, av.	2.5	0.006	0.002	0.1	0.3
Australites, av.	4.7	0.013	0.0035	0.12	0.8
Bilitonites, av.	5.6	0.055	0.035	0.14	1.0

The results are not in favor of Spencer's theory (C. A. 27, 2011), nor does the general resemblance between the compns. of the tectites and that of the lithosphere favor a cosmic origin. Analyses were also made of Paucurtambo glass (Linck, C. A. 28, 2301), and of glass spheres from Colombia (Stutzer, C. A. 21, 41), showing compns. resp. differing markedly from that of the tectites, and identical with that of obsidian. C. A. S.

**The mineral production of India during 1934** Lewis L. Fernor, *Records Geol. Survey India* 69, Pt. 3, 245-335 (1935); cf. C. A. 29, 3745.

**Deposits of mixed minerals in Sardinia** L. Gerbella, *Ricerche assoc. mineraria sarda* 40, No. 7, 4-S (1935), cf. C. A. 29, 2888.

**Hills of Shah Abdul Azim (Persia)** A. Riviere, *Compt. rend. soc. géol. France* 1935, 60-2.—A stratum, a few in thick, of much crushed quartz and limestone is impregnated with Fe and galena, which extend into the limestones above and below it.

**Outline of the geochemistry of the Urals** E. C. Zakharov and S. A. Yushko, *Trans. All-Union Sci. Research Inst. Econ. Mineral.* (U. S. S. R.) No. 75, 4-42 (in English 43-6) (1935).—Late Paleozoic igneous activity in the Urals gave origin to bodies of (1) gabbro, diorite and plagiogranite with associated pyrite and Cu-Zn deposits and (2) K granites with high-temp. Au deposits and meso-epithermal Hg-Sb-Au deposits. The amts. of As, Sn, Sb, Se, Te, Cd and Tl associated with various types of

deposits and mineralization and their areal distribution are summarized. Ge and In were detected spectroscopically in Zn ores, one of which contd. 0.02% In<sub>2</sub>O<sub>3</sub>.

R. H. Beckwith

**Notes on the Balumbing mine, west coast of Sumatra** D. W. J. Grey, *Bull. Inst. Mining Met.* No. 375, 1-18 (1935).—Subsequent to propylitization and silicification of the rhyolites and tuffs by hydrothermal solns., faulting occurred, mineralization by ascending solns. followed and was at first confined to the fissures. The original filling was probably calcite and Mn with which latter the Au and Ag were associated. Descending surface waters, charged with H<sub>2</sub>SO<sub>4</sub> derived from oxidation of the pyrite in the propylite, attacked and leached the fractured surfaces of the chloritized and sericitized rocks in the elastic zone and yielded kaolin and hydrous SiO<sub>2</sub>, mostly colloidal. These waters also attacked the Au and Ag. At the surface Ag was only slightly attacked, but in depth, in the path of increasingly acid solns. as oxidation proceeded downward, the Ag went into soln. as sulfate. The soln. descended through the shattered rock where Ag was partly pptd. by unaltered orthoclase and in neutral soln. partly abstracted by colloidal kaolin. The H<sub>2</sub>SO<sub>4</sub> reacted with NaCl in the mine waters to form HCl, which with the Mn in the ore generated Cl which dissolved Au. Four possible precipitants, each of which probably had something to do with it, are considered.

The evolution of the present metallurgical flow sheet is described in detail. An old tabling and cyaniding plant was scrapped in favor of flotation. Use of a blanket strike before the classifier reduced reagent consumption to Pb(OAc)<sub>2</sub> 0.93, Na<sub>2</sub>S 0.31, Bu xanthate 0.19, Aerofloat 15.0 14 soda ash 0.90 lb per ton. In Aug. 1934, in treating an ore averaging 2.79 dwt per ton, 32.6% of the Au was recovered on blankets, flotation recovered 93.6% of the Au from an impoverished head (1.88 dwt) for an over-all recovery of 95.7%.

Alden H. Emery

**Microscopical study of copper ores from Nieder-Marsberg (Westphalia)** Friedrich Schwacke, *Chem. Erde* 9, 480-525 (1935).—A detailed account with photomicrographs, of the manner in which the various minerals, primarily chalcocite, covellite, bornite, chalcopryrite, galena, sphalerite, pyrite, marcasite, tetrahedrite, farnatite, quartz and calcite, and secondarily malachite, azurite, cuprite, tili ore, melanoconite, limonite, gypsum and native Cu and Ag, occur, with their relations to the country rock, and discussion as to their origin.

C. A. Silberrad

**Geology of the iron deposits in the lower Yangtze region** C. Y. Hsieh, C. C. Sun, Y. C. Cheng and K. Chern, *Mem. Geol. Survey China*, Ser. A, No. 13, 191 pp. (1935).—Six types of occurrence of Fe in Tangtu district are described. All are genetically related to the diorite and were formed as an after-effect of the igneous intrusion.

In the first stage of mineralization apatite, magnetite and actinolite with perhaps a small amt. of hematite were deposited under high temp. and partly pneumatolytic conditions. Not long after this, a soln. forming large bodies of hematite emanated from the same magmatic source. Almost simultaneously the veins were altered, the actinolite to nontronite, while the country rocks were sheilded, sericitized and partly aluminized or kaolinized.

At first the soln. was very rich in Fe and SiO<sub>2</sub>; subsequently it became alk. and locally sulfurous. In the third stage of mineralization specularite, hematite, quartz, chalcocite, a little apatite and barite deposited under epithermal conditions. Next Fe was deposited in the interstices of quartz and barite crystals under cold-water conditions. In the final stage, atm. agencies altered the Fe to limonite. The Fe deposit of Tungkuangshan is of contact metamorphic origin. The ore, magnetite with a small amt. of hematite, is in close assoc. with garnet. Garnet, diopside, wollastonite and forsterite have been formed in the calcareous rocks, and sericite, andalusite and cordierite in the argillaceous and siliceous rocks.

The Changlungshan Fe veins were deposited by solns. from granitic rocks at great depth and under high temps. The Sanshanchen deposits are of the contact pyrometamorphic type similar to those of Tungkuangshan. The Cheng-

menshan deposit is of hydrothermal origin and of the mesothermal type. In the Nanking Hills, 3 fields occur. The ore was formed as an igneous after-effect of the intrusive rock, probably as a replacement under mesothermal to epithermal conditions. Alden H Emery

Ferrous deposits of Berry. André Vatan. *Compt. rend. soc. géol. France* 1935, 64-5.—The origin of these deposits, consisting of an intimate assoc. of  $\text{CaCO}_3$  and limonite, is attributed to transport of  $\text{Fe}(\text{OH})_2$  from laterite in pseudo-sols in  $\text{H}_2\text{O}$  of  $\text{pH}$  less than 7, i. e., rich in  $\text{CO}_2$ , into a lake with limestone bottom. Here  $\text{CaCO}_3$  dissolved as bicarbonate and the alkyl of the  $\text{H}_2\text{O}$  increased until at  $\text{pH}$  10 the colloidal sols of  $\text{Fe}(\text{OH})_2$  became unstable, and limonite deposited in intimate assoc. with  $\text{CaCO}_3$ . C A Silberrad

Sudbury nickel field restudied. A G Burrows and H C Rickaby. *Ann. Rept. Ontario Dept. Mines* 43, Pt. II, 1934, 49 pp (1935).—The rocks of the area are described and the theories for the origin of the ore reviewed. Alden H Emery

Description of the Umshunskii tungsten ore deposit. T G Tikhova. *Razvedka Nedr. (Subterranean Prospecting)* 5, No. 3, 15-23 (1935).—A wolframite ore contg.  $\text{WO}_3$  74.62,  $\text{FeO}$  17.43,  $\text{MnO}$  7.60,  $\text{CaO}$  0.30 and  $\text{SiO}_2$  0.20% was found in Umshunsk, located about 207 km from Sretensk, Siberia. A A Boehlingk

The Khaidarkan mercury-antimony fluoite deposit. A I Suloev and A L Ponomarev. *Trans. All Union Sci. Research Inst. Econ. Mineral. (U. S. S. R.)* No. 71, 5-76 (in English 77-8) (1935).—The deposit, in the northern Alai foothills southwest of Fergana, occupies an area of 10.9 sq. km. A belt of limestones is tightly interfolded and interfaulted with sandstones and shales, and all are cut by diabases, probably of Variscan age. Granites, granodiorites and pyroxenites in the vicinity are probably of the same age. Extensive areas of breccias along faults and along the contacts between limestones and shales are cemented and replaced by fine-grained quartz, fluorite, stibnite and cinnabar and are cut by veins of the same material. The ores are of disseminated epithermal type and of hypogene origin. Estimates of tonnage of fluorite, Sb and Hg are given. The deposits are believed to merit exploitation on a large scale. R H Beckwith

Glauber salt in North Dakota. Irvin Lavine, Herman Feinstein and Earl Skene. *Chem. & Met. Eng.* 42, 681-2 (1935).—Locations, quantities and analyses of deposits of  $\text{Na}_2\text{SO}_4$  are given and methods of prospecting are described. E R Rushton

Walpole Island, S. Pacific. E Aubert de la Rue. *Compt. rend. soc. géol. France* 1935, 48-9.—The phosphate deposits here contain 55-65% Ca phosphate, and have yielded some 10,000 tons per yr. They consist of a light brown pulverulent material, and occur in pockets in coral rock. C A Silberrad

Geologic distribution of fire clays in the United States. J R Chelkowsky. *J. Am. Ceram. Soc.* 18, 367-90 (1935). C H Kerr

Kaolin deposits of the Ukraine from the newest geological exploration data. I I Melnikov. *Trans. Inst. Econ. Mineral. (U. S. S. R.)* No. 70, 35 pp (1935).—A description of the field relations and estimates of tonnage of extensive primary and transported kaolin deposits in the southwestern Ukraine. R H Beckwith

Red sands of Alpes-Maritimes. S Deb. *Compt. rend. soc. géol. France* 1935, 116.—Chem. and mech. analyses show these sands to be sufficiently refractory, plastic, coherent and permeable to gases to be suitable for foundry work or manuf. of silica bricks. The heavy constituents are zircon, rutile, anatase, brookite, magnetite, ilmenite and kyanite. C A Silberrad

The geochemistry of caustobolites. N A Orlov and V A Uspenski. *Khim. Tverdogo Topliva* 5, 663-79 (1934).—A bibliography with 37 references. A A B

A short geological description of the Kivdo Raichikhin brown coal deposit. A T Ponomarenko. *Razvedka Nedr. (Subterranean Prospecting)* 5, No. 3, 10-15 (1935).—This deposit is located 600 km west of Khabarovsk, Siberia. The coal belongs to the class of humic coals

contg.  $\text{H}_2\text{O}$  30-34, volatile substances 26, ash 6, coke 35.5, S 0.5, H 2.5, C 42 and O + N 13.5%. Its structure is that of fusain and durain coal. A A Boehlingk

Geothermal stages and the chemistry of artesian waters of northern Dagestan. I I Chebotarev. *Razvedka Nedr. (Subterranean Prospecting)* 5, No. 3, 23-6 (1935).—Geothermal and chem. investigations of the waters indicate the presence of crude oil. A A Boehlingk

Densities of rocks calculated from their chemical analyses. Reginald A Daly. *Proc. Nat. Acad. Sci.* 21, 657-63 (1935).—The method of calcn. of liddings (C. A. 14, 2457) which assumes the material to be completely crystd., nonvesicular and at room temp. was tested. The water-corrected normative  $\rho$  of the more abundant holocryst. rocks may be regarded as the actual  $\rho$ . The calcd.  $\rho$  was too low when the combined water was much in excess of 1%, and also in general for peridotites. Applications of the calcns. to extrusive and porous rocks are made. P S Roller

Viscosity of melted rocks. Michel P. Volarovich. *Compt. rend.* 202, 78-80 (1936). *cf. C. A.* 29, 4922, 7744.—The viscosities at several temps. between 1100° and 1400°, detd. by V's method, and complete analyses of the following rocks are given (figures after each indicate the percentage of  $\text{SiO}_2$  and the viscosities in poises at 1400°): basalt 43.03, 55, teschenite 50.58 (with 23.41  $\text{Al}_2\text{O}_3$  +  $\text{Fe}_2\text{O}_3$ ), 851; andesite 63.76, 1175, olivine basalt (erupted 1932) 52.4, 126; diabase 48.14, 60, obsidian 73.79, 170,000, and artificial glass 72.8 (with 15.8  $\text{Na}_2\text{O}$ ), 123. The increase in viscosity with increased  $\text{SiO}_2$  is obvious, unless modified by a large amt. of  $\text{Na}_2\text{O}$  as in glass, the high viscosity of teschenite is attributed to the large amt. of  $\text{Al}_2\text{O}_3$  +  $\text{Fe}_2\text{O}_3$ . C A Silberrad

Mixing or separation into components of silicate rock magmas. G Linck. *Chem. Erde* 10, 126-8 (1935).—Grignov's expts. (*cf. C. A.* 29, 6542) are unsatisfactory, as he has based the compn. of his melts on the constituents charged without allowing for possible volatilization. There is no evidence of silicate magmas ever sepg. into 2 phases, either as 2 layers, or as an emulsion (such as  $\text{NaCl}$  may form in a magma). On slow cooling sepn. may occur of a mineral with which the magma is satd. at a temp. slightly below its m. p. e. g., of leucite. Any differentiation must consist of a wandering of those components which crystallize out first, e. g., by gravitation (*cf. Vogt, C. A.* 18, 805). C A Silberrad

Geochemical characteristics of protocrystallization. A E Fersman. *Compt. rend. acad. sci. U. R. S. S. (N. S.)* 3, 216-20 (1935).—In the crystn. of basic and ultrabasic rocks, the minerals crystg. earliest are those of sym. crystal structures, highest coeffs. of thermal stability and highest values of lattice energy. The elements predominating in these minerals are those of even at no and esp. those with max. values of packing energy, of shortest ionic radii and of lowest at vols. and ionization potentials, i. e., elements with the most stable nuclei and with the greatest mech., thermal and electrostatic strength of the electronic shells. Michael Fleischer

Geochemical investigations of volcanoes in Japan. I. Studies on the gases and the spring waters of the volcano Asama. I Kimio Noguchi. *J. Chem. Soc. Japan* 56, 1495-510 (1935).—Results of analyses of the various volcanic products are given. II. Chemical compositions of the lavas of the volcano Oshima, Izu. Iwaki. *Ibid.* 1511-22.—The compn. of the lavas are similar. T. Katsura

Petrology of Alaid volcano, North Kurile. Hisashi Kuno. *Japan J. Geol.* 12, 153-62 (1935) (in English).—The lavas of Alaid Island are of several kinds of olivine-basalt, while those of Taketomi Island consist of a single kind of olivine-anorthite-basalt. These basalts differ markedly from those from the volcanoes of Huzi zone, central Japan. Among other differences they are more alk. and less siliceous, and the groundmass contains olivine and alkali-feldspar with silica absent. In the Alaid basalts, the olivine and augite crystd. out side by side. The olivine in the effusive stage was more ferrieritic than that crystd. in the intratelluric. The early olivine and

augite reacted with residual magma to form more ferriferous zones successively around the r.s.p. crystals

P. S. Roller

Granite of the Mont Blanc massif Paul Corbin and Nicolas Oulianoff *Bull. soc. géol. France* [5], 4, 767-70 (1935)—Two microgranites are described, showing no peculiar features

C. A. Silberrad

The andesitic rocks of Ditréu, C. A. Department, Transylvania Virgil Ivanovici *Ann. sci. univ. Jassy* 20, 80-97 (1935)—The mineral descriptions of several samples of rock are given, with chemical analyses. They are composed mainly of plagioclase, augite and olivine

John I. Milberry

A shallow intrusion of the bysmalith of Cavallo L. Glaueaud *Bull. soc. géol. France* [5], 4, 515-22 (1935)

The bysmalith at Cavallo (Constantine, Algeria) consists of vitreous and microgranular rocks, the latter a quartziferous hornblende microgranodiorite containing phenocrysts of strongly zoned plagioclase (30-40% an), green pleochroic hornblende and occasionally of orthoclase and biotite embedded in a paste of minute crystals of plagioclase with orthoclase and quartz (often poikilitic). Contact with the surrounding Eocene marls is marked in the bysmalith by the crystals being much smaller, while the marls have been metamorphosed in places to depths of a few cm to 4 m, with formation of sericite and spotted schists, chlorite, hornfels, garnet, rutile, sphene, etc. In 2 zones, one the result of mere heat, the other of pneumatolytic and hydrothermal action. Elsewhere the contact is marked only by a brecciated zone cemented by a paste of the comminuted materials or of calcite

C. A. Silberrad

Glasses and nephelite accumulations from Steinberg near Fiedbach Karl Scholtsch *Zentr. Mineral. Geol.* 1935A, 38-82—The nephelite basalts were probably formed by accumulation of crystals following differentiation. They show evidences of having been transported through the melt during the period of de-gassing. The composition of the glasses agrees qualitatively with the composition to be expected after separation of olivine and pyroxene, which are present as phenocrysts. Four new rock analyses

Michael Fleischer

The catamorphism of igneous rocks under humid tropical conditions John B. Harrison *Brochure publ. by Imp. Bur. of Soil Sci., Harpenden, Eng.*, 79 pp.—An extensive treatise is given on the chemical, microscopical and petrological aspects of the tropical catamorphism of igneous rocks. The  $\text{SiO}_2$ , Fe, Al, Mn, Mg, Ca, Ti and alkali analyses of the catamorphic products are made on a soln. obtained by treating the rock with a mixt. of 40 cc  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84), 20 cc  $\text{HCl}$  (sp. gr. 1.19) and 10 cc  $\text{HNO}_3$  (sp. gr. 1.40). The  $\text{Fe}^{3+}$  is determined on a soln. of a separate part of the material dissolved in  $\text{HCl}$  and dil.  $\text{H}_2\text{SO}_4$ . Under tropical and temperate conditions, surficial weathering of igneous rocks results in the catamorphism of the rock minerals by processes of hydration and oxidation with the production of hydrated silicates of  $\text{Al}_2\text{O}_3$  or silicoaluminic acids, hydrated silicates of Fe, hydrated oxides of Fe, with residual quartz and other resistant minerals. II. Catamorphism of basic igneous rocks under humid tropical conditions. The action of primary lateritization is due to the action of carbonated ground waters on its surface and is the setting free from combination in the rock minerals of the  $\text{SiO}_2$ , Ca, Mg, Na and K, while leaving most or all of the Al as gibbsite and the greater part of the Fe as limonite. While this primary process is the same on high and low levels, the after-processes differ materially. During the formation of primary laterite at low altitudes from basic igneous rocks, small quantities of quartz, a proportion of the  $\text{SiO}_2$  set free by catamorphism becomes crystalline during similar formation at high altitudes no change in crystalline  $\text{SiO}_2$  is indicated. On the contrary, it appears that some loss of quartz may take place at high levels. During the lateritization at low levels of similar, but quartz-free basic rocks, there is apparently little or no formation of crystalline  $\text{SiO}_2$ . Primary laterite is composed of the residue from the catamorphism of basic rock; laterite

earth consists of these residues with varying proportions of  $\text{SiO}_2$  taken up from the ground-water from rocks undergoing active lateritization; the surficial ironstones and bauxites are composed of constituents leached from the primary laterite during its conversion into lateritic earth, as well as lateritic earth after its desiccation. Primary lateritization is followed by desiccation which gradually results in vast masses of lateritic earths or argillaceous laterite. Exceptionally deposits of secondary quartz occur. Under temperate conditions, catamorphism of basic rocks is not a lateritic action, but a more gradual and less complete degradation and decomposition, accompanied by oxidation, hydration and leaching with the formation of various more or less complex hydrated silicates. III. Catamorphism of intermediate igneous rocks under humid tropical conditions. There are 5 fairly well marked zones of alteration between an epidote-quartz-diorite and its final degradation products: lateritic earth and surficial bauxite. There is (a) a gradual increase in the proportions of  $\text{Al}_2\text{O}_3$  present in the catamorphic products, (b) a gradual change from  $\text{FeO}$  to  $\text{Fe}_2\text{O}_3$ , (c) marked diminution in the proportions of CaO and MgO, and (d) very great diminution of the alkalis. IV. Catamorphism of acidic igneous rocks under humid tropical conditions. Under tropical conditions, acidic rocks, such as aplites, pegmatites or granites and granitic gneisses, do not undergo primary lateritization, but gradually change through catamorphism into pipe- or pot-clays, or more or less quartziferous and impure kaolins. Lateritic earths, and even pot-clays, may undergo desiccation, with the formation of concretionary and surficial masses of bauxite

J. R. Adams

Some differentiate and endomorphism contact rocks from Szarvasko Zsigmond Szentpétery and Kálmán Emzst *Földtan. Közlem.* 65, 305-14 (1935)—Chemical analyses and detailed petrographic description of 7 rock samples, consisting of quartz-plagioclase-pegmatite, quartz-plagioclase, lamprophyre, titanomagnetite-gabbro-peridotite and some accumulative metamorphic rocks are presented and discussed

S. S. de la Foy

The contact metamorphism of the Onawa pluton, Piscataquis County, Maine Shaler S. Philbrick. *Am. J. Sci.* 31, 1-40 (1933)—The Onawa pluton was formed by intrusion of a quartz-rich subalkali intermediate magma. Differentiation occurred with production of a basic border. Metamorphism of the slate series was begun during the intrusion of the pluton and continued through the associated igneous activity during which intermediate dikes were intruded accompanied or followed by injection of aplite veins of several compositions. Acid dikes with assimilated aluminous material were next intruded. Quartz veins carrying black tourmaline are the last phase of igneous activity. The degree and character of metamorphism were independent of the type of equigranular holocryst. igneous rock at the contact of the pluton but was much less intense in the vicinity of an apophysis of porphyritic microcryst igneous rocks. Both intensity and character produced were governed by the conditions of crystallization of the igneous rock. The aplite veins show in their modes of occurrence certain features that suggest that the rock reached a semimolten condition. The wide aureole and extensive recrystallization indicate that the superheat of the pluton was large and slow in dissipation. The lack of high-temperature minerals in the metamorphics shows that the temperature was not much above that of the country rock prior to and during intrusion. The highest degree of metamorphism is in a xenolith of plagioclase-cordierite-pleonaste-hypersthene hornfels

Alden H. Emery

Metamorphic rocks of southern Abukuma plateau. Kenichi Sugi *Japan J. Geol.* 12, 115-51 (1935) (in English)—The petrography of the following types of rocks is described: metamorphic rocks (a) from igneous and pyroclastic, (b) from sedimentaries and (c) due to retrogression. Metamorphic facies and causes of metamorphism are considered. Under (a) the rocks are divided into 2 main groups, one characterized by mineral assemblage of green schists including hornblende-bearing varieties, and the other by amphibolites. The retrogressive

rocks are widely distributed among low-grade metamorphic members of the eastern zone but are derived also from high-grade rocks of the western zone P S Roller

Metamorphic rocks of neighborhood of Viviers, Pyrénées-Orientales I Ragun *Compt rend soc géol France* 1935, 58-60 —The calcareous strata in this neighborhood show 2 distinct forms of metamorphism (a) cryst limestones and marbles contg considerable amts of silicates which often form visible crystals of scapolite, pyroxene, garnet, amphibole, quartz and sphene, and (b) less metamorphosed, contg smaller amts of quartz, feldspar and biotite, and considerable mizzonite C A S

Metamorphism on border of lustrous schists of Cerrières, Hautes Alpes Ch Pussenot *Compt rend soc géol France* 1935, 144-6 —Several strata of Rhætic and Liassic siliceous limestone in this neighborhood are characterized by a metamorphism that has produced large quantities of albite crystals C A S

Peculiar recrystallization of limestone Jean Goguel *Compt rend soc géol France* 1935, 152-3 —Near Vauluse and elsewhere in the department of that name the calcite of the Urgonian limestone has recrystd in yellow spherulites, usually about 1 mm diam, made up of cryst fibers, slightly cemented together and showing slight pleochroism Analysis gives the compn as approx  $75\text{CaCO}_3 + 1\text{MgCO}_3$ . The bed of spherulites rests on and penetrates into unaltered limestone C A S

Petrogenesis of Franconian ripple limestone Ph Schmitt *Chem Erde* 9, 321-64 (1935) —The methods of deposition of the ripple limestones of the area southeast of Homburg are examd, as also their chem compn (which throws no light on them) Sixty complete analyses are given, as a result of which the limestones are classified in 3 divisions according to the amt of clay present The bad smell on breaking the limestone is attributed to a polymerized form of  $\text{H}_2\text{S}$  C A S

Comparison of the spectroscopic analyses of Cryptozoön profluerum and the mineral waters of Saratoga Springs, New York Oskar Baudisch *Arkiv Kemi, Mineral Geol* 12B, No 9, 5 pp (1935) —Cryptozoön from the Saratoga Springs region contain Ca, Na, K, Li, Mg, Sr, Si, B, Fe, Mn, Cu and Ag All Ag lines were definite The quantities of Sr, Mn, Cu and Li contained in waters from Hathorn, Coesa, Geyser and Lincoln Springs are tabulated All the Cryptozoön contained B and much larger amts of Mn than the springs, while neither contained Ba The springs contained no B Since B is found in sea sediments and formations (Cryptozoön are of algal origin) while only traces have been found in igneous rocks and residual clays, its absence from the spring waters suggests that they are of magmatic origin A W D

Chemical weathering and soil formation in Siam E Blanck, W Credner and E v Oldershausen *Chem Erde* 9, 419-52 (1935) —A considerable no of analyses of surface soils, including laterites and "red earths," and of the immediately underlying rock in various places in Siam are given The essential differences between soils formed by rock decompn *in situ*, and those of the level areas resulting from sedimentation is emphasized, and the effects of climate are discussed C A S

Weathering of rocks under varying climatic conditions E Blanck and R Thernitz *Chem Erde* 9, 529-39 (1935) —Pieces of Muschelkalk and Bunter sandstone were exposed to the weather on the Brocken (1142 m) and at Göttingen (140 m) in 1922, and examd in 1927 and 1934, as to the extent of weathering In 1927 (cf C A 22, 4423) no definite inferences could be drawn In 1934 detailed comparison of appearances and complete analyses show that there was little difference between the weathering in the 2 places as regards the sandstone, but that the Muschelkalk suffered more in Göttingen than on the Brocken, a difference attributed mainly to the higher temp prevailing at the former place C A S

Recent and fossil Terra rossa formation in southern Franconian Alps E Blanck and E v Oldershausen *Chem Erde* 10, 1-66 (1935) —Mainly of geological interest, but the paper contains a considerable no of complete analyses of surface soils, underlying rocks, limestones, etc., and of terra rossa mainly from the valley of the Altmühl and neighborhood (30 m N. of Augsburg), showing the relations between the surface soil and mother rock, and effects of weathering and solvent action of water C A S

The chemistry of the weathering process R Schwarz *IX Congr. intern. quim pura aplicada, Madrid* 3, 329-8 (1934); cf C A 29, 78737 —Hydrolysis of alkali or alk earth aluminosilicates under mild conditions will not produce kaolin, because the soln becomes alk  $\text{SiO}_2$  sol is quite stable in alk soln, but  $\text{Al}(\text{OH})_3$  and  $\text{Fe}(\text{OH})_3$  are pptd, and laterite is formed The formation of kaolin must be due to pneumatolytic action It could not be produced from amorphous material, without hydrothermal action, by using salts or inorg or org acids in a measurable length of time C R Rushton

The distribution of the chemical elements in the universe Walter Noddack *IX Congr. intern. quim pura aplicada, Madrid* 3, 327-33 (1934); cf C A 29, 7071 —There is greater uniformity in the compn of meteorites than anywhere on the earth The mineral structure of the meteorites and the fact that even very small ones contain nearly all of the elements in approx the same proportions indicate that they condensed from the gas phase The smaller meteorites, because of their lesser gravitational fields, could not hold the more volatile elements, so there is very little Hg, N or rare gases in them, and the same elements are lacking in the spectra of the fixed stars C R Rushton

Determination of the thorium content of rocks Wm D Urry *J Chem Physics* 4, 34-40 (1936) —A new method for the detn of  $10^{-4}$  to  $10^{-2}$  g Th is described It is used to complete the data necessary for the calcn of the age of geological material The  $\alpha$ -particle activity of Th in a streaming gas is measured with a counter this procedure is more accurate than the usual ionization measurements on the Th deposit Simultaneously the Pa/Ra ratio in common rocks can be detd G M P

Formation of diamond from geologic point of view (Veres) 2 Cathodic phosphorescence for examn of minerals and thin sections of rocks (Urban) 3 Physicochem analysis of the Mondovio Luriso mineral waters—presence of Li in besmaudite (Francesconi, Bruna) 14 S bacteria in the "pink" waters of the Surukhani oil fields and their significance in the geochemistry of water (Malushev, Malian) 14

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## 9—METALLURGY AND METALLOGRAPHY

D. J. DEMOREST, OSCAR E. HARDER AND RICHARD RIMBACH

Geochemistry and the theory of metallurgy. A. F. Kapustin, *Sotsialist Rekonstruktsiya i Nauka* 1935, No. 5, 22-43.—An analogy is drawn between Tamman and Goldschmidt's theory of the structure of the earth's crust and conditions in a metallurgical furnace in regard to disson equal and the affinity of metals toward S and O. The equal between ZnS and gaseous HCl was at const. pressure, and curves of concn.-temp. for different isobars were obtained. It should be possible to fortify the behavior of a compd. in metallurgical treatment from its behavior in soln. B. V. Shvartzberg.

A note on the use of ultraviolet lamps in mines for rapid detection of scheelite in ores by fluorescence. Wm. O. Vandenberg, *Bur. Mines, Information Circ. No. 6873*, 3 pp. (1935). Alden H. Emery.

The San Antonio mine and mill. Anon. *Trans. Can. Inst. Mining Met.*, 1936 (in *Can. Mining Met. Bull. No. 285*) 1-9.—The cyanide mill is described. A. H. E.

Nickel progress during the last decade. John F. Thompson, *Rev. Met.* 32, 509-17 (1935).—A survey of recent advances. At the Frood mine in the Sudbury district the ore is ground and subjected to flotation. Gang is eliminated and Cu-rich and Ni-rich portions are sepd. from each other. The Ni-rich portion is roasted in Herreshoff furnaces placed above reverberatories into which hot calcines are fed. Reverberatories are run to produce a 25% mat and a slag with 35% SiO<sub>2</sub>. The mat is blown in Pierce-Smith converters to a compn. of Cu 42%, Ni 37% and S 20%. Converter slags return to the reverberatories. The "tops and bottoms process" (Orford method) depends on the soln. of CuS in Na<sub>2</sub>S, which is obtained by roasting Na<sub>2</sub>SO<sub>4</sub> with C and mat. After melting, the dissolved CuS floats on the top of the Na<sub>2</sub>S and is sepd. from it after cooling. Bottoms are crushed, washed and sintered. This sinter is melted with C and is cast into anodes. Anodes are placed in electrolytic cells inserted in cloth bags which serve as membranes. Pure Ni soln. contg. only Ni-SO<sub>4</sub> and boric acid is pumped into them. On leaving the cells the soln. is free from Cu by addn. of Ni powder and Fe is removed by converting it into Fe(OH)<sub>3</sub> and filtering off. At Clydach (Wales) the Mond process is applied to roasted and reduced bottoms furnishing 99.9% Ni free from Co. Statistical data on the uses of Ni are given. Forty-seven references. J. D. G.

Modern uses of nonferrous metals. C. H. Mathewson, *Am. Inst. Mining Met. Engrs.*, 1935. The new industrial development and exploitation of metals and alloys. Zay Jeffries, 1-15, cf. C. A. 29, 429. Aluminum. Francis C. Frary, 16-41. Arsenic and antimony. K. C. Li, 42-53. Bismuth. W. C. Smith, 54-64. Chromium. Frederick M. Becket, 65-93. Cobalt. B. E. Field and W. A. Wistler, 94-106. Copper. C. H. Mathewson, 107-37. Gold. F. E. Carier, 138-49. Lead. J. O. Betterton, 150-69. Magnesium. John A. Gann, 170-90. Miscellaneous alloy-forming elements. beryllium, calcium, cerium, lithium, manganese, titanium, vanadium, zirconium. J. F. Harris, 191-216. Molybdenum. W. H. Phillips, 217-28. Nickel. Paul D. Merica, 229-69. The platinum metals. I. M. Wise, 270-93, cf. C. A. 29, 1041. Quicksilver. C. N. Schuette, 294-306. Selenium and tellurium. W. E. Milligan, 307-25. Silver. Robert H. Leach, 326-42. Tantalum and columbium. J. W. Fingle, 347-55. Tin. D. J. Macnaughtan, 356-75. Tungsten. W. P. Sykes, 376-88. Zinc and cadmium. W. M. Peirce, 389-415. Alden H. Emery.

A study of the occurrence and amenability to leaching of the phosphorus compounds in some red iron ores of Alabama. Ill. S. Hertzog, *Bur. Mines, Rept. of Investigations No. 3294*, 9 pp. (1935).—Most of the P in these ores occurs as Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> mixed with small quantities of ferrous, ferric and Al phosphate. Thoroughly leached outcrop ores usually are low in P compared with unleached ores. Acid solns. are the best solvents for Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>. The acid

consumption increases with increased CaCO<sub>3</sub> content. The P in ores in which the CaCO<sub>3</sub> content is small is not removed by weak acid solns. FePO<sub>4</sub> is removed by strong acid solns., which also dissolve appreciable amts. of the Fe ore. A cheap reagent is essential. It must not introduce any objectionable element into the ore. The removal of P from the red Fe ores of Ala. by leaching does not appear to be commercially feasible. A. H. E.

Historical notes on the Paho process. T. A. Rickard, *Trans. Can. Inst. Mining Met.* 1936 (in *Can. Mining Met. Bull. No. 285*) 23-48.—NaCl, CuSO<sub>4</sub> and Hg are added at widely separated intervals to Ag ore, the whole process takes 24 to 30 days. Ag extn. is 80-85%. A. H. E.

Flotation at Government Gold Mining Areas. Anon. *S. African Mining Eng. J.* 46, 339 (1935).—Fagergren flotation machines following corduroy tables are to be used to sep. the pyrite for sep. grinding and cyanidation. Alden H. Emery.

Metallurgy of gold and platinum among the pre-Columbian Indians. Paul Bergsøe, *Nature* 137, 29 (1936).

E. H. Metallurgical stoppers and nozzles. A. M. Trahtenberg and I. A. Tatarskii, *Ogneupor* 3, 648-54 (1935).—Nozzles with magnesite inserts must be used for casting boiling metal and without them for quiet metal. The latter nozzles must be fired at over 1400° and must have a max. porosity of 23% and an alumina content of 34%. Nozzles with inserts can be produced from a batch contg. less alumina, but not under 31-2%. The porosity can attain 28%, the firing is satisfactory at 1300°. Stoppers must be fired at about 1350° and possess a max. porosity of 23%. It is recommended to connect the stopper with the shank with the aid of a bolt turning round 90°.

E. F. Stefanowsky. Qualities of pig iron. Round table. Ralph H. Sweetser, *et al.* *Am. Inst. Mining Met. Engrs., Tech. Pub. No. 669*, 16 pp. (1935). Alden H. Emery.

The influence of low-graphite and low-silicon pig iron on the mechanical properties of gray cast iron at elevated temperatures. Max Paschke and Friedrich Bischof, *Gieserei* 22, 625-7 (1935), cf. C. A. 30, 1004.—Four exptl. melts were prepd. from the following materials: a special pig iron with high C and low graphite and Si, hematite pig iron, steel scrap, FeSi and FeMn. As the special iron was increased in the mix, the hematite pig iron was decreased and the steel scrap was kept practically const. so that the resulting analyses of the 4 melts were similar. Any noticeable changes in properties therefore measures the influence of the low-graphite low-Si pig iron. Results show that with an increasing amt. of the special pig iron the strength increased at elevated temps.

C. B. Jeuni. Various phases of activity in iron and steel reviewed. Introduction. A. B. Kinzel, *Mining Met.* 17, 49 (1936); cf. C. A. 29, 3956. Development and use of alloy steels rapidly expanding. *Ibid.* 49-50.—In the Cr steels, Si or Mn is used as the modifying agent; in the Ni steels, Mo is used, in the Mn steels, V. In engineering steels, the development of modified types of the S. A. E. group is notable. Grain-size control specifications are being accepted; this control is achieved through use of Al, Zr or V. More attention given to the product of the iron blast furnace. Ralph H. Sweetser, *Ibid.* 50-1. Temperature problems engage open-hearth operators—bureaucracy only fear of steelmen. L. F. Renartz, *Ibid.* 51-2.—Thermocouples are being installed in the roofs of open-hearth furnaces. Increased roof life and more tons per hr. have been reported. Further development of open-hearth efficiency depends on high temps. in the hearth obtainable only if better refractory brick are produced. Pros and cons of furnace insulation are discussed. Pressures at which natural gas is burned are being increased; a small amt. of fuel oil may be used to increase luminosity and thus speed heat transfer. Bessemer steelmaking is reviving. G. B.



Waterhouse *Ibid* 52-3—Improvements have been made in the properties of free-machining steel used for screw stock and for use in automatic or semi-automatic machines. Great progress in x-ray examination for non-destructive testing. A. V. de Forest *Ibid* 53-4—Developments in nondestructive testing are reviewed, including x-rays, radiography, Sperry test, magnetic analysis, sonic testing, Magnallux. Grain-size control an important development in steel metallurgy. F. B. Foley, *Ibid* 54-5—Recent studies in control and effect of grain size are reviewed. Casting of camshafts and crankshafts an interesting development. James T. MacKenzie *Ibid* 55—The most outstanding development in Fe and steel foundries has been the casting of camshafts from cast Fe and crankshafts from graphitic steel. Cu seems slated to replace Ni to a considerable extent in castings. Hot- and cold rolling of wide thin sheets greatly advanced. Anon. 55-6. Alden H. Lmery

Steelmaking in 1935. Emil Githmann. *Blast Furnace Steel Plant* 24, 91, 99(1936). E. H. Some problems of steel melting. Earl C. Smith. *Trans Am Inst Mining Met Engrs* 116, 13-25(1935). Chief attention is given to refractories and slags.

Alden H. Lmery. Metallurgy of the induction furnace and the production of tool steel. S. L. Robertson. *Metal Treatment* 1, 170-85(1935).—A review. E. H.

Oil fired assay furnace—experience shows improvement possible. G. Spencer Compton and C. McHarty. *Chem Eng. Mining Rev* 28, 53-81(1935).—Three oil-fired furnaces employed, resp. at the Kalgurli School of Mines, the North Kalgurli United Mines and the lab. of the Western Mining Corp. are described. At the North Kalgurli mines the furnace is a combination cupelling and fusion one with low-pressure burner using crude oil. The usual slot in the back of the muffle is blocked up and a piece of refractory Carbolux piping cemented over a circular hole  $1\frac{1}{4}$  in. in diam. in the top of the muffle and placed as far back as possible. This pipe projects to the outside (top) of the furnace a height of 6 in. The improvement is effected by fitting a piece of seamed black-iron piping over the vertical refractory pipe. This black-iron pipe is 6 ft. long and has fitted to it a simple butterfly throttle by which the upcast draft can be regulated. This lowers cupellation time about 20 min. W. H. B.

The open hearth in 1935. K. C. McCutcheon. *Blast Furnace Steel Plant* 24, 47, 50(1936). E. H.

Steel-melting furnaces—automatic control in the open hearth. A. J. Boynton. *Iron Steel Engr* 13, 25-6 (Jan, 1936), cf. *C. A.* 29, 3271<sup>8</sup>.—Automatic control results in a decrease in fuel consumption ranging from 7 to 11%, and a notable increase in rate of production.

W. H. Boynton. Open hearth furnace operation—four million B. t. u. per ton. L. H. Schwartz and G. E. Rose. *Iron Steel Engr* 13, 18(Jan 1936).—A large no. of runs and a careful study of results indicate the great advantage to be gained in open hearth practice by the transfer of heat by radiation from the flame, and conduction and convection are relatively ineffective as compared with radiation from the flame under these conditions. In the second place, in regenerated mixed gas (coke-oven and blast-furnace gas) there is a continuous supply of open hearth fuel that is borne down heavily with C particles and that can be regulated as to richness in an ideal manner as required for the different periods of the open hearth heat. Thirdly, since open hearth furnaces must be built of refractory materials, they are necessarily more or less porous and, hence, if air infiltration is to be kept at a min. the subatm. conditions must be kept at a min. in an open-hearth furnace just as in a battery of by-product coke ovens. The transfer of heat by radiation requires a more or less static situation and shows greater effectiveness upon the bath when it is under cover of the slag.

W. H. Boynton. Waste-heat boilers in open hearth practice. R. Percival Smith, et al. *J. Iron Steel Inst. (London)* Special report No. 10, 73 pp (1935). *Blast Furnace Steel Plant* 23,

7 108-10, 719, 780-3, 800, 850-60, 862, 876(1935), 24 87-9 (1936).—See *C. A.* 30, 412<sup>7</sup>. A discussion on the report on "Waste-heat boilers in open-hearth practice." A. F. Webber, et al. *J. West Scot. Iron Steel Inst.* 43, 3 12 (1935).—The construction and advantages of the Spenser thimble tube boiler and of the Smaullo fire tube boiler are given. The use of a water tube boiler is compared to that of a fire tube boiler. A properly designed economizer will lower the temp. 55° to 80°. The results of the Park C. test are not representative of ideal conditions because the gas temp. and flow were less than the boiler was designed for and because the producer gas was not of the most desirable composition especially with respect to moisture content (38.7 grains per cu ft.). The results are representative of general experience, however, because frequently the heat available, the steam generated, and the draft-control requirements are less than estd. before the installation. F. G. N.

Service of silica brick in open hearth furnaces of the Petrovskii plant (at Dnepropetrovsk). V. Trubnikov. *Ozgonpur* 3, 492-8(1935).—Silica brick in the roofs of open-hearth furnaces were more durable in 1934 than in 1933 (up to 366 smeltings against 248 in a 100-ton furnace). This is due principally to improvements in the work of the furnace. The repairs undertaken in the furnace and phys. and chem. changes in silica brick from its roof are described. E. B. Stefanowsky

Suspended, unfired, reinforced roofs for open hearth and electric furnaces. I. S. Kainarskii, B. Ya. Pines and S. Ya. Kozlov. *Ozgonpur* 3, 661-6(1935).—Roofs of metal-cased magnesite-chromite blocks, after a run of 62 hrs., were in satisfactory condition, thus it is possible to select appropriate basic and neutral materials for the purpose. E. E. Stefanowsky

Progress at the blast furnace in '35. A. J. Boynton. *Blast Furnace Steel Plant* 24, 55-6(1936); cf. *C. A.* 29, 780<sup>7</sup>. E. H.

Some engineering aspects of the first report of the blast furnace practice sub-committee. Thomas B. Mackenzie. *J. West of Scot. Iron and Steel Inst.* 43, Pt. 2, 19-49 (1935).—Pertinent data concerning the output and furnace dimensions are presented for each of 15 basic practice furnaces, 13 hematite practice furnaces, and 14 foundry practice furnaces. One furnace is operated by each practice. When making foundry iron its output is 95% and when making hematite Fe is 93.3% of the output when making basic Fe. The coke consumption for foundry Fe is 110.1% and for hematite Fe is 115.5% of that for basic Fe. The coke consumption per sq. ft. of hearth is 25-0.06D<sup>2</sup> for basic practice, 25-0.067D<sup>2</sup> for hematite practice, and 25-0.032D<sup>2</sup> for foundry practice. D is the hearth diam. in ft. Description of the equipment of the basic furnaces includes hot blast stoves, means of cooling the bosh wall, of charging the furnace, of providing the blast, and the vol. of blast. Formulas that represent British practice relate the important furnace dimensions to the hearth diam. and to the tons per hr. production. F. G. Norris

Production and preparation of blast furnace flux. F. C. Hodges. *Am Inst Mining Met. Engrs Tech. Pub.* No. 668, 11 pp (1935). Alden H. Lmery

Metals and alloys in the chemical industry. I. Introduction and theory. Frederick A. Rohrmann. *J. Chem. Education* 13, 53 5(1936). II. Ferrous metals and alloys. *Ibid* 53-9. E. H.

Uncommon metals and their practical use. R. Freitag. *Warme* 58, 662-3(1935).—F. discusses Be, Li, Ba, Ca, Re, massium, W, Na, Cd, Ti, Hg and the uses to which they have been put. W. H. Bruckner

Atomic arrangement in metals and alloys. W. L. Bragg. *J. Inst. Metals* 56, 275-9(1935).—The theory of rearrangement in the cryst. structure of an alloy is discussed. This arrangement consists of 2 features: (1) the pattern of sites characteristic to each phase, and (2) the method of distribution of the constituent atoms among the phase-sites. X-ray analysis has made possible definite progress in this study. C. B. Jenni. Thermomagnetic analysis of metallurgical products and

its recent applications P. Chevenard. *Mitteil.* 10, 104-20(1935).—A recording thermomagnetometer has been developed in France at the Imphy Steel Works. The principles of thermomagnetic analyses are given. The magnetization field cycle characteristics, magnetization in a max. field, remanence and coercive force, are modified with rise in temp. the hysteresis falls and the magnetization decreases following the Langevin-Weiss law and becomes very weak at the Curie transformation point, at which the metal becomes paramagnetic. In a series of alloys, the variations of these characteristics with the compn. depend to a great extent on the constitution. With solid solns, the magnetic hysteresis is generally weak. The magnetization and temp. of the Curie point vary with the concn., following a definite law. In solid solns, formed by Ni only 5% Cu is required to render the alloy paramagnetic at ordinary temps. In aggregates, the Curie point remains at a const temp. Thus for aggregates iron-cementite, steels and white iron, the thermomagnetometer registers the magnetic transformation of cementite at 210°. At 750° the reaction  $\alpha\text{-Fe} + \text{Fe}_3\text{C} \rightleftharpoons \text{austenite}$  is manifested by an almost vertical drop in magnetization; the ferromagnetic  $\alpha\text{-Fe}$  disappears, with formation of paramagnetic austenite. This reaction of 3 phases is invariant, i.e., isothermal. The Faraday-Curie thermomagnetic method was employed, i.e., the magnetization produced by a force which attracts a specimen placed in a nonuniform magnetic field. The thermomagnetometer consists of an expansion pyrometer attached to a magnetic balance. The test specimen in a silica tube is placed parallel to a standard sample (paramagnetic alloy of Ni-Cr-W) which has a regular and reversible expansion. These samples are heated in an elec. furnace and the expansion pyrometer records the change in vol., and with the application of a magnetic field it registers the temp.-magnetization curve. The temp. in the test should not rise more than 300° per hr. Isothermal tests can also be made. Thermomagnetic analysis has been applied in the following cases: (1) Magnetic transformation of a 0.57% C steel. In the annealed state this steel consists of an aggregate of two ferromagnetic phases,  $\alpha\text{-Fe}$  and cementite. (2) Magnetic transformation of Fe-alloys contg. 0.20% N at 480°. This alloy consists of a mixt. of a Fe-N solid soln and Fe<sub>3</sub>N. (3) Reheating of quenched austenitic steels. A steel contg. 1.5% C and 2.25% Cr is in a state of homogeneous austenite. This unstable austenite is destroyed on heating. By means of thermomagnetic and dilatometric methods, the complicated reactions occurring during reheating are identified. Heating to 300° does not change the state of the austenite but when held for 7 hrs. at 360° the alloy becomes partially ferromagnetic. This partial magnetization can be explained by the partial transformation of  $\gamma\text{-Fe}$  into  $\alpha\text{-Fe}$ . (4) Transformation of a Hadfield steel by cold working. After a quench from 1100° the Hadfield steel consists of homogeneous paramagnetic austenite. Cold working renders this steel weakly magnetic, owing to the change austenite  $\rightarrow$  complex carbide + austenite poor in C and Mn. (5) Stabilization by heating a high Ni-Cr steel. (6) Heterogeneity of complex austenites. G. T. Motok.

Dynamic elasticity of metals A. Leblanc. *Mitteil.* 10, 20-13(1935).—The elastic deformation which is produced in a metal subjected to dynamic forces is given the name of dynamic elasticity. Elastic deformation by impact is given by  $\epsilon = 1000 E^2 / 2M = 500 E^2 / M$ , where  $\epsilon$  is the absorption capacity of impact work per cu. dm. of metal,  $M$  is the coeff. of elasticity longitudinal to the metal, and  $E$  is the elastic limit of the metal, all terms expressed in kg./sq. mm. G. T. Motok.

Methods of mechanical testing of metals at low temperatures A. S. Ialkevich. *Zavodskaya Lab.* 4, 1007-1100(1935), cf. C. A. 29, 1302.—The existing methods of mech. testing of steels, brass, bronze and Al alloys in the lab. at temps. below 0° are discussed. Chas. Blanc.

Actual problems in testing methods for light metals. M. Roß. *Aluminium* 17, 631-7(1935).—The present status of technological testing of light metals, the various

methods and their particular purposes are discussed and classified; terms are defined and the prepn. of test specimens is described; their dimensions are given in sketches. It is emphasized that material testing and experience are one indivisible unit; testing of materials in the lab., supervision during production and manu. and experience, made on the finished structure must always be correlated to obtain the greatest benefit for the progress of industry. M. Hartenheim.

Effect of test conditions on shrinkage determination O. Bauer and H. Siegerschmidt. *Metallwirtschaft* 14, 854-8(1935).—The usual method for detg. shrinkage was used, but the length and width of the test pieces, the rate of pouring, and the temp. of the mold were varied. The alloy was 16% Sb, 0.21% graphite and the balance Pb. With equal width the short pieces shrank more than the long ones. With equal length the narrow pieces shrank more than the wide ones. With slow pouring rate the size of the test piece had less influence on the shrinkage than with fast pouring rate. At 210° mold temp. the quickly poured test pieces had higher shrinkage than the slowly poured ones, but at 130° the conditions were reversed. The results are explained by the effect of the test conditions on the  $\alpha$  temp. of the metal in the mold which controls the shrinkage. Photomicrographs of slowly cooled and quenched test pieces showed segregation in the upper part of the mold, especially in the slowly cooled pieces. C. E. Macfarlane.

Untenability of the conception of an upper and lower yield point and of the breaking load of soft steel and other metals G. Welter. *Metallwirtschaft* 14, 1043-7(1935).—The stress-strain curves of soft steel produced automatically by the conventional types of testing machines are not correct, as they show a drop in load when the test piece starts to stretch at the yield point, and another drop in load after the max. has been reached and before the test piece breaks. These drops do not actually take place, but are due to the construction of the testing machines. This contention is illustrated by curves obtained by the use of a testing machine loaded with dead weight, lead shot. These curves show a deviation from the straight line at the yield point for annealed 0.15 C steel, an increase in the rate of elongation, but a slight increased instead of a reduction in the required load. The load approaches a horizontal line at the breaking point, but does not drop. C. E. Macfarlane.

Use of x-ray and microscopic analysis as an aid in rational thermal treatment of brass A. I. F. Nikov and M. M. Umanskii. *Zavodskaya Lab.* 4, 1043-6(1935).—The results of x-ray and microscopic exams of the effect of thermal treatment on the microstructure of 2 samples of brass are illustrated and described. Chas. Blanc.

Magnetic investigations of precipitation hardening Hermann Auer. *Physik. Z.* 36, 880-2(1935).—Magnetic measurements are of use in measuring the influence of various thermal treatments of temperable alloys, as the susceptibility of such alloys drop during transformation, and the fall is linear with log time. The dependence of the discontinuity in the final susceptibility on temp. of quenching allows a magnetic formulation of phase diagrams. From quant. measurements of susceptibility the stoichiometric proportions of elements in stable phases may be detd. The influence of quenching velocity on Al-Cu alloys was studied. C. E. F. Jeffreys.

Methods of hardening cast iron by heat treatment J. S. Vanick. *Metal Progress* 29, No. 1, 40-3(1936).—Chem. compn. must be such as to allow the casting, as cast, to possess fine distribution of graphite, complete freedom from massive carbides or carbide clusters, a tendency to harden evenly and deeply and be relatively easily machinable. Illustrations are given for heat treating typical cast irons. W. A. Mudge.

The use of catalysts in the pickling of metals. M. Peernaux. *Verre silicates ind.* 7, 50-1(1936). H. S. W.

Progress in the knowledge and application of plastic forming processes. E. Seibel. *Metallwirtschaft* 14, 893-7(1935).—A review of cold drawing of rods and wire, deep-drawing, and drawing tubes, covering power requirements,

distribution of forces, effect of die angles and lubrication  
Thirty-eight references C. E. McFarlane

The influence of pressure on the electric resistance of hard drawn gold wire in different stages of annealing and of soft gold wire A. Michels and M. Lennsch *Physica* 2, 591-6 (1935) —The expts were conducted in the manner previously described (C. A. 24, 2649). C. A. 29, 342 —The Au wire at 25° decreased in resistance steadily for 4 months after drawing, after annealing to 50°, 75°, 100°, 150° or 450° the relative resistance at 25° decreased from 1.03712 to 1.00000. Pressure produced a hysteresis effect, the 450° wire after application of 2000 atm at 75° had a permanent resistance increase of 1/42 000, generally, however, the change is much lower. For pressures up to 1942 kg per sq cm the pressure coeff.  $(1/R_0)(\Delta R/\Delta p)$  is about 2700 to 2900 depending on temp; tabulated data are given. The av temp coeff.  $(d/R)(1/R_0)(\Delta R/\Delta p)$  is  $-1.3 \times 10^{-4}$  sq cm per kg per deg abs, the pressure coeff.  $(d/dp)(1/R_0)(\Delta R/\Delta p) = 3 \times 10^{-11}$  cm<sup>4</sup>/kg<sup>3</sup> B. J. C. van der Hoeven

Special methods of gas firing to avoid scale and soft skin Winfield Foster *Metal Progress* 29, No 1, 51-2 (1935) —A comparison of results obtained from heating by coal and by gas W. A. Mudge

Rhodium surfaces for metal reflectors Blaus Bart *Product Eng* 6, 308-9 (1935) —By electrolytically depositing Rh metal on a master mold high optical accuracy (in the order of millionths of an in.) can be obtained in metal reflectors. Rh has a high specular reflection over a wider color range than any other reflector material. It is harder than Ag, non-oxidizing, resistant to heat, unaffected by industrial and chem. fumes, easy to deposit, requires no polishing and is not color selective A. N. H.

Elasticity of aluminum at high temperatures J. Vero *Roy Hong Palatin—Joseph Univ Tech Econ Sci, Sopron, Publ Dept Mining* 17, 163 70 (1935) —A modification of the method of Schwinning and Strobel (C. A. 26, 5524-5) was worked in which the elongations caused by the creeping phenomenon are determined more precisely. The values are 5480-5750 at 150°, 4650-4775 at 240° and 3700 at 340° for the elasticity modulus in kg/sq mm S. S. de Finily

Iron of high purity Frank Adcock and Charles A. Bristow *Proc Roy Soc (London)* A153, 172 200 (1935) —Measurements of phys. properties, made on specimens of pure Fe from 5 batches with known analysis, gave: length of side of body-centered cube,  $2.8700 \pm 0.0005$  Å at 17°,  $d_1$ ,  $7.871 \pm 0.002$ , coeff of expansion  $(\alpha \times 10^6)$  at the temps, 50°, 75°, 100°, 125° and 150°, resp., 11.2, 11.6, 12.0, 12.4 and 12.8, sp elec resistivity,  $R$ , for the temp range, 0-200°,  $R_0 = (8.9 \pm 0.051) + 0.00006 \theta \times 10^{-4}$  thermal cond. (in cgs units) at the temps, 0°, 25°, 50°, 75°, 100°, 125°, 150°, 175° and 200°, resp. (0.16), 0.18, 0.18, 0.18, 0.17, 0.17, 0.16, (0.16) and (0.16). The elec resistivity of 2 earlier (less pure) batches was slightly lower than for the higher purity batch given above. The values for the thermal cond. are higher than those usually attributed to Fe. Other factors than purity cause variation in magnetic properties, the purest Fe did not possess the highest magnetic permeability. The  $\alpha$ - $\gamma$  transformation did not occur at a definite temp but occupied a definite temp range, difficult to explain in terms of possible impurities present. The transformation is apparently not a simple phase change L. E. Steiner

The orientation of crystals in silicon iron Richard M. Bozorth *Trans Am Soc Metals* 23, 1107-11 (1935) —X-ray examn of Si iron prep'd by Goss (cf C. A. 29, 5793), shows that the component crystals are oriented so that a [100] direction is parallel to the direction of rolling and a (110) plane lies in the rolling plane. The differences in magnetic properties in different directions in the sheet are explained in terms of the properties of the single crystals W. A. Mudge

Graphitization of cast iron A. Le Thomas *Rev universelle mines* 11, 553-4 (1935) —Tests made by Le T. agree with results published by Portevin (C. A. 24, 1828), 300° must be assumed as that practical temp be-

low which castings do not undergo any further measurable change by slow graphitization Eleven references

M. Hartenbom  
The diagram iron-carbon silicon A. Dawans *Rev universelle mines* 11, 541-32 (1935) —The binary diagrams I-Fe, Fe-Si and Si-C are explained and from these the ternary diagram Fe-C-Si is derived and discussed from the particular point of the practical foundry and steel worker. Twelve references M. Hartenbom

Gases in steel T. Swinden *Iron & Steel Ind*, 9 100-6 (1935) —A review P. G. Norris

The structure method of examination of steel heterogeneity V. D. Sadovskii *Zavodskaya Lab* 4, 103-4 (1935), cf C. A. 28, 3698 Chas. Blane

The detection of oxide inclusions in steels by imprint R. Mitsche *Berg- und Hütten-männisches Jahrbuch* 11 127 33 (1935) —The practical importance of determining nonmetallic inclusions in steel is explained and the method of Niessner (cf C. A. 26, 5871) described which permits making photographic prints on gelatin paper of no oxide inclusions. The paper is impregnated with a sol of 1 HCl in 20 H<sub>2</sub>O, the polished section is then pressed onto the paper and the latter developed in a ferrocyanide soln (20 g in 1 l water). The influence of the kind of gelatin paper, developing conditions and least magnitude of inclusions which can be detected are discussed. For references M. Hartenbom

Snow flakes in forged steel Leon Gullet and Marc Ballay *Rev met* 32, 522-30 (1935) —A crit survey of the contemporary views on the nature of snow flakes in steel and on the methods for their elimination. Nineteen references J. D. G.

Formation of flakes in chromium nickel molybdenum steels I. Musatti and A. Reggioni *Rev met* 3 531-52 (1935) —Steels contg 0.30-0.40% C, 0.40-0.6% Mn, 0.30 Si, 0.6-0.8 Cr, 2.7-3.2 Ni and 0.30-0.40% Mo have a strong tendency toward flake formation. Max heats of this analysis investigated showed independence of the defect from nonmetallic inclusions, liquation segregations in ingots and dendritic segregations. Theory concerning flake formation with internal stresses caused by austenite transformations was refuted by application of heat treatments preventing this change and the failure to cure the trouble. Expts of H. Benek, I. Schenck and H. Mueller, C. A. 29, 3957, showing the relation between flake formation and H were amplified by heating flake free specimens in H<sub>2</sub>. After heating 5-25 hrs at 1150° all samples contained flakes located radially around their periphery at a certain distance from the surface. Similar heating in N did not produce any flake. Amt and size of the flakes after the same treatment increased in the following order: C steels had the least amt, then Ni-Cr steels and the worst flaking was observed in Ni-Cr-Mo alloys. Heating in H<sub>2</sub> at lower temp decreases the intensity of the action. Under 800° no flake formation was observed. Thirty-nine references J. D. G.

The aging of steel L. S. Davenport and E. C. Bain *Trans Am Soc Metals* 23, 1047-93 (1935) —Two kinds of aging are described, (A) C aging and (B) strain-aging. (A) is caused by pptn of 1 c carbide from ferrite super-sat'd with C, pptn is in a random manner throughout the grains. (B) is caused by the rejection of an Fe-C comp'd, from ferrite seemingly supersat'd with C, in the slip bands of cold worked grains W. A. Mudge

Grain size and its influence on the manufacture of steel wire B. L. McCarthy *Iron Age* 136, No 15, 14-19, No 16, 23 7, 80-2 (1935) —Choice of steel for making wire begins at the open hearth and includes consideration of the compn of the charge, the C content at melt, the rate of C elimination from the bath, the compn and character of the slag and the final deoxidation of the metal. In absence of an Al addn, the McQuaid-Chen test (C. A. 16, 1730) indicates the amt of oxides dissolved in the metal. The grain size of fine-grained steel is controlled by the judicious addn of Al in the ladle to steel that has been deoxidized in the furnace with Mn and Si. The grain size of coarse-grained steel is not subject to such close

control. The distribution of cold work depends on the microstructure and should be as nearly uniform as possible. The microstructure is developed during patenting. Coarse-grained steels have a slow rate of the decomn of austenite at Ar<sub>1</sub> and fine-grained steels a fast reaction rate. Dissolved oxides and fine grains may retard the rate of soln of the constituents. Coarse-grained steel is desirable for the manu of high-C wire because of the resulting coarse patented grain size. McQuaid-Tin tests were made on 3 samples from the same heat. The coarse-grained steel to which no Al was added in the mold has no 4 grain after carburizing at 870° and no 3 and 1 at 925° and 985°, resp. The fine-grained steel to which 6.5 oz Al per ton was added in the mold had no 6, 4 and 2 grain after carburizing at 925°, 985° and 1040°, resp. The fine-grained steel to which 13 oz Al per ton was added in the molds had no 7, 6 and 5 grain size after carburizing at 925°, 985° and 1040°, resp.

F. G. Norris. **Contributory effects of furnace atmospheres on the grain size of molybdenum high-speed steel.** Arthur Phillips and M. J. Weldon *Trans Am Soc Metals*, 23, 880-401(1935).—A steel contg Mo 7.03, Cr 3.93, V 2.05 and C 0.77% was heated in oxidizing, reducing and neutral atms and the resulting effect upon grain-size, temp of incipient melting and extent of decarburization detd. The most pronounced structural alterations are produced by oxidizing atms., particularly those contg appreciable amts of H<sub>2</sub>O vapor. W. A. Mudge.

**Correlation of failures from embrittlement of 4 to 6% chromium steel with the notched-bar impact test.** H. M. Wilten. *Trans Am Soc Metals*, 23, 915-38(1935).—Lab. temper brittleness tests can be used to predict service failures in refinery cracking stills. W. A. Mudge. **Construction steels.** L. Persoz *Metalux* 10, 216-42 (1935).—A survey is given of phys. testing, constitution of steels, heat treatments and classifications of steels. All the common phys. testing and examn. methods employed in control of construction steels are discussed.

G. T. Motok. **Endurance limit of welded boiler plate in the unannealed and annealed condition.** C. Appaly and F. Sauerwald. *Metallwirtschaft* 14, 858-61(1935).—The welded 0.09 C boiler plate was tested as welded, after annealing at 930° and after stress relieving at 650°. The endurance tests were made at 400°. The unannealed welded steel had an endurance limit of 10 to 11 kg./sq. mm. compared with 9 for the parent metal. The endurance limit and microstructure of the parent metal were not affected by the annealing and stress-relieving treatments. The 930° treatment reduced the endurance limit of the welded steel to that of the parent metal, and increased the grain size to approx. that of the parent metal. The 650° treatment reduced the endurance limit to a point half way between the welded and parent metal, while the grain size remained unchanged. The higher endurance limit of welded boiler plate is due partly to finer grain size and partly to internal strains. Some of the tests were made both by the usual long-time method and by the shortened method of Juretzek and Sauerwald (C. A. 29, 2592). Good agreement was obtained by the 2 methods. Six references. C. E. Macfarlane.

**Experience with higher-pressure steam installations.** Otto Schöne. *Warme* 58, 765-74(1935).—Part of the paper deals with the use of welded alloy steels suitable for superheater tubes above 400°. Micrographs are shown of structure of weld and transition zone of the various steels discussed. The corrosion attack on and scale deposits on steam turbine blades are discussed and shown in photographs. W. H. Bruckner.

**Modern materials for high-pressure vessels.** L. P. McAllister. *Welding Engng* 20, No. 5, 30-2(1935).—Tables are shown giving the chem. and phys. properties of Ni steels, Mn-V steels and Cromansil steel. A. N. H.

**Materials for high temperature steam piping.** Wilhelm Paul. *Warme* 58, 839-43(1935).—A discussion of C steels as compared with alloy steels with respect to tensile strength, ductility and creep limit at 400-500°. Tables are given of tensile strength, ductility and endurance limit

at intervals of temp. from 20° to 600° and charts of tensile strength, creep, endurance and improvement factor as compared with C steel, for 0.5% addns. of Ni, Co, Mn, Ti, Si, Cr, Cu, W, Mo and V. The latter charts are for 400° and 500°. The increase in cost of each steel due to the 0.5% alloy addn. is tabulated in comparison with the 0.1% C steel. Another table gives the endurance limit of various steels with 1 to 3 alloy addns. of up to 1% each and compares the improvement of the alloy steels with a 0.1 C-0.5 Mn steel. W. H. B.

**Chromium steels of high nitrogen content.** Russell Franks. *Trans Am Soc Metals* 23, 968-87(1935).—Grain refinement is due to Cr nitride functioning to promote austenite formation, or similar to that of carbides in ordinary steel. Addns. of N<sub>2</sub> are preferably recommended for low-C steels because N<sub>2</sub> will not correct the harmful effect of C in high-Cr steels. W. A. Mudge.

**Improved fabrication of 18-8 chromium steels.** Donald R. Pratt. *Welding J* (N. Y.) 14, No. 12, 16-19(1935).—Discussion of fabrication by gas welding with Cb stabilized plate and rod. Ti and Cb are considered equally effective in plate in overcoming intergranular attack, but Cb for welding rod does not burn out as readily as Ti and welds therefore have greater corrosion resistance because of larger stabilizer recovery. Cb is recommended also for welding ordinary unstabilized steels because of counteracting effect of Cb or C pickup. Where it is necessary to cross welds, the Cb bearing rod deposit is protected against heat effect of the crossing weld. In welding stabilized 18-8 use of neutral flame with flux painted on rod or seam and use of flux point on underside of weld are recommended. The weld should not be puddled because of excessive loss of Cb from such procedure. Other precautions in welding technique are given. W. H. Bruckner.

**The importance of aluminum additions in modern commercial steels.** Harry W. McQuaid. *Trans Am Soc Metals* 23, 797-838(1935).—See C. A. 30, 65°. W. A. Mudge.

**Hardening characteristics of one percent carbon tool steels.** T. G. Digges and Louis Jordan. *Trans Am Soc Metals* 23, 839-56(1935).—Com. 1% C tool steels of foreign and domestic manufacturers were prepd with 3 different initial structures, viz., spheroidized cementite, coarse pearlite and sorbite. The effect of these initial conditions upon the austenitic grain-size and grain-growth characteristics was detd. between 1425° and 1775°F. For quenching temps. below that at which all C is completely dissolved in the austenite the initial structure exerts a definite effect. Above this temp. each steel approached both a grain size and crit. cooling rate which was characteristic of the steel regardless of its initial structure. W. A. Mudge.

**Interpretation of torsion impact properties of carbon tool steel.** G. V. Luerssen and O. V. Greene. *Trans Am Soc Metals* 23, 801-77(1935).—By use of a 1.08% C steel and correlating torsion impact with other phys. properties it is shown that the 3 stages in the impact curve coincide with the 3 universally recognized structural changes traversed in tempering. These are (1) stress relief in the martensite, (2) the transformation of retained austenite to martensite and (3) the decomn. of martensite to the softer, more ductile constituents. W. A. Mudge.

**Influence of carbon content on high-temperature properties of steels.** A. F. White, C. L. Clark and R. L. Wilson. *Trans Am Soc Metals* 23, 995-1015(1935).—Short time tensile, creep and impact tests at temps. from 85° to 1400°F. with supplementary microscopic examn., show that the behavior of steel at elevated temps. is sometimes independent of the C content and may be affected by initial heat treatments or the addn. of alloying elements that would change the character of the carbides in the structure. W. A. Mudge.

**Influence of common alloying elements on properties of steel.** Edgar C. Bam. *Rev. met.* 32, 553-61(1935).—Alloying elements change only speed and intensity of the reactions found in common C steel and their temp. Factors defining the final properties of C steel are the same as

those for alloy steel, but the latter has certain limitations which are eliminated by the addn. of alloys. They dissolve in austenite and affect profoundly speed and type of transformations occurring during the final cooling. The lower the temp. at which transformations actually occur, the finer is the dispersion of carbide particles and the harder is the metal. Steel must have a certain crit. speed of cooling to carry it through higher temp. ranges of transformation to the temp. of about 150° at which martensite is formed. All alloying elements which dissolved in austenite, with a possible exception of Co, reduce this speed and permit quenching to penetrate deeper in the metal. Alloying elements refine austenitic grain either by forming fine oxides in melting and deoxidation or by forming carbides which are insol. in the matrix. Size of austenitic grain inversely affects phys. properties of steel. Deep quenching is produced here either by adding other alloys or by dissolving some of the pptd. carbides such as Mo. Stability of these carbides at higher temp. has a pronounced effect on grain size and speed of transformation. J. D. G.

**Alloy steels and ferro alloys in 1935.** James C. Vignos. *Blast Furnace Steel Plant* 24, 57-8 (1936), cf. *C. A.* 29, 1702. E. H.

**Laboratory methods of preparation and testing of high-melting alloys.** L. I. Kramp and M. A. Yur'ev. *Zavodskaya Lab.* 4, 1090-2 (1935).—The literature on the prepn. and examn. of various carbides, nitrides and borides is discussed. About 15 references. Chas. Blanc.

**Special apparatus used for testing aluminum alloys.** E. John Pike. *Metallurgia* 13, 35-7 (1935).—Special testing equipment is described and results of tests on several Al alloys are given. J. L. Gregg.

**Fatigue strength of aluminum alloys.** R. Immann. *Aluminium* 17, 639-43 (1935).—The usual defn. of fatigue strength is made on the Schenck machine with polished specimens. The fatigue strength is influenced by the structure of the alloy (heterogeneous or homogeneous), by the mfg. process (casting, rolling, heat treatment) and by notch effects (due to casting skin, inclusions, segregations, lesions of the surface, oxidation). These effects are discussed for different Al alloys, and shown in diagrams and photographs of structures. The fatigue strength is defined as that load in kg./sq. mm. which the material will stand under an alternating load of  $100 \times 10^6$  alternations for Al wrought alloys, and  $20 \times 10^6$  alternations for Al cast alloys. M. Hartenheum.

**Hot shortness of aluminum alloys in die castings.** J. Vero. *Roy. Hung. Palatin Joseph Univ. Tech. Econ. Sci., Sopron, Pub. Dept. Mining Met.* 7, 138-42 (1935).—Hot-shortness is defined as the tendency of an alloy to form cracks during cooling in the mold. It is proposed that the extent of hot-shortness be expressed numerically by the frequency of cracks occurring in castings of simple shape, given as a percentage of the cracked samples to all castings. In alloys contg. more than 12-13% of liquid at the temp. of the critical paste state possible cracks can be prevented. Data on strength and load are not always sufficient to est. hot-shortness of an alloy. The influence of temp., wt. and cooling factor of the mold and of pouring temp., etc., is discussed and possibilities of avoiding cracks are shown. S. S. de Fmily.

**Crystal structure of AuBe<sub>2</sub> and PdBe<sub>2</sub> and its relation to the cubic AB<sub>2</sub> structure.** Lore Misch. *Metallwirtschaft* 14, 897-9 (1935).—The intermetallic compds. AuBe<sub>2</sub> and PdBe<sub>2</sub> were prep'd by melting in an elec. furnace, and were exam'd by x-rays. Both have a face-centered cubic lattice with 24 atoms in the elementary cell. The lattice const. for AuBe<sub>2</sub> is 6.085 Å and for PdBe<sub>2</sub> is 5.982 Å. The lattice structure is not quite the same as the typical cubic AB<sub>2</sub> structure, such as that of MgCu<sub>2</sub>, but corresponds to that of ZnS. C. E. M.

**Beryllium iron alloys.** W. Kroll. *Metals & Alloys* 7, 24-7 (1936). D. S.

**Beryllium-copper—established properties and proved applications.** Robert W. Carson. *Product Eng.* 6, 175-7 (1935).—Photographs indicate a variety of applications. Hardness, fatigue strength, cond., ductility and stability

are controlled by heat treatment and cold work in fabrication. Ann. Nicholson. Hurd.

**Copper-lead bearings.** D. E. Anderson. *Product Eng.* 6, 363-4 (1935).—A discussion with micrographs. Ann. Nicholson. Hurd.

**The transformation in the copper-gold alloy Cu<sub>3</sub>Au.** C. Sykes and H. Evans. *J. Inst. Metals* 58, Advance copy No. 716, 29 pp. (1935).—The effect of the transformation in the alloy Cu<sub>3</sub>Au on the elec. resistance, x-ray structure and  $\alpha$  heat was investigated experimentally. It is shown that the general character of the transformation is satisfactorily predicted by the Bragg-Williams theory of the formation of superlattices by rearrangement. This process involves first the formation of small nuclei which grow to a size approximating that of the individual crystals, if const. temp. is maintained. If the alloy is continuously cooled before it attains equil., nuclei tend to grow and at the same time the degree of order in the nuclei increases because of the reduction of temp. C. B. Jenni.

**Röntgenographic and electrical investigations of the Cu-Au system.** C. H. Johansson and J. O. Lind. *Ann. Physik* 25, 1-48 (1936), cf. *C. A.* 20, 1154, 21, 2204.—In the compn. range 47-53 atoms % Au there is formed ordinarily the known tetragonal compd. CuAu (I). Under specified conditions in the ranges 36-47, and 53-65, and even 47-53% Au, there may be formed an ordered orthorhombic phase CuAu (II), as shown by superstructure lines on x-ray powder photographs. II is closely related to I and can be derived from I by increasing the b-period of I by an integral factor such as 10, the a- and c-periods remaining unchanged. The detailed structure analysis of II is given. Lattice const. and specific resistances of Cu-Au alloys over the whole compn. range and after varying heat treatments are reported. Complete literature index of recent work on this system. C. D. West.

**The various copper-nickel alloys.** Joseph Dhavernas. *Culture et l'atlon* 8, 541-3 (1935).—The alloys of Ni with Cu, Zn, bronzes and light metals are briefly reviewed. Cu-Ni alloys are now widely used as fire tubes and condenser tubes, the alloy with 20-30% Ni possesses a particularly high resistance to corrosion by seawater. The alloy with 45% Ni, "constantan," has a high thermoelec. force and a fairly const. elec. resistance at varying temps. Alloys of Cu-Ni 7n are the so-called German silvers with 15 or less % Ni and 45-55% Cu. Ni added to Sn bronzes gives a much finer grain and improves the tensile strength. All alloys with up to 4% Ni addn. show increased hardness and strength. A heat-resistant alloy ("V") is composed of Al 92.5, Cu 4.0, Mg 1.5 and Ni 2%. Another light metal used for engine parts in combustion motors is a complex Al alloy contg. Ni, Cu, Mg, Fe, Ti and Si. M. Hartenheum.

**The copper-zinc diagram.** J. Schramm. *Metallwirtschaft* 14, 995-1001, 1047-50 (1935).—A no. of disputed points in the Cu-Zn constitutional diagram were investigated by thermal and microscopic analysis of samples melted in an evacuated porcelain tube. An improved method for obtaining heating and cooling curves with a mirror galvanometer was used which produced extremely accurate results. The transformation which takes place at about 900° was found to be a peritectic reaction,  $\alpha + \text{melt} \rightleftharpoons \beta$ . The compn. of these phases is  $\alpha = 67.7\% \text{ Cu}$ ,  $\beta = 63.5\% \text{ Cu}$  and melt = 62.5% Cu. At 834° there is transformation between the phases  $\beta$ ,  $\gamma$  and melt which consists of solidification or melting, melt  $\rightleftharpoons \gamma$ . The compn. of  $\beta$  is 43.6% Cu, and  $\gamma$  and melt 40.25% Cu. The point H in the diagram does not correspond to a compd. Cu<sub>2</sub>Zn, as claimed by Bauer and Hansen (*C. A.* 22, 567), as it solidifies over a temp. interval. The point B on the peritectic line UVW has the compn. 1.70% Cu. The improved Cu-Zn diagram is given. Twenty-nine references. C. E. Macfarlane.

**High chromium alloys—centrifugal castings and tubes.** R. J. Wilcox. *Metal Progress* 29, No. 1, 44-8 (1936).—The machinery does not differ essentially from that used for cast pipe or gray Ie. Operations must be highly

coordinated and tuned, and metal temps. carefully controlled to overcome the inherent sluggishness of the alloys. Details are given for alloys contg. 3 to 65% Ni, 9 to 20% Cr with and without addns of Mo. W. A. Mudge

Tests on the determination of creep limit of lead alloys H. Hanemann and W. Hofman *Metallwirtschaft* 14, 915-17(1935) —Pure soft Pb and hard Pb contg. 1.11% Sb, in the form of strips 35.8 sq mm, were used. The elongation in tension under constant loading was measured by means of a microscope, over a gage length of 400 mm. Loads from 10 to 55 kg/sq mm were used and elongation-time curves were plotted. The creep limit for hard Pb at 20° was found to be approx. 22 kg/sq mm, but soft Pb continued to stretch even with the lowest loads used. The time required to produce 1% elongation was also plotted against load and the results were compared with those of Moore, Betty and Dollins (*C. A. 29, 3280*). The discrepancy between the 2 may be due to different test methods or difference in materials. Preliminary tests on hard and soft Pb of the same comp. but from another source indicated considerably lower results. C. E. Macfarlane

The solid-solution series MgCu, MgNiZn and MgZn-MgCuAl. Werner Döring *Metallwirtschaft* 14, 918-19 (1935) —Since MgCu, and MgNiZn are both cubic, and MgZn, and MgCuAl are both hexagonal, a series of alloys in steps of 10% was made of these groups to det. whether they formed continuous solid solns. The alloys were melted and homogenized, and their lattice const. detd. by x-ray examn. In both cases the solid solns. were found. In the MgCu-MgNiZn series the lattice const. remain the same, while in the MgZn-MgCuAl series a continuous decrease takes place. C. E. M.

Recent uses of molybdenum in the ferrous metal industry. Jean Cournot *Rev. met.* 32, 562-76(1935) —An extended list of alloy compns. contg. Mo and covering steels, steel casting and cast iron uses. Most of the items are illustrated with figures giving their phys. properties and optimum uses. J. D. G.

Nickel-alloy gear materials and their heat treatments J. W. Sands and F. J. Walls *Product Eng.* 6, 370-3 (1935) —Gears made of Ni alloys, heat-treated steel, cast-iron and bronze alloys are compared as to compn. and phys. properties. Graphs, charts and photographs are shown. Ann Nicholson Hurd

The system nickel-manganese H. S. Valentiner *Z. Physik* 97, 745-57(1935), cf. *C. A. 29, 3967* —Data are given for the changes in resistance for Mn-Ni alloys subjected to magnetic fields. Alloys with up to 20% Mn have a normal behavior, while 25-30% alloys show neg. values for  $\Delta R/R_0$  far below the Curie point even with moderate intensities. S. Tolansky

Cadmium nickel bearing alloys A. J. Phillips *Product Eng.* 6, 365(1935) —Photographs and charts of their properties are given. Ann Nicholson Hurd

Cadmium-silver-copper bearing alloy L. C. Blomstrom *Product Eng.* 6, 364-5(1935) —A discussion with a micrograph and photographs. A. N. Hurd

Properties of solum containing magnesium (solum gamma) G. Sachs and E. Scheuer *Metallwirtschaft* 14, 937-41, 972-5(1935) —This alloy, contg. 12% Si, 0.4% Mg, 0.5% Mn, balance Al, is especially suitable for large and complicated castings which require high strength. Large castings can be quenched in water during heat treating without danger of cracking, as in other Al alloys. Slight warping can be overcome by straightening after quenching, but after aging this alloy is too stiff to straighten. It was found that in castings quenched under ordinary conditions the hardness of thin sections was less than that of heavy sections. This was due to cooling of the thin sections during the transfer from furnace to quench. Uniform results were obtained when this interval was reduced to 10 seconds by unproved equipment. It is essential to use cold water for quenching solum gamma. The highest possible quenching temp. should be used, usually 525° after heating for 3 hrs. At about 530° the Al-Si-Mg-Si eutectic melts and leaves voids at the grain boundaries. Very large castings are

apt to be too soft because of the relatively large interval between Mg-Si dendrites and resulting slow diffusion. This is overcome by increasing the heating time and by the use of chills. The use of salt as a flux should be avoided as it lowers the Mg content and results in low phys. properties. The aging treatment is not crit., 20 hrs. is required at 150° and 4 hrs. at 180°. The endurance limit is quite high in the quenched state, but is not increased by aging. The endurance limit is not lowered by running water over the test piece. Test bars cut from 14 production castings averaged 31.3 kg/sq mm tensile strength, 2% elongation, 109 Brinell hardness, and 7 kg/sq mm endurance limit. Still better properties are obtained in permanent mold castings. Solum gamma is not recommended for castings which are subjected continuously to temps. higher than 150° to 170°. Twelve references. C. E. Macfarlane

The effect of thallium on the freezing point of Lipowitz alloy S. J. French and D. Saunders *Metals & Alloys* 7, 22-3(1936)

Pressed and cast hard alloys with a tungsten base I. M. Grynazov *Nefteyanoe Khimichestvo* 29, No 10, 47-57 (1935) —A review covering the phys. data, mol. vols., linear elongation factors, prepn. of hard alloys (for drilling bits), chem. compn. of various hard alloys, appearance, sp. gr., hardness, tenacity, x-ray investigations, microstructure, etching, brazing, properties of some carbides, nitrides and borides. Twenty-one references. A. B.

Bearing requirements in automotive engines V. Skillman *Product Eng.* 6, 362-3(1935) —The evolution of the different bearing alloys is discussed. A. N. Hurd

A new heat-resistant alloy S. L. Hoyt and M. A. Scheel *Trans. Am. Soc. Metals* 23, 1022-38(1935) —The alloy contains 37.5% Cr, 7.5% Al and balance Fe and is malleable both hot and cold. The alloy is suitable for operation at 2300-2400°F in resistor elec. furnaces. Details are given. W. A. Mudge

Which alloy for die castings? Herbert Chase *Product Eng.* 6, 379-82(1935), cf. *C. A. 29, 437* —A large table gives the compn. and properties of die casting alloys. Photographs are shown. Ann Nicholson Hurd

A new metal mallet K. Wisemann *Aluminium*, 17, 644(1935) —An alloy of Al-Zn with 7-15% Zn was found to be a very suitable substitute for Pb, as it keeps its shape much longer and is sufficiently deformable not to damage the metal parts on which it is used. M. H.

Summary and outlook [corrosion]. Fritz Todt *Chem. Fabrik* 1936, 1-2, cf. *C. A. 29, 4316* —The closing address before the convention on corrosion, Berlin, Nov. 18-19, 1935. J. H. Moore

The anodic behavior of corrosion- and heat-resistant iron alloys L. Harant Berg and H. Hulten-mannschke *Jahrbuch* 83, 122-7(1935) —The process of soln. of anodes of different corrosion resistant alloys in H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> was studied. Steels of approx. similar compn. and structure showed also a great similarity in the dissolving process. After the passive state has been reached the soly. depends on the c d applied, at an av. c d there exists a sharply defined zone of perfect soly. where all constituents of the steel dissolve in the same proportion in which they are contained in the steel. The carbide carbon is oxidized in the passive state of the steel and at the higher c ds so that the carbides also dissolve. Curves show the anode potentials as a function of c d. Eight references. M. Hartenheum

Comparison between the effect of hydrogen-ion concentration on the rate of corrosion of steel in pure oxygen and air at 25° J. M. Bryan *Trans. Faraday Soc.* 31, 1714-18(1935), cf. *C. A. 29, 1043* —The main effect of high O<sub>2</sub> concn. is to mask the H-ion concn. effect, and to equalize the corrosion rate over the greater part of the pH range. H<sub>2</sub> evolution is inhibited at high acidity by O<sub>2</sub>. Ferric Fe is similar in action to high concn. of O<sub>2</sub>. Ferrous Fe promotes oxidation type corrosion over the whole pH range. C. I. P. Jeffreys

Laboratory study of the atmospheric corrosion of metals II. Iron—primary oxide film W. H. J. Vernon *Trans. Faraday Soc.* 31, 1068-76(1935), cf.

C A 26, 285—Marked differences in properties of oxide film on Fe are observed according to whether the film is produced below or above a crit temp 200°. The equation  $d \log W/dt = -Q/RT$  expresses the relation between rate of oxidation and temp, but there is a change of const at 200° giving 2 intersecting straight lines with the greater slope above the crit temp when log oxidation rate is plotted against  $1/T$ . At higher temp a parabolic relation ( $w^2 = kt$ ) holds between oxidation rate and time. Below 200° the expression deviates ( $w^2 = kt$  approx). Above 200° the colors produced are consistent with interference of light in a continuous transparent sheath, and any given color may be obtained at different temp by calcn of the time required to give appropriate thickness. Below 200° the relation between color and film thickness does not hold. The ill-defined colors ultimately obtained suggest a granular film. At 25° a point of inflection in the time-oxidation curve below which deviation from the quadratic parabola is in the reverse direction, at 15 days, is believed to imply completion of unit lattice of oxide over the whole surface. A secondary inflection where ( $w^2 = kt$ ) changes to ( $w^3 = kt$ ) marks the breakdown of this primary film. Electron diffraction expts show a difference in structure of the film above and below 200°. The diffraction patterns for a sample oxidized at 135° indicates a cubic structure of side 8.4 Å. This may be due to  $\gamma\text{-Fe}_2\text{O}_3$  or  $\text{Fe}_3\text{O}_4$ . At higher temps a rhombohedral structure ascribed to  $\alpha\text{-Fe}_2\text{O}_3$  is obtained. Evidence indicates that the crystals were oriented on the surface with a 211 plane parallel to the metal surface. III Secondary product or rust (influence of sulfur dioxide, carbon dioxide and suspended particles on the rusting of iron) *Ibid* 1678-1700—In purified air of high relative humidity (RH) rusting of mild steel is reduced if the high RH is approached from zero. This is ascribed to the strengthening of the primary film in the early stages. At const RH just below satn time-corrosion curves are convex about the corrosion axis, under supersatn conditions they are concave for 70 days then nearly linear. This is ascribed to changes in primary film. In the presence of  $\text{SO}_2$  at approx 50% RH (primary crit humidity) there is a marked weight increase followed at a little higher RH with rust formation. At approx 80% RH (secondary crit humidity) there is a great increase in rate of attack. The primary crit humidity depends upon a relation between concns of  $\text{H}_2\text{O}$ ,  $\text{O}_2$  and  $\text{SO}_2$  for rusting to begin. Secondary crit humidity depends upon the properties of the initially formed rust. Differences in the behavior of Fe and Zn depend upon secondary crit humidity phenomena. The influence of suspended solid particles depends upon whether (1) they are intrinsically active, e.g.  $(\text{NH}_4)_2\text{SO}_4$ , (2) neutral, but capable of adsorbing active gases, e.g. charcoal, (3) neutral with little adsorptive capacity, e.g.  $\text{SiO}_2$ . Under supersatd conditions  $(\text{NH}_4)_2\text{SO}_4$  particles greatly increase the rate of attack.  $\text{SiO}_2$  and charcoal in purified air have little effect, but charcoal has an enormous effect in presence of traces of  $\text{SO}_2$ , because of local concn of the gas.  $\text{SO}_2$  has a definite repressive effect on the corrosion of Fe under all conditions investigated, especially marked in the presence of  $(\text{NH}_4)_2\text{SO}_4$  particles. C E P Jefferys

Testing metals for their chemical resistance to fluosilicic acid. M A Filippenko and I I Zarnig. *Mineralnue Udobreniya Insektitsionnyy I*, No 5, 90-81(1935).—Among a great variety of metals and alloys investigated a cast iron contg Cu 0.99, S 0.008, Cr 40.68 and P 0.05% was most resistant, next was a steel contg C 0.15, Ni 8, Cr 18 and Ti 0.4%. The metal samples were kept under agitation in a thermostat charged with a mixt of  $\text{H}_2\text{SiF}_6$  & 5,  $\text{Fe}_2\text{O}_3$  0.5 and  $\text{SiO}_2$  about 1.5% (corresponding to conditions existing during manu) for 5-6 days at a temp of 45°. A A Boetlingk

And corrosion of structural iron in the presence of sulfur dioxide and hydrogen sulfide. V A. Karnitzkii and N A Golubev. *J Applied Chem (U S S R)* 8, 89/4 (11 German 871)(1935).—The corrosion of structural iron in  $\text{H}_2\text{SO}_4$  and  $\text{HCl}$  is accelerated in the presence

of dissolved  $\text{SO}_2$  and  $\text{H}_2\text{S}$  gases. This acceleration is identical for both gases when dissolved in  $\text{HCl}$ , while a soln of  $\text{SO}_2$  in  $\text{H}_2\text{SO}_4$  is more active. Corrosion by  $\text{H}_2\text{SO}_4$  and  $\text{HCl}$  in the presence of dissolved  $\text{SO}_2$  is proportional to the concn of the  $\text{SO}_2$ . The highest corrosive effect by  $\text{SO}_2$  and  $\text{H}_2\text{S}$  depends upon the concn of the H ions; it is highest in 30%  $\text{H}_2\text{SO}_4$ . This accelerating action is probably due to the formation of FeS films in various parts of the metal which create a great no of local elements. A. A. Boetlingk

The effect of air velocity on the scaling of iron. W. Baukloh and O. Reil. *Metallwirtschaft* 14, 1055-7 (1935).—Samples of Armco iron were heated in a quartz tube at 600°, 700° and 800° in a stream of air which was preheated in another furnace. The velocity of the air was varied from 0.05 to 0.7 m/sec. At each temp the loss in weight due to scaling rose sharply at first with increase in air velocity, then dropped after reaching a max, and finally became const. The maxima and horizontals in terms of weight loss were higher the higher the temp, and the maxima occurred at higher velocities the higher the temp. A theoretical explanation is given. C E. Macfarlane

Testing steels for their resistance to corrosion and erosion in contact with phosphoric acid extracts from Vyatka phosphates. M A Filippenko and I I Zarnig. *Mineralnue Udobreniya Insektitsionnyy I*, No 4, 95-6(1935).—Strips of various alloys were placed in a thermostat (73-6°) and immersed in a suspension contg  $\text{Fe}_2\text{O}_3$  24.10,  $\text{SO}_3$  3.4 and P 0.91% (liquid phase) and gypsum and undecomposed solid particles of Vyatka phosphorite (solid phase). For 100-114 hrs. the following alloys resisted the corrosion and erosion completely: (a) C 0.10, Si 0.39, Cr 19.50, Ni 8.64 and Mn 0.54%; (b) C 0.10, Si 0.94, Cr 15.48, Ni 8.77, Mn 8.77 and Ti 0.6%; (c) C 0.25, Si 0.40, Cr 17.00, Ni 9.00 and Mn 0.50%. Steel contg C 0.10, Si 0.78, P 0.011, Cr 27.29 and Mn 0.24% was slightly attacked and that contg C 0.35, Si 2.50, Cr 18.00 and Ni 25.00% was badly corroded. A. A. Boetlingk

Corrosion-resisting materials for gas appliances. A. L. Ward and W. H. Fulweiler. *Western Gas* 11, No 11, 26(1935).—Parts of pilot valve and thermostats made of brass with less than 63% Cu or if made with Al do not corrode. Brass with more than this percentage of Cu corrodes by forming CuS. W. H. Bruckner

Rust damage in hot-water systems. Alex. Marx. *Gesundh.-Ing* 58, 705-8(1935).—Theories to account for rust formation are critically discussed. The fact that such troubles are much more prevalent and serious in recently developed high-pressure systems is held to be due not to the properties of the water, the type of system (high- or low-pressure), or primarily to the presence of Cu parts in the system, but rather to the fact that the ingot steel commonly used for the more recent high-pressure systems since the war is less resistant to corrosion than the weld Fe formerly used in the older low-pressure systems. M. G. Moore

Automatic apparatus for corrosion tests of metals by intermittent action of moving liquid and its vapor in the presence of air or other gases. P. Ivitzkii and N. Arnshtam. *Zavodskaya Lab* 4, 1113-15(1935).—A metal plate is immersed in a liquid contained in the top chamber of a superimposed 2-chamber unit. A no. of such units is set in a box-like thermostat heater. The top chambers of each unit are fitted with top tubes connected by means of rubber tubing to a common outlet provided with a 1-way valve. The bottom chambers are similarly connected through the side tubes and with an elec. piston pump through a system of safety flasks. By means of a circuit-breaker regulated by a clock mechanism, the liquid is intermittently forced into the top chamber and then returned by gravity to the bottom chamber. The construction and operation of the app are described in detail and illustrated. Chas. Blanc

Longer life for galvanizing furnaces. Wallace G. Imhoff. *Am. Machinist* 80, 18 20(1934).—Discussion of factors of corrosion from molten Zn on the inside and chem.

destruction of furnace on the outside by combustion atm. Forcing production by increasing the temp. of the galvanizing pot is shown to decrease the life of the pot and ultimately to decrease production due to high rate of formation of galvanizing dross at higher temps. The dross sinks to the bottom of the pot and becomes tightly packed, this increases temp. of furnace bottom and aggravates drossing because of the high temp. differential between the bottom and the liquid Zn.

W H Bruckner

Coloring metals H Krause *Metallwirtschaft* 14, 1015-17(1935). Cf C A 29, 5403<sup>1</sup>—Cd can be colored black by dipping in cold soln of 60 g KClO<sub>3</sub>, 35-40 g Cu(NO<sub>3</sub>)<sub>2</sub>, 1 l H<sub>2</sub>O, brushing, redipping, rinsing, drying and polishing. Another soln contains 30 g Cu(NO<sub>3</sub>)<sub>2</sub>, 2.5 g KMnO<sub>4</sub>, 1 l H<sub>2</sub>O, used at 60-80°. Cd is colored brown by 160 g KMnO<sub>4</sub>, 60-250 g Cd(NO<sub>3</sub>)<sub>2</sub> and 1 l H<sub>2</sub>O. Cu and Cu alloys are colored brown by repeated dipping in a hot soln of 5 g KMnO<sub>4</sub>, 50 g CuSO<sub>4</sub>, 1 l H<sub>2</sub>O, and brushing. Addn of a little FeSO<sub>4</sub> produces a more greenish color. A soln of 50-60 g KClO<sub>3</sub>, 100-125 g CuSO<sub>4</sub> or Cu(NO<sub>3</sub>)<sub>2</sub>, 1 l H<sub>2</sub>O colors Cu and its alloys a reddish brown. Various shades can be produced by addn of Ni or Fe salts. Cu is colored red by dipping in molten KNO<sub>3</sub> or NaNO<sub>3</sub>. A violet color is obtained by adding 1% KMnO<sub>4</sub>. Fe is colored black by a mixt. of molten NaNO<sub>3</sub> and NaNO<sub>2</sub>. Addn of 5-25% NaOH produces a flat black. For some purposes it is better to color Fe in a soln of 1.5 g NaOH, 25-50 g NaNO<sub>3</sub>, 5 g tannic acid, 1 l H<sub>2</sub>O at about 160°. Al is colored gray by a electrolytic bath of 100 g As<sub>2</sub>O<sub>3</sub>, 30 g NaCO<sub>3</sub>, 10 g KCN, 1 l H<sub>2</sub>O, and black by 10-20 g (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub>, 5-15 g NH<sub>4</sub>Cl, 1 l H<sub>2</sub>O. The latter can also be used for Zn, Fe, Pb Sn and Cd.

C E Macfarlane

Metallurgy of oxyacetylene welding of steel J H Critchett. *Welding Engr.* 20, No. 2, 21-4(1935)—A discussion of foundry practice and the metallurgical reactions occurring in welding.

A N Hurd

Welding the stainless steels A P Johnston *Welding Engr.* 20, No. 8, 19-22(1935)—Drawings and micrographs are shown.

Ann Nicholson Hurd

Welding aluminum with the metallic arc Arnold Meyer. *Welding Engr.* 20, No. 9, 29-31(1935)—Photographs and micrographs of welds are shown.

Ann Nicholson Hurd

Carbon electrodes in welding technic Goltner *Elektrowärme* 5, 300-1(1935)—In general, 3 carbon types are used, all with and without core of carbon powder or special addn: (1) hard, amorphous C electrodes for all purposes and normal stresses, (2) medium hard or soft, graphitic electrodes which can be used for larger currents because of their graphite content, (3) electrically graphitized carbons, having the least burning loss and highest elec. cond. They will stand twice the current load as the amorphous C and are used where very thin electrodes are required for making very fine and neat seams. All electrodes may also be used with Cu plating. Diams. required for various currents are given and the fields of application of the C are discussed.

M H

Soldering of aluminum cables and ropes with soft solder paste W Nicolini. *Aluminium* 17, 618-50(1935).—The soldering paste (Alutinol) is composed of Sn 40, Zn 40 and Cd 20%, it is used with a flux. Tests showed good mech. properties of the joint which can be well worked and has a good elec. cond. The procedure is described.

M Hartenheim

Volatilization of silica and Si as Si sulfide [applications to metallurgy of Fe, etc.] (Dolch) 6. Analyses of sulfite waste liquor [binders for molding sands] (Roll) 23. Detn. of moisture in sulfidic Cu ores (Iolsson, Fershtater) 7. Analysis of mine dusts (Clark, Reynolds) 3. Balancing mine, Sumatra (Grey) 8. Zn-dust paints (Brit. pat. 436,164) 26. Product for intensifying combustion and improving metallurgical products (Belg. pat. 407,556) 13.

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Sulfidic ores. R. Englert & F. Becker, Chem. Fabrik und Galina Finanz A.-G. (Antor Michels, inventor). Ger. 620,710. Oct. 25, 1935 (Cl. 40a. 12.70).

Sulfidic ores contg. Pb, Cu, Zn, etc., are worked up by fusing with carbonates of alkali or alk. earth metals and blowing gases contg. O onto the fused mass. Thus, rock salt, galena and soda are powd. and fused in a reverberatory furnace and O blown in until half the PbS is oxidized.

Metallic Pb seps. at the bottom of the furnace and can be tapped off below the fused salt. A 95% yield of metallic Pb is obtained. By shortening the O treatment, PbCl<sub>2</sub> is produced. Other examples describing the similar treatment of Cu pyrites and Zn blende are given.

Iron ores. Fried. Krupp Grusonwerk A.-G. Fr.



789,379, Oct. 28, 1935. The reduction and fritting of ores of Fe are carried out in a rotary tubular furnace, solid fuel being added in the proportion of 20-30% of the ore, and oxidizing gases being introduced but little external heat being added so that the reduction, preceding the fritting, takes place in a long zone of the furnace.

Apparatus for washing and (or) classifying metalliferous earth Harold W. Bearcroft. Brit. 436,435, Oct. 11, 1935

Electrodynamical separator for ores S. G. Kosinski Russ 37,652, July 31, 1934 Construction details

Ball and like mills for grinding ores in liquid Wilhelm Mauss Brit 436,534, Oct. 14, 1935.

Flotation concentration of oxidized ores Ludwig J. Christmann and Stuart A. Falconer (to American Cyanamid Co.) U. S. 2,029,156, Jan. 28 An oxidized ore is subjected to a froth flotation operation in the presence of Na mercaptobenzothiazole,  $\text{H}_2\text{PO}_4$  and a frother. Na diisopropylidithiophosphate, etc., also may be used. Numerous examples are given. Cl. C. A. 29, 3642<sup>1</sup>

Apparatus for ore flotation Randall P. Akms U. S. 2,028,179, Jan. 21. Various structural and operative details

Impeller for flotation apparatus Arthur J. Weing (to Morse Bros. Machinery & Supply Co.) Can. 355,213, Jan. 7, 1936 In a flotation process, air and pulp are supplied adjacent to each other and thoroughly intermixed by combined pumping, slicing and striking actions in a rotary movement before and during upward movement of the air and pulp.

Apparatus (with a rotating table) for separating minerals from ores by centrifugal force Paul R. Yewell U. S. 2,029,090, Jan. 28 Structural, mech. and operative details

Recovery of precious metals John A. Hatton Can. 355,124, Jan. 7, 1936 Precious metals are recovered from their ores by reduction, agitation and concn in a mixed soln. of alkali metal carbonate, alkali metal chloride and an  $\text{NH}_4$  compd., in the presence of metallic Hg. The soln. is drawn off and reused.

Precious metal recovery from ores and concentrates George J. MacKay U. S. 2,028,940, Jan. 28 See Can. 354,049 (C. A. 30, 1347<sup>1</sup>)

Tin Th. Goldschmidt A-G (Ludwig Schertel, inventor) Ger. 620,711, Oct. 25, 1935 (Cl. 40a 44 60) Material such as waste tin plate or raw Pb is treated with alkali to form stannate lye. Solid alkali stannate is obtained from the lye. This is heated with  $\text{CO}_2$  to reduce it to Sn and alkali carbonate. The reduction may be carried out at sintering temp., the finely divided Sn being burned to oxide which is then worked up to Sn blocks by a reducing flame. The  $\text{CO}_2$  may be mixed with reducing gases.

Tin recovery from residues containing tin and from Lafayette M. Hughes Brit. 436,582, Oct. 7, 1935 See U. S. 2,390,765 (C. A. 28, 4283<sup>1</sup>)

Apparatus for distillation of magnesium Fritz Hansgig (to American Magnesium Metals Corp.) U. S. 2,029,921, Feb. 4 Various structural and operative details

Purification of magnesium Fritz Hansgig and Konrad Erdmann (to American Magnesium Metals Corp.) Can. 354,354, Nov. 26, 1935 Finely divided Mg is mixed with a hydrocarbon oil and heated to carbonize the oil and bond the powder into aggregates. The Mg is vaporized from the aggregates and condensed to coherent form, the impurities remaining in the carbonized matrix of the aggregates. Cl. C. A. 29, 6874<sup>1</sup>

Purifying magnesium Walther Schmidt and Adolf Beck (to Magnesium Development Corp.) U. S. 2,029,898, Feb. 4 For removing "iron impurities" from Mg, there is dissolved in the molten metal, by heating to above 700°, sufficient Mn, Si or Mn chloride to form primary crystals on cooling of the melt while simultaneously combining with the impurities, and the melt is cooled to a temp. not exceeding about 700° and the crystals contg. the purifying substance and impurities are removed from the melt.

Magnesium Frank R. Kemmer (to Magnesium Products Inc.), Can. 354,952, Dec. 24, 1935 A charge of  $\text{MgO}$  and carbonaceous material is subjected to at least a partial vacuum to remove the gases. These gases are replaced by a protective liquid and the charge is fed into a reaction chamber and heated.

Metallurgy Hendrik J. Janssen (to Shell Development Co.) Can. 354,308, Nov. 19, 1935 Finely divided ore is mixed with liquid fuel and air is blown through the mixt. in the presence of an oxidizing catalyst to obtain fatty acids. A lye is added to the mixt. to form soaps. The fuel slurry is atomized and burned with such quantity of air that incomplete combustion takes place and the metal ore is reduced.

Refining metals and alloys Vereinigte deutsche Metallwerke A-G and Henry Lepp (to Compagnie générale d'Electrometallurgie) Brit. 436,204, Oct. 4, 1935 The mech. properties of metals, e. g., Cu, Ni and their alloys, particularly those produced by melting scrap metal, are improved by creating neutral or reducing conditions in the bath by means of a neutral or reducing atm. when the metals are melted and then rendering the atm. oxidizing and adding to the bath 1 or more oxidizing agents in quantity substantially equal to that necessary to oxidize the gaseous impurities, e. g., H,  $\text{SO}_2$ , CO, N, etc., in the bath, the oxidation taking place in the presence of a slag contg. 1 or more compds. of Ba or Sr and in the presence of a substance or substances e. g., Zn, Cd, Fe, Mn, Na, Pb, less noble than the principal metal of the bath. The oxidizing agent must not be decomposed at the temp. of the bath and is preferably a metal oxide. Cl. C. A. 29, 3643<sup>1</sup>

Metals Tréfileries et laminours du Havre Fr. 789,436, Oct. 29, 1935 Metals such as Al, Mg, Ca and Si, generally obtained by electrolysis, are obtained by first forming an alloy or a mixt. contg. at least 1 metal in the metallic state and subliming the metals 1 at a time at suitable temps. The alloy may be formed by heating together oxides and carbides of the metals to be alloyed or oxides and reducing metals.

Deoxidizing metals Société d'Electrochimie d'Electrometallurgie des acieries électriques d'Ugine Brit. 436,450, Oct. 7, 1935. The metals are treated with a very fluid slag contg. not less than 5 or more than 90%  $\text{TiO}_2$ , and acid, neutral or basic oxides usually contained in metallurgical slags and in such proportions that the slag has an acid character and is capable of oxide from the metal. In an example, a slag contains  $\text{TiO}_2$  35,  $\text{SiO}_2$  25,  $\text{Al}_2\text{O}_3$  30,  $\text{CaO}$  5 and Mn oxide 5%. Cl. C. A. 30, 73<sup>1</sup>

Extracting iron Lucien P. Basset. Brit. 436,443, Oct. 3, 1935 In the simultaneous manuf. of Fe or steel and portland cement or hydraulic lime, the charge consisting of ore, chalk, coal for reducing the ore and the  $\text{CO}_2$  from the chalk, carburizing coal and a quantity of addnl. coal approx. 5% of the reducing coal for combatting the oxidizing effects of the reducing flame used is heated in a rotary furnace by a flame obtained by the combustion of pulverized coal with air sufficient to produce chiefly  $\text{CO}_2$  and little or no  $\text{CO}$ .  $\text{H}_2\text{O}$ -vapor may be supplied to assist in removal of S from the clinker produced and air may be introduced into the furnace to burn the combustible gases therein. A cement produced by the process contains  $\text{SiO}_2$  21,  $\text{Al}_2\text{O}_3$  6-7,  $\text{CaO}$  68 and other ingredients 5%, a hydraulic lime,  $\text{SiO}_2$  16-17,  $\text{Al}_2\text{O}_3$  6-7,  $\text{CaO}$  72 and other ingredients 5%.

Lead Ernst J. Kohlmeier and Henry Hocking Ger. 620,943, Oct. 30, 1935 (Cl. 40a. 18 01). Pb and other metals are obtained from flue dust, mixts. of oxides or other starting materials by mixing them with powd. C and introducing the mixt. into fused salt or other neutral flux. A foam is maintained in the fused mass during the addn. of the mixt. Carbonates may be added to the fused mass to produce this foam. The reaction may take place in a short rotating-drum furnace. Examples are given.

Metallurgical furnace of the suspended-arch type suitable for treating lead and its alloys Karl A. Landner

(to American Smelting and Refining Co.). U. S. 2,029,492, Feb. 4. Structural details.

Melting and holding furnace for metals. John G. G. Frost (to National Smelting Co.). Can 351,204, Nov. 19, 1915. Structural details are described. The furnace is adapted to be used for metals in which a low Fe content is desired.

Furnace construction and operation for heating soaking pits. Gustav A. Merkt (to Morgan Construction Co.). U. S. 2,029,580, Feb. 4. Various details.

Charging apparatus for the Siemens-Martin furnace. N. N. Gergel. Russ 204,660, May 31, 1931. Addn. Russ 21,576, Feb. 28, 1935. Mechanical features.

Mouth lock for blast furnaces. P. D. Zaitsev. Russ 14,971, April 30, 1920. Construction details. Addn. Russ 33,981, Jan. 31, 1934.

Apparatus for passing metal plates, sheets or bars through pickling baths, furnaces, etc. The Milner-Guthrie Co. Ltd. Ir 749,283, Oct. 25, 1935.

Degasifying liquid slag. Dortmund-Hoerder Huttenverein A-G. Brit 447,095, Nov. 4, 1935. See Fr 781,653 (C A 29, 619F).

Granular blast furnace slag. L. Gothot. Belg 400,970, Jan. 31, 1935. The slag is molded and screened directly as it leaves the blast furnace, so as to obtain grains of 0.7-0.8 mm.

Preparing metal stock such as aluminum stock for working. Richard L. Templin (to Aluminum Co. of America). U. S. 2,029,800, Feb. 4. Mech. features.

Apparatus for tempering iron objects. The Linde Air Products Co. Ir 789,132, Oct. 21, 1935.

Cyanide cementation. Deutsche Gold und Silber-Scheidanstalt vorm. Roessler (Walter Beck and Klaus Bonath, inventors). Ger 620,978, Oct. 31, 1935 (Cl. 18c. 317). The cementation process is regulated in fused cyanide baths for Fe and steel by adding nonalkali or less-alk. substances whose cyanides are more readily decomposed under the reaction conditions than the cyanide comprising the bath. Thus, a bath contg. increased BaCl<sub>2</sub>, NaCl, KCl and NaCN is given an addn. of SrCl<sub>2</sub> to increase the efficiency of the cementation process when Fe or steel is immersed in the bath. Other examples are given.

Cementation of steels. Louis Renault. Fr 784,736, Oct. 15, 1935. Gas from a producer blown with air and steam is treated so as to convert the H<sub>2</sub> therein to CH<sub>4</sub> by reaction with the CO or CO<sub>2</sub>, water formed is removed and the resulting gas is used for cementation of steel.

Pickling iron and its alloys. Oscar Fakler. Fr 789,099, Oct. 22, 1935. The bath of H<sub>2</sub>SO<sub>4</sub> used contains such a proportion of FeSO<sub>4</sub> and is carried to such a temp. that the FeSO<sub>4</sub> activates the pickling action of the free H<sub>2</sub>SO<sub>4</sub>, the temp. and concn. of the free H<sub>2</sub>SO<sub>4</sub> being such that the FeSO<sub>4</sub> crystallizes with 7 mols. of water. Thus, a bath is used contg. 170-220 g. of anhyd. FeSO<sub>4</sub> per l. and the H<sub>2</sub>SO<sub>4</sub> is maintained between 120 and 60 g. per l., the temp. being about 50° F. Ir 789,100. Residual waters from HCl pickling baths are recovered by transforming the metal chlorides therein to alk. earth chlorides by adding an alk. earth base and regenerating the HCl by treating these chlorides with H<sub>2</sub>SO<sub>4</sub>.

Apparatus for pickling, scrubbing and swilling metal plates. Saml. Davies. Alexander Engineering Works and Samuel Davies. Brit 476,222, Oct. 3, 1935.

Nitriding articles of alloy steel. K. M. Boroditzkil. Russ 37,724, July 31, 1934. After the usual thermal treatment articles are nitrided with NaCN or KCN at temps. not exceeding the annealing temp., i. e., 520-650°.

Nitrided steel. Wilhelm Kroll. U. S. 2,029,724, Feb. 4. For improving the hardness of a steel contg. Be 0.1-5.0%, the steel is chilled from a temp. above that of aging and is subsequently superficially nitrided while heating it to 400-600°.

Annealing steel. Soc. anon. des établissements Marrel Frères. Ir 789,601, Nov. 4, 1935. A piece of steel hardened by tempering is annealed locally by heating to 650° by a bath of molten Al or other substance or max. m. at about 650°, kept near its solidification point.

Processing material such as in annealing strip metal for

1 springs by electric heating and treatment with a liquid such as water or oil for quenching. De Hart G. Serantom (to Western Elec. Co.). U. S. 2,029,937, Jan. 28, 1935. App. and various operative details are described.

Furnace suitable for bright annealing. Patrick J. McIntyre. U. S. 2,028,941, Jan. 28. Structural, mech. and operative details of a furnace to which gases such as non-oxidizing gas and superheated steam may be supplied.

2 Heat-treating ferrous alloy articles. Frederick R. Bonte. Brit. 476,202, Oct. 1, 1935. Addn. to 406,131 (C A 28, 47653). In making articles as described in 406,131, the articles, after heating to above the crit. point, are cooled from that temp. and then subjected to further heat treatment to harden their surfaces. The heat treatment involves heating to 900-1800°F. and quenching, and may be applied to a selected part of the article. The alloy may contain C 1.5-2.5 and Si 0.6-0.9% but, if it contains 1.5-2.0 C and up to 1% Mn, the Si may be increased to 1.3%. The alloy may also contain Ni 0.75-2.0, Mo 0.2-0.5, Al 0.5-15.0, Cr and W 0.5-18.0 and V up to 1%.

Heat-treating steel wheels or tires. Christer P. Sandberg and Oscar F. A. Sandberg (also legal representatives of Nils P. P. Sandberg, deceased). Brit 436,530, Oct. 14, 1935. In treating the wheels at above the crit. range, the surface or surfaces are cooled very rapidly by means of jets of atomized H<sub>2</sub>O so that sufficient evapn. of the H<sub>2</sub>O particles is assured, and the amt. of H<sub>2</sub>O is reduced as the surface cools. The cooling is stopped when the heat retained corresponds to an av. temp. above 500° but below the crit. temp. and the wheel is then placed in a heat-insulated container or furnace until the retained heat has evened out to produce said av. temp., the wheel being finally cooled slowly.

Heat treatment of rails. Quincy Bent and Edward F. Kenney (to Bethlehem Steel Co.). U. S. 2,029,286, Feb. 4. App. and various operative details are described.

Heat-treating electric-arc-welded chrome-alloy-steel containers such as those used in oil cracking. Harry S. Blumberg (to M. W. Kellogg Co.). U. S. 2,029,807, Feb. 4. A welded container the base metal of which is a Cr steel contg. 4-6% of Cr (the remainder being Fe and C except for incidental impurities) and the weld metal of which is an austenitic Cr-Ni-Fe alloy of low-C content contg. about 0.42% or more of a metal such as Ti, Nb or Ta, of greater carbide-forming tendency than Cr, proportioned to combine with substantially all the C in the weld metal is heated to effect recrystn. in the base metal and soln. of carbides in the weld metal, then slowly cooled to below the crit. range of the base metal so as to effect annealing of the base metal and pptn. of harmless carbides in the weld metal, then reheated to below but near to the crit. range of the base metal to effect approx. attainment of equil. and cooled.

Special steels. Cimmar Molybdenum Co. Fr 788,889, Oct. 18, 1935. Mo steels contain C 0.1-0.7, Mn 0.1-2, Si 0.5-2.5, Cr 0.01-11 and Mo 1-8%. Ni 0.01-2.5 and V 0.01-2% may also be present. An example contains C 0.45, Mn 0.55, Si 1.25, Cr 8, Mo 4.25, Ni 1.8, V 1% and Fe the rest, S and P being present in the usual amts.

Steel ingots. L'auxiliaire de l'industrie. Fr 780,895, Sept. 10, 1935. A compn. contg. C 2b, SiO<sub>2</sub> 62, Al<sub>2</sub>O<sub>3</sub> 1, Fe<sub>2</sub>O<sub>3</sub> 0.5, MgO 0.5, alkali 6 and water 4%, is introduced into fused steel immediately after its casting in the ingot mold, to improve the texture of the ingot and particularly to reduce the cavity therein.

Cast steel car wheels. Alfred W. Gregg and Raymond H. Frank (to Bonney-Floyd Co.). U. S. 2,029,819, Feb. 4. Wheels possessing differentially hardened tread and axle regions are formed from iron contg. incidental impurities such as S and P together with C 0.25-0.40, Mn 0.90-1.50, Mo 0.20-0.40, Cr 0.10-1.00 and Si 0.20-0.50%.

Steel alloys. Frederick M. Becket and Russell Franks (to Electro Metallurgical Co.). Brit. 436,184, Oct. 7, 1935. Addn. to 414,211 (C A 29, 1099). Seamless tubes and other articles are made of austenitic steel alloys

of the compn given in 414,211, particularly those contg. Cr 15-25, Ni 6-12, C up to about 0.2 and Cb and Ta up to about 4%, the Cb being present in greater amt. than the Ta

**Composite metal bodies** Frederick F. Gordon Brit. 436,401, Oct. 10, 1935 See Ger. 622,484 (C. A. 30, 1351)

**Aluminum alloys** I G Farbenund. A-G. Fr. 788,952, Oct. 21, 1935 Alloys which are useful where resistance to pressure of liquids or gases is desired contain Mg 3-7, Si 0.8-1.5, Mn 2-5% and Al the rest

**Beryllium and its alloys** Compagnie de produits chimiques et electrometallurgiques Alais, Fropes & Camargue Fr. 789,244, Oct. 25, 1935 See Brit. 435,747 (C. A. 30, 1353)

**Copper alloys** Leonard C Bannister and Imperial Chemical Industries Ltd Brit. 436,662, Oct. 16, 1935 Pipes, vessels, etc., for use in mfg., conveying and storing beer are made of Cu alloys contg. up to 10% Al. The alloys may also contain Zn 0-30, Si or Mn about 1% and Ni a small amt. The interior of the pipes, etc., may be cleaned before use by pickling in a soln. contg.  $H_2SO_4$  10 and  $Na_2Cr_2O_7$  10%.

**Iron alloys** Electro Metallurgical Co. Fr. 788,947, Oct. 19, 1935 Alloys contain Cr 50-80, N 0.35-1.8, C 1-5%, and Fe the rest. The ratio of N to Cr is preferably 1/100. The alloys may be made by melting Fe and adding a Fe-Cr-N alloy contg. Cr 60-80 and N 0.35-1%.

**Nickel-cobalt alloy** Westinghouse Electric & Mfg. Co. Ger. 620,897, Oct. 29, 1935 (Cl. 405 14) The sum of the Ni and Co contents is 80-95% and the sum of the Fe and Ti content is 5-20%. As an example, the alloy comprises Ni 73, Co 18, Fe 7 and Ti 2%.

**Alloys** Stahlwerke Röchling Buderus A-G and Alfred Kropf Ger. 620,712, Oct. 29, 1935 (Cl. 404 1 65) Addn to 616,558 (C. A. 30, 424) Alloys contg. Cr 10-40, Ta 3-50, C 0.1-1.0 and Ni 40-60% are cold-molded or shaped in stages. The alloy is heated to 950-1050° before each cold shaping stage, and is cooled to 300-600° after each stage.

**Alloy of high strength** George P. Halliwell (to Canadian Westinghouse Co. Ltd.) Can. 354,247, Nov. 19, 1935 An alloy contg. Ni 40-60, Co 20-35, Fe 5-20, Ti 0.5-10 and Cr up to 20% is quenched from approx. 900° and aged at 500-800° for approx. 1/2 hr. It has an elastic limit of 52,000-66,200 lbs. per sq. in. and a tensile strength of 105,000-120,200 lbs. per sq. in. at approx. 600° Cl. C. A. 30 74

**Hard alloys** A A Abinder Russ. 38,325, Aug. 31, 1934 Cementing metals or alloys are atomized into liquid low boiling organic substances (e.g., alc., gasoline, acetone) to which is added not over 20% of their weight of rubber, tar or guita-percha. The finely divided metal thus formed is mixed with carbides, shaped, pressed and ignited.

**Hard alloys** V D Romanov, V S Rakovskii, V I Tret'yakov, R A Trubnikov, V P L'vovskaya, N E Levina and E M Belostotskaya Russ. 37,534, July 31, 1934 Tungsten carbide or other hard compds in powder form are introduced into a chamber in which a cementing metal is being reduced and vaporized.

**Hard alloys** Furth-Sterling Steel Co. Brit. 436,255, Oct. 8, 1935 See Can. 350,602 (C. A. 29, 5063) Part of the Al may be replaced by Mg and (or) Be. Up to 5% of Fe, Ni and Co may be present.

**Improving the ductility of magnesium alloys** Robert D Lowry and Fred L Reynolds (to Dow Chemical Co.) U. S. 2,029,728, Feb. 4 A relatively non-ductile annealed rolled Mg base alloy material such as one contg. Mg together with Al 4 and Mn 0.3% is alternately bent and straightened at a temp. between about 5° and 135° so as to stress the metal without substantially changing its thickness, and is then annealed.

**Tools for cutting glass, etc.** The General Elec. Co. Ltd. and John H Partridge Brit. 436,017, Oct. 3, 1935 The tools are made by sintering at a high temp. a mixt. of finely divided  $Al_2O_3$  and a material which is,

or which becomes after sintering, softer and more ductile than cryst.  $Al_2O_3$ . Thus a mixt. of  $Al_2O_3$  with 10%  $Fe_2O_3$  (rouge) is pressed into a coherent bar of square section with sharp edges and the bar is sintered at about 1700° in an elec. furnace that has resistance elements of Mo and is filled with  $H_2$ , the  $Fe_2O_3$  being reduced to Fe.

**Alloys for bearings** General Motors Corp. Brit. 436,633, Oct. 15, 1935 An alloy consists of at least 95% Cd and 0.5-5% Ag, preferably with 0.25-1% of a hardening metal, e.g., Cu and (or) Ni. Up to 0.25% Zn may be present.

**Composite bearings** Degenhart Baron von Loe Brit. 436,705, Oct. 16, 1935 A Cu-coated bearing-shell of Fe that is not hardenable by chilling is heated in borax to 1050-1085° and inserted into a mold which is then filled with molten Cu-Pb alloy and plunged into cold  $H_2O$ .

**Permanent magnet** Robert Bosch A-G Fr. 789,426, Oct. 29, 1935 The magnets are made of an alloy contg. Ni 7-30, Al 3-20, Cu 20-40% and Fe the rest.

**Permanent magnets** Robert Bosch A-G Fr. 789,157, Oct. 24, 1935 Alloys contg. Ni 7-40, Al 3-20, Cu up to 40% and Fe the rest are used, the Ni and Cu being introduced into the molten bath in the form of com. alloys of Ni and Cu. Cf. C. A. 29, 7430 and preceding abstr.

**Composite metal products** The Mond Nickel Co. Ltd. Brit. 436,329, Oct. 9, 1935 A protective metallic film is applied to the surface of a metal or alloy contg. Cr, the film is placed in contact with the cleaned surface of a foundation or base metal and the composite unit is heated to form a bond between the base metal and the film, which is preferably of Ni or Ni alloy and preferably applied to the Cr alloy by electrodeposition. The outer layer may consist of an alloy contg. Cr 14, Fe 6 and Ni 80% or of stainless steel preferably contg. Cr 15 and Ni 8%.

**Removing aluminum from copper and copper alloys** V. A. Dubinkin, S. A. Dubinkin and I. V. Molvanov Russ. 38,324, Aug. 31, 1934 Al is removed by melting the metals in an oxidizing atm. with the introduction of Ba salts except those contg. S in the acid radical.

**Removing oxide coatings from ferrous metals** Harry S. George (to Electro Metallurgical Co. of Canada, Ltd.) Can. 354,406, Nov. 26, 1935 A surface-oxidized article contg. Fe and at least 8% Cr is cleaned by providing a box-annealed scale theoron, treating in a hot bath contg. about 20% NaOH in which Cl has been bubbled until O is liberated, and then subjecting to a hot acid bath contg. 50 g. per l. of  $HNO_3$  and 10-100 g. per l. of HCl.

**Coating with metals** Cyril H Walker, Charles Sykes and Associated Electrical Industries Ltd. Brit. 436,792, Oct. 14, 1935 Surfaces are coated by distn. of metals in a vacuum by placing the coating metal as a thin uniform layer on the surface of a heater, preferably an electrically heated strip of W or Mo, raising the temp. until the coating metal melts to form a thin uniform coating on the heater and then raising the temp. to distil the coating metal onto the surface to be coated. App. is described.

**Machine for coating metal plates with tin, tern or other metal or alloy** Saml Davies Alexander Engineering Works Ltd. and Samuel Davies Brit. 436,007, Oct. 3, 1935

**Machines for applying a layer of tin or other metals or alloys to metal plates** Saml Davies Alexander Engineering Works, Ltd. and Samuel Davies Fr. 788,440, Oct. 10, 1935

**Mold materials** International de Levant Mass. facturing Corp. Ltd. Brit. 436,196, Oct. 7, 1935 A centrifugal metal mold is lined, prior to casting Fe therein, with ferrosilicon powder of such fineness that not more than 5% is retained on a 150-mesh sieve, Tyler series, and not less than 10% on a 200-mesh sieve.

**Tungsten carbide castings** Orrin F. Marvin (to Mills Alloys, Inc.) U. S. 2,028,911, Jan. 28 In making thin castings such as rotary saw blades, the fluid material from which the casting is to be made is poured into a mold, which may be formed of graphite and is also poured around the outside of the mold. App. is described.

**Casting aluminum magnesium alloys** Aluminum Ltd.

Fr. 788,835, Oct. 18, 1975. To avoid unfavorable reaction between the alloy and the atm. or the constituents of the mold, the mold of green sand is treated with  $\text{NH}_4\text{F}$ , or  $\text{NH}_4\text{F}$  is incorporated with the sand before molding.

Welding rod. Aubrey T. Roberts and John H. Pater-son (to American Murex Corp.) Can. 354,513, Dec. 3, 1935. An electrically conductive coating is provided on the rod comprising a bonded mixt. of powd. Fe and Fe-Mn in the proportion of 6% Mn to the powdered Fe. The Fe-Mn mixt. comprises not less than 10% of the welding rod. Cf. C. A. 29, 6506<sup>1</sup>.

Electrode for welding hard alloys. N. I. Mushitzkil. Russ. 37,778, July 31, 1934. A tubular electrode con-

granular hard alloys contains also 3-20% B and 2-4% molasses to improve the hardness of the welded spot.

Paste for welding electrodes. A. A. Alov. Russ. 37,779, July 31, 1934. In a welding paste contg. coal, graphite, Ni, Cr, V, Mo, Mn, etc., is incorporated the basic component Na acetate or K acetate together with  $\text{NaOH}$  or  $\text{Ca(OH)}_2$  or soda-lime to the amount of at least 50% of the amount acetate.

Apparatus for measuring the hardness of metals. Felix Eugene. Brit. 436,155, Oct. 7, 1935.

Apparatus for degreasing metal and other articles. Joseph A. M. W. Mitchell and Imperial Chemical Industries Ltd. Brit. 436,155, Oct. 7, 1935.

## 10—ORGANIC CHEMISTRY

CHAS. A. ROUILLER AND CLARENCE J. WEST

Activation of specific bonds in complex molecules at catalytic surfaces. Hugh S. Taylor, Kiyoshi Morikawa and W. S. Benedict. *J. Am. Chem. Soc.* 57, 2735 (1935).

The activation of the C-C and C-H bonds have been followed on an active Ni surface by the study of the 2 reactions (a) the exchange reaction  $\text{C}_x\text{H}_y + m\text{D} \rightarrow \text{C}_x\text{H}_y\text{D}_m$ , where  $x + y = 6$  and  $m$  may be varied arbitrarily, (b)  $\text{C}_x\text{H}_y + \text{H}_2 \rightarrow 2\text{CH}_4$ . The former involves only the C-H, the latter the C-C bond, the other reactant in each case being H or D adsorbed on the surface in the activated form. On this catalyst the exchange reaction proceeds quantitatively at 115° under conditions in which reaction (b) is quite negligible. The production of  $\text{CH}_4$  sets in about 150° and is sensibly complete around 200°. This defines the temp., catalyst and reactant concn. conditions necessary to activated adsorption of  $\text{C}_x\text{H}_y$  molecules, producing either the C-H or the C-C bond split or both. The work is being continued. C. J. West

Method of comparison and critical analysis of the physical properties of homologs and isomers. The molecular volume of alkanes. Geo. Calingard and John W. Hudly. *J. Am. Chem. Soc.* 58, 154-7 (1936).—A graphical method is described for the crit. comparison of data on any one of the phys. properties of large groups of homologs and isomers. The illustration chosen covers the d. (mol. vol.) of the alkanes and suggests the necessity of obtaining more and better data in the range  $\text{C}_5$  to  $\text{C}_{10}$ . The effect of structure is discussed. C. J. West

Isomerization of normal heptane. Geo. Calingard and Harold A. Beatty. *J. Am. Chem. Soc.* 58, 51-4 (1936); cf. C. A. 29, 4325<sup>1</sup>.—On heating  $\text{C}_7\text{H}_{16}$  with  $\text{AlCl}_3$  at 90° (90° in the still-head) there were obtained, on the basis of 100 parts  $\text{C}_7\text{H}_{16}$  reacted, the following products: pentanes and below, 64.6; 2-methylpentane, 3.4; 3-methylpentane, 2; hexane, 0.4; 2,1-dimethylpentane, 1.5; 2,2,3-trimethylbutane, 0.5; 3,3-dimethylpentane, 0.4; 2-methylhexane, 1.2; 3-methylhexane, 1.0; polymerization products, 2.1. Phys. properties used for the identification of the products are given. The results in general suggest that naphthenes are not formed. C. J. West

Induced substitution of pentene by chlorine. T. D. Stewart and Bernhard Weidenbaum. *J. Am. Chem. Soc.* 58, 98-100 (1936).—Cl and 2-pentene react in  $\text{CCl}_4$  to yield 1-chloro-2-pentene as well as the normal addn. product. Substitution is favored by increasing the pentene concn., addn. by increasing the relative concn. of Cl. The same effect is observed in 1-phenyl-2-propene to a smaller extent but not at all in the slowly reacting 2-chloropentene. A tentative explanation is suggested, based upon competitive reactions involving a common intermediate. The substitution product was identified by its reaction rate with  $\text{Me}_2\text{N}$ , the pseudo 1st order sp. reaction rate const. in mols. per l. at 25° being: the chlorinated 2-pentene 0.10; 1-chloro 2-propene 0.015,  $\text{CCl}_4$  and 2-chloropentene no reaction in 9 hrs.

Influence of substituents on the additive reactivity of ethylene derivatives. III. Further experiments on the

addition of bromine in solution. S. V. Anantakrishnan and Christopher K. Ingold. *J. Chem. Soc.* 1935, 1396-8, cf. C. A. 29, 6566<sup>1</sup>.—The work on the relative rates of addn. of simple  $\text{C}_4\text{H}_8$  derivatives toward the electrophilic reagent  $\text{Br}^+$  (C. A. 26, 78) has been extended to a study of the systems  $\text{C}_4\text{H}_8 + \text{PhCH CH}_2$ ,  $\text{PhCH CH}_2 + \text{MeCH CH}_2$ ,  $\text{Me}_2\text{C CH}_2 + \text{MeCH CH}_2$ ,  $\text{C}_4\text{H}_8 + \text{Me}_2\text{C CH}_2$ ,  $\text{C}_4\text{H}_8 + \text{Me}_2\text{C CHMe}$ ,  $\text{C}_4\text{H}_8 + \text{OHCH CH}_2$ , and  $\text{MeCH CH}_2 + \text{OHCH CH}_2$ . The expts. were carried out at -70° in  $\text{CH}_2\text{Cl}_2$  with exclusion of light. The "reduced rates," i. e., rates of reaction for  $\text{C}_4\text{H}_8$  derivs. expressed as multiples or fractions of the rate for  $\text{C}_4\text{H}_8$  itself, are given. An accumulation of Me groups causes an increase in the "reduced rate" of addn.:  $\text{C}_4\text{H}_8$  1.0,  $\text{MeCH CH}_2$  2.0,  $\text{Me}_2\text{C CH}_2$  5,  $\text{Me}_2\text{C CHMe}$  10.1,  $\text{Me}_2\text{C CMe}$  11. The CHO group, instead of deactivating more powerfully than  $\text{CO}_2\text{H}$ , actually increases the reaction rate quite appreciably.  $\text{OHCH CH}_2$  1.5,  $\text{OHCH CHMe}$  3.0. C. J. West

Mechanism of the addition of halogens to ethylenic linkages. Richard A. Ogg, Jr. *J. Am. Chem. Soc.* 57, 2727-8 (1935).—The following mechanism is suggested for the addn. of halogens to ethylenes:  $\text{Br}^+ + \text{RCH=CHR} \rightarrow \text{RCHBr}-\text{C}^+\text{HR}$ ,  $\text{RCHBr}-\text{C}^+\text{HR} + \text{Br}^- \rightarrow \text{RCHBr}-\text{CHBrR} + \text{Br}^-$ ; i. e., the addn. is catalyzed by the halide ion and the intermediate is a carbocation. The first reaction is to be regarded as a substitution reaction of the halide ion with 1 of the electron pair bonds of the ethylenic linkage and hence as attended by optical inversion of the intermediate C atom. The carbocation thus has a *trans*-configuration and since no configuration change attends the 2nd reaction, the final addn. product should be *trans*. A very similar mechanism probably obtains for addn. of  $\text{HX}$  to ethylenic linkages, the 2nd step being replaced by addn. of a H ion to the carbocation. Acetylenic linkages should behave similarly. C. J. W.

Photolodination of the ethylenic bond at low temperatures. Geo. S. Forbes and Arthur F. Nelson. *J. Am. Chem. Soc.* 58, 182-3 (1936).—When an equimol. quantity of I was added to 1-butene (0.01 M) in  $\text{CH}_2\text{Cl}_2$  or  $\text{CHCl}_3$  and the solns. illuminated, 90% of the I disappeared in  $\text{CH}_2\text{Cl}_2$  at -60 to -90° in 3 hrs. and 95% in  $\text{CHCl}_3$  at -60 to -70°, the solns. illuminated in quartz by a spark between high W steel electrodes evolved the I quantitatively within 1 hr., in 3 stages; at 20° for 100 hrs. 81% of the I was evolved, also in stages.

Peroxide effect in the addition of reagents to unsaturated compounds. X. The addition of hydrogen bromide to methylacetylene (allene). M. S. Kharasch, J. G. McNab and M. C. McNab. *J. Am. Chem. Soc.* 57, 1663-5 (1935); cf. C. A. 30, 718<sup>1</sup>; 28, 5037<sup>1</sup>.—The "normal" addn. of  $\text{HBr}$  to methylacetylene (I) produces exclusively 2,2-dibromopropane; under the influence of added peroxides,  $\text{HBr}$  adds to I to give the 1,2-isomer. The  $\text{HBr}$  must be passed into the I, contr. the added peroxide, at a temp. of -33° to -40° in order to effect a complete reversal of the "normal" addn. XI. The solvent effect in the addition of hydrogen bromide to isobutylene.

M. S. Kharasch and W. M. Potts. *Ibid.* 58, 57-60 (1936).  
 —Pure *tert*-BuBr is formed by the addn of HBr to *vacuo* to isobutylene (I) in the presence of solvents and antioxidants. *iso*-BuBr is formed in amts varying from 100 to 75% by the addn of HBr to I in the presence of solvents, air and peroxides. The conclusion is reached that, under rigidly maintained antioxidant conditions, solvents *per se* have no effect upon the direction of addn of HBr to I but affect markedly the rate of addn. This is interpreted as indicating that the apparent directive solvent effect observed under other than antioxidant conditions is in reality an effect of the solvent upon the relative rates of the 'normal' and peroxide-catalyzed addn reactions.  
 C. J. West

Reaction of butylacetylene with oxygen. Charles A. Young, R. R. Vogt and J. A. Nieuwland. *J. Am. Chem. Soc.* 58, 55-61 (1936). cf. *C. A.* 29, 1771.  
 Butylacetylene (I), which has been carefully purified to remove olefin impurities, forms a peroxide readily. Evidence is presented which indicates that the peroxide is not formed by a polymer or allene form of I.  $C_4H_5CO_2H$  has been identified as 1 of the decompn. products of the peroxide of I. This peroxide is quite unstable in a coned form, decomps to an acidic, viscous, dark brown, strongly lachrymatory liquid.  
 C. J. West

Alkyl acetylenes and their addition compounds. VII. Preparation of some ketals of alkyl acetylenes with the higher alcohols. D. B. Killion, G. F. Hennon and J. A. Nieuwland. *J. Am. Chem. Soc.* 58, 80-1 (1936). cf. *C. A.* 29, 7939.  
 —The action of butylacetylene or amylacetylene on a soln. of an alc. and  $Cl_2CCO_2H$  in the presence of a catalyst from red  $HgO$ ,  $Et_2O$ ,  $BF_3$  and  $MeOH$  gives the following 2,2-dialkyl alkanes: 2,2-diethoxyhexane,  $b_p$  68-69°,  $d$  0.835,  $n_D^{20}$  1.4087; dipropoxy homolog,  $b_p$  95-96°,  $d$  0.836,  $n_D^{20}$  1.4160; dibutoxy homolog,  $b_p$  115-117°,  $d$  0.837,  $n_D^{20}$  1.4240; dipentoxo homolog,  $b_p$  141-143°,  $d$  0.839,  $n_D^{20}$  1.4285; dihexoxy homolog,  $b_p$  143-145°,  $d$  0.839,  $n_D^{20}$  1.4322; 2,2-diethoxyheptane,  $b_p$  81-83°,  $d$  0.834,  $n_D^{20}$  1.4132; dipropoxy homolog,  $b_p$  107-109°,  $d$  0.836,  $n_D^{20}$  1.4191; dibutoxy homolog,  $b_p$  134-136°,  $d$  0.837,  $n_D^{20}$  1.4252; dipentoxo homolog,  $b_p$  148-150°,  $d$  0.839,  $n_D^{20}$  1.4310. With branched-chain monohydric alcs. Ketals have not been isolated by this method. *iso*-PrOH, *tert*-BuOH and *tert*-AmOH show no evidence of reaction.  
 C. J. West

The reaction of acetylene upon acetyl chloride. André Cornillot and René Alquier. *Compt. rend.* 201, 837-8 (1935).  
 —Acetylene reacted with  $AcCl$  at 15° in the presence of  $AlCl_3$  to form *Me*  $\beta$ -chlorovinyl ketone (I),  $b_p$  135°,  $b_d$  40°, in 25% yield. I polymerized to a resin on standing. The semicarbazone of I m. 180°. The oxime and phenylhydrazones were not formed. I in boiling alc. NaOH gave 50% triacetylbenzene.  
 E. W. Scott

Chlorination of methane. I. Preparation of carbon tetrachloride. A. I. Kiprianov and T. S. Kusner. *J. Appl. Chem. (U. S. S. R.)* 8, 673-84 (1935).  
 —Up to 90% of  $CCl_4$  was obtained when passing a mixt. of  $CH_4$ ,  $HCl$  and  $Cl_2$  through an activated C catalyst, at a reaction temp. of 525-80°. The ratio of the ingredients was  $Cl/HCl = 1/2$ . 160 l.  $Cl_2$  can be handled hourly by 100 g. activated C, and the approx. ratio, depending upon the purity, of  $Cl_2/HCl = 16/64$ . 80-100 l. A detailed description of the exptl. procedure is presented.  
 A. A. Bochtchik

Allylic rearrangements. I. Crotyl and methylvinylcarbinyl bromides. Saul Winstein and Wm. G. Young. *J. Am. Chem. Soc.* 58, 104-7 (1936).  
 —Details are given of results reported in *C. A.* 29, 7939.  
 C. J. West

Initial formation of methyl alcohol in the oxidation of methane. William A. Bone. *Nature* 136, 910 (1935). cf. Newitt and Srego. *C. A.* 29, 1770.  
 —Explosions of  $5CH_4 + 2O_2$  mixts. yielded among the suddenly cooled products 0.13%  $MeOH$  and 0.03%  $HCHO$ . Not the faintest trace of peroxide formation could be detected. The  $MeOH$  is formed before the  $HCHO$ .  
 O. E. S.

Action of sulfuric acid on 2-butanol. Nathan L. Drake and Fletcher P. Veitch, Jr. *J. Am. Chem. Soc.* 57,

2623-5 (1935).  
 —The action of 75%  $H_2SO_4$  on 2-butanol at 80° for 48 hrs. in an autoclave gives, among other products, 3,4-dimethyl-2-hexene (I) and di-*sec*-butyl ether (II). I was identified by the action of  $O_3$ , giving *Me* *sec*-Bu ketone (III) (2,4-dimethylphenylhydrazones, m. 71-72°), alk. HOBr transforms III into  $Me(CH_2CO)_2H$  (*p*-phenylphenyl ester, m. 70-6°). II b. 121°,  $d_4^{20}$  0.79,  $n_D^{20}$  1.3930, HBr gives *sec*-BuBr. The theory of the mechanism of polymerization by acids suggested by Whitmore (*C. A.* 28, 1327) explains the observed facts better than that of Kline (*C. A.* 29, 1057).  
 C. J. West

Synthesis of several octadecyl alcohols with branched chains. Otto Brunner and Gertrud Wiedmann. *Monatsh.* 66, 438-42 (1935).  
 —*Li*  $\alpha$ -ethylpalmitate, b. 198-203° (95% yield), amide, m. 111-12°, reduction of the ester with Na in  $EtOH$ -BuOH gives 70% of  $\alpha$ -ethylhexadecyl alc., b. (high vacuum) 100-10°, m. 15°, the phenylurethan is an oil, the 4'-chlorophenylcarbamate m. 105-6°. Di-*Et* butyldodecylmalonate, b. 224-6° (70% yield).  $\alpha$  Butylmyristic acid, b. 214-19° (77%), *Et* ester, b. 184-192°; amide, m. 10-10°;  $\alpha$ -butyltridecyl alc., b. 125-30° (high vacuum), m. 1°; 4'-chlorophenylcarbamate, m. 102-3°. Di-*Et* hexyldodecylmalonate, b. 222-5° (70%),  $\alpha$ -hexylauric acid, b. 218-20°, *Et* ester, b. 195-8°; amide, m. 110-11°;  $\alpha$ -hexyldodecyl alc., b. 120° (high vacuum), m. -7-5°; 4'-chlorophenylcarbamate, m. 77-8°. Di-*Et* dodecylmalonate, b. 220-3° (60%),  $\alpha$ -octylcapric acid, b. 212-18°, *Et* ester, b. 195-7°; amide, m. 122-5-3-5°;  $\alpha$ -octyldodecyl alc., b. 110-20° (high vacuum), m. -8°; 4'-chlorophenylcarbamate, m. 90°.  
 C. J. West

Formation of amides from nitriles by the action of hydrogen peroxide. L. McMaster and C. R. Noller. *J. Indian Chem. Soc.* 12, 652-3 (1935).  
 —Data for optimum conditions for the Radziszewski reaction as ded. from 2-8 expts. for 13 nitriles, using 3, 6, 12 and 30%  $H_2O_2$  in the presence or absence of  $EtOH$  are summarized. In general, the  $H_2O_2$  was added to a weighed amt. of the nitrile and sufficient 95% alc. to effect soln. was added (cf. Murray and Cloke, *C. A.* 29, 729). The soln. was made alk. with 6 N NaOH and was kept at 60° for 4 hrs. The alk. mixt. was cooled, neutralized with  $H_2SO_4$ , evapd and extrd. with  $CHCl_3$  or crystd. from  $H_2O$ . Conditions for the conversion of *Li*, *Pr*, *Bu*, *iso*-*Bu*, *Ph* $CH_2$ , *Ph* $CH$ ,  $CH_2$ , *CPh*,  $\alpha$ - $O_2NC_6H_5$ , *p*- $O_2NC_6H_5$ ,  $\alpha$ - $MeC_6H_4$ ,  $m$ - $MeC_6H_4$ ,  $(CH_3O)_2C_6H_3CH_2$  and  $\alpha$ - $O_2NC_6H_4C_6H_4CH_2$  nitriles into 49.6, 64.9, 62.5, 56.5, 59.5, 61.0, 83.0, 50.4, 91.2, 93.0, 84.8, 97.4 and 85.4% yields of the corresponding amides, m. 80-1°, 114.6°, 104.1°, 135.4°, 153°, 166.5°, 202-6°, 176.5°, 201.6°, 141.5°, 93.8°, 168.6° and 199.6° (all m. ps. cor.) are given (cf. *Org. Synthesis*, XIII, 94).  $H_2O_2$  in concns. up to 30% has no effect on neutral solns. of  $EtCN$  or  $\alpha$ - $O_2NC_6H_4CN$  at temps. up to 100° when decomposed catalytically by  $MeNO_3$ ,  $Co(OH)_3$ ,  $Co_2O_3$  or  $Ni_2O_3$ . The  $OH$  ion is evidently necessary as a catalyst in this reaction. C. R. Addinall

Vinylidiazomethane. Charles D. Hurd and S. C. Lu. *J. Am. Chem. Soc.* 57, 2076-7 (1935).  
 —Vinylidiazomethane (I) results in 22-5% yields by the method of Nurdlinger and Acree (*C. A.* 4, 1616). *Allyl*nitrosourea in red oil, decomps. on attempted distn. at 3 mm.,  $d_4^{20}$  1.051,  $d_4^{25}$  1.047 (94% yield). I and  $BrOH$  in  $Et_2O$  give, after 2 days, 65.7% of allyl benzoate. I (0.33 g.) in 100 cc.  $Li_2O$  gives, after 2 days, 0.72 g. pyrazole (cf. Adamson and Kenner, *C. A.* 29, 2910, 5413).  
 C. J. West

New method for the preparation of alkory boron halides and their reaction with metals. Corlies R. Kinney, H. Theron Thompson and Lee C. Cheney. *J. Am. Chem. Soc.* 58, 2396-7 (1935).  
 —Dissomethylboron chloride (I), b. 110-15°, results in 15% yield from *iso*-AmOH and  $BCl_3$  in dry  $CHCl_3$  at -20° but in 45% yield from (*iso*-AmOH) $B$  and  $BCl_3$  after standing 24 hrs. ( $EtO$ ) $B$  reacts readily with Na and Zn dust on warming slightly, gases being evolved and ( $EtO$ ) $B$  being isolated in about 75% yield. I does not react with "mol. Ag" but reacts readily with Na or Zn on warming, the reaction is exothermic and becomes violent unless cooled. The gases evolved at 70°

contain HCl and probably II and iso-C<sub>11</sub>H<sub>22</sub>; the liquid product contains only (iso-AmO)<sub>2</sub>B and B<sub>2</sub>O<sub>3</sub>.

**C. J. West**  
Preparation of dichlorodiethyllead A. I. Yakubovich and I. Petrov. *J. prakt. Khim.* 144, 67 8(1935).—Repetition of Gilman and Robinson's exp't (*C. A.* 24, 2981) on the action of HCl on I in PhMe gave about 50% Ph<sub>2</sub>CH<sub>2</sub>Cl, 30% Ph<sub>2</sub>CHCl and 20% Ph<sub>2</sub>CH<sub>2</sub>Cl. Pure Ph<sub>2</sub>CH<sub>2</sub>Cl results from the action of Cl upon Ph<sub>2</sub>CH<sub>2</sub> at -60° to -20° (Krause and Gruttner, *C. A.* 11, 976).

**C. J. West**  
Constitution of the bisulfite addition compounds of aldehydes and ketones. Walter M. Lauer and Carl M. Langhammer. *J. Am. Chem. Soc.* 57, 2360-2 (1935). The H<sub>2</sub>SO<sub>4</sub> addn comp'd of HCHO and presumably of all aldehydes and ketone bisulfites are salts of R<sub>2</sub>C(OH)SO<sub>3</sub>Me. The prep'n of C<sub>6</sub>H<sub>5</sub>SO<sub>3</sub>K from C<sub>6</sub>H<sub>5</sub>CHO and K<sub>2</sub>SO<sub>4</sub> is described (73% yield). Fusion with AcOK gives AcOCH<sub>2</sub>SO<sub>3</sub>K (I) (20.5% yield), also prep'd from HOCH<sub>2</sub>SO<sub>3</sub>K, AcO and AcOH, both decompose 211.6°, both form plates and flattened prisms from 95% 1:10H<sub>2</sub>O, the index of refraction corresponding to light vibrating parallel to the long direction of the prism is 1.491  $\pm$  0.002. C<sub>6</sub>H<sub>5</sub>SO<sub>3</sub>K and K<sub>2</sub>SO<sub>4</sub> give C<sub>6</sub>H<sub>5</sub>SO<sub>3</sub>K, reduction of C<sub>6</sub>H<sub>5</sub>SO<sub>3</sub>K gives MeSO<sub>3</sub>K, identified by the amide and amide I and KCN give AcOCH<sub>2</sub>SO<sub>3</sub>K, while PhNH<sub>2</sub> gives 85% of PhNHCH<sub>2</sub>SO<sub>3</sub>K.

**C. J. West**  
Reduction of aldehydes with aluminum isopropoxides. Wm. G. Young, Walter H. Hartung and Frank S. Crossley. *J. Am. Chem. Soc.* 58, 100 2(1936).—Al(iso-PrO)<sub>3</sub> is effective for the reduction of aldehydes to the corresponding alcs., other products are esters, aldol derivs. and resinous material. Details are given of the prep'n of crotyl alc. in about 51% yield in iso-PrOH or in 40.5% yield in C<sub>6</sub>H<sub>5</sub>l, citronellol was obtained in 32% yield and cinnamyl alc. in 68% yield. Reduction of PhCHO with Al(Oi-Pr)<sub>3</sub> in PhOH at 25° and in refluxing C<sub>6</sub>H<sub>5</sub>l gave 0 and 6% BuOH, while Al(iso-PrO)<sub>3</sub> gave 30 and 28% BuOH. With C<sub>6</sub>H<sub>5</sub>CHO only 14% crude crotyl alc. was obtained. The reaction mechanism is more complicated than previously reported.

**C. J. West**  
Synthesis of deuterioacetaldehyde. J. F. Ziegler and Darrell V. Sackman. *J. Am. Chem. Soc.* 57, 2735 (1935).—C<sub>2</sub>D<sub>2</sub> in an acid soln of Hg salts in D<sub>2</sub>O gives deuterioacetaldehyde, with a vapor pressure of 327 mm. at 0°; it shows the usual aldehyde reactions.

**C. J. West**  
 $\beta$ -Chloropropionaldehyde and several of its derivatives. A. Kirmann, M. Goudard and M. Chahudaz. *Bull. soc. chim.* [5], 2, 2143-52(1935).—The acetal and several other derivs. of ClCH<sub>2</sub>CH<sub>2</sub>CHO (I) have been prep'd and the comparative mobility of the Cl atom in the presence of the various groups has been studied. Distn. at 19 mm. of the product formed by the action of dry HCl on ClCH<sub>2</sub>CHO at -10° gave I and its trimer, which on distn. yielded I, b. 170-1°, d<sub>4</sub> 1.40°, d<sub>20</sub> 1.268, n<sub>D</sub> 1.175, M. R. 20 55 (calcd. 20.94). The mixt. formed by the passage of 2 mols. of dry HCl into ClCH<sub>2</sub>CHO in a large excess of MeOH was decanted from the aq. HCl layer, dist'd and dried over CaCl<sub>2</sub>. The crude product was freed from the di-Cl deriv. by the addn. of a large excess of MeOH and, on distn., gave ClCH<sub>2</sub>CH<sub>2</sub>CH(OMe) (II), b. 51°, d<sub>4</sub> 1.061, n<sub>D</sub> 1.427, M. R. 33 42 (calcd. 33.45). Treatment of II with dry HCl and distn. of the product yielded ClCH<sub>2</sub>CH<sub>2</sub>CH(OMe)Cl (III), b. 55°, d<sub>4</sub> 1.180, n<sub>D</sub> 1.450, M. R. 32 19 (calcd. 32.05). Similar treatment with dry HBr and rectification gave 3 fractions: trimethoxypropane, b. 45.6°, b. 140°, d<sub>4</sub> 0.942, n<sub>D</sub> 1.400, M. R. 34 49 (calcd. 34.60). ClCH<sub>2</sub>CH<sub>2</sub>CH(OMe)Br, b. 75°, d<sub>4</sub> 1.806, and BrCH<sub>2</sub>CH<sub>2</sub>CH(OMe), (IV), b. 58 60°, d<sub>4</sub> 1.311, n<sub>D</sub> 1.475, M. R. 36 49 (calcd. 36.35). The enhanced activity of the Cl atom does not show itself in double decomp. reactions but by favoring the elimination of H with the consequent reestablishment of the double link of ClCH<sub>2</sub>CHO. On refluxing with AcONa in AcOH, I regenerates ClCH<sub>2</sub>CHO. Treatment with AcO converted I into the diacetate, ClCH<sub>2</sub>CH<sub>2</sub>CH(OAc)<sub>2</sub>, in 47%, and gave also a mixt. of ClCH<sub>2</sub>CH(OAc) and ClCH<sub>2</sub>CHClOAc, identified

by their Raman spectra. A further fraction consisting of a polymer of I, b. 170-75°, was also isolated. The extreme instability of the Cl group was also manifested in the attempts to prep. I by decarboxylation or by hydrolysis of the hemiacetal. A NaHSO<sub>4</sub> complex can be isolated but the aldehyde loses the hydrolysis on sapon. of the complex with either dil. H<sub>2</sub>SO<sub>4</sub> or NaHCO<sub>3</sub>. Treatment of I with RMgX compds. leaves the Cl atom intact and the trimer, in 36%, is totally inert to these reagents. II and IV also contain a very mobile halogen atom and, on treatment with PtONa, AcONa or even AgO in the cold, split off HCl or HBr. However, the action of KCN on IV gave NCCl<sub>2</sub>CH<sub>2</sub>CH(OMe)Ph, b. 91°, d<sub>4</sub> 0.992, n<sub>D</sub> 1.416, M. R. 32 67 (calcd. 33.02), converted by treatment with MeMgBr into isonic acid, AcCH<sub>2</sub>CH<sub>2</sub>CH(OMe)<sub>2</sub>, b. 85°, d<sub>4</sub> 0.983, n<sub>D</sub> 1.421, M. R. 37 66 (calcd. 37.83). semicarbazone, m. 126°. Boiling IV with NH<sub>3</sub> in Me<sub>2</sub>CO produced L<sub>1</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(OMe)<sub>2</sub>, b. 85°, d<sub>4</sub> 0.888, n<sub>D</sub> 1.415, M. R. 50 34 (calcd. 51.0). IV was unreactive to refluxing with MeMgBr for 8 hrs. Agitation of II with a 4-fold excess of benzene in the presence of AlCl<sub>3</sub>, distn. of the product and recrystn. from 95% alc. yielded PhCH<sub>2</sub>CH<sub>2</sub>CH(OMe)Ph, C<sub>14</sub>H<sub>18</sub>O, m. 31°. III reacted vigorously with MeMgBr in the cold to give ClCH<sub>2</sub>CH<sub>2</sub>CH(OMe)Me, b. 125°, d<sub>4</sub> 0.964, n<sub>D</sub> 1.515, M. R. 31 82 (calcd. 31.81), and with PhMgBr to form the analogous ClCH<sub>2</sub>CH<sub>2</sub>CH(OMe)Ph, b. 113 4°, d<sub>4</sub> 1.075, n<sub>D</sub> 1.516, M. R. 51 76 (calcd. 51.31), which reacted with benzene in the presence of AlCl<sub>3</sub> to give a crude fraction, b. 170-40°, contg. PhCH<sub>2</sub>CH<sub>2</sub>CH(OMe)Ph, m. 31°. Attempts to cyclize III by boiling with Mg turnings in Et<sub>2</sub>O and in Bu<sub>2</sub>O failed. The results are discussed from the viewpoint of induced polarities, and the conclusion is drawn that the notion of the mobility of a group as a function of the structure of a mol. has little validity except in the surroundings of a well-defined reaction medium.

**C. R. Addison**  
Enolization as directed by acid and basic catalysts. III. The acid-catalyzed enolization of some secondary butyl ketones. Paul D. Bartlett and Charles H. Stauffer. *J. Am. Chem. Soc.* 57, 2580-9(1935), cf. *C. A.* 28, 3051f. —The competitive rates of enolization in the 2-EtMe-CHCOR, where R is Me, Et, cyclohexyl and benzyl, in glacial AcOH with HNO<sub>3</sub> as catalyst. The method has been checked with the observation that under these same conditions the rates of iodination and racemization of d-MeEtCHCOR are identical. The relationship between alkyl substitution and competitive enolization rate of an active II does not bear out any simple hypothesis of enolization mechanism previously advanced; it is opposite in menthone to what it is in the acyclic sec. Bu ketones.

**C. J. West**  
Chemistry of the lipides of tubercle bacilli. XLII. The composition of the timothy bacillus wax. The isolation of d-2-eicosanol and d-2-octadecanol from the unsaponifiable matter of the timothy bacillus wax. Mary C. Pangborn and R. J. Anderson. *J. Am. Chem. Soc.* 58, 10-14(1936); cf. *C. A.* 30, 501f. —The wax of the timothy bacillus (*C. A.* 23, 941) was purified by pptn. with Me<sub>2</sub>CO from Et<sub>2</sub>O (8 times), it contains 0.29% P, 0.41% N, 1.7% ash, m. 45°, 1 no. 20.5, sapon. no. 60.0, [a]<sub>D</sub> 15.1° (CHCl<sub>3</sub>). Sapon. of 50 g. wax gave 10.9 g. fatty acids giving alc-sol K salts, 1.7 g. fatty acids giving H<sub>2</sub>O-sol K salts, 8.5 g. acids giving alc-insol but Cl<sub>2</sub>-sol K salts, 12.0 g. diacid alc., C<sub>20</sub>H<sub>40</sub>O<sub>4</sub> (I), 0.5 g. higher optically active alcs., 1.5 g. crude glycerol and 2.3 g. crude triolol. I is obtained from the Cl<sub>2</sub>-insol portion of the K salt and m. 60-7°, [a]<sub>D</sub> 6.1°, mol wt 518, 1 no 15.2, the presence of a H<sub>2</sub>O group is shown by the formation of the Ac deriv., m. 41°, [a]<sub>D</sub> 8.1° (CHCl<sub>3</sub>). Me ester, amorphous, in 49°, [a]<sub>D</sub> 6° (CHCl<sub>3</sub>). Crystn. of the optically active alc. fraction from MeOH (9 times) gives d-2-eicosanol, m. 62.5-3°, [a]<sub>D</sub> 4.2° (CHCl<sub>3</sub>). Ac deriv., m. 37-7°, [a]<sub>D</sub> 1.5° (CHCl<sub>3</sub>). Me deriv., m. 39-40°, acid phthalate, m. 60-1°, oxidation of the alc. gives 2-eicosanone (II), m. 58.9°, semi-

carbazone,  $m$  128°, oxime,  $m$  73-4°. The mother liquors give *d*-2-octadecanol,  $m$  66°,  $[\alpha]_D^{25}$  5.7° (CHCl<sub>3</sub>), 7.3° (C<sub>6</sub>H<sub>6</sub>), phenylurethan,  $m$  72-3°, solidifies 66° and then  $m$  70-7°,  $[\alpha]_D^{25}$  7.9° (CHCl<sub>3</sub>), oxidation gives 2-octadecanone (III),  $m$  52°. II and III are identical with the products prepd. by Morgan and Holmes (C. A. 19, 2807).

C. J. West  
The sapropels as a source of high molecular carboxylic acids. D. A. Shvedov, S. A. Kuzin and A. I. Andreeva. *Mosk. Obshch. Ispyt. Prirodoz.* 1934, No. 5, 30-2, cf. C. A. 28, 59611. The sapropels can be readily oxidized with HNO<sub>3</sub> to high-mol. carboxylic acids with a yield of 30-60%.

L. Bielous  
Isotopic exchange reactions of organic compounds. I. The intermolecular nature of three-carbon tautomerism. D. J. G. Ives and H. N. Rydon. *J. Chem. Soc.* 1935, 1735-42, cf. C. A. 29, 432. The "equilibration" of vinylacetic (I) and crotonic acids (II) in the presence of 1.05 mols. of NaOH in dil. D<sub>2</sub>O at 100° has been studied, as a check, the behavior of PrCO<sub>2</sub>H (III) under the same conditions has also been examined. A method of isotopic analysis depending on d. dectms accurate to 1 part per million, applicable to small samples of H<sub>2</sub>O and involving no loss, is described. The exptl. results show that, although there is no detectable interchange with II and III, substantial interchange occurs in the case of I, on the basis of the necessarily tentative assumption that no isotopic discrimination occurs, the results indicate that 1 of the propene H atoms of I undergoes isotopic interchange with the solvent. It is concluded that purely intramolecular mechanisms for this type of 3-C tautomerism are unacceptable.

C. J. West  
Relative rates of ozonation of unsaturated compounds. C. R. Noller, J. I. Carson, H. Martin and K. S. Hawkins. *J. Am. Chem. Soc.* 58, 24-6 (1936).—Curves are given showing the relative rates of ozonation of a no. of unsatd. compds. Whereas a double bond, unaffected by the presence of other groups, adds O<sub>3</sub> extremely rapidly, the rate is markedly decreased when the double bond is conjugated with CO groups, 3 or more Ph groups or 2 Cl atoms attached to the doubly bound C atoms also decrease the rate of addn. Where 2 or 3 double bonds are conjugated with each other, 1 bond adds O<sub>3</sub> rapidly while the others add only slowly. In the case of *cis-trans* isomers, where the rate of addn. is decreased by other groups, the *trans*- is found to add more rapidly than the *cis* form. The procedure should prove valuable in the investigation of unsatd. compds. of unknown structure. Compds. studied include oleic acid, Me oleate, elaidic acid, 1-phenyl-1-cyclohexene, (CICH<sub>2</sub>)<sub>2</sub>, (PhC)<sub>2</sub>, PhC<sub>2</sub>CHPh, stilbene, vinylacetic acid, crotonic acid, cinnamic acid, crotonic aldehyde, itaconic acid, diphenylbutadiene, maleic acid, sorbic acid, citraconic acid, Me maleate, maleic acid, mesaconic acid and Me fumarate.

C. J. West  
Decenoic acid, C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>, in sperm head oil. Yoshiyuki Toyama and Tomotaro Tsuchiya. *J. Chem. Soc. Japan* 56, 1313-15 (1935), cf. C. A. 30, 315, 316. Decenoic acid (I) is sepd. from sperm-head oil. Consts. bp. 143-8°, d<sub>4</sub><sup>20</sup> 0.9238, n<sub>D</sub><sup>20</sup> 1.4507, n<sub>D</sub><sup>25</sup> 1.4488, M. R. 49.56 (theory 49.45). Me decenoate in acetone is oxidized with KMnO<sub>4</sub> and the acid esters are saponified. The free acids liberated by sapon. are azelaic and formic acid. Therefore, the structure of I is Δ<sup>9</sup>-decenoic acid. Also in *Bull. Chem. Soc. Japan* 11, 26-9 (1936). K. Kutsuta.

Highly unsaturated alcohols in sperm-blubber oil. Yoshiyuki Toyama and Goroku Akiyama. *J. Chem. Soc. Japan* 56, 1316-19 (1935).—From the higher-boiling fraction of unsaponifiable substances of sperm blubber oil, an ether-insol. bromide (I) is prepd. Dehydrobromination of I gave a mixt. of highly unsatd. alcs (II). II was converted into an acetate, analysis of which indicated that it consisted of C<sub>26</sub>H<sub>44</sub>O and C<sub>28</sub>H<sub>46</sub>O. Also in *Bull. Chem. Soc. Japan* 11, 29-34 (1936). K. Kutsuta.

Catalysis in organic chemistry. V. Decomposition of esters and acids by anhydrous zinc chloride. H. W. Underwood, Jr., and O. L. Baril. *J. Am. Chem. Soc.* 57, 2720-30 (1935), cf. C. A. 25, 3619.—The Me, Et,

Pr and Bu esters of monobasic aliphatic acids were not affected by the catalyst; Am esters decomposed very slowly into an unsatd. hydrocarbon and the aliphatic acid, the rate of decompn. increases with the no. of C atoms in the alc. group. The aliphatic monobasic acids themselves are not affected by the catalyst. Esters of aliphatic acids are all decomposed, the dibasic acid reacting with the ZnCl<sub>2</sub> to form the Zn salt with evolution of HCl; the HCl forms the alkyl halide with the unsatd. hydrocarbon. Esters of aromatic acids decompose into an unsatd. hydrocarbon and the aromatic acid, which decomposes into CO<sub>2</sub> and a satd. hydrocarbon (if the acid is monobasic), dibasic acids form the anhydride; benzyl esters decompose into the acid and the PhCH<sub>2</sub> groups polymerize. All unsatd. hydrocarbons evolved, whether aliphatic or aromatic, polymerize. Esters and acids which did not dissolve the ZnCl<sub>2</sub> were not themselves decomposed.

C. J. West  
Constitution of dimeric ketene. W. R. Angus, A. H. Leckie, C. G. Le Ièvre, R. J. W. Le Ièvre and A. Wassermann. *J. Chem. Soc.* 1935, 1751-5.—This study is an attempt to distinguish between the 3 possible formulas CH<sub>2</sub> CO CH<sub>2</sub> CO (I) = C(OH) CH CO CH<sub>2</sub> (II) = C(OH) CH C(OH) CH (III). The observed dipole

moment (3.16 in C<sub>6</sub>H<sub>6</sub>, 3.30 in CCl<sub>4</sub>, both × 10<sup>18</sup>) and the mol. refraction ([R<sub>D</sub>]<sub>D</sub> = 20.14) are incompatible with I and III is excluded by the heat of combustion (5321 = 1.5 cal./g. (const. vol.)). The dimeric ketene must therefore exist predominantly in the keto-enol form (II). Forms I and III could nevertheless be present in comparatively small concns. Dissoln. of the ketene has no great effect on the Raman spectrum and, therefore, presumably, on the constitution. Raman spectra are given for the pure liquid and the soln. in CCl<sub>4</sub>.

C. J. West  
New method of preparation of acetic anhydride and homologs. V. M. Rodionov, A. I. Smirnov and T. A. Aleksova. *Khim. Farm. Prom.* 1935, No. 2, 102-6.—The method is based on the following scheme. 2AcONa + N<sub>2</sub>O → Ac<sub>2</sub>O + NaNO<sub>2</sub> + NaNO, L. N. Stability of pinacولات in liquid ammonia solution. Charles B. Wooster and Donald S. Latham. *J. Am. Chem. Soc.* 58, 76-8 (1936), cf. C. A. 29, 707.—The di-Na derivs. of pinacol (I) and acetophenone pinacol (II) may be prepd. by the action of NaNH<sub>2</sub> on these glycols in liquid NH<sub>3</sub>. These pinacولات give no evidence of dissociation into metal alkyls. Na in liquid NH<sub>3</sub> replaces only 1 of the H atoms of I, it reacts extensively with II, not only replacing H but also cleaving the C—C bond.

C. J. West  
1,4- and 1,5-Oxido compounds. Synthesis of 1,5-oxidodecane. Adolf Franke, Alfred Kroupa and Otto Schmid. *Monatsh.* 66, 400-32 (1935); cf. C. A. 26, 4031.—Details are given of the prepn. of Me(CH<sub>2</sub>)<sub>9</sub>COCH<sub>2</sub>CO<sub>2</sub>Et in 80% yield, it bp. 109-12°. The Na deriv. with (CH<sub>3</sub>)<sub>2</sub>Br<sub>2</sub> gives Me(CH<sub>2</sub>)<sub>9</sub>COCH[(CH<sub>3</sub>)<sub>2</sub>Br]CO<sub>2</sub>Et, hydrolyzed by dil. HCl to Me(CH<sub>2</sub>)<sub>9</sub>CO(CH<sub>2</sub>)<sub>2</sub>OH (II), which is difficult to isolate at this stage and is converted to anhydride 1,5-ol-5-one (II), bp. 190-202°. With H<sub>2</sub>O II readily gives pure decan-1-ol-5-one (I), bp. 144-8°, m. 24-5-3°, the mol. refraction corresponds to the open-chain formula. Oxidation of 1.254 g. I with CrO<sub>3</sub> in AcOH gives 0.5 g. δ-ketocaproic acid (III), m. 56-4° and 0.27 g. of a neutral product, in 52°, an ester of I and III. Reduction of I with Na-Hg in LiOH yields 43% of decan-1,5-diol (IV), bp. 155-5-7°, m. 23-4°. Catalytic reduction of II with Pt gives 1,5-oxidodecane (V), bp. 198-5-200-5°, Br in CHCl<sub>3</sub> gives 1,5-dibromodecane, bp. 150-1-5°, which was transformed into the dinitrile and then to α-amylpimelic acid, whose diamide m. 161-5°. IV and 30% H<sub>2</sub>SO<sub>4</sub> give some V but largely the unsatd. alc., C<sub>10</sub>H<sub>18</sub>O, b. 228-31°, which yields 40% V with 57% H<sub>2</sub>SO<sub>4</sub> at 133°. (CH<sub>3</sub>)<sub>2</sub>Br<sub>2</sub> (70 g.) and BrONa, heated 9 hrs. at 200°, give 25 g. 6-bromohexan-1-yl benzoate (VI), bp. 187-9°, and 10 g. hexamethyleneglycolbenzoate, m. 55-5-6°. VI and AcCHNaCO<sub>2</sub>Et, followed by hydrolysis, give nonan-1-ol-8-one, m. 23-3-5°, this does not yield an oxide with

$H_2PO_4$ , but a high-mol. compd. of unknown constitution.

C. J. West

**Reduction of organic compounds by ethanalamines.** M. Meltner, C. Wohlbeg and M. J. Kleiner. *J. Am. Chem. Soc.* 57, 2554 (1935).—The ethanalamines (preferably the diethanalamine) reduce anthraquinone to anthranol,  $Me_2CO$  to  $Me_2CHOH$ , azobenzene to  $PhNH_2$ , chrysoidin to 1,2,4- $C_6H_3(NH_2)_3$ ,  $PhNO_2$  and  $o$ - $O_2NC_6H_4Me$  to azo, azoxy and amino compds. The yields from the last 2 compds. under various conditions are given. The reaction depends on the decomn of the  $NH_2$  alc into  $NH_3$  and an aldehyde.

C. J. West

**Some derivatives of  $\beta$ -sulfolpropionic acid.** Lucius A. Bigelow, Hugh W. Sigmon and David H. Wilcox, Jr. *J. Am. Chem. Soc.* 57, 2521-4 (1935).—The Ag salt of  $HO_2SCH_2CH_2CO_2H$  (I), from I and  $Ag_2O$ , is anhyd, contrary to Rosenthal (*Ann.* 233, 15 (1889)), heating with  $MeI$  at  $100^\circ$  for 6 hrs gives 78% of the *Me ester*,  $b_p$  172-3°,  $n_D^{20}$  1.7°,  $Et ester$ ,  $m$  4° (78%), *Pr ester*,  $m$  -7° (86%), *Bu ester*,  $m$  -25° (96%), phenylethyl ester,  $m$  80° (64%).  $PhCH_2Br$ ,  $PhCH_2I$  or phenacyl bromide did not react. The acid chloride (92% pure)  $m$  -9° ( $\pm 1^\circ$ ).  $PhNH_2$  gives the dianilide,  $PhNHCOCH_2CH_2SO_2NHPh$ ,  $m$  161°.  $NH_3$  in  $C_2H_5$  gives 53% of *NH*,  $\beta$ -sulfolpropionamide,  $m$  164-8°.  $Ba(OH)_2$  gives the *Ba salt* in a quant yield, with dil.  $H_2SO_4$  it appears to give the di-*NH*, salt of I, pyrolysis of which at  $111^\circ$  and 3-4 mm gives a quant yield of the acid *NH*, salt of I.

C. J. West

**Preparation of some of the lower alkyl sulfonic acids.** D. L. Vivian and E. Emmet Reid. *J. Am. Chem. Soc.* 57, 2559-60 (1935).—*Propylsulfonic acid*,  $b_p$  136°,  $n_D^{20}$  1.2516, *Bu homolog*,  $b_p$  147°,  $n_D^{20}$  1.252°, *i* *Am homolog*,  $b_p$  163°,  $n_D^{20}$  1.259°, *n* 1.226, *hexyl homolog*,  $b_p$  174°,  $n_D^{20}$  1.261°, *d* 1.047. The *Bu* compd was prep'd by oxidation of  $BuSH$  with concd  $HNO_3$ , the others were obtained from *Ba* salts with  $H_2SO_4$ . The *m* ps are compared with those of  $RCO_2H$ . *Butanesulfonyl chloride*,  $b_p$  90°,  $d_4^{25}$  1.2140,  $d_4^{25}$  1.2404. The *Bu* acid did not react with  $C_2H_5$  at  $80^\circ$ ; the  $PhNH_2$  salt of the acid,  $m$  162°, did not undergo any change at  $130^\circ$  in several hrs.

C. J. West

**Preparation and properties of  $\beta$ -alkylcholone chlorides and their acetyl esters.** Randolph T. Major and Howard T. Bonnett. *J. Am. Chem. Soc.* 58, 22-4 (1936).—1-*Chlorononan-2-ol*,  $b_p$  114.5-6.5°. 1-*Dimethylammonioalkan-2-ols* were prep'd from the appropriate chlorohydrin with 2 mols.  $Me_2NH$  in  $C_2H_5$  at  $115-20^\circ$  for 15 hrs. *Mer*- $NCH_2CH(OH)R$ , where *R* is *Et*,  $b_p$  142-4°, *Pr*,  $b_p$  73-4°, *Bu*,  $b_p$  89-90°,  $C_6H_{11}$ ,  $b_p$  99-101°,  $C_6H_{13}$ ,  $b_p$  104-6°. The methiodides were prep'd in the usual manner. *Me*- $NCH_2CH(OH)R$  *MeI*, where *R* is *Et*,  $m$  162-3°, *Pr*,  $m$  193-200°, *Bu*,  $m$  90-2°, *Am*,  $m$  98-100°,  $C_6H_{11}$ ,  $m$  109-10°,  $C_6H_{13}$ ,  $m$  122.5-3.5°. The corresponding chlorides: *Et*,  $m$  174-6°, *Pr*,  $m$  115-17°, *Bu*,  $m$  100.5-2°, *Am*,  $m$  72-4°,  $C_6H_{11}$ ,  $m$  69-71°,  $C_6H_{13}$ ,  $m$  97-9°. *Ac derivs* of the chlorides *Et*,  $m$  144-6°; *Pr*,  $m$  163-9°; *Bu*,  $m$  188-7°; *Am*,  $m$  182-4°;  $C_6H_{11}$ ,  $m$  169-71°;  $C_6H_{13}$ ,  $m$  178-7°. A preliminary report upon the pharmacol. action of these compds is presented.

C. J. West

**Experiments on the direct esterification of higher fatty acids with glycerol and with ethylene glycol.** T. P. Hilditch and J. G. Rigg. *J. Chem. Soc.* 1935, 1774-8.—In the direct esterification of higher fatty acids with  $C_2H_5(OH)$  or  $C_2H_4(OH)_2$ , the yield of monoglycerides can be very much increased by employing a medium in which both the alc and acid are freely sol, such as  $PhOH$  or  $MeC_6H_4OH$ . In expts using a wt. of  $PhOH$  equal to the wt. of acid much higher yields of monoglyceride are obtainable, according to the temp., time of reaction and the ratio of  $C_2H_5(OH)$  to acid in the reaction mixt.; in the most favorable circumstances the product may contain about 90% of monoglyceride. The presence of  $\beta$ - $CuI_2SO_4H$  considerably increases the total yield of esters but causes some diminution of the proportion of monoglyceride. Reichler's camphor- $\beta$ -sulfonic acid is a more efficient catalyst but it also lowers somewhat the percentage of monoglyceride. The higher the temp. the

greater the total yield but the percentage of monoglyceride again falls. The variations in the conditions of reaction have less effect on the yield of monoglycerides in the case of stearic and oleic acids than in the case of acids of lower mol wt. With  $C_2H_5(OH)$ , the total yield of esters and the proportion of monoacylated glycol therein were larger than in expts with  $C_2H_4(OH)_2$ , under the same conditions. Pure monolaurin or monopalmitin may be prep'd by crystn from  $Et_2O$  at  $0^\circ$ . Almost exclusive formation of a monoglyceride takes place in this reaction. Details of many expts are given.

C. J. West

**A new methylxethyl.** Joseph Wiemann. *Compt. rend.* 201, 1399-4 (1935). cf. *C. A.* 29, 3651<sup>r</sup>.—Treatment of crude vinylpropenyl glycol (I) with  $PhNCO$  gave a phenylurethane which, on sapon with  $Ba(OH)_2$ , yielded a liquid (II),  $b_p$  111°,  $d_4^{20}$  0.9344,  $n_D^{20}$  1.4800, *M* 36.57 (calcd. 36.64) corresponding to that of a vinylpropenyl glycol. Treatment of 1.8 g of II with 1.7 g of  $AgClO_4$  in 250 cc. of  $H_2O$  in the presence of 0.05 g of  $O_2O$ , gave 0.5 g of a methylxethyl (III),  $Me[CH(OH)]_2CH_2OH$ , *m* 127°, which formed a mixt of dibenzoz and tribenzoz acetals, *m* 151°, on heating with  $BzH$ . I is evidently a mixt. of 2 diastereoisomers which yields 2 phenylurethans and 2 methylxethyls, *m* 190° and 127°. III is the more fusible and is derived from the glycol forming the least sol. phenylurethane, and it is concluded that it has the steric formula  $MeCH(OH)[CH(HO)]_2CH_2OH$  identical from the 3rd C atom with that of allitol.

C. R. Addinall

**The preparation of pure sorbitol and sorbose and the influence of this ketose on the conductivity of boric acid.** J. Boeseken and J. L. Leefers. *Rec trav chim* 54, 851-5 (1935).—With a special catalyst ( $Ni$ ,  $Co$  and  $Cr$  on  $Ca_3(PO_4)_2$ ), glucose (100 g) in 500 cc. of  $EtOH$  was reduced completely to sorbitol (I) in 1-2 hrs. at  $150^\circ$  with 150 atm of  $H_2$ . I was purified by prep'd monobenzoxy-sorbitol, *m* 163-8°, from which I was obtained by hydrolysis. I in the presence of a culture of *Acetobacter suboxydans* was oxidized to sorbose (II) under carefully controlled conditions. The sp cond of II (0.1 M) was  $4.2 \times 10^{-4}$  mho, changing to  $6.1 \times 10^{-4}$  mho in 21 min. The sp cond of II (0.1 M) in  $H_2BO_3$  (0.1 M) soln was  $229 \times 10^{-4}$  mho, a change which can be observed to a lesser extent with fructose.

E. W. Scott

**$\alpha$ -Caprolactone.** F. J. Van Natta, J. W. Hill and W. H. Carothers. *J. Am. Chem. Soc.* 58, 183 (1936).—A reply to Stoll and Rouve's criticisms (*C. A.* 30, 1361<sup>r</sup>) of recent work (*C. A.* 28, 1603<sup>r</sup>).

C. J. West

**Odor and constitution.** Some  $\gamma$ -substituted  $\gamma$ -butyrolactones. B. Rothstein. *Bull soc. chim* [5], 2, 1936-44 (1935), cf. *C. A.* 29, 3308<sup>r</sup>.—The condensation of a series of monoalkyl-substituted ethylene oxides with  $NaCH_3CO_2Et$  (I) has provided a new method of synthesis of  $\gamma$ -substituted  $\gamma$ -butyrolactones, of which several are already known and used in perfumery. A series of chlorohydrins,  $RCH(OH)CH_2Cl$ , was prep'd by the method of Detoeuf (*C. A.* 16, 2113), which gave 40-60% yields contaminated with the difficultly separable *di*- $Cl$  derivs, and in which the rate of reaction diminished with increasing mol wt of the ethylene hydrocarbon. The following phys data are tabulated for the designated *R* substituent: *Bu*,  $b_p$  73-5°,  $n_D^{20}$  1.4478,  $d_4^{20}$  0.9139, *M* 36.02 (calcd. 36.30), *iso-Am*,  $b_p$  85-7°,  $n_D^{20}$  1.4475,  $d_4^{20}$  0.9310, *M* 40.62 (calcd. 40.91), *Am*,  $b_p$  91-2°,  $n_D^{20}$  1.4489,  $d_4^{20}$  0.9316, *M* 40.70 (calcd. 40.91);  $Me_2CH-$  ( $CH_3$ ),  $b_p$  99-100°,  $n_D^{20}$  1.4508,  $d_4^{20}$  0.9774, *M* 45.30 (calcd. 45.53). The ethylene oxides,  $RCH_2O$

where *R* is *Bu* (II), *iso-Am* (III), *Am* (IV) and  $Me_2CH-$  ( $CH_3$ ) (V) were prep'd by heating the corresponding chlorohydrins with  $NaOH$  (36% *Bz*) at  $40^\circ$  for 2 hrs with stirring. Theoretical yields of oxides where *R* is  $Me(CH_2)_n$  (VI),  $Me(CH_2)_n$  (VII),  $Me(CH_2)_n$  (VIII),  $Me(CH_2)_n$  (IX) and  $Me(CH_2)_n$  (X) were obtained by the action of  $Bz_2O_2H$  on a 50% excess of 1,2-ethylenes in  $CHCl_3$ . The following consts are tabulated: II,  $b_p$  123-4°,  $n_D^{20}$  1.4093,  $d_4^{20}$  0.8456, *M* 29.25 (calcd. 29.32); III,  $b_p$  54-5°,  $n_D^{20}$  1.4135,  $d_4^{20}$  0.8339, *M* 33.97 (calcd. 33.97); IV,  $b_p$  43-4°,  $n_D^{20}$  1.4148,  $d_4^{20}$  0.8362, *M* 33.97.



34.12 (calcd 33.97), V,  $b_{11}$  62-3°,  $n_D^{20}$  1.4230,  $d_4^{20}$  0.8481, M R 38.43 (calcd 38.58), VI,  $b_{11}$  75°,  $n_D^{20}$  1.4250,  $d_4^{20}$  0.8374, M R 43.36 (calcd. 43.20); VII,  $b_{11}$  64°,  $n_D^{20}$  1.4296,  $d_4^{20}$  0.8401, M R 47.93 (calcd. 47.82), VIII,  $b_{11}$  105°,  $n_D^{20}$  1.4330,  $d_4^{20}$  0.8415, M R 52.62 (calcd. 52.44), IX,  $b_{11}$  124-5°,  $n_D^{20}$  1.4355,  $d_4^{20}$  0.8402, M R 57.20 (calcd. 57.04), X,  $b_{11}$  139-9°,  $n_D^{20}$  1.4380,  $d_4^{20}$  0.8414, M R 61.77 (calcd. 61.68). A soln of 1 mol of ethylene oxide in an abs alc soln of 1 mol of I was kept at room temp for 2 hrs and was then boiled for several hrs. After the addn of the necessary NaOH the reaction mixt was saponified by boiling for several hrs longer. The alc was removed and after diln and extrn with Et<sub>2</sub>O the soln was acidified with dil H<sub>2</sub>SO<sub>4</sub>. The acid was extrd with Et<sub>2</sub>O and the washed and dried ext was evapd and distd *in vacuo*, giving 70% yields of the lactones, 1,4-octanolide,  $b_{11}$  127°,  $n_D^{20}$  1.4451,  $d_4^{20}$  0.9796, M R 38.58 (calcd 38.79), 1,1-nonanolide,  $b_{11}$  136°,  $n_D^{20}$  1.4462,  $d_4^{20}$  0.9672, M R 43.02 (calcd 43.23) (hydrazinolactone, m 84°), odor of coco faintly tinged with amineod, 7-methyl-1,4-octanolide,  $b_{11}$  136°,  $n_D^{20}$  1.4452,  $d_4^{20}$  0.9620, M R 43.17 (calcd 43.21), odor of essence of angelica seed, 8-methyl-1,4-octanolide,  $b_{11}$  145°,  $n_D^{20}$  1.4462,  $d_4^{20}$  0.9513, M R 47.63 (calcd 47.83), peach odor, 1,4-undecanolide,  $b_{11}$  162°,  $n_D^{20}$  1.4512,  $d_4^{20}$  0.9494, M R 52.21 (calcd 52.44) (hydrazinolactone, m 94-5°), strong peach-like odor, on which account and its relatively low price it is used as a base in many perfumes, 1,4-dodecanolide,  $b_{11}$  130°,  $n_D^{20}$  1.4522,  $d_4^{20}$  0.9393, M R 56.95 (calcd 57.07), slightly musk, peach like odor but heavier than the previous lactone, 1,4-tridecanolide,  $b_{11}$  142-3°,  $n_D^{20}$  1.4532,  $d_4^{20}$  0.9312, M R 61.57 (calcd 61.68), 1,4-tetradecanolide, m 29°,  $b_{11}$  140°,  $b_{12}$  193°, 1,4-pentadecanolide, m 32°,  $b_{12}$  159°. Sapon of the corresponding lactones gave hydroxydodecanic and hydroxytridecanic acids, m 63° and 66°, resp. I opens the O-bridge of the substituted ethylene oxides and fixes the HC(CO<sub>2</sub>Et)<sub>2</sub> group on the least substituted C atom. The above series shows that in a homologous series there are about 3 members which have fundamentally the same odor, modified by slight nuances. The branched-chain isomer has generally the same odor as the straight-chain compd but with a decided note of fresh green leaf. The  $\gamma$ -substituted butyrolactones from C<sub>11</sub> to C<sub>14</sub> are similar to their  $\alpha$  isomers in possessing an odor of musk and pear though the latter are stronger and more lasting. C R A

**Multivalent amino acids and peptides V. Cystine** cyamidine Jesse P Greenstein *J Biol Chem* 112, 75-8 (1935), cf. C A 29, 7761<sup>1</sup>.—The cyamidine deriv of cystine, anhydro- $\alpha,\alpha'$ -disulfanodisulfide ( $\beta$ -thiopropionic acid) is easily prepd by heating diguanidocystine with concd HCl, then adding ice-cold 3 N NH<sub>4</sub>OH to the soln of the HCl salt, it darkens above 240°. HCl salt, m 160°, half m 188°. It is extremely sensitive to alkali like all ring compds involving the disulfide linkage, and yields Na<sub>2</sub>S, pyruvic acid and guanidine, the latter substance being destroyed during the reaction with evolution of NH<sub>3</sub>. VI The action of proteolytic enzymes on certain synthetic substrates *Ibid* 57 22 (1936).—The diketopiperazine, anhydro-1-lysyl-1-glutamic amide, is completely resistant to pepsin, trypsin and papain-HCN and anhydrominotricarballylic tetramide is not attacked by papain HCN. While these results are not sufficient in themselves to discredit the diketopiperazine hypothesis, they do imply that considerable caution should be exercised in applying the anhydride structure to the proteins L-lysyl-1-glutamic acid and L-lysyl-1-histidine are hydrolyzed by yeast peptidase, the latter more slowly than the former. Glycyl- $\beta$ - $\alpha$ -aminocarballylic acid is slowly split by yeast and intestinal erepsin but is resistant to carboxy peptidase. A P Lothrop

**Synthesis of fumaric acid A M Bulligina *Maifobolno Zhivoe Delo* 1934, No 4, 43**—The prepn of C<sub>4</sub>H<sub>2</sub>(CO<sub>2</sub>H)<sub>2</sub> by oxidation of furaluril with NaClO<sub>2</sub>, with V<sub>2</sub>O<sub>5</sub> as catalyst, was carried out on a semi-com scale. Yield 74%. F Bielowski

**Amino acids and related compounds X Electrolytic oxidation of aspartic acid and malonic acid Yoshitane**

1 Takayama and Saburo Miduno. *J. Chem. Soc. Japan* 56, 1490-3 (1935); cf. C. A. 29, 7973<sup>1</sup>.—Use of a PtO<sub>2</sub> anode and aspartic acid (I) in dil H<sub>2</sub>SO<sub>4</sub> at 35° gives 10.8 mol % of HCO<sub>2</sub>H, 1.4 mol % malonic acid (II) and a small amt. of succinic acid (III), NH<sub>3</sub> and CO<sub>2</sub>. The same treatment on II gives HCO<sub>2</sub>H and CO<sub>2</sub>. The course of decompn. of I is the first oxidation product is malonic semialdehyde which decomposes to give II, and II gives HCO<sub>2</sub>H through HCHO. It is possible that 2 mols of II give a mol. of III. K Kutsuta

2 Allomucic acid and a new tetrahydroxydicarboxylic acid Théodore Posternak. *Helv Chim Acta* 18, 1233-7 (1935), cf. C. A. 29, 5817<sup>1</sup>.—The m p of synthetic dl-talomucic acid (I) is not lowered on mixing with dl-talomucic acid prepd by epimerization of mucic acid with pyridine (Fischer's allomucic acid). 0.7 g I dissolves in 100 cc H<sub>2</sub>O, the antipodes are sol. in 3-4 parts H<sub>2</sub>O. Di Et ester of I, prepd by each of the above methods, m 133-141°, mixed m p 138-140°. d-Allomucic acid (II) (cf. Levene and Jacobs, C. A. 5, 507) was prepd by the method of Austin and Hummoller (C. A. 28, 4383<sup>2</sup>). The new tetrahydroxydicarboxylic acid (III) was obtained in 0.6 g yield by HNO<sub>3</sub> (d 1.15) oxidation of 1 g of the lactone of II. III, m 197-8° (decompn.) (heating slowly), is slightly sol. in H<sub>2</sub>O, insol in alc and ether. The Na salt of III is quite sol. in H<sub>2</sub>O, this soln is optically inactive. III forms a di Et ester, m 153-4°, a monolactone, m 200-1° (decompn.), sol in H<sub>2</sub>O, slightly sol. in alc. and EtOAc, and a diphenylhydrazide, m. 227-229° (decompn.). W. Gordon Rose

3 The formation of hydrocyanic acid from organic compounds, in the presence of ammoniacal cupric sulfate Jacques Parrot *Compt rend* 201, 993-5 (1935).—Several compds were studied by use of the method previously described (C. A. 29, 5818<sup>1</sup>). The following nos express the no of mols HCN obtained from 100 C atoms in the substance, in 8 hrs (1) at 58-62°,  $\alpha$ -glucoheptose 2.6, d-fructose 17.5, d-sorbitose 15.6, d-glucose 5.0, d-mannose 3.1, d-galactose 0.3, glucosamine-HCl salt 12.7, l-arabinose 3.0, l-xylose 3.3, mesoxalic acid 16.6, tartaric acid 13.0, glyoxal 3.5, glyoxylic acid 18.4, lactose, maltose, sucrose, rhamnose, gluconic acid, tartaric acid, oxalic acid, formaldehyde and pyrogallol 0.0, (2) at 80-85°, lactose 2.5, maltose 2.6, rhamnose 8.5, glyoxylic acid 20.2, sucrose, gluconic acid, pyruvic acid, oxalic acid, formic acid, glycerol, alanine, formaldehyde, pyrogallol and hydroquinone 0.0. HCN is formed by the action of NH<sub>3</sub>-CuSO<sub>4</sub> on reducing sugars, and similar substances. W. Gordon Rose

4 Mechanism and heat of polymerization of fulmic acid V Fulmic acid Kurt Sennewald and Lothar Breckenbach *Ann* 520, 201-34 (1935), cf. C. A. 29, 123<sup>1</sup>.—S and B measure the rate and heat of polymerization of fulmic acid (I) at room temp in H<sub>2</sub>O solns and with various concns of H<sub>2</sub>SO<sub>4</sub>. I polymerizes by a sec order reaction,  $\gamma$ -decylsuccinylsuccinimide (II), and II reacts further, mostly with I to give a trimer (HONC), (III), and partly with itself to give isocyanic acid (IV). With decreasing concns of mineral acids, the rate of formation of II and III increases, but more in the case of III, thus decreasing the amt of IV formed. In the total absence of mineral acid, only III is formed, and the reaction appears to be trimol. The formation of II instead of isomitosuccinonitrile oxide, as postulated by Wieland (C. A. 19, 2807<sup>1</sup>) is demonstrated by the addn of halogen acids. AgONC in H<sub>2</sub>O is treated with HCl, the soln filtered, extrd with Et<sub>2</sub>O, and after distn the residue is washed with H<sub>2</sub>O, and dried, giving chloroglyoxime, m 152.5°. Similarly, bromoglyoxime, m 158° (anti-diacetate, m 100°), and iodoglyoxime, m 163°, are prepd. G. Calmgaert

5 Dicyanodiamide F Chastelain *Helv Chim Acta* 18, 1287-1302 (1935).—A crit. review is given of the literature on dicyanodiamide (I). Alk hydrolysis of I is believed to be a bimol reaction, since the rate of liberation of NH<sub>3</sub> is the same when the mol proportions of NaOH and I are reversed. C believes that I exists in a tautomeric equl between the forms HN C NH C (NH) NH

(II) and  $\text{H}_2\text{NC}(\cdot\text{NH})\text{NHCN}$  (III). Only II is present in alk. solns., and this is the preferred mode of representing I in the solid state and in neutral solns. III exists in acid solns.

Several derivatives formed by the action of phosgene, chloroformates and chloromethyl carbonates on cholesterol. André Kling and Maurice Rouilly *Compt. rend.* 201, 782-4 (1935).—Cholesterol (I) with  $\text{COCl}_2$  gave only cholesterol chloroformate, in 114%  $\text{CICO}_2\text{Me}$  did not react with I. The ester formed by treating I with  $\text{CICO}_2\text{CH}_2\text{Cl}$  was unstable. I with  $\text{CICO}_2\text{CHCl}_2$  and with  $\text{CICO}_2\text{CCl}_3$  gave dichloromethyl cholesterol carbonate, in 85%, and trichloromethyl cholesterol carbonate, in 107%. The facility of reaction of the chlorinated Me chloroformates with I increased as the no. of Cl atoms was increased.  $\text{CO}(\text{OCCl}_2)_2$  was still more active. These results confirmed the explanation offered by K. (C. A. 28, 1793) for pulmonary effects caused by inhalation of suffocating gases.

E. W. Scott  
Application of xanthate reaction to dehydration of binary  $\alpha$ -glycols. V. A. Fomin *J. Gen. Chem.* (U. S. S. R.) 5, 1192-4 (1935).—The absence of isomerization in the dehydration of secondary and tertiary alcs. by the thermal decarboxylation of the corresponding ethers of xanthic acid (cf. Chugaev, *Ber.* 34, 2276 (1901), 35, 2470, 2012 (1902)) made the use of this method of dehydration of  $\alpha$ -glycols interesting, because all other methods result in a radical change of the hydrocarbon skeleton. The pinacol deriv. of Me xanthate,  $\text{Me}_2\text{C}(\text{OCS}_2\text{Me})\text{C}(\text{OH})\text{Me}$ , is decomposed in the process of formation without  $\alpha$ -pin of COS and with the formation of MeS and the heterocyclic

stable compd.  $\text{S} \cdot \text{C} \cdot \text{O} \cdot \text{CMe}_2 \cdot \text{CMe}_2 \cdot \text{O}$  (I), in 176% ( $\text{Me}_2\text{CO}$ ) (sealed tube) I, heated with concd. KOH, gives pinacol hydrate,  $\text{K}_2\text{CO}_3$  and  $\text{K}_2\text{S}$ . The hydrate and I, heated with  $\text{H}_2\text{SO}_4$ , give pinacolone. A mixt. of 50 g. of dry pinacol in 500 g. xylene with 20 g. K. is refluxed for 25 hrs. The cold soln. of K pinacolate is treated with 85 g. of  $\text{CS}_2$  with shaking and then heated on a water bath for 4 hrs. After addition of 80 g. of MeI and refluxing for 6 hrs., the reaction mixt. is steam-distd. to expel the xylene and the I in the distn. flask is boiled with C. Chas. Blanc.

Reduction of nitroguanidine. II. Preparation and properties of nitroguanidine. V. J. Sabetta, David Hummelford and G. B. L. Smith *J. Am. Chem. Soc.* 57, 2478-9 (1935); cf. C. A. 26, 2442.—Reduction of nitroguanidine with Zn in 1.5%  $\text{NH}_4\text{Cl}$  gives 40-60% of the NO deriv. (I). Soln. of I in g. per 100 g.  $\text{H}_2\text{O}$ : 5°, 0.093; 15°, 0.118; 20°, 0.154; 25°, 0.185; 30°, 0.216; 40°, 0.305; 50°, 0.527; 72°, 1.23; 73°, 1.41. The intensity of the yellow color of an soln. is proportional to the concn. at const. temp. and  $p_H$  between 6 and 10; at 10° the color intensity is 70% of that at 40° and at  $p_H$  2 and 12 it is 80% of that at  $p_H$  7. I detonates at 161°, it may be detonated on the hand without injury. The isoelec. point of I is near  $p_H$  7. The decomposition in acid, neutral and basic soln. is monomol or pseudo-monomol. The energy of activation in acid and basic solns. is about 19,000 cal. per mol. and is probably somewhat higher in neutral solns. I may be analyzed by titration with  $\text{KMnO}_4$ ; 4 other analytical methods are indicated. III. Synthesis of aminoguanidine. G. B. L. Smith and Edward Anzelmi *Ibid.* 2730.—Me isothiourea sulfate and  $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$  in  $\text{H}_2\text{O}$  at 10° give 90% of aminoguanidine sulfate, with 1 mol.  $\text{H}_2\text{O}$ , in 206% (decompn.), it is a strong base,  $\text{A}_9$  estimated as  $1 \times 10^{-4}$ . IV. Preparation of nitroguanidine by catalytic hydrogenation. Eugene Lieber and G. B. L. Smith. *Ibid.* 2749-80.—Catalytic reduction of nitroguanidine with Adams' Pt oxide catalyst gives 55-60% NO deriv. and with Raney Ni catalyst 36-44%.  $\text{H}_2\text{O}$  is the most satisfactory for the Pt catalyst and is suitable for the Ni catalyst, though MeOH gives somewhat higher absorption rates with the latter. The optimum temp. is 25-35° and a decrease or increase of 10° results in a lowering of the rate of  $\text{H}_2$  absorption of 40-70%. With the Ni catalyst the optm. ratio is 0.5 g. of catalyst mass to 1 g.  $\text{NO}_2$  compd.

C. J. West

Colored compound formed in the Sullivan reaction for guanidine. M. X. Sullivan and W. C. Hess. *J. Am. Chem. Soc.* 58, 47-8 (1936).—The N detn. and the products of acid hydrolysis indicate that the colored compd. formed in the Sullivan reaction for guanidine is 4-guanidino-1,2-naphthoquinone. Hydrolysis yields guanidine and hydroxy-naphthoquinone. The guanidine deriv. of  $\beta$ -naphthoquinone,  $\text{C}_{10}\text{H}_8\text{N}_2\text{O}$ , m. 265-7° (decompn.), and on hydrolysis yields more or less  $\alpha$ - $\text{C}_{10}\text{H}_6\text{OH}$ .

C. J. West

Barbituric acid derivatives. II. A comparison of 2-mercapto compounds of 4-imino-5-methylthio-barbituric acid and 5-methylthio-barbituric acid. Takeichi Nishikawa *J. Chem. Soc. Japan* 56, 1497-04 (1935); cf. C. A. 29, 7947.—Compds. are prepd. by replacing the 2-H of 4-imino-5-methylthio- and 5-methylthio-barbituric acid by Me, Et, Pr, Bu and iso-Bu groups and their m. p., d, soly., cond.,  $p_H$  and the extent of enolization are estd. The differences in these properties are explained by the fact that the imino group gives stronger basicity and has larger space to be occupied than the keto group when the corresponding 4-imino and 4-keto compds. are compared. In a similar manner, the change in properties of the homologous series of 4-imino and 4-keto compds. are explained from the facts that the pos. character and the vol. to be occupied by the alkyl group in the 2-position are increased with an increase in the no. of C atoms.

K. Kutsuta

Synthesis of 1-d-glucosidocytosine. Guido E. Hilbert and Eugene J. Jansen *J. Am. Chem. Soc.* 58, 60-2 (1936).—2,4-Diethoxypyrimidine (50 g.) and 50 g. acetobromoglucose, heated at 65° overnight, give 28 g. of 1,2-dihydro-2-keto-4-ethoxy-1-tetraacetyl-d-glucosidopyrimidine (I), in 206% (cor),  $[\alpha]_D^{25}$  36.1° (c 12 in  $\text{CHCl}_3$ );  $\text{EtOH-HCl}$  and I form 1-d-glucosidouracil, heating I (3 g.) with  $\text{EtOH-NH}_3$  (satd. at 0°) at 80° for 96 hrs. gives 2.06 g. 1-d-glucosidocytosine (II), with  $\frac{1}{2}$   $\text{EtOH}$ , in 197-9% (decompn.), the hydrate m. about 128° and readily loses  $\text{H}_2\text{O}$  at 100° and 1 mm., anhyd. II is extremely hygroscopic and in 24 hrs. gains 0.6% of its wt. in  $\text{H}_2\text{O}$ , crystn. from 90-5%  $\text{EtOH}$  gives a product with both  $\text{EtOH}$  and  $\text{H}_2\text{O}$ , m. 194-5° (decompn.),  $[\alpha]_D^{25}$  23.7° ( $\text{H}_2\text{O}$ , c 2.8), anhyd.,  $[\alpha]_D^{25}$  25.6° ( $\text{H}_2\text{O}$ , c 1.8); picrate, m. 216-8° (decompn.), nitrate, with 1 mol.  $\text{H}_2\text{O}$ , m. 143° (decompn.),  $[\alpha]_D^{25}$  21.3° ( $\text{H}_2\text{O}$ , c 2.3). II hydrate and  $\text{Ac}_2\text{O}$  with  $\text{C}_2\text{H}_5\text{N}$  give 1-tetraacetyl-d-glucoside-7-acetylcytosine, m. 225°,  $[\alpha]_D^{25}$  38.1° ( $\text{CHCl}_3$ , c 1.7). The chem. properties of II are in general quite similar to those of cytidine.

C. J. West

Cardiac glucosides. XII. Empirical formulas of scillardin A and its derivatives. A. Stoll, A. Hofmann and J. Feyer *Helv. Chim. Acta* 18, 1247-51 (1935).—From mol. wt. detns., by titrimetric methods, of various acids of the bile and scillardin groups it is concluded that scillardin A (I), the aglucon of scillaren A, contains only 24 C atoms and is in agreement with the simple bile acids. The formula,  $\text{C}_{24}\text{H}_{40}\text{O}_8$ , formerly proposed for I is, accordingly, altered to  $\text{C}_{24}\text{H}_{40}\text{O}_8$ . The titrations were carried out by dissolving the compd. in 95% alc. and titrating with 0.1 N NaOH in the presence of phenolphthalein, allowance being made for the alkyl. of the solvent (0.1-0.3 cc. of 0.1 N NaOH). Details of exptl. results for allocholanolic, isoscillardinic (A), cholanic, dehydrocholanic and hydroxydehydroxycholanic acids are given. Values agreeing for mol. wt. corresponding to  $\text{C}_{24}\text{H}_{40}\text{O}_8$ ,  $\text{C}_{24}\text{H}_{40}\text{O}_9$ ,  $\text{C}_{24}\text{H}_{40}\text{O}_{10}$ ,  $\text{C}_{24}\text{H}_{40}\text{O}_{11}$  and  $\text{C}_{24}\text{H}_{40}\text{O}_{12}$  were found. New formulas for the following compds. previously described in this series of investigations during the years 1933-5 are: scillaren A,  $\text{C}_{24}\text{H}_{40}\text{O}_9$ , proscillardin A,  $\text{C}_{24}\text{H}_{40}\text{O}_{10}$ ; anhydroscillardin A,  $\text{C}_{24}\text{H}_{40}\text{O}_8$ , isoscillardinic acid A,  $\text{C}_{24}\text{H}_{40}\text{O}_9$ , methyl-scillardinic acid A,  $\text{C}_{24}\text{H}_{40}\text{O}_{10}$ , anhydroscillardinic acid A Me ester,  $\text{C}_{24}\text{H}_{40}\text{O}_9$ , scillanic acid,  $\text{C}_{24}\text{H}_{40}\text{O}_{10}$ ; octahydroscillardin A,  $\text{C}_{24}\text{H}_{40}\text{O}_{10}$ , octahydrodeoxy-scillardinic acid A,  $\text{C}_{24}\text{H}_{40}\text{O}_{11}$ , methyl scillarenic acid A,  $\text{C}_{24}\text{H}_{40}\text{O}_{11}$ , isoscillarenic acid A,  $\text{C}_{24}\text{H}_{40}\text{O}_{11}$ , hexahydrodeoxy-scillarenic acid A,  $\text{C}_{24}\text{H}_{40}\text{O}_{11}$ , hexahydrodeoxy-proscillardinic acid A,  $\text{C}_{24}\text{H}_{40}\text{O}_{12}$ .

C. R. Addinall

Ring-closure studies in the sugar benzoates. M. L. Wolfson and Clarence C. Christman. *J. Am. Chem. Soc.*

58, 39-43 (1936).—This work is a continuation of the ring closure studies in acylated sugar derivs (C. A. 29, 744<sup>9</sup>, 3313<sup>9</sup>). *l*-Arabinose (10 g) and 35 cc  $\text{BzCl}$  in 75 cc  $\text{C}_6\text{H}_5\text{N}$  (the soln of the sugar in the  $\text{C}_6\text{H}_5\text{N}$  is allowed to stand 24 hrs at room temp before the  $\text{BzCl}$  is added) give  $\alpha$ -*l*-arabinose tetraacetate (I), m 160-1°,  $[\alpha]_D^{25}$  112.5° ( $\text{CHCl}_3$ , c 4), the  $\beta$ -isomer in 173-4°,  $[\alpha]_D^{25}$  125° ( $\text{CHCl}_3$ , c 4). A mixt of 25 g I and 100 g  $\text{HBr}$  in 200 cc  $\text{AcOH}$ , allowed to stand overnight, gives 10.5 g benzobromo-*l*-arabinose (II), m 114-5°,  $[\alpha]_D^{25}$  204° ( $\text{CHCl}_3$ , c 2). *l*-Arabinose di-Me mercaptal (17 g) and  $\text{Ph}_2\text{CCl}$  in  $\text{C}_6\text{H}_5\text{N}$ , allowed to stand at room temp for 30 hrs and then treated with  $\text{BzCl}$  and  $\text{C}_6\text{H}_5\text{N}$ , give 35 g of *trityl-l*-arabinose di-*l*-*l* mercaptal triacetate, m 111-12°,  $[\alpha]_D^{25}$  -25° ( $\text{CHCl}_3$ , c 4), hydrolysis with  $\text{HBr-AcOH}$  followed by the action of  $\text{HgCl}_2$ ,  $\text{CaCO}_3$  in  $\text{Me}_2\text{CO}$  gives *l*-arabinose triacetate, m 162-3°,  $[\alpha]_D^{25}$  236° ( $\text{CHCl}_3$ , c 4), in  $\text{C}_6\text{H}_5\text{N}$  (c 4) the value changes from 143° to 196° in 12 hrs, the same compd results by hydrolysis of the Br atom in II the action of  $\text{BzCl}$  gives I. *d*-Galactose di-*l*-*l* mercaptal triacetate, m 127-8°,  $[\alpha]_D^{25}$  -16.5° ( $\text{CHCl}_3$ , c 4), results in 13 g yield from 25 g of the *trityl deriv*,  $\text{HBr}$  in  $\text{CHCl}_3$  gives the *6-Br deriv*, m 103-4°,  $[\alpha]_D^{25}$  -1.4° ( $\text{CHCl}_3$ , c 4), *d*-galactose tetraacetate methyl alcoholate, m 112-13°,  $[\alpha]_D^{25}$  6.5° ( $\text{CHCl}_3$ , c 4),  $\alpha$ -*d*-galactose pentaacetate, m 124-0°,  $[\alpha]_D^{25}$  187° ( $\text{CHCl}_3$ , c 4). *Trityl-d*-mannose di-*l*-*l* mercaptal triacetate, m 105-6°,  $[\alpha]_D^{25}$  0°,  $[\alpha]_D^{25}$  -10.5° ( $\text{CHCl}_3$ , c 3.5), the corresponding triacetate, m 133.5-4.5°,  $[\alpha]_D^{25}$  36° ( $\text{CHCl}_3$ , c 4). *d*-Mannose di-*l*-*l* mercaptal triacetate, m 110-17°,  $[\alpha]_D^{25}$  -5° ( $\text{CHCl}_3$ , c 2), *d*-mannose tetraacetate, amorphous,  $[\alpha]_D^{25}$  -115° ( $\text{CHCl}_3$ , c 2.3). The prepn of aldehyde-*d*-glucose pentaacetate, m 81-2°,  $[\alpha]_D^{25}$  40° ( $\text{EtOH}$ , c 3), from the di-*l*-*l* mercaptal is described. C. J. West.

Constitution of osazones. Lewis L. Engel. *J. Am. Chem. Soc.* 57, 2419-23 (1935).—The spectral absorption characteristics of the sugar osazones are practically identical and in concert with the structures originally suggested by Fischer. The bisphenylhydrazones of methylglyoxal and dimethylglyoxal have practically coincident absorption curves, the differences from those of the sugar osazones are ascribable to the presence in the latter of an O atom on the neighboring C atom. Osazones are readily susceptible to hydrolysis under mild conditions, the mutarotation of sugar osazones appears to depend upon the establishment of an equilibrium between them and their hydrolysis products. It was hoped that the question of the ring could be settled by a study of the methylation products of glucose phenylsazone but in no case was it possible to obtain homogeneous cryst products,  $\text{Me}_2\text{SO}$ , and alkali in dioxane yielded sirupy products with  $\text{MeO}$  contents from 16.3 to 22.6% and cryst mixts having 14 to 20.7%  $\text{MeO}$ , basic products, comprising *N*-methylated osazones and  $\text{PhNHNHMe}$ , were always formed. Methylation of partially methylated material with  $\text{MeI}$  and  $\text{Ag}_2\text{O}$  or  $\text{Ag}_2\text{CO}_3$  caused considerable decomposition. Neither methylation of partially acetylated glucose phenylsazone with  $\text{Me}_2\text{SO}$ , and alkali nor treatment of a partially methylated sirup with  $\text{Na K}$  alloy followed by  $\text{Me}_2\text{SO}$ , led to satisfactory results. Methylation of fructose methylphenylsazone (I) by the liquid  $\text{NH}_3$  method led to cleavage of the *N-N* linkage instead of methylation of the  $\text{NH}$  groups I and  $\text{PhNHNHMe}$  in  $\text{MeOH}$  after 1 week give glucose methylphenylsazone, m 192-1°,  $\beta$ - $\text{O}_2\text{NC}_6\text{H}_5$ ,  $\text{NHNHMe}$  gave the methylphenyl-*p*-nitrophenylsazone, orange, m 223.5-4.5° (m ps cor). Galactose phenylsazone yields a monoacetate deriv, yellow, m 183.5-4.5°, on standing with  $\text{P}_2\text{O}_5$  in  $\text{Me}_2\text{CO}$  at 0° for 1 hr. Glucose phenylsazone and  $\text{Ac}_2\text{O}$  in  $\text{C}_6\text{H}_5\text{N}$  give a tetra-*Ac* deriv, m 102-4°,  $[\alpha]_D^{25}$  -58.5° (97%  $\text{tOH}$ , c 0.431). The tetra-*Ac* deriv of I, orange-yellow, m 126-7°,  $[\alpha]_D^{25}$  -184.8° ( $\text{tOH}$ , c 0.403). C. J. West.

The structure of *d*-xylofomethylose. P. A. Levene and Jack Compton. *J. Biol. Chem.* 112, 775-83 (1936), cf. C. A. 29, 2923<sup>9</sup>.—Acetone-*d*-xylofomethylose on methylation by Purdie's method gave the *3-Me deriv* (I),  $b_p$  58.40°,  $n_D^{20}$  1.4777,  $[\alpha]_D^{25}$  -49.4° ( $\text{H}_2\text{O}$ ). Removal of the  $\text{Me}_2\text{CO}$  residu from I by 1%  $\text{H}_2\text{SO}_4$  gave

3-methyl-*d*-xylofomethylose (II),  $b_p$  100.2°,  $[\alpha]_D^{25}$  81° ( $\text{H}_2\text{O}$ ), phenylsazone, m 128-30°. II (2.4 g), refluxed with 1%  $\text{MeOH-HCl}$  (50 cc) for 1 hr, gave on distn 3-methyl- $\alpha$ -methylxylofomethyloside (III) (0.9 g),  $b_p$  58-62°,  $n_D^{20}$  1.4410,  $[\alpha]_D^{25}$  121.5° ( $\text{H}_2\text{O}$ ), and 3-methyl- $\beta$ -methylxylofomethyloside (IV) (1.0 g), m. 45-60°,  $b_p$  72-5°,  $[\alpha]_D^{25}$  -127.0° ( $\text{H}_2\text{O}$ ). III with Purdie's reagents gave 2,3-dimethyl- $\alpha$ -methylxylofomethyloside,  $b_p$  33-41°, m. 31-5°,  $[\alpha]_D^{25}$  151.0° ( $\text{H}_2\text{O}$ ). This, on oxidation with concd  $\text{HNO}_3$  for 7 hrs, during which the temp was raised to 100° in the 1st 30 min, gave *d*- $\text{H}_2\text{O}_2\text{CCH}(\text{OMe})_2\text{CO}_2\text{H}$  (V). Similarly from IV 2,3-dimethyl- $\beta$ -methylxylofomethyloside,  $b_p$  38-40°,  $n_D^{20}$  1.4201,  $[\alpha]_D^{25}$  -102.4°. This with  $\text{HNO}_3$  also gave V. *d*-Xylofomethylose (VI) (1 g) in  $\text{H}_2\text{O}$  (50 cc) and  $\text{Ag}_2\text{O}$  (12 g) were heated at 80° for 8 hrs and gave  $\text{AcOH}$ . Polarimetric data for the rate of lactonization of *d*-gulonemethyloside are given and indicate the formation of both  $\gamma$ - and  $\delta$ -lactones. These observations confirm the structure previously assigned to VI with the deoxy group at position 5 (C. A. 30, 84<sup>9</sup>). R. C. Elderfield.

The action of phosphate on hexoses. II. Ryuzaburo Nodzu and Kiyotada Matsui. *Bull. Chem. Soc. Japan* 10, 467-71 (1935); cf. C. A. 29, 4737<sup>9</sup>.—In the previous paper it was shown that when glucose was distd with 40%  $\text{K}_2\text{HPO}_4$ - $\text{K}_2\text{H}_2\text{P}_2\text{O}_7$  soln about 5% of the glucose was converted into acetol. To see if other salts had the same action 5 g of glucose was distd in the same way with 50 cc  $\text{Na}_2\text{HPO}_4$ - $\text{NaH}_2\text{PO}_4$  soln,  $p_H$  6.2-6.5,  $b_p$  106-0°. Acetol and a trace of  $\text{Ac}_2\text{O}$  were isolated as semicarbazones. Glucose (3 g), distd with an arsenate soln (40 g  $\text{KH}_2\text{AsO}_4$  + 5.2 g  $\text{KIIO}_3$  + 62 g  $\text{H}_2\text{O}$ ),  $p_H$  6.2-6.4,  $b_p$  104.5°, gave 1.2% acetol. Glucose and 40%  $\text{Na}_2\text{SO}_4$ ,  $p_H$  6.3-6.4,  $b_p$  104-6°, gave 3% acetol, as detd by iodometry, but some  $\text{SO}_2$  may have distd over. Some acetol semicarbazone was isolated. Solns of  $\text{KCl}$ ,  $\text{NaCl}$ ,  $\text{K}_2\text{SO}_4$ , and  $\text{Na}_2\text{SO}_4$ ,  $b_p$  103-6°, gave no acetol in Pyrex glass but did give traces of  $\text{CH}_3\text{I}$  forming compds in common glass. To det the effect of certain metallic ions on the rate of acetol formation the rate curve for purified glucose and phosphate soln was plotted and the same curve was plotted for similar solns contg 0.003-0.01  $M$  solns of salts. It was found that  $\text{Ca}^{++}$ ,  $\text{Mg}^{++}$ , and  $\text{Fe}^{+++}$  had little effect while  $\text{Zn}^{++}$  and  $\text{Cu}^{++}$  retarded the reaction.  $\text{Fe}^{++}$  alone accelerated the reaction. John E. Milbery.

The behavior of carbohydrates toward hot alkali solutions. R. S. Hillert and A. Wolter. *Angew. Chem.* 49, 54-5 (1935).—11 even sugars were heated between 100° and 170° with  $\text{Na}_2\text{CO}_3$  solns. This resulted in the evolution of  $\text{CO}_2$ , whose amt gave a measure of the alkali used and the amt of acid formed. The influence of temp and the quantity of soda upon the decomposition process were investigated. I xptl data are presented. Karl Kammermerer.

An improved preparation of diacetoneglucose. D. J. Bell. *J. Chem. Soc.* 1935, 1871-5.— $\alpha$ -Glucose (190 g) in 2 l  $\text{Me}_2\text{CO}$  contg 80 ml of concd  $\text{H}_2\text{SO}_4$ , on standing 4-5 hrs, gives 72% (102-106 g) of diacetoneglucose and 20 g of the monoacetone deriv. C. J. West.

Esters of the aldehydrol form of sugars. M. L. Wolfrom. *J. Am. Chem. Soc.* 57, 2198-500 (1935).— $\alpha$ -Hydo-Galactose pentaacetate  $\beta$  hemiacetal (I) (5 g) and 50 cc hot  $\text{AcCl}$ , cooled and poured into 1 l ice- $\text{H}_2\text{O}$ , give 3 g  $\alpha$ -aldehyde-1-chloro- $\alpha$ -galactose hexaacetate, m 174-5°,  $[\alpha]_D^{25}$  -41° ( $\text{CHCl}_3$ , c 4); this also results from the free carbonyl or aldehydrol forms of aldehyde-galactose pentaacetate.  $\text{AcBr}$  gives the 1-*Br deriv*, m 179-81°,  $[\alpha]_D^{25}$  -79° ( $\text{CHCl}_3$ , c 4), shaking with  $\text{Ag}_2\text{CO}_3$  and crysts from  $\text{EtOH}$  give I, thus showing that the acetyl halide was attached to C 1.  $\text{AcI}$  gives the 1-*I deriv*, yellow, m 152-3°,  $[\alpha]_D^{25}$  -111° ( $\text{CHCl}_3$ , c 2). These substances are the open-chain analogs of the cyclic acetalohexal sugars.  $\alpha$ -Aldehyde-Glucose pentaacetate and  $\text{Ac}_2\text{O}$  in  $\text{C}_6\text{H}_5\text{N}$  give aldehyde- $\beta$ -glucose heptaacetate, m 118.5-9.5°,  $[\alpha]_D^{25}$  8° ( $\text{CHCl}_3$ , c 4). It does not decolorize  $\text{Br}$  in  $\text{CCl}_4$  and on deacetylation with 1  $\text{N NaOH}$  followed by reacylation with  $\text{Ac}_2\text{O}$   $\text{AcONa}$  yields  $\beta$  glucose pentaacetate, lower yields are obtained from the pentaacetate and  $\text{Ac}_2\text{O}$  with  $\text{NaCl}$ , aldehyde-*l*-Arabinose hexaacetate,

m. 80.5°,  $[\alpha]_D^{25}$  -27° (CHCl<sub>3</sub>, c 1). These compounds may be expected to be formed in reactions involving the cyclic forms of the sugars, rather than the free carbonyl or aldehyde structures.

C. J. West

**Theory of sugar absorption by hydroxyanthraquinones.** II. *m*-Containing derivatives of hydroxyanthraquinone glycosides. Sándor Mállér. *Magyar Kémiai Folyóirat* 41, 9-15 (1935), cf. C. A. 29, 4001<sup>1</sup>—Hydroxyanthraquinone glycosides which retain the 1,2,9- or 1,8,9-dihydroxyl carbonyl grouping take up ammonia on their one carbonyl group with formation of the imino group. The specific effect of the O rich sugar radical makes this possible. The structure of the derivs. is discussed on the basis of decomposition expts.

S. S. de Mirly

**Multiplanar cyclohexane rings.** R. D. Dean and R. F. Hunter. *Nature* 136, 953-4 (1935), cf. C. A. 30, 436<sup>1</sup>. D and H confirm their former contention that only 2 stereoisomers of this type of ring are formed.

Gerrit M. Evans

**Ketones obtained by condensation of saturated hydrocarbons with acetyl chloride in the presence of aluminum chloride.** Costin D. Nemitzescu and Grigore G. Vantu. *Bull. soc. chim.* [5], 2, 220 (20) (1935)—In the presence of AlCl<sub>3</sub>, cyclohexane (I) is transformed by the action of AcCl into methylcyclopentane which loses H<sub>2</sub> and then adds AcCl to form a Cl ketone. In the presence of inactive AlCl<sub>3</sub> this intermediate splits out HCl and gives 1-methyl-2-acetylcyclopentene (II) but with active AlCl<sub>3</sub> hydrogenation takes place simultaneously and produces the said 1-methyl-2-acetylcyclopentane (III), b. 161.5° (cor). The distn. by the aid of a Golodetz column of 1500 g. of ketones obtained from I and AcCl failed to isolate any compd. with a b. p. in the range of that of acetylcyclohexane, b. 179.5-80.5°, and, contrary to the findings of Zelinskii and Tarnasova (C. A. 28, 3381<sup>1</sup>), proved that hexacyclic ketones are not formed in this reaction. The structure of II was established by heating 21 g. of methylcyclopentanecarboxyl chloride (IV) with 22.5 g. of Br in 3 scaled tubes at 125° for 5 hrs. The combined product was vacuum-distd. and yielded 22 g. of product, bp. 94-103°. This was taken up in a 3 fold amt. of benzene contg. 2 mols. of NaN<sub>3</sub> activated with (NH<sub>4</sub>)<sub>2</sub>H<sub>2</sub>O (C. A. 28, 2320<sup>1</sup>) and heated for 40 min. at 80°. The cooled soln. was saponified with 2.5 mols. KOH, neutralized with HCl and steam-distd. The distillate was extd. with Et<sub>2</sub>O and redistd., yielding a fraction between 130-40° consisting of 1-methyl-2-cyclopentanone (semicarbazone, m. 173°). The distillate contained also a solid, 1-methyl-2-cyclopentanecarboxylic acid, m. 131.5°, formed by the elimination of HBr from the brominated acid. The formation of III from I by the action of AcCl in the presence of AlCl<sub>3</sub> provides a stirring material for the ready synthesis of a great no. of derivs. The Clemmensen reduction of 40 g. of III with 400 g. of Zn-Hg and an excess of concd. HCl produced a mixt. of 78.7 and 21.3% of the trans and cis forms (C. A. 26, 1111<sup>1</sup>) of 1-methyl-2-ethylcyclopentane, b. 121°, d<sub>4</sub><sup>20</sup> 0.7728, n<sub>D</sub><sup>20</sup> 1.42835, M. R. 37.36 (calcd. 36%). Reduction of III with Na and moist alk. gave a secondary alk. which was dehydrated by the aid of oxalic acid. The loss of H<sub>2</sub>O can also take place by enlargement of the ring (C. A. 13, 1831) and, accordingly, the dehydration was carried out by Frank's modification of the method of Chugaev (C. A. 27, 3552) which cannot produce catenary isomerization. To a mixt. of 120 cc. Et<sub>2</sub>O, 10 cc. CCl<sub>4</sub>, 25.6 g. of carbonyl and 81 g. of finely powd. KOH was added 15.2 g. of CS<sub>2</sub> over a period of 1 hr., maintaining the temp. at 30°. After 3 hrs. 50 g. of MeI was added dropwise and the stirring was continued for 6 hrs. The KI was filtered off and the xanthogenate was vacuum-distd., yielding 20 g. of crude yellow liquid, bp. 80°. After redistn. and washing with KOH and with HgCl<sub>2</sub>, the distn. over Na gave 1-methyl-2-ethylidenecyclopentane (V), b. 123-4°, d<sub>4</sub><sup>20</sup> 0.7915, n<sub>D</sub><sup>20</sup> 1.44421. V, contaminated with a small amt. of a hexacyclic isomer, was also obtained by dehydration of 24 g. of the alk. with 13 g. of KHSO<sub>4</sub>. Condensation of 12 g. of III with 10.6 g. of HBr in the presence of 3 cc. of dil. alk. MeON gave 1-(1-methyl-2-

cyclopentyl)-1-keio-3-phenyl-2,3-propylene, b. 153°, d<sub>4</sub><sup>20</sup> 1.0180, n<sub>D</sub><sup>20</sup> 1.57220, di-Br deriv., m. 107°. Oxidation of III with NaOBr readily gave the carboxylic acid whose *l*-ester, C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>, b. 179-80°, d<sub>4</sub><sup>20</sup> 0.9397, n<sub>D</sub><sup>20</sup> 1.43130, was reduced with Na and alc. to 1-methyl-2-hydroxymethylcyclopentane, bp. 171-2°, d<sub>4</sub><sup>20</sup> 0.9146, n<sub>D</sub><sup>20</sup> 1.4512. By heating with 120 cc. of 45% HBr for 4 hrs. on the steam bath, 20 g. of the alc. was converted into 17 g. of the Br deriv. (VI), b. 65°, d<sub>4</sub><sup>20</sup> 1.2608, n<sub>D</sub><sup>20</sup> 1.4835, which, on treatment with the calcd amt. of Mg, yielded 1,2-di-methylcyclopentane, b. 93-4°, d<sub>4</sub><sup>20</sup> 0.7013, n<sub>D</sub><sup>20</sup> 1.41745. Sapon and distn. of the condensation product of VI and NaCH(CO<sub>2</sub>H)<sub>2</sub> produced 1-methyl-2-cyclopentyl-β-propiionic acid, b. 147-8°, acid chloride, b. 98°, amide, m. 94°. The catalytic hydrogenation of 20 g. of IV in 100 g. of xylene in the presence of 4 g. of Pd charcoal for 16 hrs. until the evolution of HCl ceased and isolation of the alkide with 10% NaHSO<sub>4</sub> gave 1-methyl-2-formylcyclopentane, b. 118-50°, d<sub>4</sub><sup>20</sup> 0.9060, n<sub>D</sub><sup>20</sup> 1.43558, semicarbazone, m. 121.5°.

C. R. Addinall

**Phosphoric acid as catalyst for alkylation of aromatic hydrocarbons.** V. N. Ipatieff, Herman Pines and V. I. Komarevsky. *Ind. Eng. Chem.* 28, 222-3 (1936). Benzene, naphthalene and tetralinonaphthalene were alkylated with C<sub>2</sub>H<sub>5</sub>I, and naphthalene and fluorene were alkylated with propene, using 85-95% H<sub>3</sub>PO<sub>4</sub> as catalyst. Among the products in the last case were identified: mono-, di-, sym-tri- and tetra-ethylbenzene, diethyl-naphthalene, mono- and di-ethyltetralinonaphthalene, in the 2nd case propylnaphthalene and propylfluorene.

O. W. Wilcox

**Behavior of ketene in the Friedel-Crafts reaction.** F. S. Spring and T. Vickerstaff. *J. Chem. Soc.* 1935, 1873-4.—Ketene and C<sub>6</sub>H<sub>6</sub> with AlCl<sub>3</sub> give PhAc and p-*l*-C<sub>6</sub>H<sub>4</sub>Ac (probably due to the presence of C<sub>6</sub>H<sub>5</sub>I), whose semicarbazone m. 106°. Ketene, passed into PhOH at 80°, causes quant. acetylation. C. J. West

**Kinetics of the Friedel-Crafts reaction and activity of mixed catalysts in the reaction of benzoyl chloride with toluene.** Lawrence F. Martin, Philip Pizzolito and Lynn S. McWaters. *J. Am. Chem. Soc.* 57, 2584-9 (1935).—A correct method of calcg. velocity constants, from data obtained by Steele's method has been applied to expts. on the Friedel-Crafts reactions of BrCl and PhCH<sub>2</sub>Cl with PhMe. The relative order of activity of various catalysts depends upon the type of reaction and in the case of BrCl the activity increases with increasing percentage of FeCl<sub>3</sub>. Mixed catalysts covering a wide range of compn. of AlCl<sub>3</sub> and FeCl<sub>3</sub> have been investigated in respect to the reaction of BrCl with PhMe. At comparable concns. the activity increases with increasing mole percentage of FeCl<sub>3</sub> and reaches a max. at percentages in excess of 50 mole %. The mixed catalysts have been shown to produce less than 1 mole of product for each mole of total metal chlorides present. Evidence is given in support of the explanation that this is due to the removal of the catalyst in the form of a bimetal complex with the reaction product, such as Br<sub>2</sub>AlCl<sub>2</sub>·C<sub>6</sub>H<sub>5</sub>·AlCl<sub>3</sub>·FeCl<sub>3</sub>. Variation of the reaction velocity over a 10-fold range of concn., the PhMe being always in large excess, has been detd. and is of the same order of magnitude as that reported in a similar reaction by Olivier. The present work furnishes evidence in support of the explanation of this effect as being due to assocn. of the reacting complex. The new exptl. data and method of calcn. lead to results in satisfactory agreement with a unimol. order for the reaction, and thus lends support to the mechanism proposed by Steele (*J. Chem. Soc.* 83, 1470 (1903)).

C. J. West

**Oxidation in the benzene series by gaseous oxygen.** V. The oxidation of tertiary hydrocarbons. H. N. Stephens and Feliciano L. Roduta. *J. Am. Chem. Soc.* 57, 2380-1 (1935); cf. C. A. 22, 3883.—Oxidation of tertiary hydrocarbons always takes place at the α-C atom; the reactions are not inhibited by H<sub>2</sub>O. MeI<sup>+</sup>PhCH<sub>2</sub>, MeI<sup>+</sup>PhCH and MeI<sup>+</sup>PhCH<sub>2</sub> give PhAc, MePhCH<sub>2</sub>, PhCH and PhCH<sub>2</sub> give PhCO, PhCH<sub>2</sub>, is not oxidized in the presence of H<sub>2</sub>O.

C. J. West

Reaction of paraffins with aromatic hydrocarbons (destructive alkylation). Aristid V. Grose and V. N. Ipatieff. *J Am Chem Soc* 57, 2415-19 (1935).— $\text{Me}_2\text{CCH}_2\text{CHMe}_2$  (I) and  $\text{C}_6\text{H}_6$  with  $\text{AlCl}_3$  or  $\text{ZrCl}_4$  give  $\text{Me}_2\text{CCH}_2$  and  $\text{PhCMe}_2$  (II). It then reacts with more I to give  $\text{Me}_2\text{CCH}_2$  and  $p\text{-C}_6\text{H}_4(\text{CMe}_2)_2$ . With  $\text{AlCl}_3$  the reaction takes place at 25-50° and the ordinary pressure during about 4 hrs., with  $\text{ZrCl}_4$  a temp. of 50-75° is required.  $\text{HCl}$  gas promotes the action of the chlorides (II), and  $\text{MgCl}_2$  have no action. Complete details of the reaction are given. The reaction also occurs with other aromatic hydrocarbons and with other paraffins.

C. J. West  
The hydrocarbon  $\text{C}_{11}\text{H}_{12}$ . H. Ikenmeyer and H. Loebeck. *Helv Chim Acta* 18, 1461-6 (1935).—Different values have been reported for the b. p. and m. p. of  $\text{C}_{11}\text{H}_{12}$  (I) prepd by different methods. Chemo and McQuillen (C. A. 29, 5820) report  $\text{C}_{11}\text{H}_{12}$  in -1.0°, b. 82.5°, Wilson (C. A. 29, 7155), m. 6.5°, b. 0.8° lower than Chemo. I was prepd by heating  $\text{Ca}(\text{CO}_3)_2$  with  $\text{Ca}(\text{OD})_2$  (cf. von Bayer, *Ann Suppl* 7, 1 (1870)).  $\text{Ca}(\text{OD})_2$  was prepd by slaking  $\text{CaO}$  with  $\text{D}_2\text{O}$  in *vacuo*. Comparative details were made of the b. p. and m. p. of  $\text{C}_{11}\text{H}_{12}$  and  $\text{C}_{11}\text{H}_{14}$ , b. 79.2°, m. 5.5°,  $\text{C}_{11}\text{H}_{14}$  b. 78.5°, m. 6.8°. I and L discuss the sources of error inherent in the detn. of C and D in org. compds. W. Gordon Rose

Monodeutenobenzene [benzene-d] Noriyoshi Morita and Toshio Tani. *Bull Chem Soc Japan* 10, 537-8 (1935).—PhD was prepd from  $\text{CaO}$  and 97%  $\text{D}_2\text{O}$  by heating the resulting  $\text{Ca}(\text{OD})_2$  at 300° with  $(\text{D}_2\text{O})_2\text{Ca}$  and distg off and drying the PhD with  $\text{P}_2\text{O}_5$ .  $\text{C}_{11}\text{H}_{12}$  prepd in the same manner using  $\text{H}_2\text{O}$  was compared with this product.  $\text{C}_{11}\text{H}_{12}$  has d<sub>4</sub> 0.8754 and  $\text{C}_{11}\text{H}_{12}\text{D}$  0.8809. If the mol. vols. are the same 97% PhD should have d<sub>4</sub> 0.8804. The m. p. of PhD was about 0.1° higher than for  $\text{C}_{11}\text{H}_{12}$ . n. of PhD for white light at 23° is about 0.00065 smaller than for  $\text{C}_{11}\text{H}_{12}$ . Oden E. Sheppard

Polymethylbenzenes XIII Mercuration Lee I. Smith and I. Lowell Taylor. *J Am Chem Soc* 57, 2370-2 (1935), cf. C. A. 29, 5820.—The following acetoxymercuric acids were prepd by refluxing the hydrocarbon with  $\text{Hg}(\text{OAc})_2$  in  $\text{MeOH}$  or  $\text{AcOH}$  for varying periods.  $\text{C}_{11}\text{H}_{12}\text{Me}$ , m. 180°, 80% yield, 1,2,4,5- $\text{C}_{11}\text{H}_7\text{Me}_2$ , m. 158-9°, 60% solv., 12 g per 100 cc boiling  $\text{MeOH}$ , 3 g at 0°, 1,2,3,5- $\text{C}_{11}\text{H}_7\text{Me}_2$ , m. 105°, 30% 1,2,3,4- $\text{C}_{11}\text{H}_7\text{Me}_2$ , m. 147°, 15%, 1,3,5- $\text{C}_{11}\text{H}_7\text{Me}_2$ , m. 102.3°, 30%, 1,2,4- $\text{C}_{11}\text{H}_7\text{Me}_2$ , m. 115-7°, 30%. The action of  $\text{HCl}$ ,  $\text{NaBr}$  or  $\text{NaI}$  on the above compds gave halomercuric acids.  $\text{C}_{11}\text{H}_{12}\text{Me}$ ,  $\text{Cl}$ , m. 205°,  $\text{Br}$ , m. 195.4°,  $\text{I}$ , m. 188-9°,  $\text{Br}$ , m. 174°,  $\text{I}$ , m. 161.2°. Isodurene,  $\text{Cl}$ , m. 174°,  $\text{Br}$ , m. 160°,  $\text{I}$ , m. 163.4°. Prehnitene,  $\text{Cl}$ , m. 216.15°,  $\text{Br}$ , m. 213.14°,  $\text{I}$  m. 200°. Digestion of the iodides with 4 mols  $\text{NaI}$  in  $\text{LiOH}$  gives bis(poly)methyl phenylmercury compds.  $\text{C}_{11}\text{H}_{12}\text{I}_2$ , m. 278° durene, m. 242-3° isodurene, m. 217.18°, prehnitene, m. 200°.

XIV Reaction between organomercury compounds and nitrosyl compounds. Ibid. 2403-3.—The action of  $\text{NOCl}$  upon organomercury compounds of polymethylbenzenes gives  $\text{NO}$  compds. Thus, 20 g of the acetoxymercuric deriv. of  $\text{C}_{11}\text{H}_{12}\text{Me}$  in 100 cc  $\text{CHCl}_3$ , treated with 5 cc  $\text{EtNO}$ , 15 cc concd  $\text{HCl}$  and 20 cc  $\text{AcOH}$  gives 50% of nitrosopentamethylbenzene, m. 160°, reduction gives  $\text{H}_2\text{NCMe}$ . Nitrosodurene (I), m. 160° (decompn.), 73% yield, nitrosodurene, m. 132°, 61% the 3f $\text{OH}$  addn product, m. 135°, nitrosoprehnitene, green, m. 72°, 70%  $\text{NO}_2$  also forms  $\text{NO}$  compds. Oxidation of I or the action of  $\text{HNO}_3$  on the  $\text{Hg}$  compd gives nitrodurene, m. 112-13° (40 and 80% yields, resp.), nitrosodurene, m. 39°. XV The Jacobsen reaction 4 Lee I. Smith and Clarence L. Moxley. *Ibid* 58, 1-10 (1936).—Chlorodurene, chlorosodurene and chloroprehnitene rearrange in contact with  $\text{H}_2\text{SO}_4$  to give chloropentamethylbenzene (I) and 3-chloropseudocumene-5-sulfonic acid (II), a Me group migrating in such a manner that the same chlorotrimethylbenzene deriv. resulted. Chlorosodurene also gives a small quantity of a compd.,  $\text{C}_{11}\text{H}_{12}\text{Cl}$ , m. 209.5°, the structure of which is not known. 5-Chloropseudocumene and the 6-isomer give II, chloro-

mentylene and 4-chlorohemamelitene were stable toward  $\text{H}_2\text{SO}_4$ . Bromomesitylene gives mesitylenesulfonic acid and di- or tribromomesitylene, depending upon the temp., 5-bromopseudocumene gives largely 3-bromopseudocumene-5-sulfonic acid, together with a small quantity of tribromopseudocumene; no pseudocumene-5-sulfonic acid was found. The following did not rearrange with  $\text{H}_2\text{SO}_4$ : hemamelitene, 5-nitropseudocumene, 5-pseudocumidine, Me pentamethylbenzenesulfonic acid, pentamethylcyclohexane, 2,3- $\text{C}_{11}\text{H}_7\text{Me}_2$  and  $p\text{-BrC}_6\text{H}_4\text{Ph}$ . Yields are given, together with the conditions used. I was identified by analysis and conversion into  $\text{C}_{11}\text{H}_{12}\text{Me}$ ; II was identified by conversion into the amide, m. 183°. 3-Chloro-5,6-dinitropseudocumene is reduced by  $\text{SnCl}_4$  in  $\text{EtOH}$  to the di-NH<sub>2</sub> deriv., m. 130.5°, stable in the air for some time, phenanthrenequinone (III) in  $\text{AcOH}$ - $\text{EtOH}$  gives 12-chloro-10,11,13-trimethylphenanthrophenazine, yellow, m. 330.5-1°. Reduction of the di- $\text{NO}$  compd with  $\text{SnCl}_4$  in  $\text{AcOH}$ - $\text{HCl}$  yields 6-chloro-2,4,5,7-tetramethylbenzimidazole, m. 250-1°. 5-Chloro-1,3-diaminomesitylene, m. 137-8°, this does not react with  $\text{HCl}$  or form a benzimidazole. Nitration of Na 4-chlorohemamelitene sulfonate gives 4-chloro-5,6-dinitrohemamelitene, m. 152-2.5°.  $\text{SnCl}_4$  in  $\text{AcOH}$  yields 7-chloro-2,4,5,6-tetramethylbenzimidazole, m. 280.5-7.5°. 10,11,12-Trimethylphenanthrophenazine, yellow, m. 253°. Many other details are given of the prepn of products used for the identification of the compds formed in the rearrangement, as well as starting materials. Na chloromethylsulfonate crystallizes with 0.5 mol  $\text{H}_2\text{O}$ , as does the Br deriv. bromomethylsulfonamide, m. 160-60.5°. 3-Chloro-5,6-dibromopseudocumene, m. 224°, 6-chloro-3,5-dinitropseudocumene, m. 163°. 6-Bromo-5-pseudocumidine, m. 60° (56.8% yield). 4-Chloro-5,6-dibromomesitylene, m. 229-30°, 13-chloro-10,11,12-trimethylphenanthrophenazine, yellow, m. 346.5-7°. 7-Chloro-2,4,5,6-tetramethylbenzimidazole, m. 288.5°. 10,11,12-Trimethylphenanthrophenazine, orange, m. 311°. Pentamethylbenzenesulfonamide, m. 91-1.5°. The ease of migration of groups present in the chloro- and bromotrimethylbenzenes is in the order  $\text{Br} > \text{Me} > \text{Cl}$ , in case of the corresponding derivs of the  $\text{C}_{11}\text{H}_{12}\text{Me}$ , the order is  $\text{Br} > \text{Cl} > \text{Me}$ . Attempts to find mild conditions which would cause Jacobson rearrangements without producing amorphous by-products were unsuccessful. Dln of the  $\text{H}_2\text{SO}_4$  with 10% of  $\text{H}_2\text{O}$  or with  $\text{H}_2\text{PO}_4$  or  $\text{AcOH}$  inhibited the rearrangement and the side reaction as well. The use of  $\text{CaCl}_2$ ,  $\text{Mg}(\text{ClO}_4)_2$ ,  $\text{PhSO}_3\text{H}$ ,  $\text{AcOH}$  or  $\text{H}_2\text{PO}_4$  merely caused by hydrolysis of the sulfonic acid to the hydrocarbon and no rearrangements were found, other than those already known, which would cause any rearrangements to take place. Little can be said with regard to the mechanism of the reaction. C. J. West.

Rearrangements of polyynes VII Formation of allenenes J. H. Ford, C. D. Thompson and C. S. Marvel. *J Am Chem Soc* 57, 2619-23 (1935); cf. C. A. 28, 1117.—*tert*-BuCOPh (2,4-dinitrophenylhydrazine, yellow, m. 194-5°) in  $\text{Et}_2\text{O}$ , added to the Grignard reagent from *tert*-BuCOPh in  $\text{Li}_2\text{O}$ , gives 68% of phenyl-*tert*-butyl-*tert*-butylethynylcarbinol (I), b. 125-8°, n<sub>D</sub><sup>20</sup> 1.5025, d<sub>4</sub><sup>20</sup> 0.9334 heating with  $\text{AcOH}$  compd a little  $\text{H}_2\text{SO}_4$  gives 2,2,6,6-tetramethyl-3-phenyl-3-hepten-5-one, b. 93-7°, n<sub>D</sub><sup>20</sup> 1.5003, d<sub>4</sub><sup>20</sup> 0.9300, m. 30.5° (2,4-dinitrophenylhydrazine, bright orange red, m. 150-1°), I and PhBr in petrol ether give 91% of phenyl-*tert*-butyl-*tert*-butylethynylbromomethane (II), b. 115-17°, n<sub>D</sub><sup>20</sup> 1.5400, d<sub>4</sub><sup>20</sup> 1.081, boiling in  $\text{Me}_2\text{CO}$  contg  $\text{Ag}_2\text{O}$  gives I. The Grignard reagent from II, decompt by  $\text{H}_2\text{O}$ , gives 82% of 2,2,6,6-tetramethyl-3-phenyl-3,4-heptadiene (III), b. 78-80°, n<sub>D</sub><sup>20</sup> 1.5031, d<sub>4</sub><sup>20</sup> 0.890, heating at 145° in a sealed tube for 90 hrs gives 85% of unchanged III, and 2 products, m. 145-2°, to be described later, ozonolysis of III gave *tert*-BuCOPh and  $\text{Me}_2\text{CCO}_2\text{H}$ . *tert*-BuCOPh (OH)Ph was converted into the bromide (93% yield), the Grignard reagent of which yielded 13% of phenyl-*tert*-butylethynylcarbinol, m. 105°. It gave no *tert*-BuCOPh. With  $\text{CICO}_2\text{Me}$  the Grignard reagent from II gives 64% of 2,2,6,6-tetramethyl-3-phenyl-3-carbomethoxy-3,4-heptadiene (IV), b. 116.20°, n<sub>D</sub><sup>20</sup>

1,5029, d<sub>4</sub><sup>20</sup> 0.9584; with O<sub>2</sub> this yields Me trimethylpyruvate (2,4-dinitrophenylhydrazone, m. 205-6°, also prep'd. synthetically) and probably *tert*-BuCOPH, the action of CO<sub>2</sub> upon the Grignard reagent gives 72% of the free acid corresponding to IV, m. 160-1°, also prep'd. by hydrolysis of IV. II reacts with 40% Na-Hg but does not give a stable Na alkyl. The products are III and 2 isomeric hydrocarbons, C<sub>11</sub>H<sub>16</sub>, m. 148-9° and 115-25°, which are thought to be diallenic hydrocarbons. Ag and Cu had no effect on solns of II, Li gives the same products as Na-Hg, reduction with Zn in EtOH gives the same mixt of hydrocarbons. Trimethylpyruvic acid 2,4-dinitrophenylhydrazone, m. 169-71° (decompn) VIII. Formation of diallenes E. D. Farley and C. S. Marvel. *Ibid* 58, 29-31 (1936).—The hydrocarbon prep'd from Me<sub>2</sub>CC CCl<sub>2</sub>Br, previously assumed to be a bundenyl (C. A. 26, 2452), is 1,1,6,6-tetraphenyl 3,4-di-*tert*-butyl-1,2,4,5-hexatetraene (I), m. 152°. Oxidation of I with O<sub>2</sub> in boiling C<sub>6</sub>H<sub>6</sub>Me<sub>2</sub> gives 52% Ph<sub>2</sub>CO, O<sub>2</sub> gives BuOH in addn. to products previously reported. Reduction of I with Na and AmOH gives the hydrocarbon C<sub>11</sub>H<sub>16</sub>, m. 182° (previously reported as C<sub>11</sub>H<sub>14</sub>) and a small quantity of an isomer (?), m. 132°. Reduction of I with HI in AcOH gives a comp'd, m. 210-11°, probably a dimer (II) of 3-phenylindene. I is rearranged to the isomer C<sub>11</sub>H<sub>16</sub>, m. 176°, which is stable toward KMnO<sub>4</sub> in AcOH, does not form a metal deriv with 40% Na-Hg and could not be reduced with Na and AmOH, reduction with HI in AcOH gives II. The Na deriv of I gives with CO<sub>2</sub> an acid, m. 171°, identical with acid prep'd by the action of CO<sub>2</sub> on Na diphenyl-*tert*-butylethynylmethyl, ClC<sub>6</sub>H<sub>4</sub>Me gives the comp'd C<sub>11</sub>H<sub>16</sub>(CO<sub>2</sub>Me), m. 173°, hydrolysis gives an acid, m. about 160° and decompn 180° to the comp'd C<sub>11</sub>H<sub>16</sub>CO<sub>2</sub>Me, m. 162°, H<sub>2</sub>O gives the comp'd C<sub>11</sub>H<sub>16</sub> (III), m. 163°. The Na deriv and (CH<sub>3</sub>)<sub>2</sub>Br<sub>2</sub> give the isomeric C<sub>11</sub>H<sub>16</sub> (IV), m. 179-80°. Oxidation of III with KMnO<sub>4</sub> in Me<sub>2</sub>CO gives the comp'd C<sub>11</sub>H<sub>16</sub>O<sub>4</sub>, m. 217-18°, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in AcOH gives the comp'd C<sub>11</sub>H<sub>16</sub>O<sub>4</sub>, m. 169°, reduction with Na and AmOH gives the comp'd C<sub>11</sub>H<sub>16</sub> (V), m. 197°, and a lower-melting isomer, treatment with 40% Na-Hg and then with H<sub>2</sub>O gives V. Oxidation of IV with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> gives the comp'd C<sub>11</sub>H<sub>16</sub>O<sub>4</sub>, m. 169°, neutral KMnO<sub>4</sub> was without action, reduction with Na and AmOH gives the C<sub>11</sub>H<sub>16</sub>, m. 197°, and an isomer, m. 155-6°. IV, treated with 40% Na-Hg and then with H<sub>2</sub>O, gives the hydrocarbon C<sub>11</sub>H<sub>16</sub>, m. 163°. 1,1-Diphenyl-1,4-dimethyl-1,2-pentadiene with 40% Na-Hg in Et<sub>2</sub>O, treated with (CH<sub>3</sub>)<sub>2</sub>Br<sub>2</sub>, gives the comp'd C<sub>11</sub>H<sub>16</sub>, m. 143-4°; the Na comp'd. with H<sub>2</sub>O does not give a cryst product. The dimer, treated with Na-Hg and then with (CH<sub>3</sub>)<sub>2</sub>Br<sub>2</sub>, gives only the unchanged material, H<sub>2</sub>O gives the comp'd C<sub>11</sub>H<sub>16</sub> (VI), m. 133°. Reduction of the dimer with Na and AmOH probably gives VI, as does HI in AcOH, oxidation of the dimer gives the comp'd C<sub>11</sub>H<sub>16</sub>O<sub>4</sub>, m. 227°. C. J. West.

Decomposition of di-*o*-tolylodoniun iodide H. J. Lucas, F. R. Kennedy and C. A. Wilmut. *J. Am. Chem. Soc.* 58, 157-60 (1936).—(*o*-MeC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>I<sub>2</sub> decomposes at 155° into *o*-MeC<sub>6</sub>H<sub>4</sub>I and not into a mixt of isomers. The nature of the reaction product indicates that the (*o*-MeC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>I<sub>2</sub> ion splits into *o*-MeC<sub>6</sub>H<sub>4</sub>I and positively charged *o*-C<sub>6</sub>H<sub>4</sub>Me ion by a scission of the C—I bond. This ion and the neg I ion produce *o*-MeC<sub>6</sub>H<sub>4</sub>I. In the formation of *o*-(*o*-MeC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>IO from *o*-MeC<sub>6</sub>H<sub>4</sub>IO and *o*-MeC<sub>6</sub>H<sub>4</sub>IO, Ag<sub>2</sub>O acts catalytically. The following phys. consts. of MeC<sub>6</sub>H<sub>4</sub>I are given *o*, b<sub>p</sub> 95.5°, b<sub>m</sub> 205.5°, d<sub>4</sub><sup>20</sup> 1.0700, n<sub>D</sub><sup>20</sup> 1.6030, *m*, b<sub>p</sub> 210°, *d* 1.6381, *n* 1.6012, *n*<sub>D</sub><sup>20</sup> 34.5-5° (cor). The *o*-MeC<sub>6</sub>H<sub>4</sub>I was identified by conversion into *o*-MeC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H through the Grignard reagent. C. J. West.

2,4,6-Trichloro-3-amino-1-methylbenzene and some of its derivatives I. Bureš and M. Trpšovská. *Časopis Českoslov. Léčnického 15*, 174-81 (1935).—By the action of Cl on *m*-acetotoluene in glacial AcOH there was obtained 2,4,6-trichloro-3-acetamino-1-methylbenzene, m. 192°. The sapon. of this product gave 2,4,6-trichloro-*m* toluidine, m. 85°. By the nitration of 2,4,6-trichloro-

there was obtained 2,4,6-trichloro-3-nitrotoluene, m. 50° and by the nitration of 2,3,4,6-Cl<sub>4</sub>C<sub>6</sub>HMe the resulting product was 2,3,4,6-tetrachloro-5-nitrotoluene, m. 149-50°. V. D. Karpenko.

The coloration of aniline Tatsuo Yamanaka. *Bull. Inst. Phys.-Chem. Research* (Tokyo) 14, 396-405 (1935).—(Abstracts in English) published with *Sci. Papers Inst. Phys.-Chem. Research* (Tokyo) Nos. 573-5).—The causes for the coloration of PhNH<sub>2</sub> have been worked out; it is attributed to the effects of impurities including water, air (O<sub>2</sub>), materials of the vessels, light of wave lengths 5100-3000 Å, especially 4537-4300 Å. Cu has the strongest coloring effect, when kept with PhNH<sub>2</sub> at 60° for 10 hrs. In the semi-industrial scale expts., Pb or Sn cooling tubes and enameled Fe reaction app. gave the same result as glass app. in the lab expts. K. K.

Production of aromatic amines by hydrogenation II. Kiyoshi Yoshikawa, Tatsuo Yamanaka and Bannosuke Kubota. *Bull. Inst. Phys.-Chem. Research* (Tokyo) 14, 409-11 (1935) (Abstracts in English) published with *Sci. Papers Inst. Phys.-Chem. Research* (Tokyo) Nos. 573-5), of C. A. 29, 7957.—The manuf. of PhNH<sub>2</sub> by the hydrogenation with poisoned Ni catalysts has been tried in the semi-industrial scale. The enameled Fe reaction tubes were used. The single tube app., 51 cm in diam. and 158 cm long, filled with 380 g Ni-Cu-Al catalyst on a Cu support, and fed with PhNO<sub>2</sub> at a velocity of 180 cc. per hr at 180°, gives 52 kg PhNH<sub>2</sub> in 370 hrs with a 97.1% yield of the theoretical. The multi-tube app., consisting of 19 reaction tubes, 32 cm in diam and 81 cm long, immersed in a PhNO<sub>2</sub> bath to control the temp., and supplied with the mixt of H<sub>2</sub> and PhNO<sub>2</sub> vapor at a velocity of 800 cc per hr, yields 200 kg PhNH<sub>2</sub> in 250 hrs. III. Tatsuo Yamanaka, Sakae Yamada, Bannosuke Kubota and Kiyoshi Yoshikawa. *Ibid* 412-23.—The preceding hydrogenation method with a thiophene-poisoned catalyst has been applied to the manuf. of aromatic nitro and azo compds and quinones. For less volatile substances, the proper solvents were used for their vaporization. In general, these catalysts are very well adapted to the hydrogenation of such compds, and the reactions proceed at lower temps., in almost all cases without decompn. The simple NO<sub>2</sub> derivs. of aromatic hydrocarbon are very easily hydrogenated to the corresponding amines, while with derivs. with substituted radicals other than NO<sub>2</sub>, the results differ with the nature of the radical. Thus *o*- and *p*-toluidine, *m*- and *p*-phenylenediamine, tolylenediamine, *α*-naphthylamine are obtained pure in theoretical yields. The manuf. on a semi tech. scale has also been carried out for toluidine, xylidine and naphthylamine with good results. K. K.

Halogenation of phenolic ethers and anilides. V. Alkyl and *ω*-substituted alkyl ethers Brynmor Jones. *J. Chem. Soc.* 1935, 1831-5, of C. A. 28, 2689.—Earlier studies of the interpretation of the Arrhenius expression,  $k = ae^{-E/RT}$ , as applied to the C<sub>6</sub>H<sub>5</sub>-substitution problem (cf C. A. 26, 1203), showed that the term  $a$  is const. within the error of expt and that in compds of the type *p*-ROC<sub>6</sub>H<sub>4</sub>X the groups OR and X each contributed a characteristic quota to the activation energy of further substitution. The series of ethers studied has been further enlarged, the additive relationships receiving further illustration. Velocity coeffs for the chlorination of compds of the type *o*- and *p*-XC<sub>6</sub>H<sub>4</sub>OR in 9% AcOH at 20°, [Cl<sub>2</sub>] 0.0075, [Et<sub>2</sub>O] 0.0225, [HCl] 0.0375, were det'd. as follows: X = *p*-CO<sub>2</sub>H, R = Me 0.444, C<sub>6</sub>H<sub>5</sub> 0.966, C<sub>6</sub>H<sub>4</sub> 0.960, C<sub>6</sub>H<sub>3</sub> 0.947, C<sub>6</sub>H<sub>2</sub> 0.919, C<sub>6</sub>H<sub>11</sub> 1.114, Ph(CH<sub>3</sub>)<sub>2</sub> 0.528, Ph(CH<sub>3</sub>)<sub>3</sub> 0.760; X = *p*-Cl, R = Me 1.236, C<sub>6</sub>H<sub>5</sub> 2.735, C<sub>6</sub>H<sub>4</sub> 2.770, C<sub>6</sub>H<sub>3</sub> 2.743; X = *p*-Br, R = Me 1.256, *iso*-Bu 2.738, C<sub>6</sub>H<sub>5</sub> 2.665, Br(CH<sub>2</sub>)<sub>2</sub> 0.266, Br(CH<sub>2</sub>)<sub>3</sub> 0.819, X = *o*-CO<sub>2</sub>H, R = Me 3.463; X = *o*-Cl, R = Me 4.41, *p* 10.05, *iso*-Pr 16.16, Bu 10.07, Am 9.33. Tables are given showing the relative directive powers of the groups OR in compds. of the above type with R = Me as 100 and also of the groups CO<sub>2</sub>H, Cl and Br, taking *p*-CO<sub>2</sub>H as 100. As an example the relative directive powers of *n* alkyl groups are: Me 100, 1 *t* 199, *p* 223, Bu 223, Am 221, C<sub>6</sub>H<sub>5</sub> 221, C<sub>6</sub>H<sub>11</sub>

219,  $C_{11}H_{17}$  207,  $C_{11}H_{19}$  201; iso-Pr has a value of 440 (which cannot be explained at present), while iso-Bu is 216. The introduction of Br into Et produces a marked decrease in reactivity, the intervention of an addl  $CH_3$  group diminishes the effect but slightly, the values relative to the Me ether = 100 being 21 and 65 for  $(CH_3)_2Br$  and  $(CH_3)_3Br$ . The Ph group exerts a similar but much weaker effect than Br. MeO 100,  $PhCH_2O$  70, EtO 199,  $Ph(CH_2)_2O$  119, PrO 223,  $Ph(CH_2)_3O$  171. *p*-Chlorophenyl Am ether,  $b_{11}$  132-3°, *hexyl ether*,  $b_{11}$  172°, *heptyl ether*,  $b_{11}$  162°, *cetyl ether*,  $b_{11}$  49°, *p*-bromophenyl cetyl ether,  $b_{11}$  49°, *o*-Chlorophenyl Pr ether,  $b_{11}$  119°, iso-Pr ether,  $b_{11}$  103°, Bu ether,  $b_{11}$  117°, Am ether,  $b_{11}$  117°. *p*-8-Phenylthioxybenzoic acid,  $m$  160°, *p*-7-phenylpropoxybenzoic acid,  $m$  166°, *p*-Bromophenyl  $\gamma$ -bromopropyl ether,  $b_{11}$  169°,  $m$  49.5°, the by-product is trimethylene glycol bis-*p*-bromophenyl ether,  $m$  143°. VI Benzyl and substituted benzyl ethers. *Ibid* 1835-40.—The following are velocity coeffs for the chlorination of compds of the type  $o$ - and  $p$ - $XC_6H_4CO_2H$  in 90% AcOH at 20°, using conditions given in part V.  $X = p$ - $CO_2H$ ,  $R = Me$  (I) 0.444,  $PhCH_3$  (II) 0.314,  $p$ - $FC_6H_4CH_3$  (III) 0.262,  $p$ - $ClC_6H_4CH_3$  (IV) 0.200,  $p$ - $BrC_6H_4CH_3$  (V) 0.203,  $p$ - $MeC_6H_4CH_3$  (VI) 0.441,  $m$ - $FC_6H_4CH_3$  (VII) 0.157,  $m$ - $ClC_6H_4CH_3$  (VIII) 0.153,  $m$ - $BrC_6H_4CH_3$  (IX) 0.159,  $m$ - $MeC_6H_4CH_3$  (X) 0.475,  $o$ - $FC_6H_4CH_3$  (XI) 0.160,  $o$ - $ClC_6H_4CH_3$  (XII) 0.152,  $o$ - $MeC_6H_4CH_3$  0.465,  $X = p$ - $Cl$ ,  $R = I$  1.226,  $II$  0.837,  $III$  0.674,  $IV$  0.489,  $V$  0.483,  $VI$  1.153,  $p$ - $OC_6H_4CH_3$  (XIII) 0.174,  $VII$  0.394,  $VIII$  0.372,  $IX$  0.377,  $X$  0.203,  $NC_6H_4CH_3$  (XIV) 0.195,  $XII$  0.346,  $o$ - $OC_6H_4CH_3$  (XV) 0.175,  $X = p$ - $Br$ ,  $R = I$  1.256,  $II$  0.850,  $IV$  0.521,  $VI$  1.170,  $XIII$  0.177,  $XIV$  0.201,  $X = o$ - $CO_2H$ ,  $R = I$  3.483,  $II$  2.700,  $III$  2.192,  $IV$  1.827,  $V$  1.855,  $VI$  3.710,  $X = o$ - $Cl$ ,  $R = I$  4.441,  $III$  2.481,  $IV$  1.861,  $V$  1.850,  $VI$  4.201,  $XIII$  0.642,  $XV$  0.628,  $X = o$ - $Br$ ,  $R = I$  5.457,  $IV$  2.155,  $XII$  0.750,  $XIV$  0.884,  $XV$  0.753. The relative effects of the groups OR and X are independent, both contributing additively to the energy of activation of chlorination. Tables give the results of calcn of the relative directive powers of the various groups studied. In the acid series the variations in the ratios for the different groups are 2-3 times as great as in the Cl and Br series. The velocity coeffs for the chlorination of the series of *p*-substituted benzyl ethers fall into the expected order, the reactivities of the halobenzyl ethers being intermediate between those of the nitro- and the methyl benzyl ethers. The general polar series  $Me > H > halogens > NO_2$  is thus again observed. This order also obtains in the *o*- and *m*-series but the order of the halogens among themselves is not that of their inductive effects, the *p/m* ratios are  $F$  1.68,  $Cl$  1.27,  $Br$  1.24. The main results are summarized as follows. For a wide range of compds of the type  $ROC_6H_4X$ , the 2 groups OR and X contribute characteristically and additively to the energy of activation of chlorination, with ethers of  $o$ - $HOC_6H_4CO_2H$  and iso- $PrOC_6H_4Cl$ , minor anomalies are observed. The const reactivity characteristic of long-chain compds is observed from Pr to heptyl, the octyl and cetyl ethers exhibiting slightly lower reactivities, the introduction of a Ph or Br into the Et and Pr groups produces a marked decrease in reactivity. For polar groups in the  $PhCH_2$  radical, the order of reactivity for *p*-substituents is  $Me > H > I > Cl > Br > NO_2$ , in the *m*-position the halogens show almost identical effects. To account for the relative reactivities of the halobenzyl ethers, a mesomeric effect in the order  $F > Cl > Br$  would appear to be necessary. *p*-Chlorophenyl ethers: *p*-fluorobenzyl,  $m$  60°, *p*-chlorobenzyl,  $m$  81°, *p*-bromobenzyl,  $m$  93°, *m*-isomer,  $m$  43°, *p*-methylbenzyl,  $m$  97°, *m*-fluorobenzyl,  $b_{11}$  223-5°, *m*-chlorobenzyl,  $b_{11}$  222-4°, *o*-isomer,  $b_{11}$  191°, *o*-nitrobenzyl,  $m$  73°, *o*-Chlorophenyl ethers: *p*-fluorobenzyl,  $b_{11}$  170°,  $m$  35°, *p*-chlorobenzyl,  $m$  69°, *p*-methylbenzyl,  $m$  76°, *o*-nitrobenzyl, pale yellow,  $m$  84.5° *p*-Bromophenyl ethers: *p*-chlorobenzyl,  $m$  98°, *p*-methylbenzyl,  $m$  105°, *o*-Bromophenyl ethers: *p*-chlorobenzyl,  $m$  69°, *o*-nitrobenzyl,  $m$  107°, *m*-isomer,  $m$  100°, *p*-(*p*'-Fluorobenzoyloxy)benzoic acid,  $m$  213°, *p*-Cl deriv,  $m$  218°, *p*-Br deriv,  $m$  231° (max soly

in AcOH at 20° 0.3-0.5 g/100 cc). *p*-(*p*'-Methylbenzoyloxy)benzoic acid,  $m$  212°; *m*-isomer,  $m$  157° *p*-(*m*'-Fluorobenzoyloxy)benzoic acid,  $m$  194°, *m*-Cl deriv,  $m$  194°, *m*-Br deriv,  $m$  202° *p*-(*o*'-Fluorobenzoyloxy)benzoic acid,  $m$  181°, *o*-Cl deriv,  $m$  183° *o*-Me deriv,  $m$  169° *o*-Benzoyloxybenzoic acid,  $m$  77° *o*-F deriv,  $m$  87°; *p*-Cl deriv,  $m$  115°, *p*-Me deriv,  $m$  111°. C J West

Geometric isomerism of asymmetric quaternary ammonium salts and betaine hydrates derived therefrom and a study of the reactions yielding them. Mime M Guaisnet-Pilaud *Ann chim* [11], 4, 365-448(1935)—*MePhPrNCH\_2CO\_2Et* (I),  $b_{11}$  145-6°, was prepd from *MePhNH* and  $ClCH_2CO_2Et$  or  $IClCH_2CO_2Et$ , *EtPhNCH\_2CO\_2Et* (II),  $b_{11}$  149-50°, and *EtPhNCH\_2CO\_2Me* (III),  $b_{11}$  147-8°, from *EtPhNH* and the suitable halo acetate and *PhPrNCH\_2CO\_2Et* (IV),  $b_{11}$  161-2° from *PhPrNH*. *EtMePhN* on standing with  $IClCH_2CO_2Et$  gave the quaternary *NH* salt *EtMePhN(Pr)CH\_2CO\_2Et* (V), which was treated with moist  $AqO$  to produce the betaine *EtMePhNCH\_2COO* (VI), in the form of 2 hydrates Th

"metastable monohydrate" (VIA) is  $C_{11}H_{19}O_3N \cdot H_2O$  tiny needles,  $m$  175° (decompn), very sol in  $H_2O$ , HOAc, sol in EtOH, difficultly sol in EtOAc,  $n_D^{20}$  1.583  $\pm$  0.003,  $n_D$  1.549  $\pm$  0.003 (method of Becke). Heating VIA to 100-10° at 16 mm for 24 hrs, then increasing the temp to 180-90°, resulted in loss of water and decompn into *EtMePhN* and I, identified by conversion to *MePhNCH\_2CONH\_2*,  $m$  164°. Treatment of aq VIA with  $Ba(OH)_2$  and pptn of the excess Ba ions with  $CO_2$  filtration and distn to dryness *in vacuo* yielded *EtMePhN* and a residue of  $(HOCH_2CO_2)_2Ba$ . VIA formed the following salts: chloroplatinate, orange,  $m$  134°, picrate, yellow,  $m$  158.5° (decompn); *d*-camphorsulfonate,  $m$  214-15°,  $[a]_D^{20}$  8.45°, acid oxalate,  $C_{11}H_{19}O_3N \cdot H_2C_2O_4$ ,  $m$  124.5°. Acidification of the picrate with HCl and subsequent treatment with  $Ag_2O$  gave the 2nd hydrate of VI, a "stable dihydrate",  $C_{11}H_{19}O_3N \cdot 2H_2O$  (VIB). VIA and VIB were obtained from V in proportions of 1 or 1.5 to 10. VIB exists as monoclinic crystals (goniometric measurements given),  $m$  79.5°, resolifies, in aq above 100°, decomposes 145-50°. Heating of VIB first at 70-80° at 17 mm, then increasing the temp to 200-205°, produced *EtMePhN* and III, identified by conversion to *EtPhNCH\_2CONH\_2*,  $m$  108°, and, at the highest temp, a fluorescent liquid,  $b_{11}$  195-205°, apparently *MePhNCH\_2CO\_2CH\_2CO\_2Et*. After treating aq VIB with  $Ba(OH)_2$ , then  $CO_2$ , 96% of the VIB was recovered. The salts of VIB with  $H_2PtCl_6$ , picric acid, *d*-camphorsulfonic acid were identical with those obtained from VIA. VIB yielded a neutral oxalate,  $m$  166°, and an acid oxalate,  $m$  69.5°. Treatment of II with MeI gave V, which reacted with moist  $Ag_2O$  to give VI in the form of a "stable hydrate" (VIE), along with VIB and *EtMePhN*. VI was obtained in a purer state similarly from III obtained by the isomerization of VIB. VIC is apparently a monohydrate,  $C_{11}H_{19}O_3N \cdot H_2O$ ,  $m$  104°, orthorhombic, strongly birefringent,  $n_D$  1.549  $\pm$  0.003,  $n_D$  1.585  $\pm$  0.003,  $n_D$  1.658  $\pm$  0.003, on heating it is converted to I (predominantly) and III, identified by conversion to the amides,  $m$  164° and 108°, resp. Treatment with  $Ba(OH)_2$  and  $CO_2$  results in the recovery of VIC unchanged. VIC forms the following salts: chloroplatinate, orange,  $m$  169°, picrate, yellow,  $m$  186° (decompn), from which VIC may be recovered, *d*-camphorsulfonate,  $m$  190-91°,  $[a]_D^{20}$  8.48°, neutral oxalate,  $m$  173° (decompn), acid oxalate (probably),  $m$  134.5° 230 g of *MePhPrN* on standing 10 days with 475 g of  $IClCH_2CO_2Et$  yielded *MePhPrNCH\_2CO\_2Et* (VII), which reacted with moist  $Ag_2O$  to produce, after repeated crystns from EtOH, 30 g of *MePhPrNCH\_2COO* (VIII) in the form of a "metastable monohydrate" (VIIIa), and 66 g in the form of a "stable dihydrate" (VIIIb). VIIIa,  $m$  171° (decompn),  $n_D$  1.595  $\pm$  0.003,  $n_D$  1.555  $\pm$  0.003, sol in  $H_2O$ , slightly sol in abs EtOH, very slightly sol in MeOAc, was weakly acidic, was converted by heating 12 hrs at 100°.

then increasing the temp. to 170–90°, to MePhPrN and 1  
 $\text{MePhNCH}_2\text{CO}_2\text{Pr}$ ,  $b_p$  150–2°, identified by conversion  
 to the amide, m. 164°. Treatment of VIIa with  $\text{Ba(OH)}_2$ ,  
 then  $\text{CO}_2$ , converted it to MePhPrN and  $(\text{HOCH}_2\text{CO}_2)_2\text{Ba}$ .  
 VIIa yielded the following salts. chloroplatinate, yellow,  
 powder, m. 198° (decompn.), picrate, yellow, m. 127°  
 (decompn.), neutral oxalate, m. 203°, very slightly sol  
 in 95% EtOH, *d*-camphorsulfonate, m. 227–27.5° (de-  
 compn.),  $[\alpha]_D^{20}$  8.63°. The dihydrate, VIIb, was ob-  
 tained also by treatment of the picrate of VIIa with  
 HCl, then with moist  $\text{Ag}_2\text{O}$ , m. 81.5°, resolidifies and  
 again m. approx 160° (decompn.), is much more sol in  
 abs EtOH than is VIIa,  $n_D^{20}$  1.595,  $n_D^{25}$  1.555 = 0.003.  
 The action of heat converts VIIb to MePhPrN, Me-  
 PhNCH<sub>2</sub>CO<sub>2</sub>Pr (predominantly), and PhNCH<sub>2</sub>CO<sub>2</sub>Me,  
 identified by conversion to their amides, m. 164° and 80°,  
 resp. Treatment with  $\text{Ba(OH)}_2$ , as above results in  
 almost complete recovery of unchanged VIIb, the salts  
 formed are identical with those derived from VIIa. A  
 3rd "stable hydrate" (VIIc), m. 109° (accompanying by  
 some  $\text{Me}_2\text{PhN CH}_2\text{CO}_2\text{O}$ , m. 124°, chloroplatinate, m.

196°), was obtained by treatment of PhNCH<sub>2</sub>CO<sub>2</sub>Et  
 with MeI,  $n_D^{20}$  1.595 = 0.003,  $n_D^{25}$  1.555 = 0.003, picrate,  
 m. 189° (decompn.). The reaction of *p*-MeC<sub>6</sub>H<sub>4</sub>NEt<sub>2</sub>  
 with  $\text{ICl}_3\text{CO}_2\text{Et}$ , and subsequent treatment with  $\text{Ag}_2\text{O}$   
 gave *p*-MeC<sub>6</sub>H<sub>4</sub>NEt<sub>2</sub> CH<sub>2</sub>CO<sub>2</sub>O (IX), very sol in H<sub>2</sub>O  
 and EtOH, very hygroscopic, oxalate, m. 138–9° IX,  
 like Et<sub>2</sub>PhN CH<sub>2</sub>CO<sub>2</sub>O and Me<sub>2</sub>PhNCH<sub>2</sub>CO<sub>2</sub>O, exists

in only 1 form. Attempts to resolve these betaines,  
 carried out with VIIb, VIIc and VIIb by recrystn. of the  
*d*-camphorsulfonates, and on VIIb, VIIc and VIIa with  
 a culture of *Penicillium glaucum* were unsuccessful. The  
 aryl betaines studied produce stable gels with org. acids  
 as pptd. by silicotungstic acid,  $\text{Si}_6\text{W}_{12}\text{O}_{48}\text{H}_{24}$ , in the  
 form of definite cryst. silicotungstates, while the asym.  
 betaines form only tiny spheroidal particles of the silico-  
 tungstates. In prep. the quaternary NH<sub>4</sub> salts, gel  
 formation took place as the mixt. of tertiary amine and  
 halogen compd. stood; the progress of the reaction could  
 be followed by the changing thickness of the gel layer  
 and by the changing total vol. of the reaction mixt. The  
 rate of reaction varied with conditions of illumination,  
 EtMePhN, prep. from MePhNH and EtI, differed in  
 reaction rate from that prep. from EtBr, although both  
 samples of EtMePhN, carefully purified, had the same  
 constn. and produced identical Raman spectra. G-P  
 concludes from the existence of the betaines in several  
 forms that the asym. quaternary NH<sub>4</sub> salts exist in 3  
 different inactive stereoisomeric (geometric) forms, and  
 explains their existence on the basis of the bipyramidal  
 bipolar and monopyratidal monopolar formulas of  
 H. D. Jones (*Proc. Cambridge Phil. Soc.* 11, 111, *J.*  
*Chem. Soc.* 83, 1400(1903), 87, 135(1905)), Willgerodt  
 (*J. prakt. chem.* 41, 291(1890)) and Brechhoff (*Ber.* 23,  
 1972(1890)), indicating that changing the order of attach-  
 ing the different groups to the N atom produces the  
 different isomers. Other compds. prep. during the  
 study of the reaction giving rise to the quaternary NH<sub>4</sub>  
 salts are: EtMe<sub>2</sub>PhN<sub>2</sub>, m. 134°; *p*-MeC<sub>6</sub>H<sub>4</sub>Et<sub>2</sub>N<sub>2</sub>,  
 m. 156.5°, sol in H<sub>2</sub>O, 1 EtOH, nearly insol in EtOAc, *p*-  
 MeC<sub>6</sub>H<sub>4</sub>Et<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>Et, m. 160–2°; *p*-MeC<sub>6</sub>H<sub>4</sub>Et<sub>2</sub>NCH<sub>2</sub>-  
 CO<sub>2</sub>Et, m. 123°. G. R. Yohle

Condensation of  $\omega$ -bromocetophenone with 1- $\alpha$ -amino-  
 phenyl-3-phenylthiocarbamide. Tejendra N. Ghosh,  
*Current Sci.* 4, 312(1935). The product obtained by  
 G. (cf. C. A. 25, 4625) is not identical with that described  
 by Pathak (cf. C. A. 30, 4379). W. J. Peterson

Phenylthiocarbamides. Triad NCS. I. Aniline  
 thiocyanate. Hans Krall and Rameshwar Dayal Gupta,  
*J. Indian Chem. Soc.* 12, 629–34(1935).—Aniline thio-  
 cyanate (I) has been prep. in theoretical yields by the  
 direct union of PhNH<sub>2</sub> (II) and HSCN (III). A measured  
 quantity of III, prep. by passing H<sub>2</sub>S through H<sub>2</sub>(SCN)<sub>2</sub>  
 in Et<sub>2</sub>O and estd. by Volhard's method, was added to 1

equiv. of freshly distd. II in Et<sub>2</sub>O. On cooling a heavy  
 liquid sepd. which crystd. to colorless shining scales  
 of I, m. 80–1°. Prepn. of I by the double decompn. in  
 concd. aq. soln. of Ca(SCN)<sub>2</sub> and PhNH<sub>2</sub>, HCl gave 30%  
 yields. The conditions must be carefully controlled or  
 various double compds. sep. The isomerization of I to  
 PhNHSCNH<sub>2</sub> (IV) takes place over a wide range of  
 temp. with considerable evolution of heat. The trans-  
 formation never exceeds 80% and suggests an equil.  
 Up to 110°, IV does not revert to an appreciable rate.  
 This apparent equil., the sepn. of I in liquid form during  
 its prepn. in Et<sub>2</sub>O, and the presence of a H<sub>2</sub>O-sol sub-  
 stance with the properties of a thiocarbamide in the prepn.  
 of I from Ca(SCN)<sub>2</sub> and PhNH<sub>2</sub>, HCl indicate the existence  
 of a 3rd unknown isomer of I and IV which undergoes  
 desulfurization with ease and which is freely sol. in H<sub>2</sub>O.

II. Action of hydrolytic agents on phenylthiocarbamide.  
 Santaran Mehta and Hans Krall. *Ibid.* 635–9.—Phenyl-  
 thiocarbamide (I), PhNHSCNH<sub>2</sub>, is not hydrolyzed  
 normally to give NH<sub>3</sub> or PhNH<sub>2</sub> with the simultaneous  
 production of a OHI compd. In concd. alkali I is dissocd.  
 into H<sub>2</sub>S and PhNH<sub>2</sub> (II). Some NH<sub>3</sub> is produced  
 together with CO<sub>2</sub> by the hydrolysis of II to PhNH<sub>2</sub> in  
 secondary reactions. I (5 g.) was refluxed with 1.0, 0.5,  
 0.25, 0.125, 0.0625 and 0.00 equivs. of KOH in 132 cc.  
 for 1 hr. Depts. of H<sub>2</sub>S and HSCN (III) by titration  
 with alk. 7n soln. and by acidification with H<sub>2</sub>SO<sub>4</sub>, de-  
 compn. of H<sub>2</sub>S with SO<sub>2</sub>, oxidation of excess SO<sub>2</sub> with  
 HNO<sub>3</sub>, and titration with 0.01 N AgNO<sub>3</sub>, showed the  
 presence of 23.20, 8.67, 2.29, 0.86, traces and 0.00% of  
 H<sub>2</sub>S and 5.7, 6.8, 7.0, 7.9, 8.7 and 8.8% of III. Thus,  
 diminishing alk. favors a disocn. into III and PhNH<sub>2</sub>.  
 Similar results were obtained by hydrolysis with 1 equiv.  
 of Ba(OH)<sub>2</sub>, which gave 13.21% of H<sub>2</sub>S and 0.89% of  
 III in comparison to the 29.6 and 0.66% obtained with  
 1 equiv. of KOH. Hydrolysis with AcOH and HCl  
 gave no H<sub>2</sub>S, 16.08 and 0.185% of III together with  
 faint and strong smells of PhNCS. Thus the C–S bond  
 is ruptured in the presence of alkali and the C–NHPh  
 linkage by treatment with acids. The C–NH<sub>2</sub> bond is  
 only cleaved in the presence of strong mineral acids.  
 III. Action of nitrous acid on phenylthiocarbamide.  
*Ibid.* 640–6.—In the presence of acids HNO<sub>2</sub> reacts with  
 PhNHSCNH<sub>2</sub> (I) to give a base (II) (cf. Hefner, *Ber.*  
 22, 1170(1889)) and NO or PhNCS (III) and N<sub>2</sub> accord-  
 ingly as a strong or weak acid ionizer is present in the  
 soln. To obtain an idea of the progress of the 2 reactions,  
 expts. were carried out in Allen's modification of Lunge's  
 nitrometer and other depts. were made on a larger scale  
 to est. the yields of solid and liquid products. The  
 gradual addn. of 1 equiv. of pure NaNO<sub>2</sub> to a soln. of  
 I in excess dil. HCl pptd. the yellow nitroso deriv. of  
 I. Addn. of a 2nd equiv. of NaNO<sub>2</sub> acted upon the  
 unchanged I. The soln. smelled of PhNC from the  
 beginning of the reaction which finally yielded 82–5%  
 of II, 10% of III and a gas composed of 90.5% NO and  
 9.5% N. In the presence of dil. AcOH, equimol. quanti-  
 ties of HNO<sub>2</sub> and I gave 66.5% of III, 23% of I and a  
 gas contg. 80% N and 20% NO. Under any conditions  
 the 1st change is an oxidation producing II and NO,  
 followed by the normal reaction of amines forming N and  
 III. The evolution of N in the presence of AcOH shows  
 that I contains an NH<sub>2</sub> group and that its configuration  
 must be represented by PhINC(S)NH<sub>2</sub> or PhN:C-  
 (SH)NH<sub>2</sub>. In the presence of HCl the configuration  
 changes to that of a mol. which has no NH<sub>2</sub> group. Oxidation  
 eliminates 1 S and 4 H atoms from 2 mols. of I.

C. R. Addinal  
 Possible rearrangement reactions of monochloramine  
 and certain Grignard reagents. Geo. H. Coleman and  
 Robert A. Forrester. *J. Am. Chem. Soc.* 58, 27–8  
 (1936).—PhClEtMgCl and C<sub>2</sub>H<sub>5</sub>NH<sub>2</sub> yield 92% of Ph-  
 CH<sub>2</sub>NH<sub>2</sub>;  $\alpha$ -C<sub>2</sub>H<sub>5</sub>MgCl gives 47% of  $\alpha$ -C<sub>2</sub>H<sub>5</sub>NH<sub>2</sub>; if  
 $\alpha$ -MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> or  $\alpha$ , $\beta$ -MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> are formed in these  
 reactions, they are present in less than 1% of the reaction  
 product. PhCH<sub>2</sub>CHClMgCl yields 14% of PhCH-  
 CHClNH<sub>2</sub>. Thus no indication of rearrangement  
 products was found.  
 C. J. West



The preparation of *m*-phenylenediethylamine, *p*-phenylenediethylamine and benzohexamethylamine from the three phenylenediacetonitriles Paul Ruggli, B. B. Bussemaker, Wilhelm Müller and Alfred Staub. *Helv. Chim. Acta* 18, 1388-95(1935) —By employing pressures of 70-75 atm  $H_2$  at temps of 90-100°, it is possible to obtain 50-60% yields of primary amines by reduction of nitriles, since the higher temp. and pressure shortens the time of reaction, and of aldime formation. Ten g.  $m$ - $C_6H_4(CH_2CN)_2$  (cf. Tuttle, *C. A.* 20, 1794) in 100 cc. abs. alc. salted with  $NH_3$  was reduced with  $H_2$  (70 atm) at 90-100°, with 20 g. Ni on clay (Rupe, *C. A.* 13, 2196) as catalyst. Removal of alc. in *vacuo* (the amine protected from  $CO_2$ ) and distn. gave 6 g.  $m$ - $C_6H_4(CH_2CH_2NH_2)_2$  (I), b.p. 152-3°. The residue contains secondary amine as shown by analysis of the HCl salt and Pt salt. The following derivs. of I were prep'd: *di*-HCl salt, m. 300-302° (decompn.) (black); *Pt* salt, m. 242°; *di*-Bz deriv., m. 181°. Similarly, 40 g.  $p$ - $C_6H_4(CH_2CH_2NH_2)_2$  (cf. Tuttle, *C. A.* 20, 1794) (40 g. catalyst) gave 24 g.  $p$ - $C_6H_4(CH_2CH_2NH_2)_2$ , b.p. 130°, m. 36°, *di*-HCl salt, m. only in a free flame; *Pt* salt, darkens 280°; *picrate*, m. 246° (decompn.); *di*-Bz deriv., m. 225°; *di*-Ac deriv., m. 210°; 15 g.  $o$ - $C_6H_4(CH_2CH_2NH_2)_2$  (III) (Moore and Thorpe, *C. A.* 2, 1443) in 100 cc. solvent was reduced in the same manner, using 80 g. catalyst. 12.5 g. distillate was obtained, 7.6 g. b.p. 120-9°. Redistn. yielded a product, b.p. 120-7°, which was shown to be  $o$ - $C_6H_4(CH_2CH_2NH_2)_2$  (IV) (cf. von Braun and

Reich, *C. A.* 20, 1413), and not  $o$ - $C_6H_4(CH_2CH_2NH_2)_2$ , prep'd. in 20% yield by reduction of III with Na and alc. (von Braun, *et al.*, *C. A.* 11, 2676). Several mechanisms are proposed to account for the formation of IV.  
W. Gordon Rose.  
Some remarks with regard to the communication of J. Reilly and P. J. Drumm on the mechanism of diazotization J. Böeseken and H. Schoutissen *Rec. trav. chim.* 54, 956-8(1935) —B and S criticize the paper of Reilly and Drumm (*C. A.* 29, 6588\*), stating that R and D have paid no attention to the earlier work of B and S (*C. A.* 15, 1023, etc.) and that R and D fail to give details of their procedure for the detn. of the quantity of diazotized amino salt with the colorimeter so that it is impossible to discuss the results on the basis of B and S expts.  
Oden E. Sheppard.  
Condensations between maleic anhydride and phenylhydrazones Guido la Parola *Gazz. chim. ital.* 65, 624-7(1935) —The condensation reaction of  $OC(=CH)CH(=O)CO_2$  (I) with Schiff bases (cf. *C. A.* 29, 3315\*) was extended to phenylhydrazones of aromatic aldehydes to learn whether, besides the expected formation of an acyl group in the NH group, the HC N group would be hydrolyzed. However, the only reaction was the following:  $I + PhHN=CHAr \rightarrow ArCH=NNHPhCOCH_2CH_2OH$ . In each case when equimol. quantities of the 2 reagents in  $C_6H_5$  were mixed at 70°, the products were pptd. directly on cooling. Since the reaction seems to be a general one, it should be of utility as a means of identification of phenylhydrazones. When the NH group in the phenylhydrazone contains an alkyl group, as in  $PhMeNN$  CHPh, there is no reaction with I, even after prolonged refluxing. *N*-Maleylbenzophenylhydrazone-carboxylic acid,  $PhCH=NNHPhCOCH_2CH_2OH$ , m. 123-4°; *N*-*p*-Tolual homolog,  $p$ - $MeC_6H_4CH=NNHPhCOCH_2CH_2OH$ , m. 107°. Aromatic compd., m. 136°. Salicylic compd., m. 126°. All 4 acids are hydrolyzed by hot aq. 0.5 N NaOH to the corresponding phenylhydrazones. C. C. Davis.

Constitution of some additive compounds of tertiary phosphines W. Cule Davies and W. P. Walters *J. Chem. Soc.* 1935, 1786-92 —The disocn. of the compd.  $R_2R_3P$  CS<sub>2</sub> results in CS<sub>2</sub> (gas) +  $R_2R_3P$  (liquid)  $\rightleftharpoons$   $R_2R_3P$  (vapor). The empirical index of stability is obtained by comparing the temps. at which the disocn. pressure of a deriv. of  $PhMeP$  (taken as unity) and of the standard become 50 mm. The following give the index of stability, the m. p. (sealed tube) and color of the finely powd. compd.:  $p$ -OMe 1.34, 119°, deep orange;  $p$ -Me 1.11, 118°, light brown;  $PhMeP$  1.00, 102°, terracotta;  $p$ -PhO 0.97, 88°, salmon-pink;  $p$ -Br 0.92, 96°, orange; 2,5-Me<sub>2</sub> 0.81, 71.5°; 2,4,6-Me<sub>3</sub> 0.55, 46°;  $PhEtP$  derivs.:  $p$ -Me<sub>2</sub>N 1.27, 103°, deep orange;  $p$ -PhO 0.77, 69°, brown;  $p$ -Me 0.62, 55°, chocolate, unsubstituted 0.49, 45°, brown;  $p$ -Br <0.4. The stability of the compd. is increased by an electron-releasing group ( $p$ -Me) and diminished by an electron-attracting group ( $p$ -Br). Comparison of the CS<sub>2</sub> compds. of  $p$ -tolyl-,  $p$ -xylyl- and 2,4,6-trimethylphenyldimethylphosphines shows that the introduction of  $o$ -Me groups decreases the stability. Change from arylidimethyl to aryldiethylphosphines considerably depresses the stability of the additive compd. The mechanism of the formation of the CS<sub>2</sub> compds. is discussed. Values are given for the disocn. consts. of the above compds.; also, for the vapor pressures of  $PhMeP$  and  $p$ - $MeC_6H_4PMe_2$ .  $Et_3P$  formed the following compds.: *phenyllithiocarbamate*, pale yellow, m. 61°; *p*-tolylthiocarbamate, pale yellow, m. 85-90°; *p*-nitrophenyllithiocarbamate, lemon-yellow, m. 97°, *p*-benzoquinone, black, m. about 180°, *tributylphosphine*-*p*-benzoquinone, cream, m. about 180-90°, *p*-tolylmethylphosphine-*p*-benzoquinone, cream, does not m. 250°. Mol.-wt. detns. showed the following degree of disocn.:  $Et_3P$  CS<sub>2</sub> 0.13, 0.07,  $PhPMe_2$  CS<sub>2</sub> 0.59-0.77,  $p$ -MeOC<sub>6</sub>H<sub>4</sub>PMe<sub>2</sub> CS<sub>2</sub> 0.54-0.69,  $Et_3P$  CS<sub>2</sub> (in  $C_6H_6$ ) 0.72,  $PhNCS$   $Et_3P$  (ebullioscopic in  $Me_2CO$ ) 0.38,  $p$ -O<sub>2</sub>N $C_6H_4$  NCS  $Et_3P$  0.06;  $p$ - $MeC_6H_4$  NCS  $Et_3P$  0.52.  $Et_3P$  CS<sub>2</sub> has a mol. const. of 0.015 and 0.019 for 20 and 100 l./g.-mol. The reaction between a *tert* phosphine and S is practically instantaneous. There is no evidence of combination between  $Bu_3P$  and the following compds. in  $Et_2O$  at -80°:  $PhCO$ , dimethyl- $\gamma$ -pyrone, thiourea, thioisamine, thiocarbamide, 4,4'-bisdimethylamino bisbenzophenone, Na diethyldithiocarbamate and *p*-dimethylamino benzylidenehydrazine. C. J. West.

Two methods for iodinating phenols C. J. West. *Ann. sci. univ. Jassy* 20, 131-8(1935) —Two new more or less general methods have been developed for iodinating phenols. In the 1st, I<sub>2</sub> in MeOH was added drop by drop to a soln. of the phenol in MeOH contg. dry  $NH_3$ . The reaction was rapid at first but slowed up near the end. The yields were better by this method than with aq. NaOH although arisols, if formed at all, were formed by either method. Methylamines, even  $NMe_3$ , could be substituted for  $NH_3$ . The I entered para and then ortho to the OH group. In this method the  $NH_3$  served to neutralize the HI and may have also formed intermediate products such as  $NH_4I$  and  $NH_3$ . A soln. of 13 g. I<sub>2</sub> was added to 6 g. 3,4-Me<sub>2</sub>C<sub>6H<sub>3</sub>OH in  $NH_4$  MeOH.  $H_2O$  was added, the soln. was extd. with  $Et_2O$ , the  $Et_2O$  was washed with aq.  $SO_2$ , dried and evapd., leaving 3,4-dimethyl-5-iodophenol, m. 71° (from petroleum ether). 3,5-Me<sub>2</sub>C<sub>6H<sub>3</sub>OH when treated in the same way gave a diodo-3,5-dimethylphenol, m. 176-7° (decompn.), *acetate*, m. 153-4°. This diiodophenol gave an arisol with the liberation of I<sub>2</sub> when heated with 10% NaOH in the presence of  $K_2S_2O_8$ . In the 2nd method the acetoxymercuri deriv. of the phenol was dissolved in NaOH soln. and slowly poured into dil. aq. AcOH contg. I<sub>2</sub> and KI. The reaction  $ArHgOAc + I_2 \rightarrow ArI + IHgOAc$  was instantaneous and quant. In fact the reaction could be used for detg. acetoxymercuri compds. by using excess standardized I<sub>2</sub> soln. and titrating back. The advantages of the method are that the acetoxymercuri derivs. are easy to prep., that no arisols are formed and that the I enters the ring in the same position as the AcOH group, thus orienting the I deriv. 4,6-Diacetoxymercuri-2,5-dimethylphenol (64 g.) was mixed with 180 cc. 15% NaOH, dild. to 1 l. and slowly poured into 52 cc. I<sub>2</sub> and 60 g. KI in AcOH. After standing the soln. was decanted, the HgI<sub>2</sub> was dissolved in KI soln. and the phenol was taken up in petrol ether and treated with  $SO_2$ . Yield, 93% of 4,6-diiodo-2,5-dimethylphenol, m. 63° (from AcOH). When heated with NaOH or NaI soln. this gave an arisol 2-Acetoxymercuri-6-bromophenol,</sub></sub>

when treated in the same way, gave 92% 2-iodo-6-bromo-*thymol*, an oil. When heated with 10% NaOH this liberated Br<sub>2</sub> and formed an arisol. John E. Milbrey

Identification of phenols with 2,4-dinitrochlorobenzene. R. W. Bost and Frank Nicholson. *J. Am. Chem. Soc.* 57, 2368-9 (1935).—The following 2,4-dinitrophenyl ethers were prep'd from the Na salt of the phenol and 2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>Cl in 95% EtOH. PhOH, pale yellow, m 69°, *o*-MeC<sub>6</sub>H<sub>4</sub>OH, pale yellow, m 90°, *m*-isomer, pale greenish yellow, m 74°, *p*-isomer, m 93.5°, *thymol*, pale yellow, m 67°, *guaiacol*, pale yellow, m 97°, *C<sub>6</sub>H<sub>5</sub>OH*, pale yellow, m 128°, *β*-isomer, m 95°, *p*-HOC<sub>6</sub>H<sub>4</sub>Ph, pale greenish yellow, m 118°, *resorcinol*, buff, m 101°, *isoeugenol*, *S*-yellow, m 130°, *o*-O<sub>2</sub>N<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH, buff, m 142°, *m*-isomer, light yellow, m 138°, *p*-isomer, light yellow, m 120°, 2,5-Cl<sub>2</sub>(HO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>Me, pale greenish yellow, m 112°, *o*-ICl<sub>2</sub>OH, pale yellow, m 93°, *o*-ClC<sub>6</sub>H<sub>4</sub>OH, pale greenish yellow, m 99°, *m*-isomer, pale yellow, m 75°, *p*-isomer, pale yellow, m 126°, 2,4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH, pale yellow, m 110°, 2,4,6-Cl<sub>3</sub>C<sub>6</sub>H<sub>2</sub>OH, pale yellow, m 136°, *o*-BrC<sub>6</sub>H<sub>4</sub>OH, pale greenish yellow, m 89°, *p*-isomer, pale yellow, m 141°, 2,4-Br<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH, pale yellow, m 135°, 2,4,6-Br<sub>3</sub>C<sub>6</sub>H<sub>2</sub>OH, pale greenish yellow, m 135°, *p*-ICl<sub>2</sub>OH, light yellow, m 166°.

Carvacrol V Benzyl ethers of acetylated methylisopropylphenols. Hanna John and Paul Beetz. *J. prakt. Chem.* 144, 49-52 (1935). cf. *C* 29, 7038.—*p*-Acetothymol (I) and PhCH<sub>2</sub>Cl with EtOH-KOH give about 85% of *p*-acetylthymyl benzyl ether, m 71°, *p*-propionyl homolog, m 64°, *p*-butyryl homolog, m 47°, *p*-isovaleryl homolog, m 40°, *p*-acetylthymyl 4-nitrobenzyl ether, pale yellow, m 104°, *p*-propionyl homolog, pale yellow, m 110°, *p*-butyryl homolog, yellow, m 97°, *p*-isovaleryl homolog, yellow, m 84°, *p*-benzoyl homolog, yellow, m 91°, *p*-Acetocarvacryl benzyl ether, m 64°, 4-nitrobenzyl ether, yellow, m 114°, *p*-propionacarvacryl 4-nitrobenzyl ether, yellow, m 128°. I and ClCH<sub>2</sub>(CH<sub>2</sub>)Cl, with EtOH-KOH give glycerol *α,α'*-bis(*p*-acetylthymyl) ether, m 100°; the *o*-nitro-*p*-acetylthymyl ether, m 95°.

Synthesis of certain alkyl and aryl cryptophenols. Martin E. McGreal and Joseph B. Niederl. *J. Am. Chem. Soc.* 57, 2625-7 (1935).—Condensation of Me<sub>2</sub>(iso-Pr)<sub>2</sub>COH and PhOH with ZnCl<sub>2</sub> gives *p*-(2,2,3-trimethylpropyl)phenol, m 105°, PhOH coeff (P. C. 45, Me<sub>2</sub>BuCOH gives *p*-tert-heptylphenol, b<sub>10</sub> 280°, (iso-Bu)<sub>2</sub>COH and PhOH did not give the expected *p*-tert-octylphenol but *p*-tert-butylphenol. 1-Methyl-2-cyclohexanol and PhOH give *p*-2-methylcyclohexylphenol, m 107°, P. C. 55; *β*-Me isomer, m 101°, P. C. 105; *α*-Me isomer, m 108°, P. C. 70. PhCH<sub>2</sub>OH gives *p*-PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OH, m 82°; PhMeC<sub>6</sub>HO or PhCH<sub>2</sub>CH<sub>2</sub>OH yields *p*-PhMeC<sub>6</sub>H<sub>4</sub>OH, m 64°, P. C. 40, PhMeEtCOH gives 2-phenyl-2-(4-hydroxyphenyl)butane, b<sub>10</sub> 145-8°, P. C. 33, PhMe(iso-Pr)COH gives 2-methyl-3-phenyl-3-(4-hydroxyphenyl)butane, b<sub>10</sub> 157-60°.

Thyroxine from hydroquinone monomethyl ether and 3,4,5-triodonitrobenzene. A. Ya Savitzkii. *Med. eksptl. (Ukrain.)* No. 1, 39-49 (1934).—To make 3,4,5-IC<sub>3</sub>NO<sub>2</sub>, *p*-nitroaniline was iodated in MeOH with ICl and 2,6-diiodonitroaniline thus obtained was diazotized with nitrosylsulfuric acid in acid, and treated with KI and Na. Ihydroquinone mono-Me ether and 3,4,5-IC<sub>3</sub>NO<sub>2</sub> were condensed in MeCOEt, producing pure 3,5-diiodo-4-(4'-methoxyphenoxy)nitrobenzene. The nitro group was reduced to amino, diazotized to CN, changed to imino, and hydrolyzed to 3,5-diiodo-4-(4'-methoxyphenoxy)-benzaldehyde, which, condensed with hippuric acid, produced 4-[3',5'-diiodo-4'-(4'-methoxyphenoxy)benzylidene]-2-phenyl-5-oxazalone. It was possible to eliminate the etherification and obtain directly 3,5-diiodo-4-(4'-hydroxyphenoxy)phenyl-*α*-aminopropionic acid, which, after introduction of 2 I atoms, produced thyroxine with much higher yield.

Leo Nazarek. The action of hydrogen sulfide on acid chlorides. Ludwik Szperl and Lucjan Oziolek. *Roczniki Chem.* 15, 408-12 (in French 413) (1935).—By the action of H<sub>2</sub>S

on *as*-*o*-xyloyl chloride, di-*as*-*o*-xyloyl sulfide, m. 110-20°, and di-*as*-*o*-xyloyl disulfide, m. 127-8°, are formed. The disulfide was sep'd. after decompn. of the monosulfide by NH<sub>3</sub>. Di-*as*-*o*-xyloyl sulfide, treated with alc. NH<sub>3</sub>, gives 3,4 Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CONH<sub>2</sub> and 3,4 Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>COSNH<sub>2</sub>. 3,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>COH was obtained by the Grignard reaction in 60% yield. M. Wojciechowski.

Phenyl ether series IV. Phenoxybenzene-4,4'-di-thiol and related compounds. C. M. Suter and Paul H. Scrutcheild. *J. Am. Chem. Soc.* 58, 545 (1936), cf. *C* 24, 2751.—Reduction of phenoxybenzene-4,4'-disulfonyl chloride with SnCl<sub>4</sub>-HCl-AcOH gives 71% of phenoxybenzene-4,4'-dithiol (I), m. 103-4°, with Cl-CH<sub>2</sub>CO<sub>2</sub>H in 5% KOH there results 83% of phenoxybenzene-4,4'-dithioglycolic acid, m. 165-6°, various amts. of ClSO<sub>2</sub>H in Ac<sub>2</sub>O or in H<sub>2</sub>SO<sub>4</sub> either did not affect this acid or produced H<sub>2</sub>O-sol derivs., SOCl<sub>2</sub> gives an oily material, C<sub>6</sub>H<sub>5</sub>SO<sub>2</sub>Cl<sub>2</sub>, which yields with AlCl<sub>3</sub> a red tar. I and Me<sub>2</sub>SO<sub>4</sub> in NaOH give 89% of 4,4'-dimethylthioldiphenyl ether, m. 81-5°.

C. J. West. Reaction of aldoloxime derivatives with bases. II. The reaction of carboxy-*α*-benzaldoximes with sodium hydride. Charles R. Hauser, Earl Jordan and Ruth O'Connor. *J. Am. Chem. Soc.* 57, 2150-8 (1935), cf. *C* 28, 1281.—The relative yields of nitriles and oximes formed in the reactions of carboxy-*α*-benzaldoximes with NaOH are a function of the temp. Contrary to the previously accepted view that certain carboxy-*α*-benzaldoximes have *β*-configurations, it has been shown that they are all of the *α*-type. The carboxy-*α*-benzaldoximes with NaOH give higher yields of nitrile and lower yields of oxime than the corresponding Ac derivs. Data are given for 30° and 100°.

C. J. West. Removal of hydrogen and acid radicals from organic compounds by means of bases. II. The removal of acetic acid from acetyl aldoloximes by alkalis. Charles R. Hauser and Earl Jordan. *J. Am. Chem. Soc.* 57, 2150-6 (1935), cf. *C* 28, 1281, 29, 5001.—Acetyl-*β*-benzaldoximes and 2 N NaOH give both nitrile and oxime, the yield of oxime in most cases predominating at 0° and those of the nitrile at 30°. With acetyl-*α*-benzaldoximes 2 N NaOH gives both nitrile (or the corresponding acid) and oxime at 100° and, in certain cases, even at 30°; with the exception of the *o*-NO<sub>2</sub> deriv., the yields of oxime were much greater than those of the acid, the yields of acid were extremely small at 30°. *β*-3,4-Methylene-dioxybenzaldoxime acetate is hydrolyzed somewhat more rapidly than the corresponding *α*-isomer, but it is probable that there is no very great difference in these rates. It is concluded that *α*- and *β*-aldoloxime acetates undergo fundamentally the same types of reaction with alkali, they form oxime by hydrolysis and nitrile by elimination of AcOH. They differ primarily in the ease with which they eliminate AcOH, the *β*-isomers undergoing this reaction much more readily than the corresponding *α*-isomers. These results are discussed on the basis of the hypothesis that the formation of nitrile from acetyl aldoloximes consists of the removal of a portion of alkali, followed by the release of acetate ion. In the presence of Na<sub>2</sub>CO<sub>3</sub> certain acetyl-*β*-benzaldoloximes give high yields of nitrile and low yields of the corresponding oxime. Certain acetyl-*α*-benzaldoloximes with KNH<sub>2</sub> in liquid NH<sub>3</sub> form both nitrile and oxime, the yields of nitrile being greater. Tables give the yields of products.

C. J. West. Nitro and bromonitro derivatives of *p*-aminoacetophenone. Chr. W. Raadsweld. *Rec. trav. chim.* 54, 813-27 (1935).—The nitration and bromination of several derivs. of *p*-aminoacetophenone were studied. The following new comp'ds were prep'd: Me *p*-acetylphenyl carbamate, m 162°, Et *p*-acetylphenyl carbamate, m 159°, N-(4-acetylphenyl)-N'-ethylurea, m 157°, Me 2-nitro-4-acetylphenyl carbamate, m 107°, Me 2,6-dinitro-4-acetylphenyl carbamate, m 213°, Et 2-nitro-4-acetylphenyl carbamate, m 111°, Et 2,6-dinitro-4-acetylphenyl carbamate, m 176°, N-(2-nitro-4-acetylphenyl)-N'-n-ethylurea, m 91°, N-(2,6-dinitro-4-acetylphenyl)-N'-n-ethylurea, m 120°, N-(2,6-dinitro-4-

acetylphenyl)-*N'*-phenylurea, m 191°, 3,5-dinitro-4-aminoacetophenone (I) (*N*-Ac deriv, m 176°, 4-(bromoacetyl)aminoacetophenone, m 157°, 3-bromo-4-amino-5-nitroacetophenone (II) (*N*-Ac deriv, m 203°), m 181°. I and II could be diazotized only by Witt's procedure ( $K_2S_2O_8$  and abs.  $HNO_3$ ). When the diazotized II was heated, 3,5-dinitrobenzene was obtained, the Ac group thus being displaced by the  $NO_2$  group.

E. W. Scott

Some derivatives of *p*-aminoacetophenone Chr W. Raadsvelde *Rec trav chim* 54, 827-32 (1935).—The following derivs of *p*- $H_2NC_6H_4CO_2Me$  were prep'd: *N*-(4-acetylphenyl)urea, m 183° (148° reported), *N*-(4-acetylphenyl)-*N'*-phenylurea, m 195°, *N*-(4-acetylphenyl)-*N'*- $\alpha$ -naphthylurea, m 209°, 2,4-dinitro-4'-acetylphenylamine, m 192° (185° reported); 2,4-dinitro- $\alpha$ -naphthyl-4'-acetylphenylamine, m 162°. Derivs of acetophenone ketazine 3-nitro-4-amino, m 350°; 3-nitro-4'-acetylamine, m 270°, 3,5-dinitro-4-amino, m 345°, 3-bromo-4'-acetylamine, m 315°; 3-nitro-4-amino-5-bromo, m 315°; 3-nitro-4'-acetylamine-5-bromo, m above 350°. Derivs of acetophenone phenylhydrazones 3-nitro-4-amino, m 135-8°, 3,5-dinitro-4-amino, m 245°, 3-nitro-4-amino-5-bromo, m 80°, 3-nitro-4'-acetylamine, m 160°.

E. W. Scott

Hydrazones and semicarbazides from *p*-thioanophenylhydrazine Zenichi Horii *J Pharm Soc Japan* 55, 880-7 (in German 195-8) (1935).—Reduction of diazonium compds of *p*-thioanophenylamine with  $SnCl_4$  gave *p*-thioanophenylhydrazine-HCl (I), decomp 188°, the free base,  $C_6H_4N_2S$ , m 95-6°. Condensation of I (in 95% alc) with the following ketones and aldehydes gave the corresponding *p*-thioanophenylhydrazones whose m ps are given:  $Me_2CO$ , colorless needles, m 123-5-9°, acetyl-*p*-thioanophenylhydrazine,  $C_6H_4N_2S$ , yellow needles, m 217°,  $Ac_2O$ ,  $CO_2H$ , yellow needles, m 191-15°,  $AcPh$ , yellow plates, m 109-10°,  $BzH$ , yellow needles, m 135-6°,  $\alpha$ - $HOC_6H_4CHO$ , yellow needles, m 172-3°, *m*-isomer, colorless needles, m 167°, *p*-isomer, yellow needles, m 154°,  $\alpha$ - $MeOC_6H_4CHO$ , yellow plates, m 147-8°, *p*-isomer, yellow-brown needles, m 129-9-6°, heliotropin, colorless needles, m 153-4°, veratraldehyde, yellow plates, m 117°, isovanillin, yellow brown plates, m 148-9°, 3- $ClO_4$ - $MeOC_6H_4CHO$ , yellow needles, m 113-14°, resorcyaldehyde, yellow needles, m 191-2°, 2,4-( $MeO$ ) $_2$  $C_6H_3CHO$ , yellow needles, m 129-9-5°, *p*-tolualdehyde, yellow needles, m 118-19°, cumaldehyde, yellow prisms, m 140°,  $\alpha$ - $O_2NC_6H_4CHO$ , orange red needles, m 171°, *m*-isomer, yellow crystals, m 161-2°, *p*-isomer, orange-yellow needles, m 185-6°, *p*- $Me_2NC_6H_4CHO$ , yellow flakes, m 158-9°,  $m$ - $Cl_2C_6H_3CHO$ , yellow needles, m 125-5-5°, 5- $Cl$ -2- $HOC_6H_3CHO$ , yellow needles, m 217-18°, 3,5- $Cl_2$ -2- $HOC_6H_3CHO$ , yellow prisms, m 223-4°,  $PhCH_2CHCHO$ , yellow prisms, m 139-40°, 2-furaldehyde, yellow prisms, m 124°, *p*-naphthalaldehyde, yellow needles, m 207-8°,  $\delta$ -galactose, colorless needles, m 181-5°,  $\delta$ -mannose, colorless plates, m 185-6°,  $\delta$ -arabinose, colorless needles, m 160-0-5°. 1-*p*-Thioanophenylsemicarbazide, m 217° (thio deriv, m 187°), 4-phenyl deriv, m 239-9-5° (thio deriv, m 190-1°), 4- $\alpha$ -tolyl deriv, m 188-9° (thio deriv, m 163-4°), *m*-isomer, m 230° (thio deriv, m 177-8°), *p*-isomer, m 238-9° (thio deriv, m 170-1°).

F. I. Nakamura

2',6'-Dihydroxy-4'-methoxy- $\beta$ -phenylpropionophenone, extracted from the oil of *Populus balsamifera* Albert Goris and Henri Canal *Compt rend* 201, 1435-7 (1935).—Elimination of free acids, phenols, phenylethylcinamic and cinnamic esters from the coned essence obtained by the extrn of fresh buds of *Populus balsamifera* produced a viscous yellow-brown liquid partially sol in cold benzene. Recrystn of the insol fraction from alc gave fine pale yellow needles of a dihydrochalcone (I),  $C_{15}H_{12}O_4$ , m 168°, cleaved by boiling for 3 hrs with concd  $HCl$  into 1 mol of  $PhCH_2CH_2CO_2H$  (II), m 49°, and 1 mol of 3,5-( $HO$ ) $_2$  $C_6H_3OMe$ , m 78°. Demethylation with boiling  $HCl$  formed equimol amts of  $MeI$ , II and 3,5-( $HO$ ) $_2$  $C_6H_3OH$ . In its compn. I resembles the

flavones from poplar buds, chrysol and tectochrysol (Piccard, *Ber* 10, 176 (1877)), but its resistance to alk. sapon suggests its formulation as 4',2',6'- $MeO(HO)_3C_6H_2COCH_2CH_2C_6H_4Ph$  C. R. Addinall

Diphenylmalonic acid. II. J. Mossman. *Helv. Chim. Acta* 18, 1460-8 (1935).—Sapon of  $Ph_2C(CO_2H)_2$  and  $Ph_2C(COCl)_2$  (I) with dil  $NaOH$  yields  $Ph_2CHCO_2H$  (II) (Staudinger, *et al*, *C. A.* 8, 1112). 4.1 g I, b.p. 150-2°, was agitated 17 hrs with 20 g  $H_2O$ . The acid was extd with ether. After removal of the ether *in vacuo*, the residue was triturated with  $CaH_2$  and the cryst. product (III) dried in a high vacuum at room temp. III loses  $CO_2$  below its m p, and m at the same temp as II. The mixed m p of II and III shows no depression. Analysis of III gives results in agreement with the theoretical values for  $Ph_2C(CO_2H)_2$ , 195 mg III and  $CH_4N_2$  in ether gave 120 mg.  $Ph_2C(CO_2Me)_2$ , m 93.0-3.5°.

W. Gordon Rose

Higher benzenoid hydrocarbons II The isomeric bromofluorenones Harry F. Miller and C. Bryant Bachman *J. Am. Chem. Soc.* 57, 2443-6 (1935); cf. *C. A.* 28, 3671-2.—2-(4-Bromobenzoyl)benzoyl chloride, m 162-3°, results in 92% yield from the acid and  $PCl_5$  in  $CaH_2$ ; liquid  $NH_3$  gives 95% of the amide, m 184-5°,  $NaOBr$  gives 82% of 2-(4-bromobenzoyl)aniline (I), m 106-7°. 2  $C_6H_5SO_2NH_2$ ,  $COCl_2$  and  $PhBr$  with  $AlCl_3$ , followed by hydrolysis of the impure sulfonamide with  $H_2SO_4$ - $AcOH$ , give 28% of I. The Osterlin reaction (*C. A.* 26, 5559) gives only 1% of I from 2-(4- $BrC_6H_4CO$ ) $C_6H_4CO_2H$ . Through the diazo reaction I gives 92% of 3-bromofluorenone, light yellow, m 162°, the over-all yield from  $\alpha$ - $C_6H_4(CO)_2O$  is 55%, from  $\alpha$ - $HOC_6H_4NH_2$ , 21%. 2-Benzoyl-6-bromobenzoyl chloride, light brown, m 119-20° (85.9%), 2-bromo-6-benzoylbenzamide, m 135-40° (87%); 2-bromo-6-benzoylaniline, m 128-30° (57%), the diazo reaction gives 70% of 4-bromofluorenone (II), light yellow, m 185-7° (25% over-all yield from the anhydride).  $\alpha$ - $BrC_6H_4I$  and  $\alpha$ - $IC_6H_4CO_2Me$  with  $Cu$  powder give 14% of 2-bromo-2'-carboxyfluorenyl, m 186-7°; with concd  $H_2SO_4$  it gives a quant yield of II. III The isomeric monobromo 9-fluorenols and monobromofluorenones *Ibid.* 2447-50.—Bromofluorenones (I) are reduced to bromofluorenols by Zn in  $EtOH$ - $NH_4OH$ , 2-, m 130° (97%); 3-, m 142-5° (85%); 4-, m 149-50° (82%); with I and red P in glacial  $AcOH$  these are reduced to the bromofluorenols (II) 2-, m 110° (82%); 3-, m 90-1° (quant); 4-, m 165° (80%) II are obtained from I by reduction with amalgamated Zn in  $EtOH$ - $HCl$  (practically quant yield). Satn of II in  $EtOH$  with dry  $HBr$  gives dibromofluorenones 2,9-, m 107-5-8° (quant), 3,9-, m 124-5° (92%), 4,9-, m 194-5° (83%);  $HCl$  gives the bromo-9-chlorofluorenones 2-, m 102° (quant), 3-, m 116-17° (87%), 4-, m 141-2° (86%). In the presence of  $AcOH$  fluorene mercurates predominantly in the 4-position, in the absence of solvents the 3-isomer is also formed, the sepn of the isomers was not attempted because of their insolv. Treatment of the products with  $PhOH$ - $CaCl_2$  and subsequent bromination gave the Br derivs.

C. J. West

Free radicals E. Darmon *Bull. soc. chim.* [5], 2, 2053-67 (1935).—Lecture on free radicals of various types, long-life radicals such as  $Ph_3C$ , short life of the type  $Me$  and  $Et$ , and radicals discovered by mass and ordinary spectrographic methods. The use of these methods for the detn of mol dimensions and vibrations and the calcn of energies of disson was discussed and the importance of the study of free radicals was stressed. Thirty references C. R. Addinall

Hydroxy polyketones II Dibenzoylcarbinol A. H. Blatt and W. Lincoln Hawkins *J. Am. Chem. Soc.* 58, 81-4 (1936), cf. *C. A.* 29, 5097.— $Bz_2CHOAc$  (I) in 95%  $EtOH$  shows 5.1% enol content; it is not affected by  $AcCl$ , by  $p$ - $MeOC_6H_4COCl$  in  $CaH_2N$  or by  $AcO$  alone or with  $H_2SO_4$ , it does not form a  $Cu$  deriv. with  $CuSO_4$ . In aq.  $Na_2CO_3$  I gives  $BzOH$ ,  $AcOH$  and  $Bz_2CH_2OH$ , aq.  $NaOH$  gives  $BzOH$  and  $AcOH$ ;  $Na_2CO_3$  in  $MeOH$  gives  $BzOH$ ,  $AcOH$  and  $BzOMe$ ,  $EtOH$  gives  $BzOEt$ ,

this cleavage must involve the addn. of alc. followed by cleavage of the addn. product and, from the color changes during the course of the reaction, it is probably the ene-diol which adds alc. Acid hydrolysis ( $H_2SO_4$ ) gives 25% of  $Bz_2CHOH$ , m. 111–12°.  $AcCl$  gives the acetate,  $Cu(OAc)_2$  in  $AcOH$  gives benzal,  $Na_2CO_3$  gives  $BzOH$  and  $Bz_2CHOH$ , aq.  $NaOH$  gives  $BzOH$ , in  $MeOH$  these results Br $Me$ . Distn at 4 mm gives *benzoylcarbinol benzoate*, m. 120°.  $Bz_2CHOH$  does not give a color test with  $NH_4$  acids.

**Salt formation of mononuclear naphthalene derivatives** Herbert H. Hodgson and Reginald L. Elliott. *J. Chem. Soc.* 1935, 1850–4.—2 Nitroaceto-1-naphthalene (20 g.) and  $Cl$  in  $AcOH$  at 100° give 11 g. of the 4- $Cl$  deriv., m. 219°, hydrolysis of 10 g. with  $H_2SO_4$ ,  $EtOH-H_2O$  gives 8.4 g. of 4-*chloro-2-nitro-1-naphthylamine*, orange m. 202°, removal of the  $NH_2$  group through the diazo reaction gives 1-*chloro-3-nitro-naphthalene*, dark brown, m. 127°, reduced by  $SnCl_2$  to 4-*chloro-2-naphthylamine*, m. 68° (stannichloride, light brown plates). The 4- $Br$  deriv. was similarly prepd (Ac deriv., m. 186.5°, stannichloride, buff-brown). 2-Nitro-1-naphthylamine 4-*mercurioacetate* (II), orange-red, m. 242°, *mercurichloride*, brown, m. 280°, *mercuribromide*, red brown, m. 248°, *mercuriodide*, deep scarlet, m. 237°, *mercuriodibromide*, scarlet, m. 240° (decomp.). I and  $KI$  give 4-iodo-2-nitro-1-naphthylamine, which was transformed into 4-iodo-2-naphthylamine, brown, m. 78° (Ac deriv., m. 201°,  $Bz$  deriv., m. 145°, stannichloride, light brown). Reduction of 2-chloro-4-nitro-1-naphthylamine with  $SnCl_2$  in  $HCl-EtOH$  gives the  $HCl$  salt, needles, of 2-*chloro-1,4-naphthylendiamine*, pale green, m. 114°, oxidation gives 2-chloro-1,4-naphthoquinone,  $N,N$ -di-Ac deriv., m. 314°, di- $Bz$  deriv., m. 235°. Reduction of 2-chloro-4-nitroaceto-1-naphthalene gives 2-*chloro-1-N-acetyl-1,4-naphthylendiamine*, pink, m. 215° (stannichloride, cream). 2-Bromo-1,4-naphthylendiamine, greenish yellow, m. 110° (di-Ac deriv., m. 318°, di- $Bz$  deriv., m. 239°, mono- $HCl$  salt, needles; stannichloride, needles). 2-Bromo-4-nitroaceto-1-naphthalene, pale green, m. 230°, 2-bromo-4-nitro-1-naphthol, pale yellow, m. 236°, 2-bromo-1-N-acetyl-1,4-naphthylendiamine, light brown, m. 217° (stannichloride, pink needles). 2-Iodo-1,4-naphthylendiamine, pale green, m. 119° (mono- $HCl$  salt, needles; di-Ac deriv., pink, m. 322°, di- $Bz$  deriv., pale purple, m. 282°). 2-Iodo-1,4-naphthoquinone, yellow, m. 120°. I with  $AgO$  yields the 4-nitroaceto deriv., mustard yellow, m. 270°. 1- $KI$  gives 2-*iodo-4-nitroaceto-1-naphthalene*, greenish yellow, m. 220°, 2-iodo-4-nitro-1-naphthol, pale yellow, m. 249°, 2-iodo-1-N-acetyl-1,4-naphthylendiamine, m. 235° (stannichloride, cream needles). 4-*Chloro-1,2-naphthylendiamine*, pink, m. 93° (mono- $HCl$  salt, light brown plates; 2-N-Ac deriv., brown, m. 233°; 1-N- $Cl$  deriv., cream, m. 243° (stannichloride); di-Ac deriv., cream, m. 174°). 4-*Chloro-1,2-naphthoquinone*, maroon, m. 188°. 4-Bromo-1,2-naphthylendiamine, cream, m. 97° (mono- $HCl$  salt, needles; 2-N-Ac deriv., m. 225°; 1-N-Ac deriv., m. 248° (stannichloride, needles); di-Ac deriv., brown, m. 225°). 4-Bromo-2-nitroaceto-1-naphthalene, pale green, m. 229°. 4-Iodo-1,2-naphthylendiamine, purple, m. 98° (mono- $HCl$  salt, pale brown needles; 2-N-Ac deriv., m. 238°; 1-N-Ac deriv., m. 271° (stannichloride, needles); di-Ac deriv., m. 167°).

C. J. West

**Methylethynaphthalenes III** Synthesis of 2-methyl-6-ethynaphthalene Otto Brunner and Franz Grot-Morawski. 66, 433–7 (1935); cf. *C. A.* 28, 3070.— $p$ - $EtCH_2CHO$  (50 g.) and 67 g.  $MeCH_2RbCO_2Et$  with 53 g.  $Zn$  in  $CaH_2$  give 45 g.  $Et$  2-methyl-6-ethynaphthalene, b. 159–60° (purified from the  $HIO$  ester by transformation into the  $HBr$  addn. product and removal of  $HBr$  by  $PhNMe_3$  at 180°); redn. of 4.2 g. of the ester with  $Na$  and  $EtOH$  gives 23 g. of 3-( $\beta$ -ethynaphthyl)-2-methylpropyl- $I$ , b. 140°;  $HBr$  gives with 20 g. of the alc. 20.5 g. of the bromide, b. 141–2°; action of  $KCN$ , followed by hydrolysis with 10%  $EtOH-KOH$ , gives from 10 g. bromide 15 g. 7-( $\beta$ -ethynaphthyl)-3-methylbutyric acid, b. 185–7°, concd.  $H_2SO_4$  on the  $H_2O$  bath for 2 hrs.

gives from 14 g. acid 9.2 g. 2-methyl-6-ethyl-4- *keto*-1,2,3,4-tetrahydronaphthalene, b. 143–8°;  $Zn$  and  $HCl$  give 2-methyl-6-ethyl-1,2,3,4-tetrahydronaphthalene, b. 140–5°; heating with  $S$  at 270–300° gives 2-methyl-6-ethynaphthalene, b. 145–30°; *picrate*, golden yellow, m. 100–1°; *strophane*, yellow, m. 138–9°.

C. J. West

**Estimation and purification of  $\beta$  naphthylamine in the presence of  $\alpha$ -naphthylamine and properties of some sulfonyl derivatives** Herbert H. Hodgson and Ernest W. Smith. *J. Chem. Soc.* 1935, 1854–6.—The solubilities of  $\alpha$ -toluene- $\alpha$  (I) and  $\beta$ -naphthylamine (II) in 2% aq.  $NaOH$  at 16° are 125 g. and 3 g. per l., resp.; II is practically insol in 10%  $NaOH$ , in 2%  $KOH$  the values are 185 g. and 110 g. per l.  $\beta$   $C_6H_5NH_2$  may be estimated in the presence of the  $\alpha$ -isomer by the complete pptn. of II in the presence of a high concn. of  $Na$  ions; however, when the concn. of I exceeds 20%, II is only partly pptd.; for concns. of I between 25 and 50%, the soly. of II is almost exactly proportional to the quantity of I in soln. On the addn. of aq.  $KOH$  solns. of II to solns. concg.  $Na$  ions, the  $Na$  can be detected with ease when the concn. is 2% or higher. When an aq. acid soln. contg. both  $\alpha$ - and  $\beta$ - $C_6H_5NH_2$  is gradually neutralized the  $\beta$ -isomer is pptd. first, which is used as a means of purifying the  $\beta$ -isomer. The  $Na$  salt of II crystallizes in silky needles, m. 370°, the  $NH_2$  salt could not be isolated owing to hydrolysis.  $m$ -Nitrobenzenesulfonyl- $\alpha$ -naphthalide yields a  $Na$  salt with 4 mols.  $H_2O$ , indescnt scarlet needles; at 50° it forms a chocolate-brown *monohydrate* and at 120° the anhyd. salt is formed as pale orange needles, m. 236°; the  $Ac$  salt reps. as a *dihydrate*, brick-red needles; at 120° the  $H_2O$  is lost, giving pale orange needles, m. 232°. The  $\beta$ -isomer, pale cream, m. 166.5°;  $Na$  salt, as the tetrahydrate, pale cream, m. 77°, at 60° it forms a pink *monohydrate*, and at 120° the pale yellow anhyd. salt, m. 200°, the  $Ac$  salt forms a trihydrate, pale yellow, m. 77°, forming a monohydrate at 60°, pink-orange, and at 120° the pale yellow anhyd. salt, m. 210°;  $Bz$  salt, pale orange dihydrate and light brown anhyd. salt, chars about 280°. The definite m. ps. indicate that these salts are coordinated compounds.

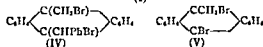
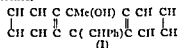
C. J. West

**Sulfonation of naphthalene. II. Determination of the mechanism of monosulfonation.** Robert Lantre. *Bull. soc. chim.* [5], 2, 2092–2108 (1935); cf. *C. A.* 30, 16941.—Energetic sulfonation was carried out by heating 0.1 g. of naphthalene (II) with 0.5 cc. of  $H_2SO_4$ , varying in concn. from 51.4–70.3%, at 180° for 10 hrs. in sealed tubes. The unchanged I was detd. by  $CrO_3$  oxidation and the proportions of  $\alpha$ - and  $\beta$ -sulfonic acids (II and III) was estd. by methods previously described. After II and III had been eliminated by filtration, I was removed by boiling with  $H_2O$  and the sulfones were detd. by  $CrO_3$  method. Similar expts. were carried out with  $\beta$ - $C_6H_5SO_2Na$  (IV) in place of I. Equil. between I and IV exists only when the rate of sulfonation is equalled by that of hydrolysis due to  $H_2O$  formed in the sulfonation process. To elucidate the sulfonation mechanism it was found necessary to study the rates of sulfonation of I and of desulfonation and to det. their variations under various conditions. Sulfonation at 140° of 0.1 g. of I with 2 cc. of 70.3, 64.0 and 57.7%  $H_2SO_4$  and expts. with 70.1, 76.1 and 82.2%  $H_2SO_4$  at 90° and 82.2, 88.3 and 94.4%  $H_2SO_4$  at 60° were carried out which showed the considerable influence of the concn. of the acid on the rate of sulfonation. Desulfonation at 140° of  $\alpha$ - $C_6H_5SO_2Na$  (V) in the presence of 2 cc. of 45.1, 51.4, 57.7 and 64.0%  $H_2SO_4$  showed that the rate increases with the concn. of the acid. Similar expts. at 100° with 51.4, 57.7, 64.0 and 70.3% acid showed that the rate of hydrolysis is an exponential function of the concn. (cf. Crafts, *Fer.* 34, 1350 (1901)). Analogous results were obtained with IV. A study of the action at 140° of 16 cc. of 64.2 and 45.0%  $H_2SO_4$  on 2 g. of V proved that sulfonation produces both  $\alpha$ - and  $\beta$ -sulfonic acids since the transposition of the  $\alpha$ - into the  $\beta$ -acid is the result of a desulfonation followed by re-sulfonation. It was further demonstrated that in the elementary process of sulfonation of I, 80–90% of the sulfonic acids produced is the  $\alpha$ -modification. By sulfona-

tion at 100° with 81.7 and 85.1% acids it was shown that at a given temp., the transformation of the  $\alpha$ - into the  $\beta$  form accelerates with increase in concn of the acid. This results from hydrolysis and resulfonation, both of which reactions are favored by increasing concn. Sulfones are only found in small quantities (2-4%) on desulfonation of IV or sulfonation of I for 39 hrs at 140° with acids varying from 45.1-70.3%  $H_2SO_4$ . The above expts prove that in the presence of acids contg. 53.0-68%  $H_2SO_4$  there is an equilibrium between I and the sulfonic acids, I decreasing as the acid concn increases, that this equilibrium is the result of the opposed reactions of sulfonation and desulfonation whose rates increase with the concn. of the  $H_2SO_4$ , the former more quickly than the latter; that the relative quantities of  $\alpha$ - and  $\beta$ -sulfonic acids formed are independent of the concn of the  $H_2SO_4$  but that desulfonation destroys the  $\alpha$  form more readily. This explanation of the phenomena of transformation of  $\alpha$  into  $\beta$  by sulfonation and desulfonation makes explicable the various problems presented by the process of monosulfonation of I and, in particular, explains the methods utilized for the prepn of each of the isomeric monosulfonic acids. Details are given for the prepn of the sample for the detn. of I by the  $CrO_3$  method. To est.  $C_{12}H_{10}SO_3$  in the presence of the  $\beta$  isomer the sulfonation mixt. is poured into a 10-fold amt. of  $H_2O$  and is neutralized by pure  $BaCO_3$ . The neutral soln is boiled, filtered and washed and the combined filtrate is evapd to dryness and pulverized. A portion is used for the estn. of the total sulfonic acids by the  $CrO_3$  method. The remainder is brominated for 6 hrs in the presence of  $H_2SO_4$  to detn. the  $\alpha$  sulfonic acid content. C. R. Addinall

Preparation of  $\alpha$  naphthoic acid. D. J. Loder and F. C. Whitmore. *J. Am. Chem. Soc.* 57, 2727 (1935).— $\alpha$ - $C_{12}H_9MgBr$  (5 mols) in 2.5 l. abs.  $Et_2O$ , slowly added to 7.5 mols  $PtCl_2$  in 500 cc.  $Et_2O$ , and the mixt. treated with a slight excess of 30%  $H_2SO_4$ , gives 70%  $\alpha$ - $C_{12}H_9CO_2H$ . Hydrolysis gives 90%  $\alpha$ - $C_{12}H_9CO_2H$ . C. J. West

Additions to conjugated systems in the anthracene series. III. Factors influencing the mode and extent of reaction of the Grignard reagent with ketones. Percy L. Julian, Wayne Cole and Thomas F. Wood. *J. Am. Chem. Soc.* 57, 2508-13 (1935). cf. C. A. 29, 7320. Benzanthrone (14 g) and  $MeMgI$  give, on decomp. the product with  $NH_4Cl$  and passing  $O_2$  through the  $Et_2O$  soln, in addn to 12 g of I, 0.3 g of a compd. (III), decomp. at 74° with sepn of anthraquinone (III). I, m 148°, in the Grignard machine it gives 1 mole gas and consumes 1 mole of reagent, oxidation of I gives III and  $BzOH$ . I (3.6 g) and 1.6 g  $Br$  in  $CHCl_3$  give 4 g of IV, m 168° (decomp.), further action of  $Br$  gives V, yellow, m 200°, with  $AcOH$   $AcONa$  IV yields a diacetate, light yellow, m 188°. Ethyldieneanthrone (VI) is obtained as a red oil by destructive distn of ethyloxanthrone in  $N_2$ , it b.p. 245-7°, distn at atm pressure yields III and  $CH_2$ . VI exists in an enolic form involving a 1,7-shift of  $H$ , the anthranol suffering oxidation to a peroxide, which cleaves spontaneously to III and vinyl ac. Anthraphenone undergoes 1,5-dimol reduction on treatment with  $PhMgBr$  or  $PhMgI$  and no reaction with  $MeMgI$ . Dihydroanthraphenone gives normal 1,2-addn. Definite products could not be isolated from the reaction of phenylanthraphenone (VII) and  $PhMgBr$ , the dihydro deriv. and  $PhMgBr$  yield VII and unchanged material. Striking analogies are given between *meso*-unsatd anthracene ketones and  $\alpha,\beta$ -unsatd ketones. The analogies are also maintained when the unsatn. is removed in both types of ketones.



C. J. West

Dissociable anthracene oxides. The influence of *meso*-naphthyl groups. Antoine Willemart. *Compt. rend.* 201, 1201-2 (1935).—9,10-Di- $\beta$ -naphthylanthracene (I), m 378-0°, was obtained by the method of Guyot and Stachling from 9,10-di- $\beta$ -naphthylhydroxydihydroanthracene and  $KI$  in  $HIOAc$  soln. I and 9,10-di- $\alpha$ -naphthylanthracene are thermochromes. A soln of the hydrocarbons, exposed to sunlight in the presence of air gives, after evapn of the solvent, a residue which when heated at 180-200° yields pure  $O$ , about 70% of the amt. calcd for 1 mol of  $O$  per mol of hydrocarbon.

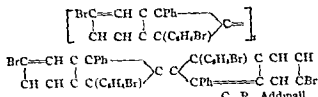
Rachel Brown

Preparation of 9,10-dihydrophenanthrene and its derivatives. Alfred Burger and Frich Mosettig. *J. Am. Chem. Soc.* 57, 2731-2 (1935).—Catalytic reduction of phenanthrene in abs.  $PtOH$  with catalyst 37 KAF (C. A. 26, 2428) at pressures from 2000-4000 lb./sq. in for 24-36 hrs gives 60-80% of crude 9,10-dihydrophenanthrene, with the Friedel Crafts reaction there results 90% of the 2-*Ac* deriv., m 51-2° (oxime, m 146-7.5°, semicarbazone, m 236-7°).  $CrO_3$  gives 2-acetyl-9,10-phenanthrene-quinone, m 223-4°, while  $NaOCl$  yields 9,10-dihydrophenanthrene-2-carboxylic acid, m 211.5-12.5° (*Me* ester, an oil), transformed by  $Se$  at 300° in 20 hrs to phenanthrene-2-carboxylic acid, 9,10-dihydro-2-bromoacetylphenanthrene, m 93.5-5°.  $C_{12}H_{10}$  on high pressure hydrogenation with  $Cu$  chromite is reduced nearly quantitatively to the 9,10-dihydro deriv.; this is completed at 160° within a few hrs. C. J. West

Synthesis of certain hydrogenated phenanthrenes. F. E. Gruber and Roger Adams. *J. Am. Chem. Soc.* 57, 2555-6 (1935); cf. Barnett and Lawrence, C. A. 29, 7319°. The synthesis of 1,1'-dihydroxy-1,1'-bicyclohexyl, di- $\Delta^4$ -cyclohexene (I) and the anhydride of II is described.  $\Delta^4$ - $\Delta^4$ -Dodecahydrophenanthrene-9,10-dicarboxylic acid (II), from the anhydride in 5%  $NaOH$ , m 242° (bloc Maquenne), the anhydride and  $NH_4OH$  in 250-300° give the imide of II, m 182-3°. I and acrolein in  $C_6H_6$ , heated 22 hrs. at 50-60°, give 20% of  $\Delta^4$ - $\Delta^4$ -dodecahydrophenanthrene-9-ol, b.p. 130-7°, semicarbazone, m 170-80°. C. J. West

Dissociable organic oxides. A tetra bromotetraphenylrubene and its dissociable oxide. Charles Dufrasse and Henri Rocher. *Bull. soc. chim.* [5], 2, 2235-40 (1935).—Only limited yields of the carbinol (I), ( $\beta$ - $BrC_6H_4$ ) $_2$  $C(OH)CPh$ , were obtained by the action of  $\beta$ - $BrC_6H_4$  $_2$  $CO$  (II) was used as starting material. II was prepd. by the  $CrO_3$  oxidation of ( $BrC_6H_4$ ) $_2$  $CH_2$  (*Am. Chem. J.* 30, 418 (1907)) according to the method of Goldwithe. Into a Grignard reagent from 1 g  $Mg$ , 6 cc anhyd  $Et_2O$  and 3 cc 1 tBr to which 4.6 cc of  $PhC$   $CH$  had been added dropwise was introduced gradually, with const stirring, 12.40 g of powd. II, m 177°. After refluxing the mixt for 1 hr, the product was worked up and the resulting oil was taken up in a min. amt. of petroleum ether (b.p. 80-100°). The pptd. carbinol was recrystd. from petroleum ether and yielded colorless, cryst. I,  $C_{24}H_{18}Br_4O$ , m 170-1° (mixed m p with II, 142°), sol in concd  $H_2SO_4$  with purple color; *Me* ether,  $C_{24}H_{18}Br_4O$ , m 103°. Refluxing a mixt of 1 g of I in 25 cc alc and 2.5 cc concd  $H_2SO_4$  for 25 min. gave, after the usual treatment, clear yellow crystals of the ethylene ketone, ( $\beta$ - $BrC_6H_4$ ) $_2$  $C(COPh)_2$  m 112-13°. The  $HCl$  ester of I, m 122-3°, is extremely sensitive to moisture and accordingly is not isolated when used as an intermediate in the prepn of the rubene. A cold soln of 3 g of I in 60 cc of anhyd  $Et_2O$  was treated with 0.9 cc of  $PCl_5$  for 6 hrs. at -15° to -20° in a well-stoppered flask. The solvent was evapd *in vacuo* and the residue was heated *in vacuo* with 1 cc of quinoline at 115° for 50 min. The mass was disintegrated with benzene and, after removal of the resin, the quinoline was washed out with 5%  $HCl$ . The benzene was distd. off and the residue was worked up and, on crystn., yielded 30% of deep clear or clear orange crystals of tetra bromotetraphenylrubene (III),  $C_{24}H_{18}Br_4$ , m 391-10°. The absorption spectrum of III is similar to that of tetraphenylrubene except that the summits of the

curves are less pronounced. Solns of III are fluorescent and are decolorized by light. A soln. of 0.6 g. of III in 50 cc of benzene and 200 cc of  $\text{Et}_2\text{O}$ , on exposure to sunlight and evapn of the non-fluorescent colorless solid, gave a cryst. colorless photolabile,  $\text{C}_{18}\text{H}_{14}\text{Br}_2\text{O}$ ,  $\text{C}_{18}\text{H}_{14}$ , which dried, at  $105^\circ$ , liberates O with a titer of 95%, and in yields of the order of 50%. III is a new rubene with characteristic properties and, from its origin and behavior, should have 1 of the 2 formulas



Dissociable organic oxides. Naphthalene formula of rubenes. Synthesis of 9,10,11,12-tetraphenylnaphthalene, its identity with tetraphenylnaphthalene (formerly rubrene). Charles Dufrasse and Leon Vellaz. *Compt rend* 201, 1374 (1935). cf. *Bull soc chim* [5], 2, 1546 (1935).—Under the conviction that the rubenes are naphthalenes and not fulvenes, an attempt has been made to synthesize tetraphenylnaphthalene (formerly rubrene) by phenylation of naphthalene in positions 9, 10, 11 and 12. The action of  $\text{PhMgBr}$  on 1,12-dihydroxy-9,10-naphthaquinone (Br 31, 1150 (1935)) yielded yellow crystals of 1,12-diphenyl-9,10-naphthaquinone (II), m.  $284^\circ$ , which was phenylated to a tetraphenyldiquinol (II) converted by dehydration to a hydrocarbon  $\text{C}_{24}\text{H}_{18}$ , identical with deshydrorubene (C. 26, 2189). Elimination of the 2 OH groups of II by reduction yielded the predicted tetraphenylnaphthalene as a red-orange hydrocarbon, 9,10,11,12-tetraphenylnaphthalene,  $\text{C}_{24}\text{H}_{18}$ . Together with I an isomeric compd., bis(9,10-diphenyl-9,10-naphthaquinone) (III) was formed. III has the fulvene skeleton previously assigned to the rubenes and the series of reactions carried out on I which should convert III into the fulvenic isomer of tetraphenylnaphthalene has been anticipated by the publication of Eck and Marvel (C. A. 29, 7971\*), who have shown that the phenylation of III does not lead to products belonging to the rubene series. The naphthalene formula is more satisfying in regard to the phenomena of dissociation and reversible oxidation since the C atoms in positions 9, 10, 11 and 12 have 3 aryl linkages and are in reality triarylmethyl groups. The same conception holds good for the structure of *meso*-diarylanthracenes whose reversible oxidation has recently been established (C. A. 29, 6889\*).

Rubene problem. A. Schönberg. *J. Am. Chem. Soc.* 58, 182 (1936).—Comments on a recent paper of Eck and Marvel (C. A. 29, 7971\*).

Preparation of tetrahydrofuran. I. T. Strulov. *Khim. Form. Prom.* 1935, No. 1, 35 (1935).—Tetramethylene glycol is treated with SOCl<sub>2</sub>, heated on a water bath 0.5 hr. and distd. at  $75^\circ$ . The distillate is dehydrated over NaOH and redist. at  $64^\circ$ .

2,6-Dimethylphenyl  $\alpha$  naphthylcarbamate. Ch. D. Hurd and M. A. Pollack. *J. Am. Chem. Soc.* 58, 181 (1936).—The xylene obtained on hydrolysis of furural (C. A. 26, 3792) is shown to be 2,6-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>OH by the prepn. of the  $\alpha$ -naphthylcarbamate, m.  $176.5^\circ$ , identical with that from an authentic specimen. C. J. West.

A stable catalyst for the oxidation of furfural. V. Ya. Serdyukov. *Moskoben-Zhivost* Delo 1934, No. 4, 43.—In the oxidation of furfural  $\text{V}_2\text{O}_5$  can be replaced with advantage by the alloys of V with Fe or Al (8% V). These alloys are also useful in the oxidation of alcs. and aromatic hydrocarbons. E. Bielouss.

The preparation of furfural from the hulls of sunflower seeds. G. V. Yuzvich. *Moskoben-Zhivost Delo* 1934, No. 5, 40-4.—The yield of furfural is enhanced by coarsely grinding the sunflower seeds to a size of 0.25-0.50 mm and using  $\text{H}_2\text{SO}_4$  in amts. of not less than 4% of dry hulls. E. Bielouss.

Reduction-oxidation phenomena observed in the de-

hydration of alcohols with a furanic nucleus. R. Paul. *Bull soc. chim.* [5], 2, 290-7 (1935); cf. C. A. 29, 4359\*.—In the presence of metallic oxides alcs. are dehydrogenated by heat and certain alcs. are directly reducible to hydrocarbons. It has been shown that both these reactions take place in the course of the dehydration of furanic alcs. by heating with  $\text{Al}_2\text{O}_3$  at  $400^\circ$  and that in addition to the expected ethylenic furans, notable quantities of alkyl furans and furyl ketones are formed. Com.

$\text{Al}_2\text{O}_3$ , partially dehydrated by heating for 3-4 hrs. at  $450^\circ$  was heated at  $400^\circ$  with the alc. in a N. atm. and the products were collected in a flask contg. a trace of hydroquinone. The condensed liquids were satd. with  $\text{K}_2\text{CO}_3$  and the upper layer was decanted, dried over  $\text{Na}_2\text{SO}_4$  and distd. in  $\text{N}_2$  or  $\text{CO}_2$ . The furyl alkanes and alkenes were sep'd in this way in 35% yields but the heat-unstable ketones were isolated as semicarbazones from the aq. alc. ketone layer. Dehydration of 60 g. of  $\text{C}_6\text{H}_5\text{OCH}_2\text{OH}$  (free from  $\text{C}_6\text{H}_5\text{CHO}$ ) gave 2 b. f. (57%) of 2-methylfuran (xylene), b.  $63-64^\circ$ ; d<sub>4</sub><sup>20</sup> 0.915, n<sub>D</sub><sup>20</sup> 1.4387, M. R. 23.35 (calcd. 23.80) ( $\text{H}_2\text{C}_2$  deriv., m.  $134^\circ$  (C. A. 27, 4794\*), and 2-furaldehyde (I) (semicarbazone, m.  $214-15^\circ$ ). Condensation of I (2 mols.) with 2.5 mols. of  $\text{MeMgBr}$  gave 1-furyl ethanol which was dehydrated to produce  $\text{C}_6\text{H}_5\text{OCH}=\text{CH}_2$ , b.  $91-93^\circ$ , a small quantity of impure  $\text{C}_6\text{H}_5\text{OCH}=\text{CH}_2$ , d<sub>4</sub><sup>20</sup> 0.936, n<sub>D</sub><sup>20</sup> 1.48168, M. R. 28.61 (calcd. 27.95) (C. A. 21, 2895\*), and a fraction, bp.  $90^\circ$  (xatm), n<sub>D</sub><sup>20</sup> 0.91\* which may be that of  $\text{C}_6\text{H}_5\text{OCH}=\text{CH}_2$ ; Bouvart gives m.  $104^\circ$  for  $\text{C}_6\text{H}_5\text{OCH}=\text{CH}_2\text{NOH}$ . The dehydration of 65 g. of 1-furylpropanol yielded 4.5 g. of  $\text{C}_6\text{H}_5\text{OCH}=\text{CH}_2$  (C. A. 27, 5738), 15 g. of  $\text{C}_6\text{H}_5\text{OCH}=\text{CHMe}$ , b.  $132-33^\circ$ ; d<sub>4</sub><sup>20</sup> 0.951, n<sub>D</sub><sup>20</sup> 1.51148, M. R. 34.05 (calcd. 32.57); and a fraction bp.  $78-8^\circ$ , contg. 1-furylpropanone (semicarbazone, m.  $100-1^\circ$ ). A large quantity of 1-furylbutanol was dehydrated and sample rectification of the product gave furylbutane, bp.  $48-49^\circ$ ; d<sub>4</sub><sup>20</sup> 0.890, n<sub>D</sub><sup>20</sup> 1.4553;  $\text{C}_6\text{H}_5\text{OCH}=\text{CHEt}$ , b.  $59-60^\circ$ ; d<sub>4</sub><sup>20</sup> 0.932, n<sub>D</sub><sup>20</sup> 1.5028, M. R. 36.68 (calcd. 37.18); and 1-furylbutane, bp.  $92-4^\circ$  (semicarbazone, m.  $182^\circ$ ). The furyl alkenes have an agreeable and tenacious odor reminiscent of the  $\text{C}_6\text{H}_5\text{OCH}$  radical. They are unstable to light and air but can be kept unchanged in N-filled sealed tubes in the presence of traces of hydroquinone. The mol. exaltation is equiv. to that caused by conjugation in a category mol. The formation of glycols from 2-furanol shows that the formation of the alkyl furans is not due to the reduction of the ethylenic furans resulting from the dehydration but is brought about by the direct reduction of the alc. function. The abnormal phenomena are the results of an autoxidation of 2 mols. of  $\text{RCH}(\text{OH})\text{R}'$  into  $\text{RCOR}'$ ,  $\text{RCH}_2\text{R}'$  and  $\text{H}_2\text{O}$ .

Preparation and reactions of tertiary tetrahydrofurylcarbinols. A. L. Doucne, Ralph H. Wardlaw and Ralph Connor. *J. Am. Chem. Soc.* 57, 2550-9 (1935).—Et tetrahydrofuroate (I) and  $\text{PhMgBr}$  gave 53% of tetrahydrofurylphenylcarbinol (II), m.  $74-80^\circ$ . With EtMgBr I gives 76% of tetrahydrofuryl-diethylcarbinol, b.  $200-3^\circ$ ; d<sub>4</sub><sup>20</sup> 0.9744, n<sub>D</sub><sup>20</sup> 1.4532; the yield on catalytic reduction of furyl-diethylcarbinol with Raney Ni at  $150^\circ$  is 57%. Tetrahydrofurylphenylcarbinol (III), bp.  $151-51^\circ$ , m.  $251-2^\circ$ ; d<sub>4</sub><sup>20</sup> 0.9219, n<sub>D</sub><sup>20</sup> 1.4543, 53-45% yield, also in 58% yield by reducing furyl-diethylcarbinol, bp.  $128-31^\circ$ ; d<sub>4</sub><sup>20</sup> 0.9251, n<sub>D</sub><sup>20</sup> 1.4708 (78% yield from Et furate and EtMgBr). Dehydration of II with  $\text{MgSO}_4$  gives 56% of 1,1-disubstituted-2,5-epoxy-1-pentene, bp.  $195^\circ$ , m.  $107-8^\circ$ , which yields with  $\text{O}$ ,  $\text{Ph}_2\text{CO}$  and  $\gamma$ -butyrolactone. The hydrazide of  $\gamma$ -hydroxybutyric alc., m.  $89-90^\circ$ . Dehydration of III gives a mixture of alcs. which could not be sep'd. II is not cleaved by  $\text{PhMgBr}$  under "forced" conditions. C. J. West.

Chalcones and chalcone oxides. I. Phenyl 3,4-methylenedioxyethyl ketone. R. P. Doddawmath and T. S. Wheeler. *Proc. Indian Acad. Sci. A*, 24, 438-51 (1935).—The variation of the marked activity of the chalcones and chalcone oxides caused by the presence of other groups in the Ph. nuclei has been studied by an examn. of the reactivities of ketones contg. electron

sources and sinks such as the  $\text{CH}_2\text{O}$  and  $\text{NO}_2$  groups. The bromination of  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{CH}_2\text{Br})$  (I) with 1 mol of Br gave  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CHBrCHBrBr}$  (II) which, on boiling with EtOH or MeOH, yielded  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{OEt})\text{CHBrBr}$ , m 143-4°, or  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{OMe})\text{CHBrBr}$ , m 115-16°, since the nucleus containing the activating group may be expected to influence the nearer Br atom. The addition of 2 mols of Br to I by adding 32 g of Br in 50 cc of glacial AcOH to 25 g of I in 150 cc of hot AcOH and heating the mixture at 100° for 30 min gave 30 g of  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{BrCHBrCHBrBr}$  (III), m 174-5°, converted into  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{BrCH}(\text{OEt})\text{CHBrBr}$ , m 126-7°, and  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{BrCH}(\text{OMe})\text{CHBrBr}$ , m 131-2°, by boiling with the corresponding alc. On heating Me<sub>2</sub>CO solutions of II and III with KI in H<sub>2</sub>O and Me<sub>2</sub>CO the corresponding chalcones, I and  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{CHBr})\text{CHBrBr}$  (IV), m 146-7°, were formed. IV was also produced by heating a mixture of 6-bromopropional (4.6 g), AcPh (2.4 g), alc (100 cc), and 10% NaOH (2 cc) at 80-100° for 15 min. Chlorination of I with 1 and 2 mols of Cl<sub>2</sub> gave  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CHClCHClBr}$  as a pasty mass (which gave, on crystals from EtOH,  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{OEt})\text{CHClBr}$ , m 118-19°), and  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CHClCHClBr}$ , m 127-8°. Refluxing 20 g of II and 10 g of KCN in 100 cc of alc for 3 hrs gave 8 g of  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{CN})\text{CHBrBr}$  (V), m 132-3°, hydrolyzed by heating for 8 hrs with alc 10% NaOH to  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{CO}_2\text{H})\text{CHBrBr}$  (VI), m 143-4°. The formulations of V and VI were confirmed by the production of PhCH(CN)CHBrBr, m 126-7° (identical with a specimen prepd by the action of HCN on PhCH<sub>2</sub>CHBrBr) from PhCHBrCHBrBr and alc KCN, and by the hydrolysis of this nitrile to the known carboxylic acid (J. Chem. Soc. 85, 1359 (1904)). The bromination of 3,4,6- $\text{CH}_2\text{O}$ ,  $(\text{O}_2\text{N})_2\text{C}_6\text{H}_3\text{CHCHBrBr}$  (VII) gave only  $\text{CH}_2\text{O}$ ,  $(\text{O}_2\text{N})_2\text{C}_6\text{H}_3\text{CHBrCHBrBr}$ , m 174-5°, reconverted into VII by treatment with KI in Me<sub>2</sub>CO. Chlorination of VII in AcOH and treatment of the pasty product with NaHCO<sub>3</sub> solution yielded  $\text{CH}_2\text{O}$ ,  $(\text{O}_2\text{N})_2\text{C}_6\text{H}_3\text{CHClCHClBr}$ , m 151-2°. A mixture of 0.8 g of Na in 25 cc abs alc, 8 g of AcCH<sub>2</sub>CO<sub>2</sub>Et, 10 g of I and 100 cc abs alc was refluxed at 80-90° for 3 hrs and produced 13 g of Et 2-(3',4'-methyleneedioxyphenyl)-4-phenyl-4-cyclohexenecarboxylate,  $\text{C}_{21}\text{H}_{24}\text{O}_4$ , m 151-2°. VII did not react. I was catalytically hydrogenated in the presence of Pd to the corresponding alc  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}_2\text{CH}_2\text{CH}(\text{OH})\text{Ph}$ , m 95-6°, but the said ketone, dihydropyrenonalacetophenone, m 39-40°, was not obtained. VII could not be reduced. With  $(\text{NH}_4)_2\text{H}_2\text{O}$ , I gave unstable white crystals of 3-phenyl-5-(3',4'-methyleneedioxyphenyl)pyrazoline, HCl salt, m 197-8°, picrate, m 185-6°. Ac deriv, m 153-4°. I reacted with PhNHNH<sub>2</sub> to give the corresponding 1,3-diphenyl-5-(3',4'-methyleneedioxyphenyl)pyrazoline,  $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4$ , m 129-30°, which was easily sol in concd H<sub>2</sub>SO<sub>4</sub> and gave an intense blue color on the addition of FeCl<sub>3</sub> and gave a NO deriv which decomposed when dry. Treatment of 20 g of I in a warm mixture of 250 cc of alc and 150 cc Me<sub>2</sub>CO with 120 cc of 6% H<sub>2</sub>O<sub>2</sub> and 20 cc of 10% NaOH produced 18.5 g of white prismatic needles of the oxide of Ph 3,4-methyleneedioxy-1-ketone (VIII),  $\text{C}_{16}\text{H}_{16}\text{O}_4$ , m 99-100°, hydrazone, m 173-4°, converted into the corresponding pyrazole,  $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_4$ , m 194-5°, by boiling with NaOEt or Ac<sub>2</sub>O. Treatment of a suspension of VIII in MeOH or EtOH with a little concd H<sub>2</sub>SO<sub>4</sub> ruptured the oxide ring with the formation of  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{OMe})\text{CH}(\text{OH})\text{Ph}$ , m 117-18°, or  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{OEt})\text{CH}(\text{OH})\text{Ph}$ , m 93-4°. Both these alkoxy compounds were transformed by boiling with alc NaOH into the diketone,  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}_2\text{COBr}$  (IX), m 114-15°. IX was best prepd by boiling alk VIII with NaOH for 30 sec. Boiling alk IX with  $\text{C}_6\text{H}_5\text{NH}_2$  for 5 min. produced 2-phenyl-3-(3',4'-methyleneedioxybenzyl)quinoxaline,  $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4$ , m 137-8°. Refluxing 10 g of VIII in 75 cc. of alc for 4 hrs with 5 g NaOH in 15 cc H<sub>2</sub>O and acidifying the filtrate with dil HCl gave 6.2 g of white needles of phenyl(3,4-methyleneedioxybenzyl)glycolic acid (X),  $\text{C}_{17}\text{H}_{18}\text{O}_5$ , m 149-50°, which was readily oxidized by K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in AcOH to  $\text{CH}_2\text{O}$ ,

1  $\text{C}_6\text{H}_5\text{CH}_2\text{Br}$ , m 70-1° (C. A. 24, 2450), proving the structure of X. The oxidation of VII with 6% H<sub>2</sub>O<sub>2</sub> gave the oxide of Ph 3,4-methyleneedioxy-6-nitrosostyryl ketone, (XI), m 159-60° (C. A. 7, 3748). Unlike VIII, XI did not react with MeOH, EtOH or NaOH but with HCl and AcOH gave  $\text{CH}_2\text{O}$ ,  $(\text{O}_2\text{N})_2\text{C}_6\text{H}_3\text{CHClCHCl}(\text{OH})\text{Ph}$ , m 183-4°. With  $(\text{NH}_4)_2\text{H}_2\text{O}$  in AcOH, VII gave  $\text{CH}_2\text{O}$ ,  $(\text{O}_2\text{N})_2\text{C}_6\text{H}_3\text{CH}(\text{CHClPh})\text{NNHAc}$ ,  $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4$ , m 244-5°. The phenylhydrazones of VII,  $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4$ , m 159-60°, was cyclized by boiling for 1 hr to give 1,3-diphenyl-5-(3',4'-methyleneedioxy-6-nitrophenyl)pyrazoline,  $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4$ , m 203-4°, which responded to Knorr's test and was converted by heating with AgNO<sub>3</sub> into the corresponding pyrazole,  $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4$ , m 163-4°, which was better obtained by treating XI in hot AcOH with PhNH-NH<sub>2</sub>. The results indicate that the NO<sub>2</sub> group removes the enhanced activity conferred on I of the Br atoms in the chalcone dibromide by the presence of a nuclear alkoxy group. It also reduces the reactivity of the oxide toward EtOH, MeOH or alkali but not toward HCl.

C. R. Addinall

ω Benzyl derivatives of acetophenone and their reduction products. G. Albert Hill and A. J. Colfrancesco. J. Am. Chem. Soc. 57, 2426-8 (1935).—The preps of BzCH<sub>2</sub>CH<sub>2</sub>Ph and BzCH<sub>2</sub>CH(Ph)<sub>2</sub> (I) (oxime, m 157°) and their reduction products are discussed. 1,3-Diphenyl-2-benzylpropan-1-ol phenylurethan, m 185°. I and BzCl with NaNH<sub>2</sub> in PhMe gave 68% of tribenzylacetophenone, m 127-8°, Na and abs EtOH reduce this to 1,3-diphenyl-2,2-dibenzylpropan-1-ol, viscous, light yellow liquid, decomps on heating at low pressures; heating the ketone with H<sub>2</sub> and red P at 200° for 4 hrs gives 13% of tetra-benzylpropane, m 164°.

C. J. West

Reduction reactions and oxidation reactions. Vincenzo Paoloni. Gazz. chim. ital. 65, 630-2 (1935).—The work of Tollens and of others has indicated that aromatic aldehydes, unlike aliphatic aldehydes, do not react with Fehling's solution. Nevertheless when BzH, anisaldehyde or cinnamic aldehyde is agitated with Fehling's solution (2 mols of CuO), clear odorless liquids are obtained, which contain no Cu<sub>2</sub>O. From the reaction products are obtained, by extrn with Et<sub>2</sub>O, PhCH<sub>2</sub>OH, anisic alc. and cinnamic alc., resp., while the residual liquids yield, on treatment with H<sub>2</sub>SO<sub>4</sub>, BzOH, anisic acid and cinnamic acid, resp. Fehling's solution thus reacts only in virtue of its alkali, as follows: 2BzH + NaOH → PhCH<sub>2</sub>OH + BzONa. The reaction is therefore similar to the Cannizzaro reaction: o-HOC<sub>6</sub>H<sub>4</sub>CHO and vanillic aldehyde do not react with Fehling's solution, nor do they give the Cannizzaro reaction, probably because of the OH group. This is supported by the fact that PhCH<sub>2</sub>OH, anisic alc. and cinnamic alc. can be prepd from the resp aldehydes by the Cannizzaro reaction, whereas salicylic alc. and vanillic alc. cannot be prepd thus, but only by the action of nascent H on the corresponding aldehydes.

C. C. Davis

Preparation of 1,2 (dimethylaminomethylmethylmethyleneoxy)benzene. Jean Druey. Bull. soc. chim. [5], 2, 2261-4 (1935).—The ω-aminomethylbenzodioxans are physiologically interesting and the prepn of their isomers, the phenylene acetals of N-substituted amino ketones was undertaken. The addition of 22 g of P<sub>2</sub>O<sub>5</sub> to a solution of 11 g of o-(HO)C<sub>6</sub>H<sub>4</sub> in 25 g of MeCOCH<sub>2</sub>Cl, b 119-20°, gave 40-45% of 1,2-(chloromethylmethylmethyleneoxy)benzene (I),  $\text{C}_{12}\text{H}_{13}\text{ClO}_2$ , b 104-5°, d<sub>4</sub><sup>20</sup> 1.2285, n<sub>D</sub><sup>20</sup> 1.5265, M<sub>r</sub> 180.13 (calcd 102); o-N deriv, m 80-1°. By heating with 3 mols of N<sub>2</sub>H<sub>4</sub> at 180° for 43 hrs 9.3 g of I was converted into 1.3 g (13%) of 1,2-(dimethylaminomethylmethylmethyleneoxy)benzene,  $\text{C}_{12}\text{H}_{17}\text{N}_2\text{O}_2$ , b 115-17°; HCl salt, m 217-18° (decomp.). The di-Et and isopropylidene analogs,  $\text{C}_{14}\text{H}_{19}\text{N}_2\text{O}_2$ , b 123-5°, and  $\text{C}_{14}\text{H}_{19}\text{N}_2\text{O}_2$ , b 150-1° (HCl salt, m 224-5° (decomp.)) were similarly prepd. Contrary to the aminomethylbenzodioxans, which have a strong action on the sympathetic nervous system, these new compounds are absolutely inactive in this respect.

C. R. A.

Sulfarsphenamine. A new method of preparation. W. J. C. Dyke and Harold King. J. Chem. Soc. 1935, 1745-7, cf. C. A. 29, 5822<sup>1</sup>.—Arsphenamine base (3.66

g.) with  $\text{HOC}_2\text{H}_4\text{SO}_3\text{Na}$  in  $\text{H}_2\text{O}$  at  $60^\circ$  ( $\text{CO}_2$  atm.) gives 1.64 g. di Na 3,3'-diamino-1,1'-dihydroxyarsenobenzene-*N,N'*-dimethylsulfite (I), yellow, 2.5 g. I in 25 cc.  $\text{H}_2\text{O}$  ( $\text{CO}_2$  atm.), treated with 0.7 cc. 40%  $\text{HClO}$  and then with concd.  $\text{NaHSO}_3$  (3 mols.), gives 2.6 g. sulfarysphenamine, it is not necessary to isolate the I in the prepn. 2,5-HO(O-N) $\text{C}_6\text{H}_4\text{NHCH}_2\text{SO}_3\text{Na}$  with  $\text{HClO}$  and  $\text{NaHSO}_3$  gives 1 nitro-2-hydroxyaniline *N,N'*-dimethylsulfite, isolated as the tri Na salt, this also results by the action of  $\text{HClO}$  and  $\text{NaHSO}_3$  in succession on 4,2-O $\text{N}(\text{H}_2\text{N})\text{C}_6\text{H}_3\text{OH}$  in  $\text{HCl}$  soln. C. J. West

Iodine substitution products of vanillin and some of their derivatives. L. Chas. Raiford and Eugene H. Wells. *J. Am. Chem. Soc.* 57, 2000 3 (1935). 2-Aminovanillin through the diazo reaction gives 54% of 2-iodovanillin (I), m. 155-6°. *Ac. deriv.* m. 70-2°, *oxime*, with 0.5 mol.  $\text{H}_2\text{O}$ , solvents 122° (anhyd.), m. 142-5 3.5°, semicarbazone, in 190-200° p. nitrophenylhydrazone, m. 211-12°, bis-benzidine, with 2 mols.  $\text{C}_6\text{H}_5\text{N}$ , brown, m. 228-30° (decompn.) I and  $\text{Me}_2\text{SO}$ , in  $\text{KOH}$  give 84% of 2-iodo-3,4-dimethoxybenzaldehyde, pale yellow, m. 82°. Reduction of 5-nitrovannillin gives 88% of the  $\text{SnCl}_4$  salt of 5-aminovanillin di  $\text{HCl}$ , orange,  $\text{H}_2\text{S}$  gives 5-aminovanillin  $\text{HCl}$ , yellowish brown,  $\text{AcO}$  gives 165% of 3-methoxy-4-acetoxy-5-acetylanilino-benzaldehyde, m. 174-6°, the corresponding di-Bz deriv. m. 101-2°, the diazo reaction gives 79% of 5-iodovanillin, light brown, m. 179-80°, identical with Carles' product (*Bull. soc. chim.* 17, 14 (1872)), *Ac. deriv.* (II), m. 105-6°, *Bz. deriv.* m. 135-5-6 5°, *acetyl diacetate*, m. 132-3°, *oxime*, m. 178-9°, semicarbazone, yellow, m. 205-5 5°, p. nitrophenylhydrazone, red, m. 242-3° (decompn.), bis-benzidine, brown, m. 242° (decompn.), *Ac. deriv.* m. 69-70°. Nitration of II with fuming  $\text{HNO}_3$  at 6° gives 53% of the *Ac. deriv.* m. 124-5°, of 2-nitro-5-iodovanillin (III), m. 140-7°, *oxime*, yellow, m. 128-9°, semicarbazone, yellow, m. 187-8° (decompn.), p. nitrophenylhydrazone, red, with 0.5 mol.  $\text{H}_2\text{O}$ , in 228-30° (decompn.), bis-benzidine, yellow, does not m. 335°. Reduction of III with  $\text{Fe}(\text{OH})_3$  in  $\text{NH}_4\text{OH}$  gives 69% of 2-amino-5-iodovanillin, light brown, m. 155°; through the diazo reaction or by the action of I on I in  $\text{AcOH}$ - $\text{AcONa}$  there results 2,5-diiodovanillin (IV), m. 200°, *Ac. deriv.* m. 127-8°, *oxime*, m. 174-5-5 5°, semicarbazone, m. 235° (decompn.), p. nitrophenylhydrazone, orange, m. 252-3° (decompn.), bis-benzidine, no m. p. With  $\text{Me}_2\text{SO}$ , IV yields 33% of 2,4-diiodo-3,4-dimethoxybenzaldehyde, m. 91°. 6-Iodovanillin could not be prep'd from acetylvanillin with I or  $\text{ICl}$ ; 3-methoxy-4-acetoxybenzal diacetate also failed to give this product. C. J. West

Reaction of carbonyl chlorides with metallic hydrides Otto Neunhoeffer and I. Friedrich Nerdel. *J. prakt. Chem.* 144, 63-66 (1935).—Repetition of the work of Chinoza (*Compt. rend.* 63, 632 (1852)) shows that  $\text{CuI}$  and  $\text{BiCl}_3$  do not give  $\text{BaI}_2$ , other hydrides and other chlorides gave no evidence of reduction. The formation of esters is due to the presence of alk. in the reaction mixt. C. J. West

Action of aromatic sulfonic and hydroxy sulfonic acids upon  $\alpha$ - and  $\beta$ -amino acids. Guido Machek. *Monatsh.* 66, 315-56 (1935). cf. C. A. 29, 6827. —Thiopyruvic acid (I) and  $\text{PhSO}_3\text{H}$ , heated 21 hrs. in  $\text{C}_6\text{H}_5\text{Me}$ , give 92% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 158-9° (all m. ps. cor.), heating I with  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$  for 19 hrs. in  $\text{C}_6\text{H}_5\text{Me}$ , bath gives unchanged I,  $\text{H}_2\text{SO}_4$  was not formed  $\text{PhMe}$ ,  $\text{H}_2\text{SO}_4$  and I, on heating 20 hrs., give 72% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 200-1°, in  $\text{C}_6\text{H}_5\text{Me}$  gives the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 171-5 2 6°;  $\text{C}_6\text{H}_5\text{I}$ , 25.3% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$  (II), m. 195-6°;  $\beta$ - $\text{C}_6\text{H}_5\text{SO}_3\text{H}$  gives 93.6% of II, yielding with  $\text{NaOH}$   $\beta$ - $\text{C}_6\text{H}_5\text{SO}_3\text{Na}$ . The  $\alpha$ -isomer of II, m. 170-1°. Alanine,  $\text{PhOH}$  and  $\text{H}_2\text{SO}_4$ , heated 24 hrs., give 70% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 170-5-80 5°;  $\text{PhMe}$  gives 54% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 193-5-8°;  $\beta$ - $\text{C}_6\text{H}_5\text{SO}_3\text{H}$  gives 69% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 227°. Leucine,  $\text{PhMe}$  and  $\text{H}_2\text{SO}_4$ , give 71.3% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 153-3 5°;  $\beta$ - $\text{C}_6\text{H}_5\text{SO}_3\text{H}$  gives 65% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 202-3°;  $\text{PhOH}$ ,  $\text{H}_2\text{SO}_4$  and  $\text{MeCHNH}_2\text{CH}_2\text{CO}_2\text{H}$  give the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 182-3°,  $\beta$ - $\text{C}_6\text{H}_5\text{SO}_3\text{H}$  gives 55% of the comp'd  $\text{C}_6\text{H}_5\text{O}_2\text{NS}$ , m. 157-8°,  $\text{MeC}_6\text{H}_4\text{SO}_3\text{H}$  does not react. These comp'ds do not give a color reaction with  $\text{FeCl}_3$ . C. J. West

Fictitious dihydrophenylbenzamidines. A. V. Kirsanov and Ya. N. Ivashchenko. *Bull. soc. chim.* [5], 2, 1944-50 (1935).—Treatment of  $\text{PhCH NPh}$  (I) with  $\text{NaNH}_2$  gave  $\text{PhC}(\text{NH})\text{NHPh}$  (II) and  $\text{Ph}_2\text{CH NPh}$  (III) (cf. following abstr.) III is obviously formed by the reduction of I but the reduction of II to  $\text{PhCH}(\text{NH})\text{NHPh}$  (IV) is less probable although Bernthsen (*Ber.* 13, 917 (1880)) described IV as the product formed by the reduction of II in the presence of  $\text{NaNH}_2$ . The prep'n was repeated under various conditions and a careful check was made of the starting material and the products of the reaction. The reduction of 25 g. of II (m. 114-15°) in 250 cc. of alc. with 194.7 g. of 3% Na  $\text{H}_2\text{g}$  gave sufficient amts of BzH,  $\text{PhNH}_2$ , III, and  $\text{Ph}_2\text{CH NPh}$ , together with unchanged II, to account for 90-95% of the starting material. It is concluded that the material obtained by Bernthsen was unchanged II. When recrystd from alc., II m. 111-12°, rising to 114-15° on recrystn. from petroleum ether. The yield of II from I and  $\text{NaNH}_2$  can be increased to 27% by slowly adding I to a large excess of powd.  $\text{NaNH}_2$  in boiling toluene. I is immediately reduced and the side reactions are appreciably diminished. C. R. Addinall

Mechanism of amination with sodamide. New procedure for the preparation of substituted amides. A. V. Kirsanov and Ya. N. Ivashchenko. *Bull. soc. chim.* [5], 2, 2109 21 (1935).— $\text{NaNH}_2$  (I) acts on comp'ds contg. a pyridine nucleus by adding to the C-N linkage, the Na and  $\text{NH}_2$  components attaching themselves to the N and C atoms, resp. The product loses  $\text{NaH}$  (II) and is transformed into the corresponding amine which reacts with II or with I to give a Na amine deriv. and  $\text{H}_2$  or  $\text{NH}_3$ . This process is confirmed in a series of analogous reactions and examples. To prove that in the reaction between the pyridine ring and I only the N and the  $\alpha$ -C atoms are involved to the exclusion of the other members of the ring, the reaction of I with 3 Schiff bases,  $\text{PhCH NPh}$  (III),  $\text{PhCH NCH}_2\text{Me}$  (IV) and  $\text{CH}_3\text{O C}_6\text{H}_4\text{CH NPh}$  (V) was studied. The mixt. formed by the addn. of 24 g. (0.6 mol.) of carefully powd. I to a soln. of 48.3 g. of III in 50 cc. of dry toluene was heated under reflux with continuous stirring at 120°.  $\text{NH}_3$  was evolved and the cooled, brick-red solid reaction mass was taken up in 300 cc.  $\text{H}_2\text{O}$  and 250 cc.  $\text{Et}_2\text{O}$ . The  $\text{Et}_2\text{O}$  layer was extd. with  $\text{AcOH}$ . This ext. was decomp'd with  $\text{NH}_4\text{OH}$ , extd. with  $\text{Et}_2\text{O}$  and the ext. was dried and evapd. The bruc products were vacuum-distd and fractionated. The  $\text{Et}_2\text{O}$  ext. of neutral products was dried and evapd. and the oily residue was distd. *in vacuo*. Working up of the crude fractions gave 5.8 g. (12%) of  $\text{PhNH}_2$ , 9.93 g. (19.6%) of  $\text{PhC}(\text{NH}_2)\text{NPh}$  (VI), 7.2 g. of  $\text{PhCH}_2\text{NHPh}$  (VII) and 6.2 g. (12.8%) of lophine (IX), together with a small amt. of colorless fine needles, m. 207-8°. These results show that the action of I on a toluene soln. of III takes place in a manner exactly analogous to that of I on pyridine and similar to that of the action of organometallic comp'ds on Schiff bases. The intermediate  $\text{PhCH}(\text{NH}_2)\text{NNAh}$  is formed, which loses  $\text{NaH}$  to produce VI. The action of  $\text{NaH}$  on VI gives  $\text{H}_2$ , which reduces III to VII. By the action of VI on excess of I,  $\text{NH}_3$  is liberated which ammonolyzes III to give  $\text{PhNH}_2$  and amine which is oxidized to IX at the expense of the reduction of VII (cf. Strain, C. A. 22, 3147). No  $\text{PhC}(\text{NPh})\text{NHCH}_2\text{Ph}$  was found in the above reaction which, evidently, takes another direction to that carried out at the temp. of liquid  $\text{NH}_3$ . Similar anomalies have been noted in the reaction between I and  $\alpha$ -picoline under the action of heat and in liquid  $\text{NH}_3$ . VI was identified by comparison with a sample prep'd from the  $\text{HCl}$  salt of III and  $\text{NH}_3$  (cf. Pechmann, *Ber.* 30, 1786 (1897)). Reduction of III with alc. Na  $\text{H}_2\text{g}$  gave a test sample of VII, m. 35-6°. *Ac. deriv.* m. 56-7°, nitroso deriv. m. 56°. IX,  $\text{C}_6\text{H}_5\text{NH}_2$ , m. 273.5-4 7°, yielded a  $\text{HCl}$  salt, m. 155-60°. The amination of 9.7 g. of IV produced 2.2 g. (22.6%) of  $\beta$ - $\text{MeC}_6\text{H}_4\text{NH}_2$ , 2.2 g. (22.6%) of  $\text{MeC}_6\text{H}_4\text{C}(\text{NH}_2)\text{NPh}$ .



m 90°, 2.0 g (20.6%) of  $\text{PhCH}_2\text{NHC}_6\text{H}_4\text{Me}$ , m 19–20° (nitroso deriv., m 45.5–8.5°), and 1.2 g (12.3%) of IX. V was obtained by the reciprocal action of equimol quantities of  $\text{PhNH}_2$  and  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CHO}$  in alc. The crude product, m 60°, was dissolved in 40 cc. of toluene and, on treatment with 4 g. of finely powdered I, gave 13% of the amine,  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}(\text{NH}_2)\text{NPh}$ , m 138–9°, and a minute quantity of  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}_2\text{NHPH}$ , m 79–80°, identified by a mixed m.p. detn. with a sample obtained by the reduction of  $\text{CH}_2\text{O}$ ,  $\text{C}_6\text{H}_5\text{CH}$  NPh by alc. Na-Hg. Thus the amination of the C N group with I is a general reaction, of which the reaction of I with compounds containing a pyridine ring is a particular case. It is highly possible that the reaction can be broadly generalized and that, under special conditions, I reacts analogously to organometallic compounds, the  $\text{NH}_2$  group filling the role of the alkyl radical. Thus in the formation of  $\text{BaNH}_2$  and  $\text{PhCH}_2\text{OH}$  in the reaction between I and  $\text{BaH}$  the first step consists in the addition of Na and  $\text{NH}_2$  to the C O linkage, a process similar to the addition to the C N group in the amination of the anils and pyridine bases. C. R. Addinall

Preparation of  $\beta$ -n-dichloro-p-sulfonylbenzoic acid. I. G. Zil'berg *Khim. Farm. Prom.* 1935, No. 2, 114–17. The by-product of saccharin manufacture is used as a starting compound.  $\text{p-MeC}_6\text{H}_4\text{SO}_2\text{Cl}$  is treated with 25%  $\text{NH}_4\text{OH}$  (100% excess of the theory), the amide oxidized with  $\text{Na}_2\text{Cr}_2\text{O}_7$  in 75%  $\text{H}_2\text{SO}_4$  and recrystallized from alc. The available Cl = 25.7%. L. Nasarevich

Dehalogenation of  $\beta$ -bromo acids. IV.  $\beta$ -Bromophenylpyruvic acid. Ben Sobin and G. Bryant Bachman. *J. Am. Chem. Soc.* 57, 2158–60 (1935), cf. C. A. 29, 2813.  $\beta$ -Bromophenylpyruvic acid (I), yellow, m 103–4°, boiling with  $\text{H}_2\text{O}$  gives 77% of  $\text{PhCH}_2\text{CO}_2\text{H}$ , shaking I in  $\text{H}_2\text{O}$  with  $\text{Ag}_2\text{O}$  gives 94% of the same acid, in alc. 1.01  $\text{H}_2\text{O}$  gives 36% of  $\text{PhCH}_2\text{CO}_2\text{H}$ , cf.  $\text{Na}_2\text{CO}_3$  gives, after refluxing 0.5 hr.,  $\text{BzCH}_2\text{OH}$  and  $\text{BaOH}$ , cf.  $\text{NaHCO}_3$  and I, allowed to stand overnight, give about 40% of  $\text{PhCH}_2\text{CO}_2\text{H}$  and  $\text{BzCH}_2\text{OH}$ .  $\text{C}_6\text{H}_5\text{N}$ ,  $\text{PhNH}_2$ ,  $\text{PhNMe}_2$  and  $\text{C}_6\text{H}_5\text{NH}_2$  all cause evolution of  $\text{CO}_2$  but give tarry and gummy masses, with  $\text{NH}_4\text{OH}$  a little  $\text{BzCH}_2\text{OH}$  was isolated. In the formation of the  $\text{PhCH}_2\text{CO}_2\text{H}$  it is assumed that  $\text{PhCH}_2\text{C}=\text{O}$  is an intermediate product. The possibilities and some of the difficulties of utilizing the dehalogenation of  $\alpha$  keto  $\beta$ -bromo acids as a new method of synthesizing ketene derivatives are pointed out.

C. J. West  
Preparation of isovalerylaldehyde. Jean Milhiet *Bull. soc. chim.* [5], 2, 2134–5 (1935).—According to Linhorn and Sufferit, isovalerylaldehyde (I), m 45°, can be prepared from salicylic acid (II) and  $\text{Me}_3\text{CHCH}_2\text{COCl}$  in the presence of pyridine. Daliotis (C. A. 22, 2808) reported the prepn. of I, m 235°, from II and  $(\text{Me}_3\text{CHCH}_2\text{CO})_2\text{O}$  after failing to repeat the prepn. according to I and S in the presence of alc.  $\text{NaOH}$ . M. has checked the findings of I and S. C. R. Addinall

Apparent cases of liquid-crystal formation in  $\beta$ -alkoxybenzoic acids. Brynnon Jones *J. Chem. Soc.* 1935, 1874, cf. Bradfield and J. C. A. 24, 1776.—The acids were prepared from  $\text{p-HOC}_6\text{H}_4\text{CO}_2\text{H}$  and the alkyl iodide with aq. KOH.  $\beta$ -Amyloxybenzoic acid, t.p. (transition pt.) 122°, m 148°, hexyl deriv., t.p. 105°, m 150°, heptyl deriv., t.p. 91°, m 145°, octyl deriv., t.p. 100°, m 145°, cetyl deriv., t.p. 99°, m 131°. The t.p. is the temp. at which the solid changes into a cloudy liquid. The m-series does not exhibit this phenomenon.

C. J. West  
The condensation of aldehydes with malonic acid in the presence of organic bases. IV. The condensation of piperonal. Toqur Ahmad Vahidy and K. C. Pandya *Proc. Indian Acad. Sci.* 2A, 402–5 (1935), cf. C. A. 29, 5830.—The condensation of piperonal with malonic acid in the presence of small traces of pyridine, lutidine, quinoline, isouquinoline, quinaldine,  $\text{PhNMe}_2$ ,  $\text{PhNH}_2$ , piperidine,  $\text{PhNHMe}$ ,  $\text{PhNH}_2$ ,  $\text{NH}_4\text{OH}$ , aniline,  $\alpha$ ,  $\beta$ - and  $\gamma$ -toluidine,  $\alpha$  and  $\beta$ - $\text{C}_6\text{H}_5\text{NH}_2$ ,  $\text{NH}_4\text{OH}$  and  $\text{NH}_4\text{Cl}$  malonate was studied. The yields varied from 40% to theoretical. The time for completion of reaction was about 4 hrs., compared to 18 hrs. when the piperonal and malonic acid

were heated in the absence of the organic bases. The product in all cases was piperonylacrylic acid. These reactions may throw light on the specific catalytic influence of each of the bases used. L. L. Quill

The synthesis of 6-methylcoumarin. A. M. Buligin, *Moskovskoe Zhivoe Delo* 1934, No. 4, 43–4.— $\text{MeC}_6\text{H}_4\text{O}-\text{COCH}_2\text{CH}_3$  was prepared on a semi-commercial scale by condensation

of  $\text{p-secresol}$  (500 g.) and fumaric acid (650 g.) and 72%  $\text{H}_2\text{SO}_4$  (2.5 g.) Yield of pure I, m 73–4°, 40%. L. Bielous

Hydrogenation of carbonyl compounds by nickel and platinumized nickel. Influence of an alkali. Marcel Lépine and Alain Horcau. *Compt. rend.* 201, 1301–5 (1935).—The hydrogenation of pinonic acid (I) in the presence of Ni obtained by decomposition of 73% Raney Ni-Al alloy takes place readily on the addition of an excess of  $\text{NaOH}$ . I is isomerized in the presence of  $\text{NaOH}$ , and reduction of various ketones at ordinary temps. and pressures was carried out to test the theory that the enolic form was more readily reduced. Ketones such as  $\text{Me}_2\text{CO}$ ,  $\text{MeCOEt}$ ,  $\text{MeCOCH}_2\text{Me}$ ,  $\text{MeCOPh}$ , 2,4-dimethyl-2-phenylhexanone and cyclohexanone, which are reduced slowly in the presence of Ni and alc. media, are hydrogenated much more rapidly after the addition of alkali. The theory of a preliminary enolization was abandoned when it was found that  $\text{PhCO}$  and  $\text{PhCOBz}$  gave results of the same order of increased speed of reduction. Similar results were obtained with ketones of complex function such as  $\text{AcCH}_2\text{CO}_2\text{Et}$ ,  $\text{LiCH}_2\text{COCH}_2\text{CO}_2\text{Et}$  and  $\text{BzCH}_2\text{CO}_2\text{H}$ . With methyl oxide, methylheptanone and benzylidenacetone, the 1st mol. of  $\text{H}_2$  fixed itself rapidly but the 2nd was only absorbed on the addition of alkali to the reaction mixture. Crotonic, benzoic, salicylic and  $\beta$ -hydroxybenzoic aldehydes and such compounds as galactose, levulose and 1-dimethylamino-4-pentanone behaved analogously. The necessity of the presence of alkali for increasing the speed of the reaction was demonstrated by the action of  $\text{AcOH}$  in slowing up the rate of reduction. Various references to the favorable action of alkalis in hydrogenation are quoted. The activity of the Ni catalyst is enhanced by plating with Pt which can be simply carried out by shaking an aq. suspension of Ni with a dil. alc. platinumchloride soln. Similar but less effective reinforcement of the catalytic effect can be obtained by plating Ni with Pd, Rh and Ru. Thus the hydrogenation of carbonyl compounds at ordinary temps. and pressures in the presence of Raney Ni catalysts is facilitated by the addition of alkali and even by treatment with Pt. C. R. Addinall

Some urea derivatives in the terpene series. Robert L. Bateman and Allan R. Day *J. Am. Chem. Soc.* 57, 2196–8 (1935).—1-Methylamine-HCl and nitrourea with  $\text{NaHCO}_3$  in  $\text{H}_2\text{O}$  give 78% menthylurea, m 140–2–40.6° (all m. ps. cor.),  $[\alpha]_D^{25}$  –50° (95%  $\text{C}_6\text{H}_5\text{OH}$ ), sym-Ac deriv., m 118–19°,  $[\alpha]_D^{25}$  –87.3°, chloral addn. product, m 146.2–72°,  $[\alpha]_D^{25}$  –49.1°, sym-bromoacetyl deriv., m 111.8–2.2°,  $[\alpha]_D^{25}$  –66.9°, sym-cinnamoyl deriv., m 144.3–5.1°,  $[\alpha]_D^{25}$  –67.9°, sym-p-nitrobenzoyl deriv., m 158.7–0.2°, sym-p-aminobenzoyl deriv., m 205–10° (decompn.),  $[\alpha]_D^{25}$  –72.3°. d-Bornylurea results in 90% yield by the above method, m 165.7–6.3°,  $[\alpha]_D^{25}$  5.83°, sym-Ac deriv., m 129.9–5°,  $[\alpha]_D^{25}$  23.5°, chloral addn. product, m 180° (decompn.),  $[\alpha]_D^{25}$  7.3°, sym-bromoacetyl deriv., m 136.1–6.5°,  $[\alpha]_D^{25}$  16.3°, sym-cinnamoyl deriv., m 220.2–20.8°, sym-p-nitrobenzoyl deriv., m 230° (decompn.), sym-p-aminobenzoyl deriv., m 233° (decompn.),  $[\alpha]_D^{25}$  15.6°. The amine from isonitrosocamphor yields 2-keto-3-carbamidocamphane, with 0.5 mol.  $\text{H}_2\text{O}$ , m 177.8–8.4°,  $[\alpha]_D^{25}$  18.3°, p-nitrobenzoyl deriv. Preliminary tests on the narcotic effects indicated that menthylurea was the most promising and may have some value, it acted rapidly without evident after-effects. C. J. West

Pinenemagnesium chloride. Action of phthalic anhydride. René Bouisset *Bull. soc. chim.* [5], 2, 2183–7 (1935).—The action of diacid anhydrides on aliphatic and aromatic Mg compounds produces ketonic acids, disubstituted lactones or, more rarely, diuretic glycols. Thus

action has not been previously carried out with terpene  $Mg$  compounds and it has been demonstrated that pinene-magnesium chloride (I) gives almost exclusively a ketonic acid with phthalic anhydride (II). The reaction of 103.5 g. (0.6 mole) of ordinary pinene-HCl with  $Mg$  in 250 cc. of anhydrous  $Et_2O$  gave 0.5 l. of I, m. 125–6°,  $[a]_D^{25} = -33.5^\circ$ ,  $[a]_D^{25} = -39.0^\circ$ ,  $[a]_D^{25} = -64.55^\circ$ . A mixt. of 74 g. (0.5 mole) of freshly distd. II, b.p. 284° (cor.), and 750 cc. of anhyd.  $Et_2O$  was boiled for a few min. and cooled to 0° in a  $CO_2$  atm. I was added dropwise at 0° and the mixt. was occasionally stirred for a period of 20 hrs. Hydrolysis was carried out with air-free  $H_2O$  at  $-20^\circ$  and the product was decanted and filtered. The solid residue was dissolved in glacial  $AcOH$  and on diln. with ice-dil.  $H_2O$  gave 115 g. (80%) of *o*-(camphene-2-carboxyl)benzoic acid (III),  $C_{17}H_{22}O_4$ , m. 154°,  $[a]_D^{25} = 58.92^\circ$ ,  $[a]_D^{25} = 69.45^\circ$ ,  $[a]_D^{25} = 119.2^\circ$ . The total yield of III was increased to 95% by the recovery of 21 g. from the  $Et_2O$  layer which also contained traces of another acid, (IV), m. 137°, together with a little *d*-bornylene. That the formation of bornyl phthalates by the action of I on  $CaH_2/OMgCl$  formed by the exposure of I to the air was avoided in the above procedure was shown by the complete absence of bornol on refluxing 10 g. of the solid residue with 150 cc. of alc. 0.97  $N KOH$  for 5 hrs. at 125°. The position of the  $CO$  group between the benzene and camphene nuclei hinders the formation of a semicarbazone. The ease with which III can be prepd. makes the synthesis of many optically active compounds possible. It is unlikely that the action of an excess of I on II would give dibornylphthalate by the reaction of I with the intermediate Grignard reagent  $CaH_2CO_2MgCl$ , but with a different reagent  $RMgX$  (where  $R$  varies from  $Me$  to  $C_6H_5$ ) the new mol. might be fixed with the production of a disubstituted phthalide which in turn might split off the terpene nucleus as bornylene, with the formation of interesting optically active compds. C. R. Addinall

**Synthesis of "ketonopinone" (4,6-diketopinane).** P. C. Gupta and K. Gnaniapathi. *Current Sci.* 4, 312–13 (1935); cf. C. A. 29, 5418. The conversion of the *Me* ester (I) of pinonic acid (II) into ketonopinone (III) has been accomplished by means of  $Na$  in  $PhMe$  or  $MeONa$  in alc., III, m. 104°, purified through its  $Cu$  derivative, gives a violet color with  $FeCl_3$ , dissolves in  $NaHCO_3$ , decolorizes alk.  $KMnO_4$ , and absorbs  $Br_2$  in  $CHCl_3$ . The constitution has been confirmed by hydrolysis with  $Na(OH)$ , to II. W. J. Peterson

**Plurality of primary bicyclic terpene alcohols.** René Bousset. *Bull. soc. chim.* [5], 2, 2187 (1935).—Contrary to the statement of Dufont and Zacharewicz (C. A. 29, 4757) myrtanol is not the only primary bicyclic terpene alc., since 2 other members of this series, 2-methanol- (C. A. 25, 507) and 2-ethanolcamphane (C. A. 29, 1798), have been previously prepd. C. R. Addinall

The physical identity of enantiomers I. Rotatory dispersion of l-bornol, enantiomeric camphors, camphoric acids, sodium camphorates, camphoric anhydrides and camphorimides. Bawa Kartar Singh and Indramani Mahanti. *Proc. Indian Acad. Sci.* 2A, 378–98 (1935), cf. C. A. 29, 3316. The rotatory dispersions of *d*- and *l*-camphoric acids in acetone,  $EtOH$  and water, *d*- and *l*-camphor in  $EtOH$ ,  $CHCl_3$  and water, *d*- and *l*-camphorates in water, *d*- and *l*-camphoric anhydride in  $EtOH$  and  $CHCl_3$ , *d*- and *l*-camphorimide in  $EtOH$ ,  $CHCl_3$  and  $CHCl_3$ , and l-bornol in  $EtOH$  and  $CHCl_3$  were detd. for 10 wave lengths ( $\lambda$  4358 to 7600 Å). Pasteur's law of mol. dissymmetry was shown to hold in all cases. The rotatory power of *d*- and *l*-camphoric acids in different concns. of  $EtOH$  first decreases with increase of concn., and then becomes almost const., these changes indicate the identity of the magnitude of the rotatory power of the 2 opposite and active forms. The effects of cyclic structure, and of the substitution of the ketomethylene group of camphor are discussed. The methods of prepn. of the compds. studied and 20 tables of data are given. L. J. Quill

The relation of production of synthetic camphor to consumption in the United States. Robert L. O'Brien,

Thomas W. Page, Edgar H. Broward and Oscar B. Ryder. *U. S. Tariff Comm. Rept. No.* 104, 5 pp. (1935). L. H.

**Camphor series.** I. Dines Chandra Sen. *J. Indian Chem. Soc.* 12, 647–52 (1935).—A general method of synthesis of cyclic thio ketones is described by means of which this camphor (I) has been produced in good yields. I is probably formed through an unstable chlorohydrin which on conversion into chlorothiohydrin breaks down to the thio ketone which is analogous in its behavior with ketonic reagents to ketones. A soln. of 30 g. of camphor in 150 cc. abs. alc. at 0° was said. with  $HCl$  and  $H_2S$  for 6–8 hrs. The red, unstable ppt. produced by adding 25 cc. of cold  $H_2O$  was filtered, washed and taken up in benzene. The dried ext. was evapd. and yielded 15 g. of racemic I,  $C_{15}H_{18}S$ , m. 145°, phenylhydrazones, b.p. 190°,  $n_D^{20} = 1.49$  (decompn.), oxime, m. 118–19°, semicarbazone, m. 217°, all are identical in m. p. with the corresponding camphor derivs. The same material was obtained from *l*-camphor but the *d*-isomer gave *l*-thiocamphor, m. 146°,  $[a]_D^{25} = -12^\circ$ . The reduction of 5 g. of I in 25 cc. cold alc. with  $Zn$  and 25 cc. of alc.  $HCl$  and extn. with  $Et_2O$  gave a crude product which was pptd. as an insol. Pb salt from alc. Treatment with  $H_2S$  in alc. and extn. with  $Et_2O$  gave thiobornol (II),  $C_{15}H_{18}S$ , m. 120° (cf. Wuyts, Ber. 36, 803 (1903) for m. p. of 61–2°), Pb salt, minute yellow needles sol. in  $Et_2O$ . Treatment with I converted II into disbornyl disulfide,  $C_{30}H_{38}S_2$ , m. 199°. C. R. Addinall

Anomalous mutarotation of salts of Reichler's acid. IV. Comparison of 2-(*N*-phenylketimine)-4-camphene-10-sulfonic acid with *d*-camphor-10-sulfonamide. Harry Sutherland and R. L. Shriner. *J. Am. Chem. Soc.* 58, 62–3 (1936), cf. C. A. 29, 7764. *d*-Camphor-10-sulfonamide, m. 120.5–1°,  $[a]_D^{25} = 76^\circ$  ( $CHCl_3$ ), 29.5° (95%  $EtOH$ ), differs markedly from the dehydration product of the  $PhNH_2$  salt of Reichler's acid. This excludes I from consideration as the dehydration product and constitutes further proof that the ketimine structure is correct. I shows no mutarotation, it did not undergo hydrolysis on standing in aq.  $EtOH$ , refluxing with 25%  $HCl$  for 36 hrs. gives 77%  $PhNH_2$  and the acid. C. J. West

Ozonation of certain hydrogenated biphenyls. C. R. Noller and G. K. Kaneko. *J. Am. Chem. Soc.* 57, 2442–3 (1935).—1-Phenyl-1-cyclohexene (I) adds 1 mole of  $O_3$  to the double bond in the cyclohexene ring and 1,1'-cyclohexenyl adds  $O_3$  to both double bonds. This indicates that steric hindrance in the 1- and 1'-positions is not the factor involved in the failure of  $Ph_2$  to form a hexaozone. Tetrahydrobiphenyl prepd. by reducing  $Ph_2$  with  $Na$  and  $AmOH$  is chiefly I. C. J. West

**Syntheses in the biphenyl series.** David D. Hartley and Robert E. Lyons. *Proc. Indust. Acad. Sci.* 44, 121–8 (1934).—*o*-Nitrophenyl has been electrolytically reduced to *o*-azoxyphenyl, m. 155°, at a Ni cathode and to *o*-hydroxyphenyl (I), m. 183°, at a Pb cathode. Yields, 61.8 and 81% resp. *p*-Nitrophenyl has been electrolytically reduced to *p*-azoxyphenyl (II), m. 213°, at a Ni cathode. Yield 90.6%. The approx. soly. of *o*-azoxy-, *o*-hydroxy- and *p*-azoxyphenyl in g. per 100 cc. of soln. at 20° are as follows: 95% alc., 0.11, 0.03, 0.01; acetone, 2.65, 0.74, 0.15. I undergoes rearrangement to *o*,*o'*-diphenylbenzidine (II) in  $HCl$  (d. 1.18) when maintained at 55° for 24 hrs. in a sealed tube. Diazotization of II and coupling of the resulting tetrazonum salt with some common intermediates in the usual manner yielded the following 4,4'-diphenyl-4,4'-disubstituted dyes (as Na salts): bis-*o*-naphthylamine-4-sulfonic acid, bis-8-amino-1-naphthol-3,5-disulfonic acid, bis-1,8-dihydroxynaphthalene-3,5-disulfonic acid, bis-8-naphthylamine-6-sulfonic acid, bis-2-amino-5-naphthol-7-sulfonic acid, bis-2-naphthol-3,6-disulfonic acid, bis-2-amino-8-naphthol-6-sulfonic acid, bis-6-naphthol. These dyes are less sol than the corresponding *o*-toluidine dyes, have a slightly greater color intensity in equal molar concns., and give similar though not identical colors. W. J. Peterson

Complex formation between polynitro compounds and aromatic hydrocarbons. II. The system 2,4-dinitro-2'-methylbiphenyl 6-carboxylic acid benzene. Daniel J. Hammett and R. Branstetter Williams. *J. Chem. Soc.*

1935, 1856-60, cf. C. A. 29, 5337<sup>1</sup>-2,4-Dinitro-2'-methylbiphenyl-6-carboxylic acid (I) (cf. Levide and Turner, C. A. 24, 529<sup>2</sup>) forms 2 solid complexes with  $C_6H_6$ , in the mol ratios 1 and 2 and 1 and 128<sup>3</sup> and 138<sup>3</sup>, resp (decd from solid liquid eq), crystn of I from  $C_6H_6$  gives the 2:1 complex, m 138-40° (sealed tube) and 154-6° in an ordinary m p tube (i.e., m p of I). Resolution of I with d-MePhCHNH<sub>2</sub> gives a d-l with [α]<sub>D</sub><sup>20</sup> (+0.5), db dA, pale yellow, m 238°, [α]<sub>D</sub><sup>20</sup> 57.1° (Me<sub>2</sub>CO, c 1.12), the most completely resolved d l has the same rotation in EtOH as it has when measured in the same solvent in the form of its  $C_6H_6$  complex. Removal of the solvent and the  $C_6H_6$  yields the I unchanged in rotation. Either the complex does not exist in appreciable amt. in soln or else the rotatory power of I is the same per g. per cc whether it is attached to  $C_6H_6$  or not. Partially resolved I can be further resolved by crystn from  $C_6H_6$ . I also crystallizes with 1 mol H<sub>2</sub>O and 1 mol MeOH, the activity of the I is unchanged by complex formation.

C. J. West

Interaction of amines and ammonia with diphenyleneethylene L. A. Pinck and Guido E. Hilbert J. Am. Chem. Soc. 57, 2398-2402 (1935) —(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>C(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub> (I) (1 g) and 15 cc liquid NH<sub>3</sub>, heated 3 weeks at 65°, give 0.5 g of a compd, m 300° (all m ps cor), insol in Et<sub>2</sub>O, while the Et<sub>2</sub>O-sol portion yields 0.4 g of 9-amino-fluorene (II) as the HCl salt and 0.07 g of fluorene. I and EtNH<sub>2</sub>, heated 2 days at 65°, give 65% of 1-ethylaminodiphenyleneethylene (III), m 165°, HCl salt, m 210-15° (decompn), p-terate, yellow, m 228° (decompn). NO deriv, pale yellow, m 217°, does not give the Liebermann nitrosamine test. Heating III in abs EtOH at 100° for 10 days gives a mixt. of fluorene, fluorenone and 9-ethylaminofluorene (IV), in EtNH<sub>2</sub>, there result fluorene and diphenyleneethylene, which are also isolated as by-products in the prepn of II. IV, from 1.3 g of II and EtNH<sub>2</sub> at 55° for 18 hrs, gives 0.57 g of the p-terate of IV, orange, m 206° (decompn). 1-Methylamino deriv, m 151°, Ac deriv, m 232°, NO deriv, m 203°. 1-Dimethylamino deriv, m 215° (decompn). I and PhCH<sub>2</sub>NH<sub>2</sub> react after standing 6 months in a sealed tube, giving the 1-benzyl amino deriv (V), m 168°, HCl salt, m 215-18°, NO deriv, m 217-18°. V also results on heating the reactants at 100° for 6 days, together with fluorene, a hydrocarbon, m 318° (decompn), a base, m 90°, and (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>CHCH<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>). V is decompd on heating in EtOH at 100° for 10 days.

C. J. West

Formation of a single diastereoisomer in the reaction of organomagnesium derivatives with amino ketones M. Tiffeneau, Jeanne Lévy and E. Ditz Bull. soc. chim. [5], 2, 1949-55 (1935) —Experience has shown that the action of organomagnesium compds on ketones with an asym. C atom in the α-position, whether substituted or not by a OH group, leads to 1 only of the 2 possible diastereoisomers and that the other form can be obtained by inverting the order of substitution of the radicals in the tertiary alc produced. These results can be explained by postulating a controlling action on the rupture of the bonds of the CO grouping. These facts are generalized by the following study of the action of an organomagnesium compd on a ketone deriv in which the asym. C atom has an NH<sub>2</sub> group as substituent. Aryl amino ketones of the type ArCOCH(NH<sub>2</sub>)Me have been treated with various ArMgX compds in such a way that 3 pairs of diastereoisomeric amino alcs have been prepd in a series of reactions from each of which only 1 diastereoisomer resulted. The ketones ArCOCH(NH<sub>2</sub>)Me were prepd by the method of Iredell and Crafts and transformed into the isomeric derivs which, in turn, were reduced to the HCl salts of ArCOCH(NH<sub>2</sub>)Me. Pairs of diastereoisomers were then produced by reacting ArMgBr with Ar'COCH(NH<sub>2</sub>)Me and Ar'MgBr with ArCOCH(NH<sub>2</sub>)Me. A mixt. of 131 g of anisylpropanone in 550 cc Et<sub>2</sub>O was satd with a current of HCl and treated with 100 g of freshly distd Am<sub>2</sub>NO<sub>2</sub>. At the end of the reaction the product was refluxed, cooled and extrd with dil Na<sub>2</sub>CO<sub>3</sub>. Acidification of the alk extrd and recrystn of the ppt from benzene gave 80% of MeOC<sub>6</sub>H<sub>4</sub>COC( NOH)Me, m 125°. The propanones, 1-

COC( NOH)Me and MeC<sub>6</sub>H<sub>4</sub>COC( NOH)Me were similarly prepd according to this procedure of Hartung and Munch (C. A. 23, 3113). These isomeric derivs were reduced by SnCl<sub>2</sub> in concd HCl (Ber. 30, 1515 (1897)) to the corresponding HCl salts of 1-aryl-2-aminopropanones, Ph (I), m 185-9°, p-tolyl (II), m 217°, anisyl (III), m 242°. The bases are unknown and attempts to prep. them by neutralization with NaOH or NH<sub>4</sub>OH cyclized them to dihydromethylidylpyrazines which, on oxidation, were transformed into the corresponding 2,5-dimethyl-3,6-diarylpyrazines, m 125°, 168° and 196-7°, resp. The reaction of I, II and III with PhMgBr (IV), p-MeC<sub>6</sub>H<sub>4</sub>MgBr (V) and MeOC<sub>6</sub>H<sub>4</sub>MgBr (VI) presented no peculiarities and after decompn with dil H<sub>2</sub>SO<sub>4</sub>, extrd with 1 H<sub>2</sub>O and treatment with NH<sub>4</sub>OH in the presence of NH<sub>4</sub>Cl, gave, after the usual procedure, HCl salts of amino alcs from which picrates with characteristic m. ps. were prepd. I and V gave a phenyl-p-tolylaminopropanol, m 65° (HCl salt, m 249°, p-terate, m 209°), whereas II and IV yielded the β-diastereoisomer, m 95° (HCl salt, m 275°, p-terate, m 132°). Mixed m p deins gave 56°, 223° and 126°, resp, thus demonstrating the different nature of the 2 isomers. Similarly were prepd the α- and β forms of phenylaminoaminopropanol, m 77° and 74° (62°) (HCl salts, m 265° and 255° (240°)); p-terates, m 198° and 205° (188°), and of p-tolylaminoaminopropanol, m 90° and 84° (92°) (HCl salts, m 280° and 242° (234°)), p-terates, m 217° and 197° (195°). Thus the reaction of the reagent Ar'MgBr with an amino ketone ArCOCH(NH<sub>2</sub>)Me gives a unique amino alc, ArAr'C(OH)CH(NH<sub>2</sub>)Me, which is diastereoisomeric with that obtained by inversion of the radicals introduced. If the amino ketone is of the type ArCOCH<sub>2</sub>NH<sub>2</sub> and has no asym. C atom, the amino alc produced is the same whatever the order of introduction of the groups. Thus PhCOCH<sub>2</sub>NH<sub>2</sub> and MeOC<sub>6</sub>H<sub>4</sub>MgBr give the same phenylaminoaminopropanol, m 100°, as MeOC<sub>6</sub>H<sub>4</sub>COCH<sub>2</sub>NH<sub>2</sub> (HCl salt, m 195-6°), and PhMgBr. Thus, the inductive role of the asym. C atom is perfectly demonstrated. C. R. A.

Simultaneous formation of two diastereoisomeric alcohols in the action of organomagnesium derivatives on active camphenyl ketones M. Tiffeneau, Jeanne Lévy and E. Ditz Bull. soc. chim. [5], 2, 1855-66 (1935); cf. preceding abstr.—A study of the action of organomagnesium derivs on ketones with an asym. C atom in the β-position has shown that the formation of 1 diastereoisomer is not exclusive but that both forms are obtained in unequal proportions. The yields are reversed when the order of introduction of the 2 radicals neighboring on the OH group is inverted. The decompn. of the reaction product from camphenyl nitrile, [α]<sub>D</sub><sup>20</sup> 12.3°, and an excess of EtMgBr in anhyd Et<sub>2</sub>O gave 30% of Et camphenyl ketone (I), b<sub>p</sub> 107°, d<sub>4</sub><sup>20</sup> 0.9201, n<sub>D</sub><sup>20</sup> 1.468, surface tension at 19° 32.6 dynes per sq cm, [α]<sub>D</sub><sup>20</sup> 21.9°, [α]<sub>D</sub><sup>25</sup> 24.5°. Attempts to purify this product by distn caused racemization and, accordingly, another prepn. was made by the action of LiMgBr on camphenyl amide, [α]<sub>D</sub><sup>20</sup> -5°, which produced 55% of I, b<sub>p</sub> 77°, d<sub>4</sub><sup>20</sup> 0.9176, n<sub>D</sub><sup>20</sup> 1.463, [α]<sub>D</sub><sup>20</sup> 27.5°, [α]<sub>D</sub><sup>25</sup> 30.6°, semi-carbazone, m 162°, oxime, b<sub>p</sub> 145°. Similarly the action of PhMgBr on the nitrile and amide gave 2 samples of Ph camphenyl ketone (II), b<sub>p</sub> 172°, d<sub>4</sub><sup>20</sup> 1.0145, n<sub>D</sub><sup>20</sup> 1.5354, [α]<sub>D</sub><sup>20</sup> 33.1°, [α]<sub>D</sub><sup>25</sup> 37.3°, surface tension at 19° 38 dynes per sq cm, and b<sub>p</sub> 121°, d<sub>4</sub><sup>20</sup> 1.0151, n<sub>D</sub><sup>20</sup> 1.5321, [α]<sub>D</sub><sup>20</sup> 38.6°, semi-carbazone, m 167°. Treatment of II with an excess of H<sub>2</sub>O<sub>2</sub> in CHCl<sub>3</sub> gave the characteristic epoxycamphenyl Ph ketone, in 64-5°, [α]<sub>D</sub><sup>20</sup> 27.8°, [α]<sub>D</sub><sup>25</sup> 31.0°. The action of a large excess of PhMgBr on I from the nitrile and working up of the reaction mixt. gave 60% of a mixt. of the 2 isomeric camphenylphenylethylcarbinols (III), b<sub>p</sub> 148-50°, d<sub>4</sub><sup>20</sup> 0.995, n<sub>D</sub><sup>20</sup> 1.5267, [α]<sub>D</sub><sup>20</sup> -12.5°, [α]<sub>D</sub><sup>25</sup> -15.0°, surface tension at 19° 36.4 dynes per sq cm. A purer product, b<sub>p</sub> 154-60°, d<sub>4</sub><sup>20</sup> 0.910, n<sub>D</sub><sup>20</sup> 1.5220, was obtained from the ketone prepd from the amide. Treatment of this product with H<sub>2</sub>O<sub>2</sub> gave a crude, m 85-6°, which was fractionally crystd from alc and by evapn from benzene and xylene solns into a more sol prepolymerant epoxide (III), in 98°, [α]<sub>D</sub><sup>20</sup> -42.7°, [α]<sub>D</sub><sup>25</sup> -51.7°.





and evidently the absence of a double bond in  $\text{Me}_2\text{BuC-CPH O NH}$  (I) is not due to polymerization. To aid the

proposed resolution of I the group  $-\text{CH}_2\text{CO}_2\text{H}$  was attached to the N atom and, since this modification might change the structure of the functional group, the operation was carried out with the Na deriv. of  $\text{EtPhC NOH}$  (II) (absorbed normal form) and the Na deriv. of I (transparent iso form). A soln. of 1 mol. of the oxime in 1 mol. of alc. 1:1  $\text{NaOH}$  was heated for 1 hr. and the theoretical amt. of  $\text{ClCH}_2\text{CO}_2\text{Na}$  was added portionwise and the mixt. was refluxed for 3 hrs. The product was then treated with  $\text{H}_2\text{O}$  and the unchanged oxime was removed. The acidified solns. were extd. with  $\text{Et}_2\text{O}$  and the evapd. exts. gave I ( $\text{PhC NOCH}_2\text{CO}_2\text{H}$  (III), m.  $58^\circ$ ), and  $\text{Me}_2\text{BuCCPh O NCH}_2\text{CO}_2\text{H}$  (IV), m.  $70^\circ$ . Both III and IV

possess spectra analogous to those of II and I, thus establishing that the condensation with  $\text{ClCH}_2\text{CO}_2\text{H}$  does not affect the functional grouping of the present oxime. Fractional recrystn. failed to resolve IV and resolution of the brutine salt, m.  $103^\circ$ , was ineffective. To obtain better cryst. derivs. by introducing a Me radical in the  $\alpha$ -position to the  $\text{CO}_2\text{H}$  group, the Na salt of I was condensed with Na  $\alpha$ -bromoisobutyrate (V), yielding a product, m.  $55^\circ$ , transformed by recrystn. and drying over  $\text{H}_2\text{SO}_4$  into a compd.,  $\text{C}_{14}\text{H}_{19}\text{NO}_3$ , m.  $135^\circ$ . A repetition of the condensation gave different results and produced an isomeric compd., m.  $79^\circ$ . Both products gave analogous absorption curves and possess the same functions. It is supposed that V acts in 2 modifications,  $\text{MeEtBrCCO}_2\text{H}$  and  $\text{Me}(\text{CH}_2)_2\text{BrCCO}_2\text{H}$ . Biochem. sepa. with *Aspergillus niger*, *Penicillium glaucum*, etc., failed to give active modifications of IV. Optical resolution to confirm the results of spectra observations has, so far, been unsuccessfully attempted. C. R. Addinall

The unequal activity of the carbonyl group in succinic methylamide and N-methylpyrrolidone toward organometallic reagents. R. Lukes and K. Smolek. *Collection Czechoslov. Chem. Communications* 7, 476-81 (1935), cf. C. A. 23, 4784.—Succinic methylamide (I) and N-methylpyrrolidone (II) undergo the normal reaction with Grignard reagents to give a carbinol which can be dehydrated to an unsatd. compd. In addn. II undergoes a reaction in which 2 radicals are substituted for the O of the carbonyl group (cf. C. A. 25, 102). The reaction between I and  $\text{PhMgBr}$  was too rapid for an effect of time on yield to be detected between 0 and 48 hrs. In the reaction between II and  $\text{PhMgBr}$ , however, time was an important factor in the amt. of reactions as detd. by titration of the bases volatile with steam. The amide N in II exercises an important restraint on the activity of the adjacent carbonyl group. While I and II both have the form of an intramol. salt, which form best explains the high b. p. and dielec. const. of amides, I had in addn. a 2nd carbonyl group which accounts for its greater activity. W. F. Bruce

Action of the Grignard reagent on the amide group VIII. Action of organomagnesium compounds on ethyl 1-methyl-2-keto-2,3-dihydro-5-pyrrylacetate. R. Lukes and J. Preuß. *Collection Czechoslov. Chem. Communications* 7, 482-90 (1935), cf. preceding abstr.—The action of  $\text{MeMgI}$  on 1,2-dimethyl-3-carbethoxy-5-pyrrylone resulted in the evolution of  $\text{CH}_4$ , indicating an enol form. No other definite product was isolated. The carbonyl group adjacent to the N rather than the carboxy group in 1,1-dimethyl-2-keto-2,3-dihydro-5-pyrrylacetate reacted with  $\text{MeMgI}$ , giving *Et* 1-methyl-5-pyrrylacetate (I),  $\text{C}_{11}\text{H}_{17}\text{NO}_3$ , b.  $135.6^\circ$ ,  $d_4^{20}$  1.0458,  $n_D^{20}$  1.49428. On hydrolysis I gave 1,2-dimethyl-5-pyrrylacetic acid,  $\text{C}_{11}\text{H}_{19}\text{NO}_4$ , m.  $119.5^\circ$  (decompn.), which on distn. at ordinary pressure gave 1,2,5-trimethylpyrrole. By using  $\text{C}_6\text{H}_5\text{MgBr}$  the product was *ethyl* 1-methyl-2-ketyl-5-pyrrylacetate,  $\text{C}_{18}\text{H}_{25}\text{NO}_4$ , b.  $184.5^\circ$ ,  $d_4^{20}$  0.9784,  $n_D^{20}$  1.4636, which on hydrolysis gave 1-methyl-2-ketyl-5-pyrrylacetic acid,  $\text{C}_{18}\text{H}_{21}\text{NO}_5$ , m.  $81-91^\circ$ . On distn. at ordinary pressure this gave 1,2-dimethyl-5-ketylpyrrole,  $\text{C}_{11}\text{H}_{17}\text{NO}$ , b.  $171-2^\circ$ . By using  $\text{PhMgBr}$  the product was *Et* 1-methyl-2-phenyl-5-pyrrylacetate,  $\text{C}_{18}\text{H}_{21}\text{NO}_4$ , b.  $205-10^\circ$ ,  $d_4^{20}$

1.1095,  $n_D^{20}$  1.57435, which on hydrolysis gave 1-methyl-2-phenyl-5-pyrrylacetic acid,  $\text{C}_{18}\text{H}_{21}\text{NO}_5$ , m.  $157^\circ$  (decompn.). On decarboxylation this gave 1,2-dimethyl-5-phenylpyrrole,  $\text{C}_{11}\text{H}_{17}\text{NO}$ , m.  $50-1^\circ$ . All these pyrrole derivs. gave intense colors in the pine splinter test with concd.  $\text{HCl}$ . W. F. Bruce

The synthesis of amino acids. III. Tryptophan. Wm. J. Hoyle and Wm. Robson. *Biochem. J.* 29, 255-8 (1935), cf. C. A. 29, 5094, 5111.—Indole-3-aldehyde was condensed with  $\beta$ -naphthol by refluxing for 30 min. in piperidine (65% yield). The indolallydnantion was heated with  $(\text{NH}_4)_2\text{S}$  (16%) and  $\text{NH}_4\text{OH}$  (3%) in a closed vessel at  $100-3^\circ$  for 500 hrs. Tryptophan was obtained in 70% yield by filtration of the reaction mixt., followed by evapn. of the filtrate, extn. of the residue with  $\text{H}_2\text{O}$  contg.  $\text{NH}_3$  in small amts., evapn. of the ext. and pptn. with  $\text{EtOH}$ . L. W. Scott

Quinoline acids from oxindole derivatives. F. Ward Zike and H. G. Lundwall. *J. Am. Chem. Soc.* 58, 40-50 (1936), cf. C. A. 29, 1420.—Isatin,  $\text{PhCH}_2\text{CO}_2\text{H}$  and  $\text{EtNH}$  in abs.  $\text{EtOH}$  gave 71% 3-hydroxy-3-(phenylcarbethoxymethyl)oxindole, m.  $154-6^\circ$ , while 1%  $\text{NaOH}$  gives isatin, refluxing with concd.  $\text{HCl}$  contg. a little  $\text{EtOH}$  gives 90% of 3-phenyl-2-quinoline-4-carboxylic acid. The isatonomalonitrile (I) of Walter (*Ber.* 35, 1321 (1902)), on reduction with  $\text{Na}_2\text{S}_2\text{O}_4$  yields 10% of 3-(decanamethyl)oxindole, m.  $181-5^\circ$ , by hydrolysis with 33%  $\text{NaOH}$  gives 1,2,3,4-tetrahydro-2-quinoline-4-carboxylic acid (II) Isatin and  $\text{NCCCH}_2\text{CONH}_2$  give 3-(cyanomorphomide)methyl)oxindole (III), red, m.  $248-50^\circ$ . I or III with concd.  $\text{HCl}$  gives the compd.  $\text{C}_{17}\text{H}_{17}\text{O}_4\text{N}_2$  (IV), m. above  $310^\circ$ , which yields a *di-El* salt and a *di-El* ester, pale yellow, m.  $150-1^\circ$ . IV also results from the tetra-*Et* ester of oxindole-3,3-bis(methanecarboxylic acid) (C. A. 29, 3674) by hydrolysis with 10%  $\text{NaOH}$ . Reduction of IV with  $\text{Zn}$  and  $\text{AcOH}$  or  $\text{Na-Hg}$  gives II,  $\text{CO}_2$  being lost. C. J. West

Some alkyl glyoxalimides. Henry C. Chutwood and E. Emmet Reid. *J. Am. Chem. Soc.* 57, 2124-6 (1935).—The best method of prep. methylglyoxalimide (I) is to heat 30 g. ( $\text{AcNHCH}_2\text{I}$ ), with 5 g.  $\text{Mg}$  at  $270^\circ$  for 75 min., the yield being 68%. Results are given for other methods, yields varying from 8 to 44%. The higher homologs were prep. by distg. the diacyl derivs. with  $\text{Na}$  or  $\text{Mg}$  or by refluxing the hydrides with the metal and extg. the bases with  $\text{CaH}_2$ . The diacyl derivs. of  $(\text{CH}_3)_2\text{NH}_2$  (I) can be made by heating I with the  $\text{Et}$  esters, although the lower members are conveniently prep. by refluxing the I with the free acids. The following m. ps. of  $(\text{RCONHCH}_2)_2$  are given, where R is: Me  $175.6^\circ$ , *i*  $101.4^\circ$ , *p*  $192^\circ$ , Bu  $184.2^\circ$ , Am  $178.5^\circ$ , hexyl  $173^\circ$ , heptyl  $171^\circ$ , octyl  $167^\circ$ , nonyl  $164.6^\circ$ , decyl  $160.8^\circ$ , undecyl  $158.8^\circ$ . The following figures are the m. ps. of the alkyl glyoxalimides and their picrates, with the yields of the former, R in the formula  $\text{RC}_2\text{H}_4\text{N}$ , being: Me  $103^\circ$ , 20%, 68%; Et  $135.1^\circ$ , 137%, 46%; *i*  $101.4^\circ$ , 32%, 40%; Bu  $141^\circ$ , 125.8%, 27%; Am  $133.8^\circ$ , 128.4%, 31%; hexyl  $160.2^\circ$ , oil, 1.7%; heptyl  $160^\circ$ , 104.8%, 47%; octyl  $152.1^\circ$ , oil, 21%; nonyl  $171.4^\circ$ , 122.5%, 56%; decyl  $159.8^\circ$ , 82%, 22%; undecyl  $159.8^\circ$ , oil  $5^\circ$ , 24%. I and Br in  $\text{CHCl}_3$  give 82% of  $\text{C}_2\text{H}_5\text{NBr}$ , *Et-HBr*, yellow. The soly. decreases with the lengthening of the alkyl side chain, the undecyl deriv. being practically insol. in  $\text{H}_2\text{O}$ , all dissolve in  $\text{HCl}$  to give well-defined salts, which form Au and Pt double salts. The toxicity of the 1st 5 members of the series decreased with the increase in the length of the alkyl radical. Goldfish and tadpoles survived in solns. of 1:5000 but succumbed when placed in solns. of 1:1000, 10 mg. of the Me compd., administered intraperitoneally, killed mice but the higher members of the series were not so toxic; 0.5 g. per kg. of any of the compds., given by stomach, was not toxic for rabbits and did not impair their kidney functions; the Me compd. increased the acidity of the urine; 10 mg. injected intravenously in cats under ether, produced a transient fall in blood pressure but little effect on the respiration. C. J. West

Some reaction products of aromatic amidines with diketones, dialdehydes and their monoximes. John B.

Ekeley and J. Lell Elliott. *J. Am. Chem. Soc.* 58, 163-4 (1936) — Phenanthrenequinone (I) dissolves in aq. solns of amidine-HCl on addn of 50% KOH; acidification gives 80-90% of the following amidines benz, m 277-8°, nitrobenz, m 269-70°, o-isomer, m 287-5-8°. The benz- and m-tolonylamidines of the diacetylmoxime in 105-7° and 94-6°, resp. The monoxime of I forms benz, m- and p-tolonylamidines, dark green and dark red (last 2), m 186°, 165-6° and 182-3°, resp. An improved method for the prepn of substituted 2,4-diphenyl-5-hydroxypyrimidines (benzoyl, oxalines) from a glyoxal amidine addn product is described, using benzamidine and glyoxal, products were prepd from the following aldehydes: phenylaldehyde, lemon-yellow, m 259-60°, o-ClC<sub>6</sub>H<sub>4</sub>-CHO, lemon-yellow, m 260°, p-isomer, yellow, m 305-6°, dimethoxyresorcinylaldehyde (Ia), orange, m 248-9°, 2,5-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO (II), orange, m 268-9°, 3,4-isomer (III), brown-orange, m 297-60°, p-MeNC<sub>6</sub>H<sub>4</sub>CHO, red-brown, m 277-8°, p-EtOC<sub>6</sub>H<sub>4</sub>CHO (IV), lemon yellow, m 292-3°, m HOC<sub>6</sub>H<sub>4</sub>CHO, yellow, m 265-5-5°, dibromosalicylaldehyde, orange-red, m 311°, homosalicylaldehyde, bright red, m 287-8°, 5-nitrosalicylaldehyde, red brown, m above 300°, cumenol (V), orange, m 246-7°, 3,4-Me(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHO (VI), orange-yellow, m 253-4°, piperonal (VII), yellow-brown, m 285-7°, m MeC<sub>6</sub>H<sub>4</sub>CHO, lemon-yellow, m 237-8°. 5-Hydroxy-2-(3-tolyl)pyrimidines from m-tolonylamidine, glyoxal and the following aldehydes Ia, orange, m 250-1°, II, orange yellow, m 229-30°, III, orange-brown, m 238-9°, IV, yellow, m 237-8°, V, yellow, m 263-4°, o-MeOC<sub>6</sub>H<sub>4</sub>CHO, yellow, m 272-3°, p-MeOC<sub>6</sub>H<sub>4</sub>CHO, lemon-yellow, m 227-4°, VI, orange, m 237-9°, VII, yellow-brown, m 249°. C. J. West

Unsaturation and tautomeric mobility of heterocyclic compounds VI The mobility of 5 substituted 1-hydroxybenzothiazoles and the ultraviolet absorption of mobile and static derivatives of 1-hydroxybenzothiazole Robert G. Hunter and Edwin R. Parken. *J. Chem. Soc.* 1935, 1755-61, cf. C. A. 28, 6439<sup>1</sup> — Ethoxybenzothiazole, pale yellow, m 35-6°, was prepd from p-tolylthiourethan, hydrolysis with concd HCl of 1-ethoxy-5-methylbenzothiazole gives I hydroxy-5-methylbenzothiazole (I), m 168-9°. Ag salt (II), cream, decomp 180°. Na salt, decomp 260-1°, partly hydrolyzed by H<sub>2</sub>O. I (1 g) in 2 cc CHCl<sub>3</sub> and 20 cc 2a% aq NaOH with 2 cc Me<sub>2</sub>SO, refluxed 10 min, gives 1 g 1-keto-2,5-dimethyl-1,2-dihydrobenzothiazole (III), m 76-7°, methylation of the Na salt with MeI at 100° for 5 hrs also gives III, 0.5 g of II gives 0.2 g of III with MeI at 100°. The methosulfate of 1-amino-5-methylbenzothiazole with alkali gives the oily iminodimethyl base, which yields with NaNO<sub>2</sub> in AcOH at 5° 1-nitrosoimino-2,5-dimethyl-1,2-dihydrobenzothiazole, dark red brown, explodes 144°, heating in C<sub>6</sub>H<sub>5</sub>Me gives III p-Tolylthiocarbamide and MeOH with a little quinoline give Me p-tolylthiocarbamate, m 79-80°, K<sub>2</sub>Fe(CN)<sub>6</sub> gives I methoxy-5-methylbenzothiazole (IV), m 32-3°, hydrolysis gives I. The Ac deriv of I m 109° I and Br in CHCl<sub>3</sub> at 6° give the 4(or 6) Br deriv (V), m 239-40°, Me<sub>2</sub>SO, and 2a% aq NaOH give 4(or 6)-bromo-1-keto-2,5-dimethyl-1,2-dihydrobenzothiazole (VI), m 145°, III and Br also give VI IV and Br in CHCl<sub>3</sub> appear to give a mixt. of V and the Br deriv of IV, converted into V by hydrolysis with HCl o-Bromo-p-tolylthiourethan, m 32-3°, 3-bromo-1-hydroxy-5-methylbenzothiazole, m 209°, Me<sub>2</sub>SO, gives the 3-Br deriv of III, m 116° 2-Bromo-p-tolylthiocarbamide, m 44-5° 2-bromo-p-tolylthiourethan, m 106° 4(or 6) bromo-1-ethoxy-5-methylbenzothiazole, m 50-1°, hydrolysis gives V p-Phenylthiourethan, m 93-4°, 1,5-dithoxybenzothiazole, m 54°, 1-hydroxy-5-ethoxybenzothiazole (VII), m 147°, Me<sub>2</sub>SO, gives I keto-2,5-dimethyl-1,2-dihydrobenzothiazole, m 85° Me p-phenylthiocarbamate, m 68-9°, 1-methoxy-5-ethoxybenzothiazole, m 75-6°, hydrolysis gives VII p-Iodo-phenylthiourethan, m 106-7°, 5-odo-1-ethoxybenzothiazole, m 76-7°, 5-odo-1-hydroxybenzothiazole, m 225-6°, Me<sub>2</sub>SO, gives 5-odo-1-keto-2-methyl-1,2-dihydrobenzothiazole, m 135°, this also results from 5-odo-1-nitroso-

imino-2-methyl-1,2-dihydrobenzothiazole, salmon-pink, explodes 160°. Me p-bromophenylthiocarbamate, m 90-100°, 5-bromo-1-methoxybenzothiazole (VIII), m 82-3° Me phenylthiocarbamate, m 93°; 1-methoxybenzothiazole, m 88-9° Methylation of 5-bromo-1-hydroxybenzothiazole gives no trace of VIII 5-Chloro-1-ethoxybenzothiazole, m 60-1°, 5-chloro-1-hydroxybenzothiazole, m 204°, 5-chloro-1-keto-2-methyl-1,2-dihydrobenzothiazole, m 112°, also from the 5-chloro-1-nitrosoimino deriv, explodes 138-9°. 5-Nitro-1-nitrosoimino-2-methyl-1,2-dihydrobenzothiazole, salmon pink, explodes 152°, heating in C<sub>6</sub>H<sub>5</sub>Me, gives the 5-nitro-1-keto deriv, yellow, m 162-3° Phenylthiourethan disulfide is unchanged after heating at its m p for 0.5 hr, no trace of this compd could be obtained by heating phenylthiourethan and 1-ethoxybenzothiazole slightly above the m p of the disulfide Absorption spectra are given for I in MeOH, NaOH, NaOEt and KOH, also for I, III and IV, and their significance is discussed VII. Selenazole derivatives Churagh Hasan and Robert F. Hunter *Ibid* 1762-6 — Phenylselenazole and Br in CCl<sub>4</sub>-CHCl<sub>3</sub> give 1-amino-benzoselenazole (I), m 142° Heating I with Me<sub>2</sub>SO in MeOH for 2 hrs gives after heating with an excess of alkali 1-amino-2-methyl-1,3-dihydrobenzosenazole, yellow, m 104°, NaNO<sub>2</sub> in AcOH gives the 1-nitrosoimino deriv, brown, explodes 142-4° s-Phenylmethylselenazole, m 111°, turns gray on keeping, Br in CHCl<sub>3</sub>-CCl<sub>4</sub> gives the N-Me deriv of I, m 140° I and Ac<sub>2</sub>O give the 1-acetamido deriv (II), m 190°, and a compd, m 130°. s-Acetylphenylselenazole, light brown, m 195°, Br in CCl<sub>4</sub>-CHCl<sub>3</sub> gives II Diphenylselenazole and Br give 1-anilino-benzosenazole, m 170° (picrate, yellow, m 245°), methylation gives a mixt. of 1-phenylimino 2-methyl-1,2-dihydrobenzosenazole, whose picrate m 172° (about 72% of the product) and 1-phenylmethylaminobenzosenazole, whose picrate, yellow, m 200°, the synthesis of the latter is described Bis-o-aminophenyl diselenide and CCl<sub>4</sub>/Et give bis-o-urethanophenyl diselenide, brown, m 110°, Sn and HCl give 1-hydroxybenzosenazole (III), m 140°, Sn was also obtained by the action of COCl<sub>2</sub> upon the Zn salt of o-H<sub>2</sub>NCH<sub>2</sub>SeH and also by the hydrolysis of 1-chlorobenzosenazole in EtOH, methylation gives the 1-keto-2-methyl deriv (IV), m 60° IV was also prepd by heating the nitrosoimino deriv in C<sub>6</sub>H<sub>5</sub>Me 1-Thiolbenzosenazole, m 159°, was prepd by the condensation of nascent o-H<sub>2</sub>NCH<sub>2</sub>SeH with CS<sub>2</sub> from the Zn salt and CSeCl<sub>4</sub> and from 1-chlorobenzosenazole and NaSH Methylation gives an oil, which appears to be the expected 5-Me deriv, since it is different from the 1-thio-2-methyl-1,2-dihydrobenzosenazole, m 80°, obtained from IV and P<sub>2</sub>S<sub>5</sub> 1-Amino-α-naphthoselenazole, m 252°, results from 1,2-BrC<sub>10</sub>H<sub>6</sub>NH<sub>2</sub> HCl and KSeCN in H<sub>2</sub>O, Ac deriv, m 250°, fusion with NaOH gives β C<sub>10</sub>H<sub>6</sub>NH<sub>2</sub> and bis-2-amino-α-naphthyl diselenide (V), brown, decomp 120° s-Phenyl-β-naphthylselenazole, m 174°, Br in CCl<sub>4</sub>-CHCl<sub>3</sub> gives 1-anilino-α-naphthoselenazole, m 210°, fusion with KOH gives V V, NaHS, aq NaOH and CS<sub>2</sub> in a stream of H<sub>2</sub>S give on heating 1-thio-α-naphthoselenazole, yellow, m 228°. C. J. West

Some alkyl derivatives of certain aryl-substituted thiazolidones John A. Davis and F. B. Dains. *J. Am. Chem. Soc.* 57, 2627-30 (1935), cf. C. A. 27, 5075 — Thiazolidones (I) were prepd from RNHCOCH<sub>2</sub>Cl and KSCN (refluxing in alc for 1-5 hrs) or from the monoaryl thioureas (refluxing in alc for 1-3 hrs with equal mol quantities of C<sub>6</sub>H<sub>5</sub>N and CICH<sub>2</sub>CO<sub>2</sub>Et). The Na salt (hot 5% NaOH) and an alkyl halide give a mixt. of the 2- and 3-alkyl derivs, sep'd by the soly of the former in dil HCl The following yields of 2- and 3-Et derivs of I were obtained from o-IC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 68%, 1%, p isomer, 50, 1, o-MeOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 65, 0, 6, m isomer, 40, 6, p isomer, 50, 1, pseudocumidine, 56, 0, 2-amino-p-cymene, 65, 0, p-IC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 74, 16, 2,4-IC<sub>6</sub>H<sub>3</sub>NH<sub>2</sub>, 0, 85 The Na salt of the I from p-IC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> and MeI gave 60 and 75% of the 2- and 3-alkyl deriv, EtI, 74 and 16%; AmI, 34 and 4%, PhCH<sub>2</sub>Cl, 60, 4% The following thioureas were prep'd N-ethyl-N'-p-ethoxyphenyl, m 112°, o-isomer, m 88°, N-ethyl-N'-pseudocumyl, m 138°, N-Me analog,

m. 179°; *N*-ethyl-*N*'-o-methoxyphenyl, m. 77°; *p*-isomer, m. 147°; *m*-isomer, m. 112°; mono-*p*-cymyl, m. 152°; *N*-ethyl-*N*'-*p*-cymyl, m. 126°; *N*-ethyl-*N*'-2,4-diiodophenyl, m. 164°; *N*-methyl-*N*'-*p*-iodophenyl, m. 171°; *N*-Et analog, m. 147.5°; *N*-Am analog, m. 130°; *N*-benzyl analog, m. 147.5°; 4-Thiazolidones 2-*p*-ethoxyphenylamino-3-ethyl, m. 93°; 2-*o*-ethoxyphenylamino-3-ethyl, m. 99°; 3-Et deriv, m. 70°; 2-Pseudocumyl-3-ethyl, m. 77°; 3-Me analog, m. 91°; 2-*o*-methoxyphenylamino-3-ethyl, m. 114°; *p*-MeO isomer, m. 83°; 2-methoxyphenylamino, m. 105°; 2-Et deriv, m. 112°; 3-Et deriv, m. 65-67°; 2-*p*-cymylamino, m. 174°; 2-Et deriv, m. 85°; 3-Et deriv, m. 61°; 2-(2,4-diiodophenylamino), m. 233°; 3-Et deriv, m. 173°; 2-iodophenylamino, m. 226°; 1-Me deriv, m. 207°; 3-Me deriv, m. 152°; 2-Et deriv, m. 110° (II); 3-Et deriv (III), m. 103°; 2-Am deriv, m. 116°; 3-Am deriv, m. 57°; 3-benzyl deriv, m. 137°; 2-benzyl deriv, m. 170°; 2-phenylamino-3-ethyl, m. 137°; 2-benzylamino-3-ethyl, m. 74°; 5-Benzal-4-thiazolidones 2-Ethyl-2-*p*-ethoxyphenylamino, m. 210°; 3-Et isomer, m. 135°; 2-*o*-ethoxyphenylamino-3-ethyl, m. 113°; 2-Et isomer, m. 114°; 2-ethyl-2-pseudocumyl, m. 180°; 2-pseudocumylamino-3-ethyl, m. 141°; 3-Me analog, m. 130°; 2-ethyl-2-*o*-methoxyphenylamino, m. 140°; 3-Et isomer, m. 93°; 2-ethyl-2-*p*-methoxyphenylamino, m. 198°; 3-Et isomer, m. 125°; 2-ethyl-2-*m*-methoxyphenylamino, m. 135°; 3-Et isomer, m. 100°; 2-ethyl-2-*p*-cymylamino, m. 169°; 3-Et isomer, m. 73°; 2-(2,4-diiodophenylamino)-3-ethyl, m. 210-11°; 2-methyl-2-iodophenylamino, m. 218°; 2-*p*-iodophenylamino-3-methyl, m. 167°; 2-ethyl-2-iodophenylamino, m. 231°; 3-Et isomer, m. 172°; 2-Am analog, m. 202°; 2-phenylamino-3-ethyl, m. 78°; 2-benzyl-2-*p*-iodophenylamino, m. 221°; 3-benzyl isomer, m. 162°; 2-benzylamino-3-benzyl, m. 109°; *o*-Chloroacetamino-*p*-cymene, m. 85°; 2,4-diiodo-*o*-chloroacetamido, m. 153°; *N*-Ethyl-*N*'-*p*-ethoxyphenyl-*N*'-phenylurea, m. 93°; *p*-nitrobenzyl-*o*-phenetidine, m. 155°; *N*-*o*-tolyl-*N*'-*o*-ethoxyphenylamino, m. 172°; *o*-Phenetidylbenzenesulfonate, m. 61°; *Et*-*o*-amidybenzenesulfonate, m. 60°; 5-benzal-3-benzyl-2,4-thiazolidone, m. 134°; 3-Am analog, m. 74°. The hydrolysis products of the 5-benzal-4-thiazolidones are given. The I condense with *p*-ONC<sub>6</sub>H<sub>4</sub>NMe<sub>2</sub>, the compd. C<sub>14</sub>H<sub>11</sub>ON<sub>2</sub>S, from II, dark yellow, m. 246° (decomp.); that from III, red, m. 212°; C I W.

Constitution of calycotrin II S. Mahal and K. Venkataraman *Current Sci.* 4, 111-121(1935), cf. *C. A.* 29, 2534—Treatment of calycotrin (I) with AlCl<sub>3</sub> has resulted in a new flavone which exhibits the usual properties of a catechol deriv and must therefore be 5,6,4'-trihydroxy-3,7,8-trimethoxyflavone or 3,5,6,4'-tetrahydroxy-7,8-dimethoxyflavone. The second HO in I being in the 8-position, I may now be formulated as 6,4'-dihydroxy-3,5,7,8-tetramethoxyflavone W J Peterson

Pyridine-2-acetic acid. M P Oparina *Khim. farm. Prom.* 1936, No. 2, 99-101.—Pyridine-2-acetic acid very easily loses CO<sub>2</sub> (in H<sub>2</sub>O at 50-60°). The Me ester is quite stable; reduced in the presence of Pt it forms piperidine-2-acetic acid, it liberates C<sub>2</sub>H<sub>4</sub> with Grignard reagents. The nitroso deriv (in H<sub>2</sub>SO<sub>4</sub> + NaNO<sub>2</sub>) does not produce the Liebermann reaction. L Nasarevich

Iodo-hydroxyquinolinesulfonic acid S Vinaver *Khim. farm. Prom.* 1935, No. 2, 109-110.—The iodination of the hydroxyquinolinesulfonic acid is best effected by direct addn. of I to the Na salt. 7-Iodo-8-hydroxyquinoline-5-sulfonic acid is liberated with HCl L N

Preparation of *o*-hydroxyquinoline. O Yu Magidson and M V Rubtsov *Khim. farm. Prom.* 1935, No. 1, 20-3.—A practical method in which aniline is treated according to Skraup, the quinoline sulfated at 160° with 20% oleum, treated with CaCO<sub>3</sub> and the Ca salt of 8-sulfonquinoline is decomposed under pressure with NaOH at 225° (17-19 atm.). The ppt. is dissolved in dil. H<sub>2</sub>SO<sub>4</sub>, boiled, filtered and hydroxyquinoline pptd. with NH<sub>3</sub> and NaHCO<sub>3</sub> L Nasarevich

*p*-Dimethylaminobenzalquinaldine. A contribution to the action of chromophoric and auxochromic groups, Hans Rupe, Heinrich Hagendach and August Collin *Helv. Chim. Acta* 18, 1395-1413(1935); cf. *C. A.* 26,

2185, 27, 2446.—Condensation products between *p*-Me-NC<sub>6</sub>H<sub>4</sub>CHO (I) and ketones are highly colored, but their salts are colorless. A similar behavior is shown by *p*-dimethylaminobenzalquinaldine (II) (cf. Noetting and Witte, *Ber.* 39, 2750(1906)), m. 183-5°, which dyes cotton orange-red but gives a colorless di-HCl salt, C<sub>14</sub>H<sub>15</sub>Cl<sub>2</sub>N<sub>3</sub>, and a blue mono-HCl salt, C<sub>14</sub>H<sub>15</sub>ClN<sub>3</sub>, m. 239-40°; diperchlorate, C<sub>14</sub>H<sub>15</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>8</sub>, monoperochlorate, C<sub>14</sub>H<sub>15</sub>ClN<sub>3</sub>O<sub>4</sub>, red-violet, m. 229-30° (decomp.); monoperochlorate, C<sub>14</sub>H<sub>15</sub>N<sub>3</sub>O<sub>4</sub>, red-violet, m. 167-8°. With MeI II gave a light yellow monomethiodide, C<sub>14</sub>H<sub>15</sub>IN<sub>3</sub> (III), m. 250-1° (decomp.), and a dark blue-red form, C<sub>14</sub>H<sub>15</sub>IN<sub>3</sub> (IV), m. 253-4° (decomp.). To det. the point of addn. in each case, I was allowed to react with MeI to give a methiodide, C<sub>14</sub>H<sub>15</sub>INO<sub>4</sub>, m. 141° (decomp.), which was condensed with quinaldine to give III. The methiodide of quinaldine condensed with I gave IV. Upon catalytic hydrogenation II gave a colorless dihydro deriv, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>, m. 68-85°, di-HCl salt, C<sub>14</sub>H<sub>17</sub>Cl<sub>2</sub>N<sub>3</sub>, m. 210-11°, diperchlorate, C<sub>14</sub>H<sub>17</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>8</sub>, m. 157-8° methiodide, C<sub>14</sub>H<sub>17</sub>IN<sub>3</sub>, m. 158-9° nitrosamine, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O, m. 90.5-91°, *Bz* deriv, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O, m. 120.5-121.5°. By the addn. of Me<sub>2</sub>SO, II gave a colorless di-*N*-Me deriv, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>S, m. 253-3° (decomp.), and a red mono-*N*-Me deriv, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>S, m. 242-3°, which was identical with the condensation product between *N*-methylquinaldine sulfate, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>S, m. 223-4°, with I. The product, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>S, m. 201-2.5° (decomp.), from the condensation of the *N*-Me sulfate of I, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>S, m. 154°, with quinaldine, was nearly colorless. By the action of PhMgBr on II,  $\alpha$ -*p*-dimethylaminophenylethylidene-1,2-dihydroquinoline (V) was isolated as the perchlorate, C<sub>14</sub>H<sub>17</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>8</sub>, m. 192-4° (decomp.), from which was obtained the pure base VI, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>, m. 125°, di-HCl salt, C<sub>14</sub>H<sub>17</sub>Cl<sub>2</sub>N<sub>3</sub>, monomethiodide, C<sub>14</sub>H<sub>17</sub>IN<sub>3</sub>, m. 125-6° (decomp.), nitrosamine, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O, m. 105-6° (decomp.), dimethiodide, C<sub>14</sub>H<sub>17</sub>IN<sub>3</sub>, m. 206-8° (decomp.). In addn. to VI, PhMgBr and II gave a substance of unknown structure, possibly an isomer of VI, m. 191.5°, methiodide, C<sub>14</sub>H<sub>17</sub>IN<sub>3</sub>, m. 196-7° (decomp.) W. F. Bruce

Some esters of amino alcohols with 9-acridinecarboxylic acid B Sandahl and Chr. I. Wender. *Bull. soc. chim.* [5], 2, 209-16(1935).—Esters of amino alcs. have generally a more or less anesthetic action and acridine derivs. act as bactericides. Attempts have been made to make compds. with both physiol. properties by prep. a series of acridinecarboxylic esters of  $\beta$ -aminoethanol and  $\beta$ -dialkylaminoethanols. The products are very feebly anesthetic and are non-anesthetic. A mixt. of 1 part of 9-acridinecarboxylic acid (I) (C A 23, 144) and 4 parts of SOCl<sub>2</sub> was refluxed until the acid dissolved and was then distilled to remove most of the excess SOCl<sub>2</sub>. The cooled product gave 85-90% of 9-acridinecarboxyl chloride (II) (I) (C A 23, 144). To a stirred suspension of 13.9 g of II in 20 cc. of dry benzene was added dropwise 3.3 g. of H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OH, prep. by the action of NH<sub>3</sub> on ethylene oxide. The thick warm brew was dried with benzene and the mixt. was refluxed for 4-5 hrs. The cooled product was filtered off, washed with benzene and dried. It xtn. with HCl, neutralization and recrystn. from 20% alc. gave  $\beta$ -aminoethyl 9-acridinecarboxylate (III), C<sub>14</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub>, m. 234-40°, which in alc. with dry HCl yielded only the mono-HCl salt, m. 238-40° (decomp.). Esterification of Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OH, prep. by the action of NIMe<sub>3</sub> on ethylene oxide, by treatment with II proceeded directly to the di-HCl salt of  $\beta$ -dimethylaminoethyl 9-acridinecarboxylate (IV), m. 172-3° (decomp.). An equimol. mixt. of the free base and IV yielded the mono-HCl salt, m. 189-90° (decomp.). The following dialkylaminoethyl esters of I and their HCl salts were similarly prep.: *Et*, HCl salt, m. 179-80° (decomp.); *di*-HCl salt, m. 170-80° (decomp.); *Pr*, HCl salt, m. 177-8° (decomp.); *di*-HCl salt, m. 177-8° (decomp.); *iso*-Bu, HCl salt, m. 178-80° (decomp.); *di*-HCl salt, m. 169° (decomp.); *iso*-Am, HCl salt, m. 146°; *di*-HCl salt, m. 167-8° (decomp.). The dialkylamino alcs. were prep. in good yields by the action of CICH<sub>2</sub>CH<sub>2</sub>OH on the requisite amine (*Org. Synthesis* XIV, 28 (*C. A.* 28, 2078); *Pr*;







(II), very pale yellow, m 248°, pptn from di-Na-CO<sub>3</sub> soln with acid gives I. Refluxing II with EtONa-FtOH for 18 hrs or the action of KMnO<sub>4</sub> on 1-hydroxy-3-(4'-nitrophenyl)-3,4 dihydrophthalazine-4-acetic acid (III) yields 30.5 or 71.6% of 1,4-diketo-3-(4'-nitrophenyl)-tetrahydrophthalazine (IV), m 307° (C 23, 145). Ag salt, yellow needles. The Ag salt and AgCl in C<sub>6</sub>H<sub>6</sub> or boiling III with Ac<sub>2</sub>O for 3 min give the 4-keto-1-acetoxy deriv of III, pale yellow, m 232°. Mel and the Ag salt in MeOH, refluxed 0.25 hr, give the 4-keto-1-methoxy deriv of III, m 199°. 3'-NO<sub>2</sub> isomer of I, pale yellow, m 193-4°, 3'-NO<sub>2</sub> isomer of II, pale yellow, m 227-8°; this results in 59% yield on heating the isomer of I in PhNO<sub>2</sub> for 1 hr, together with 19% of 1,4-diketo-3-(3'-nitrophenyl)tetrahydrophthalazine Ag salt, pale yellow; 4-keto-1-acetoxy deriv, m 164°, 4-keto-1-methoxy deriv, m 182°. 2'-NO<sub>2</sub> isomer of I, orange yellow, m 290-4° (99% yield), 2'-NO<sub>2</sub> deriv of II, pale yellow, m 293-4°; the 2'-NO<sub>2</sub> deriv of IV, pale yellow, m 293-4°. Ag salt, yellow needles, 4-keto-1-methoxy deriv, m 176-7°. o-Carboxybenzo-4'-nitro-2'-methylphenylhydrazide, pale yellow, m 219°, phthalyl-4'-nitro-2'-methylphenylhydrazide, m 228°, 1,4-diketo-3-(4'-nitro-2'-methylphenyl)tetrahydrophthalazine, m 271°. Ag salt, yellow needles. 2'-Cl deriv of I, m 270-5°, 2'-Cl deriv of II, m 267°, 2'-Cl deriv of IV, m 271-2°, the 4-keto-1-methoxy deriv, pale yellow, m 193-4°. 2'-Br deriv of I, m 232°, 2'-Br deriv of II, m 282-3°, 2'-Br deriv of IV, m 273-4°. 4-keto-1-methoxy deriv, pale yellow, m 167-8°. 2,6-Dichloro-4-nitrophenylhydrazide, yellow, m 133°, from 2,6,4-Cl<sub>2</sub>(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>NH<sub>2</sub> (BzH<sub>2</sub> deriv, orange, m 153°). 2',6'-di-Cl deriv of I, m 192°, 2',6'-di-Cl deriv of II, m 202°, 2',6'-di-Cl deriv of IV, m 309-10°. 4-keto-1-methoxy deriv, m 178-9°. 2,6-Dibromo-4-nitrophenylhydrazide, pale yellow, turning red at 110° and gradually converted into deep red prisms by heating at 80° or leaving in EtOH for several weeks, both forms m 135-6° (BzH<sub>2</sub> deriv, orange, m 168-9°). 2',6'-Di-Br deriv of I, m 200°, 2',6'-di-Br deriv of II, pale yellow, m 230°, 2',6'-di-Br deriv of IV, pale yellow, m 327-9°. o-HO<sub>2</sub>CC<sub>6</sub>H<sub>3</sub>CONHNH<sub>2</sub>, reduced in PhNO<sub>2</sub> for 2 hrs, gives the Na-CO<sub>3</sub> insol phthalylphenylhydrazide, m 178-9° (32.3%) and the sol 1,4-diketo-3-phenyltetrahydrophthalazine (V), m 212-13° (43%). The Ag salt of V and EtI in EtOH give 33.3% of 4-keto-1-ethoxy-3-phenyl-3,4-dihydrophthalazine, m 108-9°, the 1 MeO deriv, m 109-11°. C J West

Synthesis of 4-ethyl-6-aminopyrimidine Wm T Caldwell and Wm M Ziegler J Am Chem Soc 58, 78-9 (1936) — Condensation of MeCH(OEt)CO<sub>2</sub>Et with ethylpseudourea-HBr, using MeOH-KOH, gives 80% of 2-ethylmethyl-4-ethyl-6-hydroxypyrimidine, hydrolysis of the latter gives 4-ethyluracil m 204-5°, refluxing 17 g with 70 cc POCl<sub>3</sub> for 1 hr yields 17.5 g of 2,6-di-Cl deriv, b 90-5°, 17 g of this product with III and red P gives 21 g of 4-ethyl-6-hydroxypyrimidine III, yellow, m 170.5-1.5°, 20 g of which yield 9.1 g of 4-ethyl-6-chloropyrimidine (I), b 195°, 8.5 g I and 250 cc satd FtOH-NH<sub>3</sub>, heated at 150° for 4 hrs, give 6.2 g 4-ethyl-6-aminopyrimidine, with 3 mols H<sub>2</sub>O, m 45-8°, picrate, yellow, m 204.5°, HCl salt, m 198-9°, chlorourate, yellow, m 159-1°. C J West

Dioxane series II Aryl substituted dioxanes Synthesis of p-dioxane R K Summerbell and L N Bauer J Am Chem Soc 57, 2264-8 (1935), cf C A 28, 150° — 2,3-Dichloro-1,4-dioxane (I) and PhMgBr give 80% of the 2,3-di-Ph deriv. The following 1,4-dioxanes were similarly prep'd (yields in % of dioxane) 2,3-di-o-tolyl, m 105.7-6.2° (m ps cor) (61%); m-isomer, m 85° (51%) (picrate, m 91.2°), p-isomer, m 56-7.2° (72%) (picrate, m 96.5°), bis(p-chlorophenyl), m 152.3° (49%), di-p-anisyl, m 79.80.2° (67%) (picrate, m 107°), di-o-naphthyl, b 25-28° (53%) (picrate, m 166-7°), dizenyl, m 144.5-6°, dibenzyl, m 62.2° (22%). I and EtMgBr give principally p-dioxane, CH<sub>3</sub>CH(OCH<sub>3</sub>)CH<sub>2</sub>O (II), b<sub>118</sub>, 94.2°, d<sub>4</sub> 1.083, n<sub>D</sub> 1.462, a 2nd product may be 2,3-diethylidioxane, b<sub>118</sub>, 184-75°, the gas evolved in the reaction consisted of 16.5% C<sub>2</sub>H<sub>4</sub>, 48.2% C<sub>2</sub>H<sub>6</sub>, and 3.4% C<sub>2</sub>H<sub>2</sub>. The chief reaction product of MeMgBr and I is II; BuMgBr also gives II and about an equal quantity of 2,3-dibutylidioxane (7), b<sub>120</sub>, 120-2°. II and O<sub>2</sub> give HCO<sub>2</sub>H and (CH<sub>3</sub>CO)<sub>2</sub>H<sub>2</sub>; II and Cl<sub>2</sub> give 53.4% I. Br gives 92.2% of 2,3-dibromo-1,4-dioxane, m 69-70°, which evolves HBr on exposure to the atm, but yields the 2,3-di Ph deriv. with PhMgBr. II and HCl at 0° give monochloro-p-dioxane, b<sub>118</sub>, 62-3°, d<sub>4</sub> 1.276, BiCl<sub>3</sub> is a catalyst for the reaction, it decomposes rapidly in the air, with PhMgBr it yields 49% of monophenyl-p-dioxane, m 46°. C J West

Catalytic chlorination of dioxane J J Kucera and D C Carpenter J Am Chem Soc 57, 2346-7 (1935) — ICl and SnCl<sub>4</sub> are suitable catalysts for the chlorination of dioxane to the 2,3-di Cl deriv, the yield being almost quant. Their use is not adapted to the further chlorination to the tetra-Cl deriv, because of the high temp required for the latter reaction at ordinary pressure; the yields of the sym. tetra-Cl isomers, m 70° and 143°, were 83 and 33%, resp, with or without catalyst. C J W.

Piperazine S Vinaver Khim Farm Prom 1934, No 6, 11-14 — Piperazine is made by decompn with H<sub>2</sub>SO<sub>4</sub> of diisotoluenesulfonylpiperazine, made by condensation of toluenesulfonylamine and (CH<sub>3</sub>)<sub>2</sub>Br. For medicinal purposes piperazine acid tartrate is used in preference to piperazine. L Nasarevich

Derivatives of piperazine VIII Condensation with aldehydes W T Forsee, Jr, and C B Pollard J Am Chem Soc 57, 2363-4 (1935), cf C A 29, 7990° — Aldehydes react with piperazine (I) or N-mono-substituted piperazines so that 1 mol aldehyd. reacts with 2 NH<sub>2</sub> groups, with the elimination of 1 mol H<sub>2</sub>O. I and HCHO or CH<sub>3</sub>CHO give methylenepiperazine in 97 and 98% yields, m. above 300°; BzH or PhCHCl<sub>2</sub> gives benzylidenepiperazine in 98 and 46% yields, resp, m 270° (decompn), o-HO deriv, m 210° (decompn), 44%, p-Me deriv, m 275° (decompn), 90%, o-Cl deriv, m 240° (decompn), 95%, p-MeO deriv, m 290° (decompn), 85%. The N-Ph deriv of I and HCHO or CH<sub>3</sub>CHO give N-bis(N'-phenylpiperazyl)methane, m 123-4°, 79 and 24% yields, resp. The ethane, m 121-3°, 25%, the phenylmethane m 125-6°, 90%; the o-methylphenylmethane, m 144-5°, 83%; the o-methoxyphenylmethane, m 135-6°, 67%. All the condensation products are readily decomposed by acids. The compds from the N-Ph deriv of I are readily crystd. C J West

Quinoxaline VII The interaction of 2,4-dichloroquinoxaline in alcohol with ammonia and with methylamine Edw Vopicka and N A Lange J Am Chem Soc 57, 1068-70 (1935), cf C A 27, 2957° — 2,4-Dichloroquinoxaline (I) (2 g) and 20 cc EtOH-NH<sub>3</sub>, heated 2 hrs at 150°, give 0.7 g of the 2,4-di-NH<sub>2</sub> deriv, m 230° (all m ps cor), HCl salt, m 308°, sulfate, m 359°; acetate, with 1 mol H<sub>2</sub>O, m 208°, nitrate, m 280°, oxalate, m 274°, picrate, yellow, m 304° (decompn), di-Ac deriv, m 230° I (3.5 g) and 35 cc EtOH-Me-NH<sub>2</sub> give 2 g of the 2,4-bis(methylamino) deriv, m 120°, HCl salt, m 312°, picrate, m 232°. C J West

The reduction products resulting from the condensation of sugars and aromatic amines P Karrer, H Salomon, R Kunz and A Seebach Helv Chim Acta 18, 1338-42 (1935) — A report of the prep'n and properties of several substances prep'd in connection with the flavin synthesis (C A 30, 4689°) The sugar, in H<sub>2</sub>O or MeOH, is added to the amine in alc, the soln heated 2 hrs, evap'd to dryness in vacuo, the residue washed twice with ether, dissolved in MeOH and reduced at 100° with Ni catalyst at 20-25 atm H<sub>2</sub>, or at 30-60° with Pd on charcoal. The product is obtained in 60-90% yield on tcn of the soln PhNH<sub>2</sub> and d-glucose (I) yield N-phenyl-d-glucamine, m 134°. The monobenzal deriv (II), needles, m 197-8°, is assigned the probable structure PhNHCH<sub>2</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH Acetylation of II in pyridine with

Ac<sub>2</sub>O yields a triacetate, m 118-19°. p-MeC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> and I yield N-p-tolyl-d-glucamine, m 123°. p-C<sub>6</sub>H<sub>4</sub>OC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> and I give N-p-ethoxyphenyl-d-glucamine, m 123°.

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*N*-*p*-Hydroxyphenyl-*d*-glucamine, m. 163° after repeated recryst. from MeOH, was obtained from *p*-HOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> and I. I and *p*-HOC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> yield *N*-*p*-hydroxyphenylethyl-*d*-glucamine (III), isolated as the HCl salt which melts 206° and m 208° (decomp.). Reduction of CH<sub>3</sub>OH[CH(OH)]<sub>4</sub>CH<sub>2</sub>NEt, m 116°, obtained by Irvine (C A 7, 1709), yields *N*-ethyl-*d*-glucamine (IV) isolated as the HCl salt, m 134°. Full details are given for the isolation of III and IV which are more difficult to obtain than the other compds described. III and IV have but little pharmacol activity. W Gordon Rose.

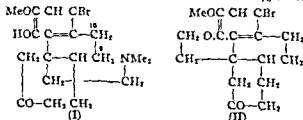
**Flavin syntheses VIII** Synthesis of 6-methyl-9-(*d*-1'-ribityl)isalloxazine and further synthetic experiments in the flavin series. P. Karrer and F. M. Stronza. *Helv. Chim. Acta* 18, 1343-51 (1935). To continue the study of the relation between chem. constitution and phys. activity of the flavins, 6-methyl-9-(*d*-1'-ribityl)-isalloxazine (I), which differs in the position of the Me group on the benzene ring from that described in C A 29, 6490°, was prep'd by the following steps: Nitration of *m*-cresol (250 g.) gave 4-nitro-3-hydroxytoluene (78 g.) which on alkylation by C<sub>6</sub>H<sub>5</sub>N<sub>3</sub>O<sub>2</sub> gave 4-nitro-3-methoxytoluene (yield 94.7%). This compd was heated with NH<sub>4</sub>OH and Cu<sub>2</sub>O at 140° to prep 4-nitro-3-aminotoluene (yield 14%), which with ClCO<sub>2</sub>Et gave 4-nitro-3-carbethoxyaminotoluene, C<sub>8</sub>H<sub>7</sub>N<sub>3</sub>O<sub>4</sub>, m 51.2° (yield 69.1%). On reduction by Pt and H<sub>2</sub> this gave 4-amino-3-carbethoxyaminotoluene, C<sub>8</sub>H<sub>9</sub>N<sub>3</sub>O<sub>4</sub>, m 120° (yield 85.3%). Reductive condensation of this compd with *d*-ribose gave 3-carbethoxyamino-4-methylphenyl-*d*-ribamine, C<sub>13</sub>H<sub>17</sub>N<sub>3</sub>O<sub>6</sub>, m 153° (yield 61.5%), 3.8 g. of which on hydrolysis by KOH and condensation with alloxan in the presence of H<sub>2</sub>BO<sub>3</sub> (cf. Kuhn and Weygand, C A 29, 6237°) gave I, C<sub>14</sub>H<sub>17</sub>N<sub>3</sub>O<sub>6</sub>, m 292°, [α]<sub>D</sub><sup>20</sup> = +84.6° = 5 (0.6 g.). The phys. action of I in daily doses of 10-20 γ was comparable to that of vitamin B<sub>2</sub>. In an attempt to prep 3,7-dimethyl-9-(*d*-1'-ribityl)isalloxazine the following syntheses were made: Nitration of 2,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHAc by an improved method gave 2,4-dimethyl-6-nitroacetamide, m 170.5° (yield 60-65%), in addition to an unidentified compd, C<sub>8</sub>H<sub>9</sub>N<sub>3</sub>O<sub>4</sub>, m 73°, probably 2,4-dimethyl-6-nitrophenol. Hydrolysis by 80% H<sub>2</sub>SO<sub>4</sub> converted the nitroacetamide to 2,4-dimethyl-6-nitroaniline, yield (60-65%). Because of steric hindrance this with ClCO<sub>2</sub>Et did not give the desired compd, 2,4-dimethyl-6-nitrocarbethoxyaniline (II). However, by condensation of 2,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>2</sub> with ClCO<sub>2</sub>Et, 1, 2,4-dimethylphenylcarbamate, m 63°, was obtained (yield 90.5%), and thus on nitration gave II, m 135° (yield 83%). Catalytic reduction of II yielded 2,4-dimethyl-6-amino-1-carbethoxyaniline, C<sub>13</sub>H<sub>17</sub>N<sub>3</sub>O<sub>4</sub>, m 99.5° (yield, 77.4%). Reductive condensation with *d*-ribose gave 2-carbethoxyamino-3,7-dimethylphenyl-*d*-ribamine, C<sub>15</sub>H<sub>21</sub>N<sub>3</sub>O<sub>6</sub>, m 139° (yield 69.5%). The further steps to the flavin were unsuccessful, since alk. hydrolysis yielded chiefly 1-*d*-ribityl-2-hydroxy-4-β-dimethylbenzimidazole, C<sub>12</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub>, m 248°. W F Bruce.

**Synthesis of lactoflavin** P. Karrer, B. Becker, F. Benz, P. Ifer, H. Salomon and K. Schöpp. *Helv. Chim. Acta* 18, 1435-48 (1935). cf. Kuhn et al. C A 30, 464°. Kuhn's synthesis of flavins is unsatisfactory because of low yield. The prep'n of *d*-ribose and lactoflavin is given in detail. From 1 kg. of Ca gluconate, 175 g. of pure *d*-arabinose was obtained (cf. Hockett and Hudson, C A 28, 5047). From 3 kg. of *d*-arabinose, 270 g. of *d*-ribose was prep'd. An improved synthesis of lactoflavin is given. 1 kg. of *o*-xylene gave 250 g. of 3,4-dimethyl-1-nitrobenzene, b. 123.30°. From 60 g. of this product 43 g. of 3,4-dimethylaniline, m 49°, was prep'd and from 30 g. of the latter, 45 g. of 3,4-dimethyl-6-aminocarbethoxyaminobenzene. From this 45 g. of 3,4-dimethyl-6-nitrocarbethoxyaminobenzene, m 54°, was prep'd, from which 3,4-dimethyl-6-aminocarbethoxyaminobenzene, m 111°, was secured. By reductive condensation of the latter (7 g.) with *d*-ribose (5 g.), 4,5-dimethyl-2-carbethoxyaminophenyl-*d*-ribamine, m 170° (9 g.), was prep'd. From the ribamine by hydrolysis with KOH and condensation with alloxan was obtained after purification by adsorption 1.8 g. of lactoflavin. W F. Bruce.

**Thiochrome.** Richard Kuhn and Hellmuth Vetter. *Ber.* 68B, 2375-85 (1935), cf. C A 29, 6242°. Vitamin B<sub>2</sub> (I) was dehydrated to thiochrome (II) in strongly alk. soln. by the action of porphorexide, a reduction-oxidation indicator, or by K<sub>2</sub>Fe(CN)<sub>6</sub>. The conditions for this reaction lead to the conclusion that a pseudo base is first formed. II does not have the phys. activity of I. The max. fluorescence of II in 0.01 N NaOH is in the region 460-470 mμ. Fluorescence is visible in dilns of 0.5 mg per l. Graphs show the relation of fluorescence to diln. and thickness of the layer observed. Comparison of II from I and from yeast showed complete identity. By hydrogenation of II in H<sub>2</sub>O or 0.1 N HCl 2 mols. of H<sub>2</sub> were absorbed and a soln. with an absorption spectrum in part identical with that of I resulted. Detn. of C-Me showed that Me was not involved in the ring closure of I to give II. The detn. of MeNH is shown frequently to be unreliable. II gave a neg. van Slyke test. Its fluorescence was destroyed by 1 hr. boiling with 2 N NaOH after which NaS was demonstrated and extn. with CHCl<sub>3</sub> gave a blue fluorescent S-free substance. W F Bruce.

**Thiochrome from vitamin B<sub>2</sub> (antixerin).** G. Barger, F. Bergel and A. R. Todd. *Ber.* 68B, 2257-62 (1935), cf. Kuhn, C A 29, 6242°, Barger, C A 29, 7329°. Conversion of vitamin B<sub>2</sub> (I) to thiochrome (II) by oxidation with alk. K<sub>2</sub>Fe(CN)<sub>6</sub> was accomplished in 30-40% yield. The HCl salt of II, C<sub>10</sub>H<sub>7</sub>N<sub>2</sub>O<sub>2</sub>·2HCl, was catalytically hydrogenated in MeOH-HCl to yield a hygroscopic substance which does not fluoresce or yield I on oxidation. By concd. HCl at 100° I was deaminated but II was not altered. W F Bruce.

**Sinomenine XLII** *d*- and *l*-Bromothebenone. Kakui Goto, Hiroshi Ogawa and Jun Saito. *Bull. Chem. Soc. Japan* 10, 481-5 (1935), cf. C A 25, 3002°; 29, 6238°. Direct bromination of thebenone gave a mixt. which could not be sep'd. In prep. the bromothebenones by indirect methods it was shown that demethoxysinomenone and thebanone and their corresponding derivs. are optical isomers and that when they are mixed they form optically inactive racemates. A soln. of 1.1 g. dihydrodes-*N*-methyl-demethoxydihydro-sinomenone in 5 cc. AcOH was brominated with 0.55 g. Br<sub>2</sub> in 10 cc. AcOH at 16° to give *d*-9,10-dihydro-des-*N*-methyl-1-bromodihydrothebenone (I), C<sub>11</sub>H<sub>11</sub>NO<sub>2</sub>Br, m 192°, [α]<sub>D</sub><sup>20</sup> 61.60°. *l*-I, m 257° (decompn.); methiodide, m 273°. *l*-I, m 192°, [α]<sub>D</sub><sup>20</sup> -62.67°, was prep'd from the thebanone deriv. in the same manner. *l*-I-*l*-I, m 257° (decompn.); *l*-I-Mel, m 273°. Equal parts of *d*-I and *l*-I were recryst'd from MeOH to give *d*-I, m. 175-7°. *d*-I-Mel (1.5 g.) was boiled 20 min. with 10 cc. 25% KOH to give *l*-1-bromothebenone (II), C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>Br, m 70°, [α]<sub>D</sub><sup>20</sup> -22.67°. *d*-II, m 70°, [α]<sub>D</sub><sup>20</sup> 23.33°, was prep'd similarly from *l*-I-Mel. *d*-II, m 191-3°. *l*-Bromodemethoxydihydro-sinomenone-Mel (3 g.) was boiled with 15 cc. of 15% NaOH for 12 min., did with 8 cc. H<sub>2</sub>O and pptd. with CO<sub>2</sub> to give *l*-des-*N*-methyl-1-bromodihydrothebenone (III), C<sub>10</sub>H<sub>9</sub>NO<sub>2</sub>Br, m 200-1°, [α]<sub>D</sub><sup>20</sup> -8.67°. methiodide, m 241° (decompn.). *d*-III, m 199°, [α]<sub>D</sub><sup>20</sup> 8.90°, was prep'd in the same way from the corresponding thebanone deriv. *d*-III-Mel, m 241°, *d*-III, m 189-92°. *l*-III-Mel (2.5 g.) was refluxed 40 min. in 30 cc. of 11% NaOH



and acidified to give *l*-1-bromo-9,10-dihydrothebenone (IV), C<sub>10</sub>H<sub>9</sub>O<sub>2</sub>Br, m. 145°, [α]<sub>D</sub><sup>20</sup> -18.80°. *d*-IV, m 148-50°, [α]<sub>D</sub><sup>20</sup> 187.33°, was prep'd in the same way from *d*-III-Mel. *d*-IV, m 159-62°. When 1.8 g. *l*-dehydrothebenone in 10 cc. AcOH was brominated with 1.1 g. Br<sub>2</sub> in 5 cc. AcOH, using ice, 1 g. of a mono-Br deriv. was obtained.

This was probably 1-(10<sup>10</sup>) bromo-9,10-dehydrothebenone (V), m 125-33°,  $[\alpha]_D^{25}$  -113.33°. Bromination of d-dehydrothebenone gave d-V, m 127-30°,  $[\alpha]_D^{25}$  112.67°, d-V, m 156-8°.

John F. Milberry

**Alkaloids from seeds of Cassia absus**, Linn. Salimuzzaman Siddiqui and Zafaruddin Ahmed *Proc Indian Acad Sci* 2A, 421 5(1935)—The small seeds of the indigenous annual, *Chakras*, contains a kernel used in the treatment of ophthalmia and skin affections and as a cathartic. The 2 bases, *chaksine* (I) and *isochaksine* (II), have been isolated as carbonates in a total amt of 1.5% of the kernel. Dil aq solns of the  $H_2SO_4$  salt of I are general depressants of the heart, respiration and nerves, 0.1 g per kg being lethal to frogs. Percolation of 3.6 g of powd kernel with cold 0.3% MeOH, neutralization with  $NH_4OH$ , acidification, removal of the alc and extrn with  $Li_2O$  gave a residual sirup. The soln of the residue in 2 l of acidulated  $H_2O$  was treated with 50 g of KI and, on working up, yielded 46 g of *chaksine iodide* (III) and about 5 g of *isochaksine iodide* (IV). A soln of 5.3 g of III in 50 cc of hot alc was treated with the freshly pptd  $Ag_2O$  from 4.5 g of  $AgNO_3$ . The mixt. was filtered and dried with 200 cc of  $Li_2O$  and 50 cc of petroleum ether. The colorless Ag free filtrates obtained were evapd in an open dish and produced hydrated *chaksine bicarbonate* (V), m 167.9°. I could not be isolated except as an impure product,  $[\alpha]_D^{25}$  32° (abs alc), but the following salts were isolated: *iodide*,  $C_{17}H_{19}N_3O_4$ , m 168° (decompn), *sulfate*, m 316° (decompn), *picrate*, m 239-40° (decompn), *chloroplatinate*, m 232° (decompn). Treatment of alc IV with freshly pptd  $Ag_2O$  and purification of the filtrate with  $Li_2O$  and petroleum ether gave II as a cream-colored hygroscopic sirup converted by  $CO_2$  to *isochaksine carbonate*, sintering at 108° and decompn at 128°. Addn of ethereal HCl to II in abs alc and  $Li_2O$  formed *isochaksine chloride*,  $C_{17}H_{19}ClN_3O_4$ , m 250-2° (decompn). I is partially isomerized when liberated from its iodide and is completely converted into II by treatment of its sulfate with  $Ba(OH)_2$ . The formation of the picrate of II from the alc- $Me_2CO$  sol iodide after the sepn of the iodide of I, showed that II is present in the seeds and is not formed by isomerization during the isolation process.

C R Addinall

**Cactus alkaloids XV Separation of the anhalonium bases** Ernst Späth and Friedrich Becke *Monatsh* 66, 327-36(1935), c f C A 29, 7989<sup>1</sup>—*Anhalonium lewisii* (1330 g) was extd with  $EtOH$  at 15-20° and the aq soln of the sirup from the evapd ext was treated with 2%  $HCl$ , the filtrate made alk and extd with  $Et_2O$ , the soln finally extd with 50% KOH. The  $Et_2O$  soln of the nonphenolic bases (2.83 g) on treatment with  $H_2SO_4$  gave mesquite sulfate (I) (0.97 g), the filtrate, made alk, yielded 0.4 g anhalonium (as the HCl salt) and 0.06 g anhalimine (I Me ether) as the HCl salt, a further quantity of I (0.194 g) was isolated from the filtrate and also 0.04 g lophophorine picrate. Details are also given of the sepn of the phenolic bases, including anhalamine, anhalonidine, pellettine and anhalidine. Formulas of the compds are given.

C J West

**Conessine series I Isomerization of conessine and its nor bases** Salimuzzaman Siddiqui *Proc Indian Acad Sci* 2A, 426-37(1935), c f C A 29, 2960<sup>1</sup>—The alkaloids of *Holarrhena antidysenterica* all bear a simple relation to conessine (I),  $C_{17}H_{19}N_3O_4$ . A study has been made of the isomerization of I and its nor bases, *isocossamine* (II),  $C_{17}H_{19}N_3O_4$ , and *conamine* (III),  $C_{17}H_{19}N_3O_4$ , by the action of cold concd  $H_2SO_4$ . The gradual addn of 50 g of I to 150 g of ice-cooled concd  $H_2SO_4$  gave a bright yellow soln which began to evolve  $SO_2$  and deepen in color to red. The soln was added slowly to 2 kg of crushed ice and the cold mixt was made alk with NaOH. The liberated base was extd with  $Et_2O$ , shaken out with AcOH and treated with concd KI soln. The cold turbid mixt yielded 5 g of *hydrindole*, m 325-6°, together with oxidation products of I. Neutralization of an AcOH soln of the hydriodide with NaOH yielded a light straw-colored sirup of *isocossamine* (IV),  $C_{17}H_{19}N_3O_4$ ,

m 239-41°,  $[\alpha]_D^{25}$  97° (1% abs. alc. soln), dissolving in concd  $H_2SO_4$  to a bright yellow soln changed to dark blue and, finally, to bright violet on the addn of  $H_2O$ , *HCl salt*, m 318° (decompn),  $[\alpha]_D^{25}$  72° (1% aq soln), *chloroplatinate*, m 271-3°, *chloraurate*, m 293.5° (decompn), *picrate*, 240-2° (decompn), *II Br salt*, (V) m 318-22° (decompn), *di-Mel deriv*, m 316-8° (decompn). Titration of  $CHCl_3$  solns of IV with Br gave V by elimination of HBr from the initial addn product and its combination with IV. The presence of a single ethylene linkage in the isocossamine series was established by titration of the di-Ac deriv. of isocossamine with Br in  $CHCl_3$  and the formation of a di-Br compd, m 185-0 (decompn). The isomerization of I to IV by concd  $H_2SO_4$  is followed by slow oxidation. A similar isomerization of II gave *isocossamine* (VI),  $C_{17}H_{19}N_3O_4$ , as a straw-colored sirup,  $[\alpha]_D^{25}$  101° (1% abs alc soln), *III salt*, m 280° (decompn), *HCl salt*, m 335° (decompn),  $[\alpha]_D^{25}$  72.80° (1% aq soln), *picrate*, m 166°, *chloroplatinate*, m 290-2° (decompn). By refluxing with a mixt of  $HCO_2H$  (1.5 mols) and  $HCHO$  (1.5 mols) and treating the product with  $H_2O$ , VI was transformed to IV. VI was converted into a *nitroso base*, *HCl salt*,  $C_{17}H_{19}N_3O_4$ , m 282-4°. The isomer of II was named "isocossamine" to avoid confusion in the nomenclature of conessamine and II and the name "norisocossamine" has been reserved for the isomer of conessamine. The isomerization of III gave *isocossamine* (VII),  $C_{17}H_{19}N_3O_4$ ,  $[\alpha]_D^{25}$  89° (1% abs alc soln), *III salt*, m 332° (decompn), *HCl salt*, m 335-6° (decompn), *picrate*, m 135°, *chloroplatinate*, m 285-6° (decompn), *dimitro deriv*,  $C_{17}H_{19}N_3O_4$ , m 123.5°, *di-Ac deriv*, m 190-1°. Methylation of VII by refluxing with 0.5 g of the base in a min of  $HCO_2H$  with 2.5 mols of  $HCHO$  for 1 hr gave IV. The optical activity of the isomers of the conessine series is about three times as pronounced in the pos. direction as that of the corresponding conessine bases. IV is also 3 times more potent than I in its action on the respiration of frogs and this potency may enhance the therapeutic value of the holarrhena alkaloids since IV may prove to be a potent aid in the treatment of amebic dysentery. C R Addinall

**Reduction studies in the morphine series. VII. Pseudocodoneone** Robert E Lutz and Lyndon Small *J Am Chem Soc* 57, 2651-6(1935), c f C A 29, 1829<sup>1</sup>—Pseudocodoneone (Knorr and Hörlein, C A 1, 2125) (10-15% yields) forms a *HCl salt*, (I), with 1 mol of  $H_2O$ , m 201.3° (decompn) (all m ps cor),  $[\alpha]_D^{25}$  -24° ( $H_2O$ , c 1), *semicarbazosemicarbazone*, with 1 mol  $H_2O$ , m 225-7° (decompn). Catalytic reduction of I in AcOH with Pt oxide gives 46% of the *dihydro deriv* (II) and 34% of the *tetrahydro deriv*, (III). II m 113°,  $[\alpha]_D^{25}$  37° ( $EtOH$ , c 0.62), *HCl salt*, with 1 mol  $H_2O$ , m 172-3°,  $[\alpha]_D^{25}$  13° ( $H_2O$ , c 0.65), *III salt*, m 250-5° (decompn),  $[\alpha]_D^{25}$  8.1° ( $H_2O$ , c 0.91), *oxime*, m 244-5°. Reduction of II with Na in  $EtOH$  gives 78% of *dihydropseudocodoneone-A*. Heating II in  $Ac_2O$ -AcONa for 3 hrs at 100° gives a nearly quant yield of *des-N-acetyl-dihydropseudocodoneone enol acetate*, m 191.5-2°. III, with 0.5 mol  $H_2O$ , m 137-8.5° (anhyd, m 170-1°),  $[\alpha]_D^{25}$  8° ( $EtOH$ , c 0.55), it does not react with  $CH_3N_3$ , AcO in  $CH_3N_3$  at 100° gives a *mono-Ac deriv*, glassy, *oxime*, m 218-9°, *HCl salt*, with 2 mols  $H_2O$ , m 165-6°,  $[\alpha]_D^{25}$  -6.2° ( $H_2O$ , c 1.29), *III salt*, with 1 mol  $H_2O$ , m 151-5°,  $[\alpha]_D^{25}$  -5.9° ( $H_2O$ , c 0.85). Reduction of either I or III with Na in  $EtOH$  (N atm) gives 62% of *tetrahydropseudocodoneone*, Na-*II* in AcOH gives the same compd. Demethylation of 4.5 g of II yields 2.45 g of *dihydropseudomorphine*, m 198°  $[\alpha]_D^{25}$  46° ( $EtOH$ , c 0.44). I (12 g) and MeMgI in  $Et_2O$  give 3.3 g of *methyl-dihydropseudocodoneone*, m 213.5-14.5°, which shows no reaction of a CO group or of a double bond. Comparison of the physical action of *dihydropseudocodoneone* and *dihydropseudomorphine* with that of *codeine* and *dilaudid* and that of the *codeine* and *morphine* isomers indicates that morphine derivs with the functional group at C-4 are more effective than those with the same group at C-3 but that spatial relationships are also very important.

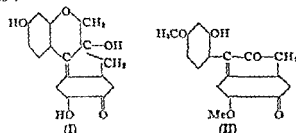
C J West

**Styrene and brucine** XXXV. Hofmann degradation of dihydrobrucine. O Achmatowicz, P. Lewi and Robert Robinson. *J. Chem. Soc.* 1935, 1855-54, cf. *C. A.* 29, 7991. *N*(b)-Methylidihydrobrucine iodide and 20% excess  $\text{Ag}_2\text{CO}_3$ , heated 20 hrs. at 100°, give the carbene (I), amorphous, deep red, hygroscopic solid, *N*(a), *N*(b)-dimethylidihydrobrucine disulfonate (II), with 16 mols  $\text{H}_2\text{O}$ , m 190-2° (decompn) with III both I and II lose  $\text{CO}_2$  and yield the iodides. Thermal decarboxylation of I yields 15% of dihydrobrucine and 5% of *N*(b)-methylidihydrobrucine (III). Heating 200 g II over a free flame in lots of 1-2 g gives the following products: 45 g III; 34 g dihydrobrucine, 19 g of *N*(b)-methylidihydrobrucine iodide (IV); 17 g dihydrobrucine-2-sulfonate, with 8 mols  $\text{CHCl}_3$ , the final fraction (12 g of a gum) probably consists largely of IV. III (methyl-4-dihydrobrucine of part V) in 221-2°, sol in concd  $\text{LiOH}$  1-2%, in boiling  $\text{FIOH}$  1-5%, in 40%  $\text{HCl}$  give a green color with  $\text{FeCl}_3$ , changing to pale yellow on warming or standing. III was not reduced electrolytically or catalytically. With  $\text{MeI}$  at 50° for 2 hrs results a dimethiodide, with 4 mols  $\text{H}_2\text{O}$ , of which 2 are lost at 130°, m 294-6° (decompn), with  $\text{MeSO}_2$ , III gives a very methoxide, converted to the  $\text{MeI}$  deriv with an  $\text{NaI}$ , dimethiodide, with 3 mols  $\text{H}_2\text{O}$ , m 206-8°, 2 mols  $\text{H}_2\text{O}$  are lost at 105°, the anhyd salt is very hygroscopic. Refluxing III with  $\text{MeI}$  gives an isomeric dihydroderivative, with 3 mols  $\text{H}_2\text{O}$ , m 145-6° (decompn), 1 mol of  $\text{H}_2\text{O}$  are lost at 105°, dimethiodide, with 4 mols  $\text{H}_2\text{O}$  and 3 mols of  $\text{MeOH}$ , m 202-4° (decompn), with  $\text{MeONa-MeOH}$  this yields 90% of *N*(b), *N*(b)-dimethylidihydrobrucine, m 165-6°. Catalytic reduction of III in  $\text{AcOH}$  and the action of  $\text{Na}$  on the acetates formed gives *N*(b)-methylidihydrobrucine- $\epsilon$  iodide (V) and the  $\epsilon$ -isomer (VI). V m 317-18°, of 2 mols of  $\text{H}_2\text{O}$  1 is not lost at 270° and 1 mm; crystals from  $\text{BuOH}$  give a product with 1 mol  $\text{H}_2\text{O}$ , the chloride, deep red, with 3 mols  $\text{H}_2\text{O}$  (2 of which are lost at 105°), m 204-6°, with  $\text{MeOH-MeONa}$  it yields III. V decomposes on heating before any  $\text{MeCl}$  is split off, VI, with 2.5 mols  $\text{H}_2\text{O}$  (1 mol retained at 105°), m 204-8° (decompn); chloride, pink, with 2.5 mols  $\text{H}_2\text{O}$ , m 273-5°; on heating extensive decarboxylation occurs and dihydrobrucine- $\epsilon$  was not formed. This chloride is resistant to boiling  $\text{MeOH-MeONa}$  but yields III on heating 0.5 hr. at 120-5°. Hydroxymethylidihydrobrucine (VII), pale yellow, m 160-1°, is recovered unchanged after refluxing with  $\text{MeI}$  and is indifferent towards  $\text{BaI}_2$ . *Ac der*, prepd in the cold, m 258-60°, it is simply saponified by  $\text{MeOH-MeONa}$ , but on heating dihydrobrucine is formed. VII with  $\text{MeSO}_2$  and 40%  $\text{NaOH}$  gives *methoxybrucine methoxybrucine methoxide*, which yielded an iodide, m 166-7° (part V). VII is not attacked by boiling 10%  $\text{H}_2\text{SO}_4$  in 3 hrs but with 25% acid about 10% of the base is converted into methylidihydrobrucine. II sulfate. *N*(b)-Methylidihydrobrucine- $\epsilon$  dione-3-MeI saps with 1 mol  $\text{CHCl}_3$ , which is replaced by 2  $\text{MeOH}$  on crys. from this solvent, methochloride, reddish brown, amorphous, m 165-70°, solidifies and then m 216-8° (decompn). *N*(b)-methylidihydrobrucine- $\epsilon$  is formed, this base gives a green color with  $\text{FeCl}_3$ ; it combines with  $\text{MeI}$  in the cold, on catalytic reduction it absorbs 2 H. On heating dihydrobrucine- $\epsilon$   $\text{MeCl}$  or  $\text{MeSO}_2$  with  $\text{MeONa-MeOH}$  in an open flask, there results dihydrobrucine- $\epsilon$  and III. J. W. Lupine alkaloids. VIII. C. R. Clemm, W. McGe Morgan and R. Raper. *J. Chem. Soc.* 1935, 1743-6, cf. *C. A.* 27, 4235. The following excerpts were carried out in an attempt to synthesize norlupine. Catalytic reduction of *El pyridyl-2-acetate-3- $\beta$ -propanolate*, b 52°, picrolonate, m 141° (picrate, yellow, m 84°);  $\text{BrCH}_2\text{CO}_2\text{Et}$  and the ester in  $\text{Me}_2\text{CO}$ , refluxed 6 hrs, give *El pyridinium-1-acetate-2- $\beta$ -propanolate bromide*, m 163°. The Diels-Alder reaction of *El pyridyl-1-acetate-2- $\beta$ -propanolate*, b 135-40°, the Diels-Alder reaction yields *3-ketobidihydroxyproline*, b 74-76° (picrate, yellow, m 145°), Clemmensen reduction

gives norlupine (octahydroproline A), b 74-76°. *El pyridyl-2-acetate*, pale greenish yellow oil, b 134-5°. (picrate, yellow, m 130-7°), *El pyridyl-2-acetate*, b 130-5° (picrate, m 125°), *El pyridyl-2-acetate-1- $\alpha$ -propanolate*, b 135-40°, *3-keto-2-methylidihydroxyproline*, b 67-9° (picrate, yellow, m 162°); *3-hydroxy-2-methylidihydroxyproline*, b 100-5° (picrate, m 157°), picrolonate, orange-yellow, m 181°. With  $\text{MeOH}$  reduction gives *2-methylidihydroxyproline*, b 32-5° (picrate, yellow, m 167°, picrolonate, light brown, m 208°).

C. J. West

**Quinoid oxidation products in the brassin series.** Paul Pfeiffer and Paul Schneider. *J. prakt. Chem.* 144, 53-62 (1935).—One of the formulas (I) proposed by Fugère, Perkin and Robinson (*C. A.* 2, 2064) is substantiated by the following work. Oxidation of 120 g trimethyldeoxybrassin gives principally trimethylbrasilone but also 4.2 g of II, red with beautiful surface luster, m 241°, sol in concd  $\text{H}_2\text{SO}_4$  with a deep pure blue color changing to deep violet,  $\text{EtOH-KOH}$  gives a green and then a violet soln., aq alkali give reddish brown solns., while  $\text{NH}_4\text{OH}$  gives a gray violet soln. II also results from the oxidation of trimethylidihydrobrassin. Oxidation of II with 33%  $\text{H}_2\text{O}_2$  in  $\text{MeOH-KOH}$  yields 2,4-HO(MeO) $\text{C}_6\text{H}_3\text{CO}_2\text{H}$ . II with  $\text{AcO-AcONa}$  yields a yellow *Ac der*, m 158-60°. The sol in concd  $\text{H}_2\text{SO}_4$  with a marine-blue color, quickly changing to violet. Reductive acetylation of II yields a *tri- $\alpha$ -*Ac der**, m 129°, giving a light orange color in concd  $\text{H}_2\text{SO}_4$ , slowly changing to violet, acid hydrolysis gives II. The oxime of II, violet powder which could not be crystd. Bromotrimethyldeoxybrassin is likewise oxidized by  $\text{CrO}_3$  in  $\text{AcOH}$  to a quinoid compd,  $\text{C}_{20}\text{H}_{20}\text{Br}$ , orange, m 215°, *Ac der*, light yellow, m 208-10°. (With H. Koles) 2-Phenylindan-1-one yields an *Ac der*, m 103-10°.



C. J. West

**Androsterone and related sterols.** Russell E. Marker, Frank C. Whitmore and Oliver Kamm. *J. Am. Chem. Soc.* 57, 2355-60 (1935); cf. *C. A.* 29, 7342. —Reduction of cholesteryl chloride, the action of  $\text{PCl}_5$  on cholesteryl chloride, or the action of  $\text{SOCl}_2$  upon  $\beta$ -cholestanol gives  $\alpha$ -cholesteryl chloride (I), m 112°, the action of  $\text{PCl}_5$  on  $\beta$ -cholestanol or the action of  $\text{SOCl}_2$  upon epicholestanol gives the  $\beta$ -isomer (II), m 102-3°. Hydrolysis of I gives epicholestanol, while II gives  $\beta$ -cholestanol. Oxidation of I with  $\text{CrO}_3$  in  $\text{AcOH}$  gives  $\alpha$ -chloroandrosterone (III), semicarbazone, m 279-81° (decompn), and  $\alpha$ -chlorocholane, m 174-5°. Hydrolysis of III with  $\text{AcOH}$  yields  $\alpha$ -androsterone.

C. J. West

**Sex hormones.** VIII. Preparation of testosterone by means of mixed esters. L. Ruzicka, A. Wettstein and H. Kägi. *Helv. Chem. Acta* 18, 1478-82 (1935); cf. *C. A.* 30, 493. *trans*-Androstene-3,17-diol 3-monoacetate in  $\text{CH}_2\text{Cl}_2$  treated with  $\text{BrCl}$  gave  $\Delta^4$ -*trans*-androsterone-3,17-diol 3-acetate 17-acetate (II), m 183-25°. It partially saponified with  $\text{alc KOH}$  gave  $\Delta^4$ -*trans*-androsterone-3,17-diol 17-benzoate (II), m 120.5-22°. This method gave much better yields of the half ester than the one previously described. II is isomated, oxidized and debrominated in the usual manner gave  $\Delta^4$ -*trans*-androsterone-3,17-diol benzoate (testosterone benzoate) (III), m 194-6°. III on sapon gave testosterone IX  $\Delta^4$ -*trans*-Dehydroandrosterone and the preparation of  $\Delta^4$ -*trans*-Dehydroandrosterone from stigmasterol, L. Ruzicka, W. Fischer and J. Meyer. *Helv.* 1863-7.  $\Delta^4$ -*trans*-Androstene-3,17-diol-17-one in abs.  $\text{Et}_2\text{O}$  treated with a satd. soln. of  $\text{HCl}$

in abs EtOH gave the chloride (I), m 156-7°. I dehalogenated gave  $\Delta^{14}$ -trans-dehydroandrosten-3-ol-17-one, m 128-5-30°. Stigmasteryl acetate (II), brominated, oxidized and debrominated, gave  $\Delta^{14}$ -trans-dehydroandrosterone, m 145° (semicarbazone, m about 270°). Stigmasteryl, treated with HCl, gave the chloride (III), m 152-3° III, treated with AcO, gave the chloride of II, m 183-3-5° II, treated with HBr, gave the bromide, m 160-5-61°. X Preparation of 17 methyltestosterone and other androstene and androstane derivatives. Relationship between chemical constitution and male hormone activity L Ruzicka, M W Goldberg and H R Rosenberg *Ibid* 1487-98.—The relative hormone activity of 20 related compds of testosterone and androstene are given. Testosterone was converted to 17-methyltestosterone, m 163-4°, by treatment with MeMgI. The following were likewise prepd: 17-Methyl- and 17-ethylandrostan-3-on-17-ol, resp, m 192-3° and 137 8°;  $\Delta^{14}$ -17-methyl- and  $\Delta^{14}$ -ethyl-trans-androstene-3,17-diol, resp, m 204° and 173°; 17-methyl- and 17-ethyl-trans-androstane-3,17-diol, resp, m 282-3° and 211°; 17-ethyl-cis androstane-3,17-diol, m 143-4°.

#### Julius White

The physical and chemical properties of biosterol and its physiological significance IX Condensation of biosterol with maleic anhydride and citraconic anhydride 2 Juro Nakamiya *Bull Inst Phys-Chem Research* (Tokyo) 14, 584-607 (1935). (Abstracts in English) published with *Sci Papers Inst Phys-Chem Research* (Tokyo) Nos 577-9). cf C A 28, 5107°.—The non-condensing part of biosterol (I) from Ischnagi (*Stereolepis sichnagi*) liver oil and citraconic anhydride gives a new satd hydrocarbon, *isacotane*,  $C_{28}H_{48}$ , m 65°. From the condensing part, a new cryst addn product, *biosterol-citraconic acid*, rhombic plates or cubes,  $C_{48}H_{76}O_8$ , m 207°, is obtained, *perhydro deriv*,  $C_{28}H_{48}O_4$ , m 65-6°, bromide,  $C_{28}H_{47}O_4Br$ , m 186-8°, HCl salt,  $C_{28}H_{47}O_4HCl$ . The crude condensation product of I and maleic anhydride (II), brominated and fractionally pptd by MeOH from the  $CHCl_3$  soln, gives the following fractions: white powder, m 247-5°, Br 33-5%, light yellow powder,  $C_{28}H_{47}O_4Br$ , m 230°, Br 42-3%, lemon-yellow powder,  $C_{28}H_{47}O_4Br$ , m 215°, Br 42-3%, and yellow powder, m 160-70°. Br 45%. A cryst addn product,  $C_{28}H_{48}O_8$ , m 247°, obtained by condensing I and II, is, on heating in pyridine with AcCl, transformed into its isomer, m 227°, no Ac deriv being obtained.

#### K Konda

The differentiation of sterols from other alcohols of the polyterpene series and the structure of lanosterol and onoceryl H Schulze *Z physiol Chem* 238, 35-63 (1936).—A no of mono and polyhydric polyterpene alcs have been classed with the sterols because they give the same color reactions and have similar empirical formulas. They differ, however, from the sterols in certain properties, especially their non precipitability with digitonin. A further distinction is the nature of the product obtained by Se dehydrogenation. The sterols, characterized by their cyclopentenophenanthrene structure, yield the diels hydrocarbon  $C_{25}H_{40}$ , whereas the polyterpene alcs yield a trimethylphenanthrene  $C_{25}H_{40}$  or a tetramethylphenanthrene  $C_{27}H_{44}$ . This dehydrogenation reaction was applied to lanosterol, the main constituent of "isocholesterol," and to onoceryl, a polyterpene alc extd from the roots of *Ononis spinosa*. Isocholesterol was heated with Se 49 hrs at 335° until evolution of  $H_2$ Se had ceased, the reaction mixt extd with  $FeO$  and the extd fractionated at 15 mm. The higher fractions distg at 200-280° yielded crystals of 1,2,8-trimethylphenanthrene, m 142-3° (picrate, m 164-5°). Oxidation of the hydrocarbon by  $CrO_3$  in AcOH gave the *quinone*, m 196-7°, which was condensed with  $\alpha$ - $C_6H_5(NH_2)_2$  to the *quinoxaline*, m 130-1°. An addl hydrocarbon, m 212-3°, was obtained from the highest fraction but was not identified. Dehydrogenation of isocholesterol with Pt at 300° was unsuccessful, the original substance being recovered unchanged. Onoceryl,  $C_{27}H_{44}(OH)_2$ , m 231-2°,  $[\alpha]_D^{25}$  5.04° (in pyridine), was prepd in 2% yield from *Ononis spinosa* roots by alc. extn and dehydrogenated by Se at 300-320°. Distn. of the

1  $FeO$  ext and recrystn of the product from MeOH gave 1,2,5,6-tetramethylphenanthrene, m 113-14° (picrate, m 152-3°), styphnate, m 162°, trimetobenzene compd, m 178°. By recrystn from  $C_6H_6$  and drying at 115°, onoceryl, m 232°, was converted into an isomer, m 202° (acetate, m 224°, benzoate, m 237-8°,  $[\alpha]_D^{25}$  21.3°; chloroacetate, m 238-44°,  $[\alpha]_D^{25}$  115°; 3,5-dinitrobenzoate, m 290-1° (decompn),  $[\alpha]_D^{25}$  19.4°; anisate, m 233-4°,  $[\alpha]_D^{25}$  10.03°). These derivs are identical in m p. and rotation with the corresponding derivs. of onoceryl, m 232°. Onoceryl is not a sterol but a triterpene deriv.

#### A W. Dox

Toad poisons VIII The dehydrogenation of cinobufagin H Jensen *J Am Chem Soc* 57, 2733-4 (1935). cf C A 29, 7902°.—Cinobufagin and Se at 310-340° for 45 hrs give a small quantity of a hydrocarbon,  $C_{28}H_{48}$ , m 120-1°, which is believed to be methylcyclopentenophenanthrene, since it gives no depression in the m p when the 2 are mixed. It appears, therefore, that cinobufagin and probably also the various other bufagins contain the same ring system as the cardiac plant aglucons and the sterols.

#### C J West

Epilcholesterol, a new isomer of cholesterol E A Evans, Jr, and Rudolf Schoenheimer *J Am Chem Soc* 58, 182 (1936).—Reduction of cholesterol with (iso  $PrO$ )<sub>2</sub>Al gives 2 isomeric sterols, that not pptd by digitonin having the compn  $C_{27}H_{46}O$ , m 84°,  $[\alpha]_D^{25}$  120.8° (2% in  $C_6H_6$ ), acetate, m 82.5°; that this is *epicholesterol* (I) is indicated by its catalytic reduction to a mixt of epidihydrocholesterol and epicoprosterol, thus the HO group at position 3 is trans to the Me group at C 10, the double bond is in position 4-5. Refluxing I 2 hrs with a dil soln of HCl in 95% EtOH gives a hydrocarbon  $C_{27}H_{46}$ , m 79°,  $[\alpha]_D^{25}$  -112.8° (2% in  $C_6H_6$ ); the absorption spectrum shows max (244, 235, 229 mμ) at shorter wave lengths than reported for cholesterol. C J West

Thermal equl of the cis trans isomers of dichloroethylene at high temps (Maroney) 2 Estrogenic activity of 1-oxo-1,2,3,4 tetrahydrophenanthrene (Butenandt, Schirum) 11F Purification and phys properties of org compds (Skau, Rowe) 2 Cause of the von Allwarden reaction (Sturm, Colle) 25 Temp dependence of the energy of activation in the decarboxylation of diacetone alc (LaMer, Miller) 2 Carotenoids of purple bacteria (Karrer, Solmsen) 11C Sulfonation of naphthalene (Lantz) 7 Absorption and fluorescent spectra of certain naphthalene hydrocarbons (Titeica) 3 Carotenoids from the anthers of *Lilium tigrinum*—anthraanthanthin (Karrer, Oswald) 11D Photochem. stability of crotonaldehyde (Blacet, Roof) 3 Thermal decompn of certain gaseous org compds (Travers) 2. Decompn of propane and n-butane and C filaments (Belchett, Rideal) 2 Cryst. derivs of vitamin E (Kimm) 11E Structure and absorption of benzocyclopentanone oximes (Ramart Lucas, Hoch) 3 Polarographic studies with the dropping-Hg cathode 11I Electroreduction of benzaldehyde (Tokunaga) 4 Effect of addn salts on the electrolytic reduction of 3,5-dinitro-o-cresol (Bradt, Linford) 4 Sepn and utilization of o and p nitrotoluenes from the mononitrotoluenes prepd from the gasoline fraction of Syukkkō crude oil II Prepn of toluidine 11I Prepn of safranine and magenta (Mizuta) 22

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**Cyclohexylphenols.** Karl Brodersen, Hermann Heinicke and Ernst Korten (to General Aniline Works). U. S. 2,025,271, Jan. 21. See Fr. 765,024 (C. A. 29, 6741).

**Cleina.** H. Th. Dulon A. G. Brit. 430,745, Oct. 9, 1935. High-mol. olefins are prep'd by heating secondary amines having at least 1 aliphatic chain of more than 8 C atoms with compounds of an acid nature that form salts with amines, until the amino group is eliminated. Among examples, (1) heptadecylphenylamine is heated in a stream of HCl, heptadecylene and heptadecyl chloride distill over, the latter may be converted into heptadecylamine by treatment with HCl, and (2) undecylphenylamine is heated with ZnCl<sub>2</sub> to give pure undecylene. **Sulfating olefins.** Robert F. LeBaron (to Standard Alcohol Co.). U. S. 2,025,230, Jan. 21. For sulfating secondary butene contained in mixed petroleum cracking products, a fraction of the cracked products is used, consisting predominantly a mixt. of branched and straight-chain olefins contg. 4 C atoms per mol. and this fraction, in liquid phase, is admixed with aq. H<sub>2</sub>SO<sub>4</sub> of 50-60% strength, the aq. acid layer formed is removed, and residual hydrocarbons, in liquid phase, are treated with aq. H<sub>2</sub>SO<sub>4</sub> of 65-70% strength.

**Ethyl alcohol.** A. A. Vansheidt and E. M. Kaganova. Russ. 38,143, Aug. 31, 1933. Glycerol is treated with 0.5-2% H<sub>2</sub>SO<sub>4</sub> soln., under slight pressure and temp. Cf. C. A. 29, 4814.

**Alcohols.** Heinrich Hoff (to I. G. Farbenindustrie A. G.). U. S. 2,025,018, Feb. 4. Alcohols such as phenethyl alcoh. are produced by acting with an alkylene oxide such as ethylene oxide in the presence of a Friedel-Crafts condensing agent such as anhydrous AlCl<sub>3</sub> on a hydrocarbon such as C<sub>6</sub>H<sub>6</sub>, a nonsubstituted heterocyclic comp'd. such as pyridine or quinoline or a monosubstituted deriv. such as a monosubstituted benzene, at temps up to 100° (usually at 6° in making phenethyl alcoh.). Numerous examples are given.

**Dehydrating alkyl alcohol.** A. A. Dymanin-Lov. Russ. 37,689, July 31, 1934. Aq. alkyl alcoh. and CCl<sub>4</sub> are continuously dist'd at 65-6° with a continuous return of a part of the distillate into the flask.

**Converting alcohols to sulfates.** Carbide & Carbon Chemicals Corp., 729,408, Oct. 29, 1955. Alcoh. contg. at least 8 C atoms are converted to sulfates by treatment with a sulfating agent such as ClSO<sub>3</sub>H or SO<sub>3</sub> in the presence of an inert liquid such as an ether which is a solvent of high vapor pressure for the alcoh. and the sulfating agent, particularly a hydrochloroether. Examples are given of the conversion of phenethyl isobutylcarbinol, (4 heptyl dromethylethylisobutylcarbinol, and 4-ethyl-3-methyl-1-undecanol, in br'd (chloroethers) ether. Temps. from -20° to 100° are used.

**Ketones from secondary alcohols.** Francis M. Archibald and Clayton M. Palmer (to Standard Alk. Co.). U. S. 2,025,267, Jan. 21. A ketone is formed by passing the vapors of a secondary alcoh. such as sec. hexyl alcoh. in contact with a catalyst of Cu<sub>2</sub>O, Zn 2S<sub>3</sub> and Sn 1.5%, Cf. C. A. 29, 1431.

**Catalytic oxidation.** N. V. de Batavische Petroleum Maatschappij. Fr. 788,021, Jan. 19, 1935. Primary and secondary unsubst. alcoh., particularly those which contain at least 4 C atoms and only 1 olefinic bond in the mol., are oxidized to unsat'd carboxylic comp'ds. by treatment with O in the presence of an oxidation catalyst at a high temp., e. g., about 300-500°. Thus, 2-methyl-1-propen-3-ol is oxidized to 2-methyl-1-propen-3-ol-2-ynoic acid, 2-methyl-1-but-3-en-1-ol to 2-methyl-1-buten-3-ol-2-ynoic acid, 3-allyl alcoh. to acrolein, 2-methyl-1-buten-3-ol to 2-methyl-1-buten-2-one, b. 117°, and 2-methyl-3-phenyl-1-propen-1-ol to 2-methyl-3-phenyl-1-propen-3-one, b. 200°. Cu, Ni and V<sub>2</sub>O<sub>5</sub> are used as catalysts.

**Secondary aromatic amines.** Waldo L. Seman (to R. T. Goodrich Co.). U. S. 2,024,642, Feb. 4. In forming a secondary amine such as *p*-phenylammonium chloride, a primary aromatic amine such as aniline is added to an aq. soln. contg. a polyhydric benzene such as hydroquinone to ppt. the polyhydric benzene as its amine salt, and the latter is heated to cause its condensation to produce a secondary amine. Several examples are given. Cf. C. A. 29, 376.

**Distilling methylamines.** F. J. du Pont de Nemours & Co. Brit. 430,874, Oct. 21, 1935. Pure NH<sub>3</sub> or NH<sub>4</sub>Me is isolated from mixts. contg. them and NH<sub>4</sub>Me by dist. the mixt. without adding NH<sub>3</sub> at elevated pressure until a portion of 1 of the amines is collected. The residue of the mixt. to distill is 2 fractions, 1 consisting of pure NH<sub>3</sub> or NH<sub>4</sub>Me, according to the pressure used and the other an azeotropic mixt. of the amines. After 1 of the amines has been isolated, the pressure is lowered and the other amine dist'd off. App. is described.

**Tertiary amines.** I. G. Farbenindustrie A. G. Brit. 436,411, Oct. 16, 1935. These are obtained by the reaction of secondary amines, or reagents that form them under the reaction conditions, with alcoh. or their carboxylic esters, or with aldehydes or ketones, at a raised temp. in the presence of a hydrogenating catalyst, at least 1 of the reagents contg. a radical of at least 8 C atoms; the reaction is effected in the presence of H if when aldehydes and ketones are treated, in presence or absence of H when alcoh. are treated. Cf. C. A. 29, 5839.

**Quaternary ammonium compounds.** I. G. Farbenindustrie A. G. Brit. 436,725, Oct. 14, 1935. Such comp'ds. containing a radical of at least 1 high-mol. salt, aliphatic radical of at least 8 C atoms which is interrupted by a S or N atom, the remaining org. radicals attached to the N being salt radicals, are prep'd by (1) causing a reactive ester, e. g., a *n*-alkyl or benzene-sulfonate ester, to react with a suitable amine, (2) treating an amine contg. the high-mol. substituent with a reactive ester of a salt, alcoh., (3) causing an appropriate thio alcoh. or amine to react with a said amine contg. a radical with a replaceable substituent, e. g., Cl, or (4) treating a said amine having a replaceable substituent, an amine or H<sub>2</sub>S group, with a reactive ester of a salt, alcoh., the product, in each case, being converted into the quaternary form if necessary. The comp'ds. possess bactericidal and fungicidal properties. Trimethyl-1-dodecylthiomethylammonium chloride is prep'd by heating dodecylthiomethyl chloride, obtained by introducing HCl into a mixt. of dodecylmercaptan and CCl<sub>4</sub>, with NH<sub>4</sub>Me; and methylated dodecylthiomethylammonium chloride is prep'd by heating MeI with diethylthiomethylamine, which results from the interaction of Na dodecylmercaptan with diethylthiomethyl chloride. In 436,723, Oct. 14, 1935, such comp'ds. contg. attached to N at least 1 high-mol. aliphatic ether radical of at least 8 C atoms in which the O is resp. from the N by at least 2 C atoms, the remaining org. radicals attached to N being said radicals are prep'd by (1) causing a reactive ester of the high-mol. alcoh. contg. an O atom in the chain to react with a suitable amine, (2) treating an amine contg. the high-mol. ether radical with a reactive ester of a said amine, (3) causing an appropriate alcoh. to react with a said amine contg. a radical with a replaceable substituent, e. g., Cl, or (4) treating a said amine contg. a hydroxy alkyl group with a



reactive ester of a satd alc, the product, in each case, being converted, if necessary, into the quaternary form. The compds are bactericides and fungicides ( $\gamma$ -Dodecyl-oxy- $\beta$  hydroxypropyl)trimethylammonium chloride is prepd by treating  $\gamma$ -dodecyl-oxy- $\beta$ -hydroxy- $\alpha$ -chloropropene with  $\text{NMe}_3$  in cold  $\text{C}_6\text{H}_6$  soln.; methylcyclohexyl-11( $\gamma$ -dodecyl-oxy- $\beta$ -hydroxypropyl)ammonium iodide is prepd by heating cyclohexylethylamine with  $\gamma$ -dodecyl- $\beta$  hydroxy- $\alpha$ -chloropropene and treating the product with MeI. In Brit 436,793, Oct 14, 1935, such compds. contg attached to N an unsulfonated aralkyl radical and a high mol aliphatic radical of at least 8 C atoms, which may be interrupted by an O, S or N atom, are prepd. by (1) causing a reactive ester of the high-mol alc to react with an aralkylamine, (2) treating an amine contg the high mol substituent with a reactive ester of an aryl alc., (3) causing an appropriate alc or amine to react with an aralkylamine having an aliphatic radical contg a replaceable substituent, e.g., Cl, attached to the N, or (4) treating with a reactive ester of an aliphatic alc. an aralkylamine having attached to the N an aliphatic radical contg an amino, OH or SH group, the product in each case being converted into the quaternary form if necessary. Among examples, (1) diethylbenzyltrimethylammonium chloride is prepd by causing benzyl chloride to react with diethylolamine, obtained from octyl bromide and  $\text{NH}_4\text{Et}$ , (2) dimethyldodecylbenzylammonium iodide is prepd by the reaction of dodecylbenzylamine with MeI. The products are insecticides and fungicides Cf C A 30, 1049.

**Unsulfonated diacylated diamines** Guillaume de Montmolin, Jacob Danuser, Gerald Bonhote and Hans Jöhner (to Soc pour l'ind chim à Bâle) U S reissue 19,842, Feb 4. A reissue of original pat No 1,899,856 (C A 27, 2963).

**Formic acid** Henry Dreyfus U S 2,028,764, Jan 28. CO and water are made to react at temps of about 100–350° in the presence of a lower aliphatic acid such as formic or acetic acid and of  $\text{Cu}_2\text{Cl}_2$ . U S 2,028,765 relates to a similar process, carried out in the presence of  $\text{Cu}_2\text{O}$ .

**Acetic acid concentration** Donald F Othmer (to Eastman Kodak Co.) U S 2,028,800, Jan 28. A still is charged with aq HOAc and just the right proportion of Pr or iso Pr acetate or propylene chloride to form an azeotropic mixt with the water in the aq. acid, water and water withdrawing agent are distd from the mixt and the distillate is condensed and the added agent is sep'd from the water of the distillate and returned for continuation of the process (various details of app and of continuous operation being described) U S 2,028,801 relates to the use of added agents such as iso-Pr iodide,  $\text{CH}_2\text{BrCl}$ , iso Bu bromide,  $\text{CH}_2\text{Br}_2$ , iso-Am chloride, BuBr or allyl iodide for removing water in a distn column, from aq HOAc. App and operative details are described.

**Hydroxy keto acid esters** Henry Gault Fr 784,101, July 22, 1935. Acyclic aldehydes or keto acid esters are fixed in aq medium without elimination of water. Examples are given of the prepn of  $\text{Ac}(\text{CH}_2\text{OH})_2\text{CO}_2\text{Et}$  (diacetate, b<sub>p</sub> 174°),  $\text{EtO}_2\text{CCH}_2\text{CH}_2\text{C}(\text{CH}_2\text{OH})_2\text{CO}_2\text{Et}$  (along with hydroxymethyl cyclohexanecarboxylic ester, m 79°),  $\text{CH}_2[\text{C}(\text{CH}_2\text{OH})\text{CO}_2\text{Et}]_n$ , in 104°,  $\text{AcCMe}(\text{CH}_2\text{OH})\text{CO}_2\text{Et}$  (oxime, m 165°),  $\text{AcC}[\text{CH}(\text{OH})\text{Me}]_2\text{CO}_2\text{Et}$ , m 33°,  $\text{AcC}[\text{CH}(\text{OH})\text{Me}]_2\text{CO}_2\text{Et}$  (its pyrazolone, m 275°) and  $\text{AcCMe}(\text{CH}_2\text{OH})\text{CO}_2\text{Et}$ , b<sub>p</sub> 118–20° Cf C A 29, 1390<sup>11</sup>.

**Aliphatic esters of carboxylic acids** William J Banister (to Commercial Solvents Corp.) U S 2,029,694, Feb 4. An ester such as Et, Me or Bu lactate, having a b. p. above 120° but not substantially less than 50° below the b. p. of the acid from which it is derived, is prep'd by introducing an alc such as Lt, Me or Bu alc into the acid such as lactic acid at a temp ranging from about the b. p. of the formed ester to approx 20° below the said b. p., and removing the resulting vapors of the formed ester, water and excess alc Cf C A 29, 692<sup>12</sup>.

**Esters of levulinic acid** Gerald J Cox and Mary L. Dodds (to Nacac Chemicals Corp.) U S 2,029,412,

Feb. 4. In forming esters such as 2 methylbutyl levulin-ate, a sirup of levulinic acid obtained by decomp. carbo-hydrates with an inorg acid such as by the action of HCl on a sucrose soln. is esterified with an alc. such as 2 methyl-butanol (suitably by heating in the presence of HCl).

**Cyanic and thiocyanic esters** Soc. anon des matières colorantes et produits chimiques de Saint-Denis and Jean Claudin Fr. 789,500, Oct 29, 1935. Such esters are prep'd by the action of CNCl on the compds RSH in which R is any radical giving to the mol the character of mercaptan, thiocarbonyl acid or their derivs. Thus, CNCl gives with a soln of Na dimethylthiocarbamate at 15–70°, tetramethylthiuram monosulfide. The formulae of a large no of compds which may be obtained are given, including S C(OX)<sub>2</sub>C(OX) S, (in which X is Me, m 54–5°, Et, m 55°, C<sub>2</sub>H<sub>5</sub>, m 54–5°, C<sub>3</sub>H<sub>7</sub>, brown oil, C<sub>4</sub>H<sub>9</sub>, yellow oil), S C(NMe)<sub>2</sub>SC(NMe)<sub>2</sub>, S m 109–103°, S C(NMe)<sub>2</sub>SC(Nt)<sub>2</sub>, S m 55°, S C(NMe)<sub>2</sub>SC(OMe) S, S C(NMe)<sub>2</sub>SC(OEt) S, S C(NMe)<sub>2</sub>SC(OCH<sub>3</sub>) S, S 41°, S C(NMe)<sub>2</sub>NHPh, m 127°, S C(NMe)<sub>2</sub>NMePh, m 79°, o-[S C(NMe)<sub>2</sub>O]C<sub>6</sub>H<sub>4</sub>NO, m 124°, o-[S C(NMe)<sub>2</sub>O]C<sub>6</sub>H<sub>4</sub>Me, m 55°, and p-[S C(NMe)<sub>2</sub>O]C<sub>6</sub>H<sub>4</sub>Me, m 93°.

**Hydrogenating cinnamic acid and its homologs** P. Ya Loshakov and D. M. Al'm-Guttsatze Russ. 37,706, July 31, 1934. Hydrogenation is effected in the presence of activated Al and in a medium of abs alc.

**Quinic acid** A. D. Lebedev, A. V. Lundkvist and I. A. Obergard Russ. 37,707, July 31, 1934. Cranberry juice is treated with chalk, filtered, the filtrate boiled and filtered while hot to sep Ca citrate. The filtrate is mixed with a satd soln of the basic Ph acetate, the ppt. of Pb quinate is filtered off and decomp'd with H-S and the obtained PbS is sep'd from the quinic acid soln which is conc'd till it crystallizes, or it may be ext'd with org. solvents.

**Cyanic acid** I. G. Farbernd A-G (Christian Steigerwald, inventor). Ger 620,906, Oct 30, 1935 (Cl 12p 10) Addn to 607,663 (C. A. 29, 4027<sup>9</sup>). The method of 607,663 is modified by carrying out the condensation in the presence of a diluent at temps. above 100° in a reflux condenser. Examples of diluents mentioned are C<sub>2</sub>H<sub>5</sub>Cl and C<sub>2</sub>H<sub>5</sub>Cl Cf C. A. 29, 480<sup>8</sup>.

**Ethers containing at least eight carbon atoms in the molecule** F. Herhard Elbel (to Jenkel & Cie. G. m. b. H.) U S 2,028,654, Jan 21. Ethers such as glycerol monododecyl ether, glycerol monotetradecyl ether and the like are readily obtained by allowing the esters of higher aliphatic or hydroaromatic acids and oxyacids to react with alcs or phenols in an alk. medium or with alcoholates or phenolates. Various examples with details and modifications of procedure are given Cf C A 29, 475<sup>7</sup>.

**The preparation of ethylene chloride and other chlorine derivatives** Gosudarstvennii Trst Rezinovoi Promyshlennosti (inventor A. I. Matsen). Russ 24,877, January 31, 1933. Cl derivs are prep'd from cracked gas (contg CO, 0.2, C<sub>2</sub>H<sub>4</sub>, 34, C<sub>2</sub>H<sub>6</sub>, 2.9, C<sub>3</sub>H<sub>8</sub>, 2.2, C<sub>4</sub>H<sub>10</sub>, 0.5, O<sub>2</sub>, 0.7, C<sub>2</sub> H<sub>2</sub>, 24.2, C<sub>2</sub>H<sub>2</sub>, 25.4, C<sub>2</sub>H<sub>4</sub>, 5.5 and N<sub>2</sub>, 2.5%) by chlorination in 2 phases. In the first phase the dry gases are admitted through the lower end of a tower heated to 35° and charged with a chemically resistant filler together with Cl<sub>2</sub> in amount insufficient to effect a complete chlorination, while liquid chlorinated hydrocarbons enter the tower through the upper end and move downward in countercurrent to the gases. In the second phase the chlorination is completed by contact in another tower of the semi finished product with Cl<sub>2</sub> dissolved in liquid chlorides, without an addnl introduction of Cl<sub>2</sub>. Details of the equipment are also covered.

**Ethylene oxide** N. V. De Bataafsche Petroleum Maatschappij Fr 788,975, Oct. 21, 1935. See Brit 434,011 (C A 30, 737<sup>9</sup>).

**Addition compounds of nitrated diphenyl ethers** Chem. Fabrik von Heyden A. G. (Armin Rost, inventor). Ger 620,761, Oct. 20, 1935 (Cl. 12p 1 01). The compds are obtained by the action of C<sub>6</sub>H<sub>5</sub>NO<sub>2</sub> or quinoline on diphenyl ethers contg more than two NO<sub>2</sub> groups. Thus, 2,4,6,2',4',6'-hexanitrodiphenyl ether is added drop by drop to C<sub>6</sub>H<sub>5</sub>NO<sub>2</sub> to give N 2,4,6 trinitrophenylpyridinium



**Decylene compounds** Carbide & Carbon Chemicals Corp. Fr 788,944, Oct. 19, 1935 1,3-Decylene aldol of the formula  $\text{CH}_3\text{CH}(\text{OH})\text{C}(\text{Ft})(\text{C}_4\text{H}_9)\text{CHO}$  is prep'd by condensing ethylhexaldehyde with AcH in the presence of an alk comp'd and subsequently neutralizing the resulting reaction mixt. This aldol is hydrogenated to give an ethylbutyl-substituted 1,3 butylene glycol of 10 C atoms, b<sub>p</sub> 132-7°, and this glycol may be esterified to form, e.g., the diacetate, b<sub>p</sub> 130-5°.

**4-Hydroxypyrene** I G Farbenind A-G Fr 789,049, Oct. 22, 1935 This is prep'd by diazotizing 3-aminopyrene-4-sulfonic acid, converting the diazo comp'd to pyrene-4-sulfonic acid by known methods and fusing this with caustic alkalis. The product, m 208°, dissolves in conc'd  $\text{H}_2\text{SO}_4$  giving an olive-brown fluorescence.

**Condensation products of aromatic hydroxy compounds with alkenols** Joseph B. Niederl U S 2,029,539, Feb. 4 Alkenols such as vinyl, allyl, crotyl, oleyl and phytlyl alcs or their aryl, halogen or nitro substitution products or the like, or substituted and nonsubstituted phenoxyoctadecyl alc such as phenoxyoctadecyl alc itself, *o*-, *m* and *p*-toloxyl-, thymoxyl, octylphenoxy-, chlorophenoxy-, nitrophenoxyl-, naphthoxyl- and such phenoxy-octadecyl alcs in which the Ph radical carries other reactive groups such as amino, hydroxy, carbonyl (aldehyde and ketonic) and carboxy groups, are condensed with  $\text{H}_2\text{SO}_4$ . Derivs of the oleyl alc such as its esters, notably its sulfuric acid ester or acetate can be used for condensation and in such cases, if hydrolysis is avoided, the corresponding hydroxyphenyloctadecyl alc esters are obtainable. Similarly the ether of oleyl alcohol can be condensed. The products are colorless or pale yellow oily or waxy solids, useful as antiseptics and germicidal agents. Their sulfonated derivs (sulfates or sulfonic acids) are wetting agents. With formaldehyde and the usual treatment they yield phenolic resins. They no longer contain the unsat'd bond of the oleyl alc. Structurally they are hydroxyaryl-octadecyl alc derivs contg one or more free phenolic hydroxyl groups. Several examples with details of procedure are given, and claim is made to hydroxyaryl stearyl alcs generally as being new compds.

**Naphthalene-1,2-dinitrile** Imperial Chemical Industries Ltd., Reginald P. Linstead and Eric F. Bradbrook Brit 436,661, Oct. 16, 1935, Fr 788,725, Oct. 15, 1935 This comp'd, m 190°, is obtained by causing the Na or K salts of 1-cyanonaphthalene-2-sulfonic or 2-cyanonaphthalene-1-sulfonic alc to react with NaCN, KCN or Na or K ferrocyanide at not less than 250°, preferably at reduced pressure and in an indifferent atm.

**Dibenzanthrone derivatives** I G Farbenind A-G Fr 789,451, Oct. 29, 1935 See Ger 621,475 (C A 30, 1067°)

**Terpene oxide solutions** I G Farbenind A-G (Stamlaus Deichsel, inventor) Ger 620,636, Oct. 24, 1935 (Cl 12o 25) Solns of the above in halogenated hydrocarbons are rendered stable by adding fat sol. dyes which absorb the chemically active light rays. Thus, a soln of the azo dye from *o*-anisidine and  $\beta$ -naphthol in  $\text{CCl}_4$  is used as the solvent for ascaridol. The soln is practically insensitive to light and will keep for 2 yr. Other examples are given.

**Organic antimony compounds** Société des usines chimiques Rhône-Poulenc Brit 436,742, Oct. 17, 1935. Na and Li antimonothiomalates, of therapeutic value, are prep'd by adding  $\text{SbCl}_3$ , preferably in  $\text{CHCl}_3$  soln, to an aq. soln of Na or Li thiomalate.

**Tetraalkyl lead** Louis S. Bake (to E. I. du Pont de Nemours & Co.) U S 2,029,301, Feb. 4 An org. halide such as  $\text{EtI}$  is brought into contact with a Pb mono Na alloy in the form of small substantially dust free particles of a uniform size (suitably about 0.03-0.12 in in diam.) Cf. C A 29, 817°

**Benzoyl persulfide** Theodore H. Rider and Robert Shelton (to Wm. S. Merrell Co.) U S 2,028,216, Jan. 21 Benzoyl chloride is caused to react with an aq. soln of Na H sulfide, the resultant reaction mixt. is oxidized with a mild oxidizing agent such as air and the resulting relatively

pure benzoyl persulfide is further purified by dissolving it in an org. solvent such as  $\text{CHCl}_3$  which is miscible with alc. but not with water, washing this soln with dil. aq. alkali, sepg. the soln and pptg. the benzoyl persulfide from it by adding an aliphatic alc. such as EtOH or MeOH which can be vaporized at a temp. below the decompn. point of the benzoyl persulfide.

**Substituted sulfides** Henkel & Cie G. m. b. H. (Eberhard Fillel and Alfred Krosthaler, inventors) Ger 620,889, Oct. 29, 1935 (Cl 12o 23 03) See U. S. 1,987,529 (C. A. 29, 1479°).

**Halogen compounds** N. V. de Bataafsche Petroleum Maatschappij Fr 789,289, Oct. 25, 1935 Polyhalo hydroxy compds or polyhalo hydrocarbons are prep'd by causing an unsat'd monohalide contg at least 4 C atoms, and in which the halogen atom is joined to a sat'd C atom to react with a reagent of the class comprising hypohalogenous acids, aq. solns of halogens, solns of hypohalogenous acids and hydrogen halides and org. hypohalides. Thus, isobutenyl chloride is converted to 1,3-dichloro-2-methyl- and 1,1-dichloro-2-chloromethyl 2-propanol, isopentenyl chloride to tertiary dichloroamyl alc., crotyl bromide to 1-bromo-3-chloro-2-butanol and a monochlorodisubstituted alkene to a comp'd  $\text{C}_5\text{H}_9\text{Cl}_2$ . The tertiary polyhalo alcs are intermediates for the prep'n of unsat'd aldehydes and ketones to be used for making resins, perfumes, etc. Fr 789,290 Unsat'd polyhalides are treated as in Fr 789,289 to give similar compds. Thus, a comp'd  $\text{CH}_2\text{Cl}(\text{CH}_2\text{Cl})\text{CHCH}_2\text{Cl}$  is converted to a tertiary tetrachloroamyl alc. of the formula  $\text{CH}_2\text{Cl}(\text{CH}_2\text{Cl})(\text{OH})\text{CHClCH}_2\text{Cl}$  Cf. C A 30, 1067°

**Polyhalogenated hydroxy compounds and hydrocarbons** Herbert P. A. Groll and George Hearne (to N. V. de Bataafsche Petroleum Maatschappij) Brit 436,337, Oct. 9, 1935 Unsat'd polyhalides are caused to react with hypohalous acids, aq. halogen solns., solns. of hypohalous acids and H halides or alkyl or aralkyl hypohalides in presence of  $\text{H}_2\text{O}$  to give polyhalogenated OH compds and (or) hydrocarbons. Among examples, (1) a cyclic system comprising reaction chamber, separator stage for breaking up emulsions in the reaction products and an absorption column for satg. circulating  $\text{H}_2\text{O}$  with  $\text{Cl}_2$  is used, the app. is charged with  $\text{H}_2\text{O}$ ,  $\text{CCl}_4$  and 1,4-dichloro 2-(chloromethyl)-2-butene, which are agitated in the reaction chamber and the  $\text{H}_2\text{O}$  circulated through the system,  $\text{Cl}_2$  being introduced continuously into the absorption column, the unsat'd halide is intermittently admitted to the reaction chamber.  $\text{H}_2\text{O}$  is introduced to keep the  $\text{HCl}$  of the circulating soln. at or below 1.13 N. The product sepg. into 2 layers and the lower, together with an  $\text{Et}_2\text{O}$  ext. of the upper, is dried and distd. to remove most of the  $\text{Et}_2\text{O}$  and  $\text{CCl}_4$  and then distd. at reduced pressure to yield a tetrachloro tertiary Am alc. and (2)  $\text{CHCl}_3$   $\text{CCl}_4$  is treated as in (1) to give  $\text{CHCl}_2\text{CCl}_3$ . Products of the process may be used as solvents and extractants, e.g., in refining mineral oils and purifying refinery and manuf'd gases. Cf. C A 30, 1067°

**Substituting halogen in the benzene ring by various groups** A. I. Poral-Koshitz, I. N. Vikhanski and I. M. Lzelev Russ 34,873, Jan. 31, 1932 Cl in the  $\text{C}_6\text{H}_5$  ring is substituted by OH,  $\text{NH}_2$ , etc., in the presence of emulsifiers, such as petroleum sulfonic acids, sulfonic acids, sulfuric acid esters of high molecular weight, fatty and aromatic alcs and hydroxy acids.

**Continuous decomposition of phenolates and naphtholates** N. V. Vitevskii Russ 37,709, July 31, 1931 The substances are first treated with  $\text{SO}_2$  traveling in the same direction and the excess of the gas is used counter-currently to the phenolates and naphtholates in a sep. app. The equipment is illustrated.

**Hydrogenation** Wilbur A. Lazier (to Canadian Industries Ltd.) Can 354,246, Nov. 19, 1935 Alkylcyclic acids, hydroterocyclic acids, or their derivs such as esters, chlorides or amides are hydrogenated under high pressure and temp. in the presence of a catalyst. E.g., high-mol. alcs are produced by reducing naphthenic acids with H in the presence of  $\text{Cr}_2\text{O}_3$  contg. a hydrogenating

metal at 200–400° and a pressure of at least 10 atm. *Cl. C. 29, 2176<sup>12</sup>*

Oxidizing unsaturated compounds. N. V. de Bontad-  
sche Petroleum Maatschappij. *Tr. 789, 633, Oct. 22, 1935*.  
Unsat. hydroxy compds. such as nonylic alcoh.,  
ethers and esters which may contain an unsat. tertiary  
C atom are heated with an acid catalyst, e. g., a mineral  
acid, or a salt of acid action in a liquid system which con-

tains an aq. or (and) org. solvent for the catalyst. Thus,  
 $\alpha$ -chlorobutanol is converted to methylacrolein with  
H<sub>2</sub>SO<sub>4</sub> as catalyst and isobutyric acid as solvent, and isopent-  
enol to methyl isopropyl ketone with H<sub>2</sub>SO<sub>4</sub> and AcOH.

Other examples are given.  
Crystallization of dextrose. Charles J. Conland (to  
International Patents Development Co.). U. S. 2,029,  
360, Feb. 4. See *Tr. 787, 060 (C. A. 30, 5895)*.

## 11—BIOLOGICAL CHEMISTRY

PAUL H. HOWE

### A. GENERAL

ARTIFICIAL DIETS

The formation of heavy hydrogen in growing organisms.  
H. O. Reitz and K. F. Bonhoeffer. *Z. physik. Chem.*  
A174, 423–434 (1935). cf. *C. A. 29, 5129<sup>1</sup>*.—This study  
was a continuation of previous work in which chlorophyl-  
litic plants were grown on nutrient salt soils. In this  
work water with a high content of D and practically  
pure D<sub>2</sub>O were used. It is possible to replace half of the  
H with D in the solid matter of the organism without in-  
jury to the latter. Further increase in content of D de-  
creases growth and at 50% growth ceases and the organism  
dies. Algae survive several days on nutrient salt  
soil in practically pure D<sub>2</sub>O and begin to grow again when  
transferred to water of low content of D. The D content  
of the water was held by a vapor pressure measurement  
described by P. Harteck. (*C. A. 29, 999<sup>2</sup>*) R. H. B.

The effect of heavy water at low concentrations on some  
microorganisms. G. Castellani. *Bull. soc. intern. micro-  
biol. Sci. nat.* 7, 396–400 (1935) (in French).—Heavy water at  
concn. 0.05–10% has no action on the growth of  
*Rhizobium radicola* and *B. fluorescent liquefaciens*.  
On the mycelium of *Penicillium herbarum* and of *Rhizoglyphus*  
sp. (isolated from the roots of *Eryngium Canadense*), it  
has a slightly stimulating action when the concn. is  
0.05–2%, but a very unfavorable action at 10%.

Isolation of nicotinamide from heart muscle. Richard  
Kuhn and Helmut Vetter. *Ber. 68B, 2374–31 (1935)*.—  
From a vitamin B concentrate free from B<sub>1</sub> and B<sub>2</sub>, ex-  
haustive extn. with CHCl<sub>3</sub>, followed by distn. of the ext.  
under a high vacuum gave a substance, C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>O, identi-  
fied as nicotinamide (I), in 12%. From 15 kg. of beef  
heart, 147 mg. of pure I was obtained. The absorption  
spectrum shows maxima at 2150 and 2900  $\mu$ . The concn.  
of I in the heart is significant because the heart stimulant  
coramine (N,N-diethylsuccinimide) has a similar con-  
stitution. W. F. Bruce.

Dielectric properties and chemical constitution of the  
phospholipids. Richard Kuhn, Isidore Hauser and Wanda  
Erdmann. *Ber. 68B, 2368–81 (1935)*. cf. *C. A. 30, 321<sup>1</sup>*.  
—The diel. const. of lecithin (I) isolated from egg yolk  
(cf. Levene and Roll, *C. A. 21, 2003*) was measured in  
alc. and in CCl<sub>4</sub> from 20° to 45°. I was dielectrically in-  
active in CCl<sub>4</sub>, but active in alc. It has a betaine form  
which in CCl<sub>4</sub> is a sood. (mol wt. about 3000) and in alc. is  
unimol. There is no good evidence for heteropolym-  
er formation. Cephalin in CCl<sub>4</sub> is also unactive.

W. F. Bruce.  
Biochemical synthesis of some  $\beta$ -glucosides. I.  
Vandeweyer, C. N. Jones and A. Kark. *Bull. soc. chim.*  
Rennes, 16, 151–61 (1934).—Correction. The  $\beta$ -gluco-  
side of ethylene glycol of C. A. 29, 4300<sup>1</sup> should be  $\beta$ -gluco-  
side of ethylene glycol mono- $\beta$ -ethyl ether, 15% mannose  
should be 15% mannitol and  $\beta$ -mannosomannoglucoside  
should be  $\beta$ -mannitolmannoglucoside.

L. T. C.  
A histological study of the effects of x-rays on frog skin.  
Amos E. Light. *Ann. Entomol.* 25, 701–714 (1935).—The ef-  
fects described are evidently due directly to the action  
of x-rays on the cells or to the action of the secondary  
cathode rays produced by the primary beam impinging  
on the tissue. According to the most modern viewpoint,  
"R" substance is liberated quite slowly from the injured

cells during a long period of time, causing the capillaries  
to dilate. Because of this gradual response of "R" sub-  
stance, the dilation persists and the power of contracting  
to the original state is lost as other cells grow and fill the  
dilated network forming the walls of the vessels. Thus  
action undoubtedly thickens the walls and makes them  
less permeable to the nutritive elements that are neces-  
sary for the growth of the tissue cells. Gradually some  
die, while others acquire abnormal forms of growth.  
X-ray shock and increased R elimination soon after a  
radiation might also be explained by the sudden release  
of "R" substance in more sensitive tissue. G. L. C.

The action of short radio waves on myxoma. A. L.  
Rosenfeld and E. S. Sumner. *J. Nat. (U. S. S. R.)*  
10, 602–704 (1935).—The action of short radio waves  
(5–10 m) was tested on blood catalase, peroxidase, trypsin,  
amylase, anisoylase and the oxidation of enzyme applied.  
No direct chem. action was observed. In cases where  
changes were noticed, these were due either to a rise in  
temp. or to the laetinal action of the radiation. The  
action of short radio waves on living tissue is due to the  
overheating of the capillaries and to the disintegration  
of the intracellular structure, which leads to the destruc-  
tion of the cells and of the organism. H. Cohen.

The protein from *Aspergillus oryzae*. P. D'achenko.  
*Trudn. Lab. Isucheniya Belka Belkovo Obmena Organizma*  
1935, No. 8, 70–81.—The "proto album" was prepd. by  
Ferry's method (cf. *C. A. 29, 6612<sup>1</sup>*) in a. by extn. with  
dil. NaOH and pptn. with AcOH at a pH of 4.6. The pro-  
tein was not obtained in a sufficiently pure form; the N  
of the various prepns. varied from 12.25 to 15.27%.

R. Cohen.  
Caseinic acid in tissue culture. V. Trutskii. *Trudn. Lab. Isucheniya Belka Belkovo Obmena Organizma* 1935,  
No. 8, 30–40.—The intravenous injection of casein in fairly  
large amts. is not accompanied by any ill effects (cf. *C. A. 29, 6613<sup>1</sup>*). Hence a more sensitive bio. method was  
needed for the study of casein. Tissues were cultured  
in a 7% soln. of casein in the "natural solvent" (cf. *C. A. 29, 6612<sup>1</sup>*), as well as in a medium containing 0.7% casein  
soln. mixed with blood plasma (1). In the latter case,  
the growth of culture was very near normal, regardless  
of whether embryo tissue was used. No harmful  
effects of the casein on the tissues and cells could be ob-  
served. Casein, in various degrees of dispersion, can be  
obtained by adding the cald. amt. of HCl to a dil. Na  
caseinate soln. to liberate the free "caseinogenic acid". The  
addn. of 2 drops of coarsely dispersed "caseinemic acid"  
(1:1000) to 10 drops of blood plasma stimulates the  
growth of the culture by 21.5% in comparison with the  
control. The addn. of a greater or smaller amt. of "casein-  
emic acid" to the plasma leads to less stimulation in growth  
or to none at all.

The "anti-complex" of egg white. S. S. Perov.  
*Trudn. Lab. Isucheniya Belka Belkovo Obmena Organizma*  
1935, No. 8, 64–74. cf. *C. A. 29, 6614<sup>1</sup>*.—The "anti-  
complex" was not isolated. Its presence was suspected,  
since the addn. of alkali to dialyzed egg white is attended  
by a decrease of the surface tension and an increase of  
the  $\eta$ . H. Cohen.

The chemical nature of the acid groups of proteins.  
M. L. Lurina. *Trudn. Lab. Isucheniya Belka Belkovo Obmena Organizma* 1935, No. 8, 75–81.—The acidity of

casein and other proteins is ascribed to the presence of aminodicarboxylic acids H Cohen

The titration curves of amino acid mixtures M. Lisitzin and P. D'yachenko *Trudus Lab. Izucheniya Belka Belkova Obmena Organizma* 1935, No. 8, 90-2 (1935), cf. preceding abstr.—Identical titration curves are obtained for casein and a mixt. of monoaminodicarboxylic acids and diamino monocarboxylic acids, in the proportion in which they occur in casein (23% glutamic acid, 1.8% aspartic acid, 5% arginine, 2.0% histidine and 6% lysine) H Cohen

The Stanek and Hausmann numbers of some "proto acids" I. Leont'ev and G. Glukharev *Trudus Lab. Izucheniya Belka Belkova Obmena Organizma* 1935, No. 8, 93-101.—The "proto acids" of casein, egg white, peas and *Phaseolus aureus* R. yield about the same amts. of ammonio, monoamino, diamino and humus N, detd. by a modification of Hausmann's method. These 4 "proto acids" likewise consume the same amts. of  $\text{HIO}_3$  on oxidation by Stanek's method (*C. A.* 26, 2141, 2941)

Yeast phosphatases Henry Albers and Irna Albers *Arkiv Kemi, Mineral Geol.* 12B, No. 3, 6 pp (1935); cf. *C. A.* 29, 7352.—A phosphatase which prefers hexosediphosphate and  $\beta$  glycerophosphate to  $\alpha$  glycerophosphate was prepd. from a Stockholm top yeast by enzyme action. The top yeast contained 8-9 times as much phosphatase as the bottom yeast. The autolyzed soln. of top yeast contained appreciable quantities of dehydrogenases. The cell residue, which contained the greater part of the phosphatases, was washed, suspended in water, and stirred with dry green malt or green malt ext. in the presence of PhMe for 2 days ( $\text{pH}$  6, temp. 34-5°). The liberated phosphatase was sep'd from the cell residue by centrifuging and clarified by filtering through diatomaceous earth. It may be kept under PhMe for months and has a tendency to increase in activity on standing. Phosphatase content is 3.6-15 phosphatase units per cc. (A phosphatase unit is the quantity that at  $\text{pH}$  3.8 and a temp. of 35° will in 1 hr. liberate from a 0.1% soln. of Na  $\beta$  glycerophosphate an amt. of phosphate equiv. to 0.1 mg. of P). On dialysis, Mg and phosphate ions are removed rapidly, but the activity remains const. for a time and then decreases gradually. Purification by dialysis is carried to the point where the activity begins to decrease. The product is practically inactive toward  $\alpha$  glycerophosphate but it splits both  $\beta$  glycerophosphate, which cannot be split by dehydrogenases, and hexosediphosphate at  $\text{pH}$  3.5-4.0. Mg ions retard its action. The name hexosediphosphatase is suggested for it.

A. W. Dexter  
The role of adenosinetriphosphoric acid in the enzymic dehydrogenation of hexoses Hans v. Euler and Erich Adler *Arkiv Kemi, Mineral Geol.* 12B, No. 6, 6 pp (1935).—In the system Robison ester (hexosemonophosphate) + dehydrogenase (from yeast) + flavin enzyme + coenzyme, buffered to  $\text{pH}$  7.6, methylene blue is decolorized in 4 min. Adenosinetriphosphoric acid cannot be substituted for coenzyme. When fructose is substituted for Robison ester, methylene blue is not decolorized unless adenosinetriphosphoric acid is present. Dehydrogenase, flavin enzyme and coenzyme must all be present also. With glucose, the action is similar but not so rapid (15 min. as compared with 4) suggesting that it may first be changed to fructose. Heating Na adenosinetriphosphate in neutral soln. at 80° for 15 min. does not destroy its activity, but heating in  $\text{N H}_2\text{SO}_4$  for 7 min. at 100° forms hydrolysis products that are inactive. A resynthesis in the sense of the Lohmann reaction (*C. A.* 29, 2185) did not occur, neither muscle- nor yeast-adenylic acid + creatinephosphoric acid, nor adenylic acid + creatine could replace adenosinetriphosphoric acid. Within broad limits, the reaction is independent of the concn. of inorg. orthophosphate. It is not retarded by 0.001 M KCN. The coenzyme can be replaced by Warburg's coenzyme but the Robison ester dehydrogenase cannot be replaced by the sp. alc. dehydrogenases. The Harden-Young ester is not an intermediate since it is not acted upon by the Robison ester dehydrogenase system.

Methylene blue can be replaced by  $\text{O}_2$ . Either aerobically or anaerobically, the speed of the reaction is dependent upon the concn. of adenosinetriphosphate. A. W. D.

Highly purified coenzyme preparations H. v. Euler, H. Albers, E. Albers, F. Schlenk and G. Günther. *Arkiv Kemi, Mineral Geol.* 12B, No. 4, 6 pp (1935).—Cryst. (unaxial needles) coenzyme preps (activity 100,000) were obtained from concd. aq. solns. by fractional pptn. with alc. Even the purest liberate  $\text{H}_2\text{S}$  on treatment with Zn and HCl. Possibly the  $\text{H}_2\text{S}$  results from the reduction of an S-S group. The KCN-Na nitroprusside test for S-S was adapted to detns. by the addn. of  $\text{NH}_3$  to stabilize the color. The relationship between the S and amt. of coenzyme values is practically const. regardless of the method used to prep. the pure coenzyme, so that the S-contg. substance seems to be a part of the coenzyme or at least very firmly attached to it. This may explain the reducing property of coenzyme toward reagents used in sugar detns. A. W. Dexter

The relationship of coenzyme and an inhibitory substance in yeast cells Hans v. Euler and Erich Adler *Arkiv Kemi, Mineral Geol.* 12B, No. 5, 6 pp (1935).—A lactic acid dehydrogenase (soln. A) was obtained from top yeast R. by autolysis with LiOAc, fractional pptn. with alc. and soln. of the second fraction in water. No coenzyme was detected in it until it had been heated at 70° for 10 min. The coenzyme in it dialyzes much less rapidly than a soln. of pure coenzyme; when the solns. are mixed, each apparently dialyzes at its own rate. A fermentation medium contg. glucose, apozymase, Na hexosediphosphate and buffered to  $\text{pH}$  6.4 was used to test its inhibitory effect. Soln. A (partially dialyzed) liberated nearly 9 times as much  $\text{CO}_2$  after its enzyme became active through heating than before. The unheated soln. also definitely inhibits the liberation of  $\text{CO}_2$  by pure coenzyme. It does not retard the coenzyme action in the alc.- or Robison ester-dehydrogenase system. A bottom yeast prep. also shows the presence of an inhibitory substance.

A. W. Dexter  
Porphyrin gelatin phosphors Fritz Bandow and Emil J. Klaus *Z. physiol. Chem.* 238, 1-13 (1936).—Phosphors are mixts. which emit an afterglow when the source of illumination has been removed. In gelatin phosphors the porphyrins show the same absorption and fluorescence spectra as in solns. The structure of the substance and state of ionization are therefore not materially altered when the pigment becomes a component of the solid basic material. The solvent used in the prepn. of the phosphor has an influence. Gelatin is *per se* alk. to the porphyrin, but by addn. of sufficient concd. HCl acid spectra can be obtained. By adsorption of the pigment the spectra are displaced to the long wave side. Influences of temp. and decompn. during the mild prepn. of gelatin phosphors are not a disturbing factor. A. W. D.

Urnary phosphatase. II Excretion in man Hajo Wolberg *Z. physiol. Chem.* 238, 23-30 (1936); cf. Kutscher, *C. A.* 29, 6615.—Men excrete 3-5 times as much phosphatase as women, because of admixt. of prostatic secretion. A further increase of 20-30% occurs during periods of fasting. A heavy intake of glucose decreases the phosphatase and increases the phosphate excretion in 75% of the subjects. After insulin injection the situation is reversed. With simultaneous administration of insulin and glucose and with const. blood-sugar level the insulin action predominates. A. W. D.

The chemical composition of the sturgeon protamines M. A. Lisitzin and N. S. Aleksandrovskaya *Z. physiol. Chem.* 238, 54-8 (1936).—Preps. of protamine sulfate obtained from the milt of 3 species of sturgeon, viz. *Acipenser huso*, *Ac. guldenstadti* and *Ac. stellatus*, showed complete agreement in elementary analyses, and in amine, arginine, histidine and lysine content. It is recommended that the protamine be designated *acipenserin* instead of sturin, husin, stellatin, etc. A. W. D.

Calcium and magnesium contents in the flesh of various animals Makoto Takamatsu *Z. physiol. Chem.* 238, 99-100 (1936).—Ca and Mg detns. were made on the muscle tissue of 20 species of animals comprising mam-

mals, birds, reptiles, fishes, mollusca and arthropods. The high Ca content of the muscle of the latter is added, snail, mussel and crab and the high Mg content of the adder and snail. The highest values, on the dry basis, were 1.3202% CaO and 2.7673% MgO in snail muscle.

**The preparation of  $\beta$ -thymonucleic acid by means of nucleotidase R. Feulgen Z. physiol. Chem. 238, 105-110 (1936), cf. C. A. 30, 1394.**—It is not possible to obtain the gelatinous  $\beta$ -form of thymonucleic acid in its native state such as occurs in the cell nuclei, since the process of extra cellular dephosphorylation. The best preps are therefore mixts of various dephosphorylated stages. 1 or prepn of the completely dephosphorylated  $\beta$ -form two procedures are applicable. If the cell nuclei have been sepd or are present in high concn, as for ex. in fish sperm, the material may be digested with pancreatin in the presence of  $\text{Na}_2\text{CO}_3$  and the filtrate after concn pptd with LiOH at alk. reaction, then reprecipitated at neutral reaction and finally dehydrated with LiOH. Where the cell nuclei are less abundant, as in the thymus gland, it is best to ext. with NaOH at 65° and ppt with LiOH, then depolymerize the gelatinous product with pancreatin and purify by extn as above. To obtain the free acid the pptd Na salt is dissolved in  $\text{H}_2\text{O}$  and pptd by HCl.

**Enzymes of fermentation. V. The phosphorylation systems of alcoholic fermentation. Antonicelli and Hilde Br. Z. physiol. Chem. 238, 111-231 (1936), cf. C. A. 30, 1397.**—Although the phosphorylation of hexose by means of yeast enzymes is a multistage process, it is extnt by hexosediphosphate (I) and by adenosinetriphosphate acid (II), the former is more effective in the case of purified enzyme system. After removal of heterophosphates a rephosphorylation is no longer effected by II, which in this system is entirely inert. The phosphorylation is therefore not identical with heterophosphorylation. Phosphorylation induced by I occurs in the absence of heterophosphates and also in the absence of II. There exist then 2 phosphorylation systems in yeast exts, which are mutually complementary. Evidence is now presented for the existence of a 3rd mechanism of phosphate transfer. The rearrangement of phosphorylserine acid to phosphorylserine acid is completely suppressed by NaF. On the other hand the phosphorylation induced by I in crude saccharose-yeast juice is only moderately inhibited by NaF, and the quantity of mobile P far exceeds the quantity of P in the added I, i. e. the action of I in the system inhibited by NaF is catalytic. If only the first 2 phosphorylation mechanisms existed this mode of reaction would be impracticable, since the catalysis by I would occur only when II could be regenerated. But the regeneration of II is completely annihilated by NaF. Hence it must be assumed that a 3rd mechanism of phosphate transfer exists, by which the mono ester is converted into di ester. Although the exts were made with cell-free exts, the utilization of inorg. phosphate induced by I may be of significance as a possible means by which the living cell meets its phosphate requirements. Moreover, the fact that this mechanism accelerates fermentation by the living cell. Phosphorylation of hexose by II is simply a rephosphorylation, not a binding of more phosphate.

**Bühselein Edgar Wemberger. Z. physiol. Chem. 238, 124-83 (1936).**—From 2.5 g of pallotones a small quantity of bilirubin was obtained by LiOH extn of the residue remaining after extn with LiOH, 5% AcOH, 5% HCl and  $\text{CHCl}_3$ . The pigment, which could not be crys'd or esterified, differs from bilirubin in its lower N content. It gives no Gmelin, diazo, Glutinski, Zn(OAc)<sub>2</sub> or pyrazole reaction. It has strong tenebral power and is undoubtedly a typical bile pigment. Occurring in stones from both Prague and Vienna it is not a local phenomenon.

**Isolation of adenosinetriphosphate acid from yeast Th. Wagner-Jauregg Z. physiol. Chem. 238, 121-30 (1936).**—From 200 g of fresh yeast some 60 mg. of adenosinetriphosphate acid was isolated as Ag salt and

found to be identical in all respects with a similar prepn from rabbit muscle. Inorg. pyrophosphate was also obtained, and a Ba salt from which 1/4 of the  $\text{H}_2\text{PO}_4$  was easily split off and which had a N/P ratio of 4.3; hence it was probably inosopyrophosphate, cf. H. W. D., 1815. **Electrolysis of oxytocin N. Das, B. N. Ghosh and U. C. Guha Z. physiol. Chem. 238, 131-2 (1936).**—Oxytocin preps were subjected to electrodialysis at various H-ion concns and with stoppage of the current at pH 9.0 and below, the hormone migrated to the cathode, at pH 10 small quantities were found both at the cathode and the anode. It is assumed that the hormone has a distinctly basic character and by adsorption on a basic carrier substance migrates to the cathode.

**Intestinal phosphatase H. G. K. Westphal Arch. uerl. med. physiol. 20, 506-90 (1935).**—The speed with which intestinal phosphatase splits off  $\text{H}_2\text{PO}_4$  from Na glycerophosphate decreases more and more with increasing reaction time. This cannot be explained by checking through the reaction products  $\text{H}_2\text{PO}_4$  and glycerol, by diminishing substrate concn, or through inactivation by contact with the buffer at pH 8.8. The speed with which  $\text{H}_2\text{PO}_4$  is split off under the influence of increasing phosphatase concn increases less rapidly than the phosphatase concn, that is, the activity of the phosphatase is not proportional to the phosphatase concn. A tonality between phosphatase concn and activity appears, if the phosphatase salts at the dilus in which they are to act on the substrate are mixed with the buffer and held for some time (1, 2 or 4 hrs.) at a constant pH. This method, however, has influence only on such phosphatase salts as are poor w. protein. There is no change in the relationships of the activities of the proton-poor phosphate salts which are prepared by a similar method from various rat intestines. For the present the customary method for the detn of intestinal phosphatase must be employed, i. e. the extn of the amt of  $\text{H}_2\text{PO}_4$  split off from a suitable substrate, over not too long a period of time. It is recommended to work in a Ca contg medium so that at least the checking action of the accumulating phosphate is eliminated.

**Repardities in the growth of tissue cultures. I. Analysis of the frequency of cell division. P. J. Gaillard. J. biopharma. 24, 282-403 (1935).**—Fr. D.

**Biological oxidations. V. Copper and hexachromogens as catalysts for the oxidation of ascorbic acid. The mechanism of the oxidation. L. S. Guzman Barron, R. H. DeLew and Frederick Klenz. J. biol. chem. 112, 623-40 (1936); cf. C. A. 29, 822.**—The so-called autooxidation of ascorbic acid (I) in carefully prep'd buffer solns, the catalysts which accelerate the oxidation, and the mechanism and chem. reversibility of the reaction have been studied under rigidly controlled exp'd conditions. I is not autooxidizable in solns up to pH 7.0. In alk. solns, it is autooxidizable, the rate increasing with pH. Salts of Mn, Ni, Fe, Cu, Ca and Zn were studied; only Cu catalyzed the reaction, its action being noticed in concns of 40 g per l. When catalytic effect is completely inhibited by 0.001 M KCN and 0.005 M  $\text{FeCl}_3$ , 95.5 mmts of CO and O. Thus the oxidation is performed in acid or neutral solns there is an uptake of 1 O per mol of I. The oxidized form of I can be completely reduced by  $\text{H}_2$  up to pH 8.0; above this amt recovered decreases until at pH 7.0 only 45% of the oxidized form can be recovered. I and O do not reduce I. The temp. coeff. for the Cu-catalyzed oxidation is 1.6. Electromotically active oxidation-reduction systems of suitable potential also act as oxidizing agents, hexachromogens of neocline, pyridine and picloric acid acting as catalysts. The mechanism of the oxidative reaction is as follows: I is oxidized to dehydroascorbic acid while the catalyst is being reduced, the reduced catalyst is then reoxidized by atm. O. The  $\text{H}_2\text{O}_2$  formed being split into  $\text{H}_2\text{O}$  and O. Addn. of amino acids retards the catalytic action of Cu, presumably owing to complex salt formation. NaCl had no effect.

**Chlorophyll derivatives. L. Marchlewski and W.**

Urbanczyk *Bull intern acad polonaise, Classe sci math nat* 1935A, 146-55(1935) (in English).—See C. A. 29, 4383<sup>4</sup>

Amylolytic and the phosphoric esters of starch and glycogen Hans Pringsheim and S. Ginsburg *Bull soc chim biol* 17, 1590-1600(1935)—By the use of a purified amylase prep'd from hog pancreas (method given) it was found possible to hydrolyze starch completely without liberating any free  $H_2PO_4$  from the phosphoric ester present in the starch. Under the same conditions glycogen was hydrolyzed with the liberation of only a small fraction of the combined  $H_2PO_4$ . L. E. Gilson

Dephosphorylation of starch by phosphatase Hans Pringsheim and Fritz Loew *Bull soc chim biol* 17, 1607-13(1935)—A very active phosphatase was prep'd from hog kidneys. By its use it was possible to set free much of the combined  $H_2PO_4$  of a boiled starch prep'n without hydrolysis of the starch. L. E. Gilson

Respiration model using sunflower-seed oil with and without carotenoids R. Retovsky *Bull soc chim biol* 17, 1614-29(1935)—When films of sunflower-seed oil, free from carotenoid pigments, were exposed to air in the dark or in bright light the oxidation rate rapidly increased to a max after a short induction period. When crude carotene was added to the oil there was almost no oxidation in the dark or in dim light even after 30 days. When the oil contg added carotene was exposed to bright indirect sunlight the induction period was prolonged several days until the carotene was bleached by the light, after which the oxidation rate increased in practically the same manner as that of the oil without carotene. L. E. G.

Isoelectric point of blood serum N. I. Joukovsky and W. A. L. Decker *Compt rend soc biol* 120, 805-8 (1935)—Bett serum was dild with physiol NaCl soln, a colloidal soln of Pt, Au, Se,  $FeO_3$  or  $TiO_3$  added, and cataphoresis observed under the microscope throughout the  $pH$  range 0-14. Despite the diversity of the substances added the curves obtained had many points in common. L. E. Gilson

Manganese in biology Ferruccio Marzetti *Ross clin terap sci affini* 34, 271-85(1935)—A comparison of various methods for the detn of a small amt of Mn showed that of Bertrand (cf C. A. 5, 2381) to be the most reliable. With this method to determine Mn, gram was shown to absorb Mn from the soil in proportion to the amt present therein. Fe was absorbed to a lesser extent and could not be substituted for Mn. Expts on rats showed that ingested Mn (in a special diet containing  $SiO_2$ ) was absorbed by the stomach and rectum but not by the other portions of the alimentary tract. The non-assimilable Mn was absorbed into the circulation and then completely excreted. Helen Lee Gruel

Peroxidase action I. The oxidation of aniline Philip J. G. Mann and Bernard C. Saunders *Proc Roy Soc (London)* B119, 47-60(1935)—Horseradish or turnip peroxidase readily oxidizes  $PhNH_2$  in the presence of dil  $H_2O_2$  at  $pH$  4.5, the soln becomes blue violet for a few sec., then brown, and a dark brown ppt. forms contg 2,5-diaminoquinoneimide, a mauveine, induline and ungreable aniline black. The primary product of oxidation is  $PhNHOOH$ , and  $p$ - $H_2NCH_2CH_2NHPh$  is an important precursor of the final products. The enzyme system converts freshly prep'd  $PhNHOOH$  into emeraldine, and 4-amino- $p$ -phenylamine first into the blue imide of Wiltstätter [quinoneimide-14(4-aminoanil)anil], then into emeraldine. Tenton's reagent ( $H_2O_2$  +  $FeSO_4$ ) converts  $PhNH_2$  into azobenzene, amino- $p$ -quinoneimide, and 2,5-diaminoquinoneimide. J. S. H.

Urinary phosphatase Waldemar Kutscher and Hayo Wolberg *Naturwissenschaften* 23, 558-9(1935), cf C. A. 29, 6615<sup>4</sup>—Detns of phosphatase at 2-hr interval in human urine showed variations: high early day meals, min at noon, rise after meal and drop toward evening. Starvation results in a const phosphatase level. Glucose diet gives a decreased phosphatase content. It is concluded that phosphatase takes part in the resorption of sugar in the kidney ducts. Abnormally high phosphatase concn is found after eating fruit or

vegetables, after use of 100 mg ascorbic acid it is likewise but not always high. B. J. C. van der Hoeven

Base-protein acid compounds M. H. Fischer and W. J. Suer *Arch Path* 20, 683-9(1935)—After citation of some of the evidence which indicates that "native" proteins are base-protein acid compds, a method for artificial production is described. The end is accomplished by working with reaction mixts contg no free  $H_2O$  and by adding to any protein, e. g., casein, first a base and then an acid or vice versa. The allowable limit of  $H_2O$  content for these systems, in which such reaction was possible, was found to be well above the normal  $H_2O$  content of living tissue, while the amt of "salt" that could thus be bound to a protein, as well as the percentage relation of the base to the acid in such "salt," proved identical with biol values. Harnet F. Holmes

Glucosulphatase XIII. Contents of glucosulphatase and phosphatase in various invertebrates Tokuro Soda and Saitaro Koyama *J. Chem. Soc. Japan* 58, 1338-9 (1935), cf C. A. 29, 4380<sup>4</sup>—The content of glucosulphatase was highest in sea-ear, horned top and scallop. The content of phosphatase was not parallel with that of glucosulphatase, but in many cases was inversely related. The activity of glucosulphatase of 1 variety was strong at  $pH$  4.3, that of another at 9.3. There was no definite relation between the dried wt of organisms and the activity of the enzyme. K. Kutsa

Tyrosinase action on mono- and dihydric substrates Mark Graubard and J. M. Nelson *J. Biol. Chem.* 111, 757-70(1935)—Tyrosinase apparently consists of only 1 enzyme as claimed by Pugh (C. A. 25, 527). Differently treated enzyme preps such as plain potato juice, enzyme obtained from the alc. ppt. in dialyzed and undialyzed states, preps obtained through further treatment by alumina adsorption and elution and finally enzyme prep'd by further treatment with adsorption on kaolin failed to change the relationship in their behavior to  $p$ -cresol and catechol. Also when the enzyme is inactivated toward catechol it is invariably inactive toward  $p$ -cresol as well. The more purified enzyme preps are less resistant than the cruder material at  $pH$  values that are more acid or alk than the optimum. The  $p$ -cresol-enzyme reaction seems to proceed differently from the catechol-enzyme reaction since catechol very rapidly inactivates the enzyme while  $p$ -cresol does not. With cresol the rate of reaction, not the total  $O_2$  consumed, is proportional to the enzyme concn but with catechol the total  $O_2$  consumed depends entirely on the amt of enzyme (and also  $pH$ ) added within the limits allowed by amt of substrate. Removal of  $o$ -quinone by addn of  $PhNH_2$  or  $NaHSO_3$  does not inhibit the reaction or change much the nature of the catechol reaction. The major difference between the action of tyrosinase on  $p$ -cresol and catechol lies in the fact that the former does not block the enzyme while catechol, though it reacts much faster initially, produces some substance (other than  $o$ -quinone) or substances which inactivate the enzyme. Richter's suggestion (C. A. 28, 6736<sup>3</sup>) of 2 enzymes is not justified. The quantitative measurement of the enzyme tyrosinase *Ibid* 112, 135-41—A new unit is suggested for measuring tyrosinase activity, the magnitude selected being the amt of enzyme under specified conditions which will bring about 10 cu mm of  $O_2$  uptake in 1 min at  $pH$  6.2° and 2°. When low enough concns of enzyme are employed, the slopes of the curve are directly proportional to the amts of enzyme used. Three alumina-dialyzed preps of tyrosinase from mushrooms showed the same relative activities toward  $p$ -cresol and catechol, indicating that the enzyme activates  $O_2$  in both cases and that tyrosinase consists of only 1 enzyme. A. P. Lothrop

Pancreatic lipase I. S. S. Weinstein and A. M. Wynne *J. Biol. Chem.* 112, 651-8(1935)—Pancreatic lipase was prep'd from fat free dried pig pancreas by extn with 50% glycerol. The dry powder retained its complete lipolytic activity for more than a year when kept in a desiccator at 5° and the clear glycerol solns were stable for 6 weeks. The initial rates of hydrolysis of tripropionin, tributyrin, tricaproin, triacetin and tri-

valent decrease in the order named. The middle ester linkage of tracetin is apparently more slowly hydrolyzed than the end linkage since the ratio of the initial rates of acid liberation from equimol amts of mono-, 1,3- and tracetin were 1 : 2 : 6. The rate of hydrolysis of esters of monophase acids increased with an increasing  $\log$  of the  $\text{pH}$  of the acid radical and decreased with an increasing length of the alkyl radical. The initial velocity of hydrolysis varied directly as the enzyme concn and with tripropion was directly proportional to the concn of the ester, in the range corresponding to 0.016 to 0.10 M. Velocities calculated from the Michaelis equation did not agree satisfactorily with the observed velocities. The initial rate of hydrolysis of tripropion and Me. Iutyrate with phosphate buffer was greatest at  $\text{pH } 7.3$  but with glycine buffer the optimum was  $\text{pH } 9.3$ . Influence of various compounds on the hydrolytic activity. *Iod.* 64-66. The optimal rate of hydrolysis of tripropion by pancreatic lipase is affected by the presence of widely different chemical compounds. "Ketones and aldehydes were definitely inhibitory when their reactive groups were free, indicating that the CO group forms some sort of attachment with reactive groups in the enzyme mol., the inhibitory capacities of the closely related ketones and of aldehydes bore a more or less direct relationship to the mol. vol. of the compounds, the inhibitory power of the aldehydes is greater than that of related ketones having the same mol. vol. Heavy metals were inhibitory, copper activated the enzyme. The mechanism of this activation is not clear but preliminary experiments indicate that it is not entirely due to removal of copper from the substrate complex, but caused activation but apparently not, as in the case of urease, by reducing toxic quonones present as impurity to the corresponding alcs. PhOH and the cresols were relatively inert but the presence of the  $\text{NO}_2$  group increased the toxicity. The inhibitory capacity of the 3,4,5-trihydroxyphenols increased with increased  $\text{pH}$  of the OH groups. Guaiacol, in which 1 of the OH groups of catechol has been replaced by an  $\text{OCH}_3$  group, was non-inhibitory whereas catechol in the same concn caused retardation. Dihydroquinol and pyrogallol were not inhibitory. Thus, of the hydroxy phenols, only the dihydroxy groups were reactive. Monohalogen derivatives of AcOH caused inhibition in the order  $\text{I} > \text{Br} > \text{Cl}$ . It is impossible to decide whether reaction with the SH or the  $\text{NH}_2$  group or both takes place during the inhibition of lipase activity by the halogen acids. The susceptibility of the enzyme to inhibition by aldehydes and heavy metals suggests that an  $\text{NH}_2$  group may be an essential constituent, if so, the halogen acetic acids may quite possibly cause inactivation by union with this group. The halogen anions were inactive in the order  $\text{F} > \text{I} > \text{Br} > \text{Cl}$ . HCl salts present in digestion mixts buffered at approx. neutrality were without appreciable influence on the activity of the enzyme." Thirty-one references.

The constitution of the prosthetic group of catalase. *Kurt G. Stern, J. Biol. Chem.* 112, 631-639 (1936), *cf.* *C. A.* 27, 4820, 4821, 4824. Catalase has been isolated in crystalline form and identified as the proteolipin of Penicillium by pyren from it of the di-Me ester of meconophosphoric acid and by its conversion into the hemoglobin of the same species by substituting nitric acid for the enzyme protein. A method is described for concg and purifying catalase from yeast prepd from horse liver, 1800 g. yielded 3-6 mg. of crude enzyme hemm, and by working up 50 lbs. in 10 hrs approx. 40 mg. was obtained.

Photochemical processes in biology. *H. G. Othman, De Toni, Biochim. terrap. spec.* 22, 577-591 (1935), *C. A.* 30, 1657. A review.

Hydrogen-transferring coenzyme, its composition and mode of action. *Otto Warburg, Walter Christen and Alfred Grebe, Biochem. Z.* 282, 157-205 (1935) (See purest prepn of the H-transferring coenzyme from red blood cells is easily sol. in  $\text{H}_2\text{O}$ , does not co-precipitate with 10% N, and 12.5% P, and yields on hydrolysis  $\text{H}_2\text{PO}_4$ , pentose, adenine and the amide of nicotinic acid.  $\text{H}_2\text{PO}_4$

wt., corresponding to  $\text{C}_{10}\text{H}_{14}\text{N}_2\text{P}_2\text{O}_8$ , is 733 (calcd. from the lowering of the i. p., 87°). One  $\text{H}_2\text{PO}_4$  group is esterified, the other 2 are easily dissociable. A reactive fluorescent component is regarded as a denatured co-enzyme formed during prepn. The coenzyme is soluble in water that obtained from yeast and probably the same as the one from heart. It cannot replace coenzyme of fermentation, but chemically the 2 are closely related. Just as coenzyme becomes the fermentation coenzyme in certain bacteria with a particular protein, so this coenzyme becomes the "intermediate" enzyme only in combination with a sp. protein, and is found in all cells so far investigated. It acts on hexosemonophosphate, converting it to phosphohexuronic acid. However, intermediate enzyme and hexosemonophosphate in the presence of a very small amt. of coenzyme do not react either aerobically or anaerobically unless the O transferring enzyme (yellow enzyme) is added also, when the reaction  $\text{Co} + \text{RCOOH} + \text{H}_2\text{O} \rightarrow \text{H}_2\text{CO} + \text{RCOOH}$  proceeds. Therefore, the intermediate enzyme + coenzyme "activate" while the yellow enzyme "oxidizes" the substrate. If an equiv. amt. of coenzyme (Co) is used instead of a very small amt. the reaction proceeds very vigorously.  $\text{Co} + \text{RCOOH} + \text{H}_2\text{O} \rightarrow \text{CoH}_4 + \text{RCOOH}$ , the reduced coenzyme not being autooxidizable, but with a catalyst like the yellow enzyme (Z) it reacts as follows:  $\text{Z} + \text{CoH}_4 \rightarrow \text{ZH}_4 + \text{Co}$ , the  $\text{ZH}_4$ , however, being autooxidizable. The reduced coenzyme, the  $\text{H}_2\text{O}$ . If 1 mol. coenzyme, is added to 20,000 mols. hexosemonophosphate, the reaction comes to a stop when 1 mol. is changed to 1 mol. phosphohexuronic acid. On adding of yellow enzyme the product is 1 mol. of reduced mol. the entire hexosemonophosphate is changed to phosphohexuronic acid. The oxidation of the substrate is thus only a reoxidation of the coenzyme. The reversible reaction between coenzyme and hexosemonophosphate depends upon the presence of intermediate enzyme, but this can be replaced by  $\text{Na}_2\text{S}_2\text{O}_4$ .  $\text{Co} + \text{Na}_2\text{S}_2\text{O}_4 + 2\text{H}_2\text{O} \rightarrow \text{CoH}_4 + 2\text{NaHSO}_3$ . The coenzyme can also be hydrogenated by Pt and H in dil. alkali, but it takes up irreversibly 3 mols. H. In this reaction only the pyridine ring is hydrogenated to piperidine and the product is catalytically inactive. Also in the reversible hydrogenation the pyridine ring takes up 1 mol. H, because if such a product is hydrogenated with Pt + H it is now found to take up only 2 mol. H and to lose its catalytic activity. The coenzyme does not absorb the long waves and even in the ultra-violet range it absorbs only 260 m $\mu$ . When the pyridine ring is irreversibly hydrogenated to piperidine the 260 m $\mu$  band is lost, but when it is partially irreversibly hydrogenated this absorption band is unaffected; this is interpreted to mean that in the latter case the combination C = N remains unchanged. The absorption band at 345 m $\mu$  appears, which disappears again on oxidation by the yellow enzyme. This presents an interesting situation because the coloration is associated with reduction and disappears when the enzyme is subjected to the usual behavior of dyes. Nicotinamide sep. from the coenzyme behaves like the coenzyme on hydrogenation with Pt + H, but cannot be partially hydrogenated either by  $\text{Na}_2\text{S}_2\text{O}_4$  or by hexosemonophosphate + intermediate enzyme. However, a methyltetraene compd. of nicotinic acid (trigonelline) can be partially hydrogenated by  $\text{Na}_2\text{S}_2\text{O}_4$  and also manifests an absorption band at 350 m $\mu$ . Both the coenzyme and this compd. manifest great sensitivity to acid, the bands (345 and 350 m $\mu$ , resp.) disappearing. The anal. of H which the coenzyme can take up per unit of wt. is expressed by the formula  $20 (\text{C}_{10}\text{H}_{14}\text{N}_2\text{P}_2\text{O}_8 + \text{H}_2\text{O})$  and has been detd. experimentally. The coenzyme can be detd. by measuring its H capacity, but even smaller quantities can be detd. photometrically, when it is reduced with an excess of hexose monophosphate by the intensity of the 345 m $\mu$  band. Still smaller amts. (a few  $\times$  only) of the coenzyme can be detd. catalytically from the oxidation of a prepn. of known H capacity. The purest coenzyme prepn. turns Congo paper blue, is very sol. in  $\text{H}_2\text{O}$ , from which it can be pptd. by alc. or acetone, and is optically active ( $[\alpha]_{\text{D}}^{25} = -24.6^\circ$ ,  $[\alpha]_{\text{D}}^{25} = -29.6^\circ$ ).

S. Morrell



The mode of action of the "intermediate" enzyme. Erwin Negelein and Erwin Haas *Biochem Z.* 282, 206-20 (1935).—In the reaction coenzyme + hexose-monophosphate, the former becomes hydrated ( $\text{CoH}_2$ ) while the latter is oxidized to phosphohexuronic acid in the presence of the "intermediate" enzyme. The aim was to determine whether this acts as a catalyst or only as a colloidal carrier of the coenzyme. The method of measuring is based upon the property of the hydrated coenzyme to develop a band in the ultraviolet at 315 m $\mu$ , the rate of hydration varies with the rate of absorption. The time intervals were measured in sec., and the coenzyme concn was measured in 0.01 mg per cc. In the reversible system: Coenzyme + "intermediate" enzyme  $\rightleftharpoons$  H $_2$ -transferring enzyme, the latter is 0.5 dissociated at a coenzyme concn of about  $10^{-4}$  mol/l. Neutral  $\text{C}_6\text{H}_5\text{O}_2$ ,  $\text{H}_3\text{PO}_4$ , and neutral coenzyme were dissolved in 0.2%  $\text{NaHCO}_3$  satd with 5%  $\text{CO}_2$ -argon, warmed to 38°, intermediate enzyme was added ( $t = 0$ ) and the light absorption measured photoelectrically. The method of calcn is discussed in detail. The course of the reaction is formulated as:  $dC/dt = RC$ , where  $C$  is the concn of the free, nonhydrated coenzyme in mg/lcc. S Morgulis

Destruction of the hydrogen-transferring coenzyme by ultraviolet rays. Otto Warburg and Walter Christian *Biochem Z.* 282, 221-3 (1935).—Radiation of 1  $\gamma$  coenzyme in 1 cc  $\text{H}_2\text{O}$  at 30 cm with a quartz lamp destroys its catalytic activity in 15 min, but if the coenzyme is in a glass tube or the soln is 1000 times more concd (1 mg/cc) there is no measurable destruction in 15 min. The photochem destruction has been studied quantitatively; the radiation intensity is varied between  $10^{-4}$  and  $10^{-3}$  cal/sq m/min and the exposure time between 20 and 60 min. The destruction const  $K = s\phi$  (1/min), where  $s$  = quantum intensity,  $\phi$  = absorption coeff and  $\phi$  = the amt of substance destroyed. The time required for  $1/2$  destruction (1 cal/sq m/min) increases with the wave length and is 520 times as large at 283 m $\mu$  as at 186 m $\mu$ . The ratio  $K/s$  is neither equal nor proportional to  $\phi$ , from which the conclusion is drawn that not the total absorption coeff, but that of a special group in the coenzyme, is responsible for the destruction of the coenzyme. S Morgulis

Structure of desoxyribose from thymonucleic acid. Katashi Makino *Biochem Z.* 282, 263-4 (1935).—The desoxyribose does not give the boric acid reaction and is neutral to phenolphthalein. The formulas for guanine, inosine and thymine-desoxyribosides are given. S M

Carbon monoxide ferrogutathione. Fritz Kubowitz *Biochem Z.* 282, 277-81 (1935).—Ferrogutathione is a dissociable compd which requires a high glutathione concn to secure combination. The binding capacity for CO likewise increases with the glutathione concn until a satn is attained, when 2 mol CO is bound for each mol Fe. The equl where the concn of glutathione is sufficient to bind all the Fe is  $K = \beta^2(1 - \alpha)/\alpha$ ,  $\beta$  being the CO partial pressure, and  $K$  the disson const of the CO compd. The CO ferrogutathione is an orange compd which is decomposed by light, most of the CO taken up in the dark being set free. S Morgulis

Phytochemical reduction of lactaldehyde. Fijn Ochiai and Komei Miyaki *Biochem Z.* 282, 293-6 (1935).—D,L-Lactaldehyde is reduced by yeast to propylene glycol. S Morgulis

Increase in cell permeability through mutagenetic radiation. Chemical demonstration of mutagenetic effect. Irv R Bakhrumov *Biochem Z.* 282, 297-302 (1935).—See C. A. 29, 3693

Colloid-chemical studies of edestin. II. K Holwerda *Biochem Z.* 282, 317-41 (1935), cf C. A. 29, 8023<sup>1</sup>.—The peptization of edestin by salt solns is not dependent upon the amt of  $\text{H}_2\text{O}$  brought in with the adsorbed ions. In peptization the adsorption was always pos., whereas it is neg. in the salting-out process. In salt concns below the peptization region the adsorption becomes even more strongly pos. The fall in viscosity on either side of its max value is also explained on the basis of adsorption; it is assumed that an increase in surface tension

tends to decrease the viscosity vol. The influence of the lyotropic series on the peptization process is directly dependent on the adsorption. S Morgulis

Affinities between proteins and fat acids, fats or lipoids. St. J. Przylecki, E. Hofer and S. Frajberg-Crynberg *Biochem Z.* 282, 302-73 (1935).—The adsorption of ovalbumin, serumalbumin, edestin, casein, gelatin and peptone by paraffin, fats, fat acids and cholesterol has been studied. Paraffin adsorbs very well ovalbumin, serumalbumin and edestin (50% of a 0.3% soln) but casein, gelatin or peptones only poorly (less than 10%). The adsorption of cholesterol is similar. This adsorption is supposed to take place through the non-polar groupings of the amino acid, especially the leucine radical. The adsorption on acids and esters, on the other hand, is thought to be through the polar groups of the proteins. In the case of combination of protein with cholesterol, this may take place either through the  $\text{CH} = \text{CH}$  or the  $\text{OH}$  groups. S Morgulis

Thermodynamics of enzymic equilibrium. Studies on the aspartase system. Kurt P. Jacobsohn and João Tapadinhas *Biochem Z.* 282, 374-82 (1935).—The equl of the system  $\text{K} \rightleftharpoons [\text{aspartate}^-]/[\text{fumarate}^-] [\text{NH}_4^+]$  depends upon  $T$ . It has been detd at 37° and 5°, and from the reaction isochore the heat of amination has been calcd.  $-\Delta H = -1.939 \times 2.303 \times \log(130/140) \times [(310 \times 278)/(310 - 278)] = +6200$  cal. The aspartase is a true catalyst. S Morgulis

Stereochemical specificity and equilibrium in the fumarase system. Biochemical preparation of  $d$ -malic acid. Kurt P. Jacobsohn and Manoel Soares *Biochem Z.* 282, 383-6 (1935); cf C. A. 29, 825<sup>1</sup>.—Comparative studies of aspartase and fumarase lead to the presence of  $d$ -malate is shifted toward  $l$ -malate. S Morgulis

Oxidation, phosphorylation and fermentation by apozymase in the presence of some reversible oxidation-reduction systems. Åke Lennestrand and John Runnström *Biochem Z.* 283, 12-29 (1935).—Suspensions of brewer's dry yeast (washed 5-6 times) were used, pure preps of cozymase were employed whose concn was given by the no. of units per cc.; methylene blue and pyocyanine solns of 0.1% were used, but the 1-naphthol 2-sulfonate indophenol was of variable concn. In one set of expts. the formation of phosphoglyceric acid by dismutation and by oxidation was calcd. Especially in the system contg the pyocyanine good correspondence was found between the formation of phosphoglyceric acid and the phosphorylation. Apparently, phosphorylation comes to an end when no more  $\text{O}_2$  is taken up. Expts with naphthol-sulfonate indophenol and methylene blue agree as to the  $\text{O}_2$  uptake, but the phosphorylation is somewhat greater in the presence of the latter. No  $\text{O}_2$  is used in the absence of cozymase, but neither is it used in the absence of hexosediphosphate. A study of the respiratory quotient indicates that practically no  $\text{CO}_2$  is formed and the oxidative process was one concerned with intermediate steps, principally with formation of phosphoglyceric acid.  $\text{CH}_3\text{COOH}$  almost completely checks the oxidative process. The "induction" period is decreased in the following order: pyocyanine > methylene blue > hexosediphosphate, and the phosphorylation is also greatest with pyocyanine. S Morgulis

Separation of diketopiperazines and amino acids in protein hydrolyzates by means of ionophoresis. II. A. S. Balabukha Popitzova, N. I. Gavrilov and A. M. Rikaleva *Biochem Z.* 283, 62-70 (1935); cf C. A. 29, 2180<sup>1</sup>.—On subjecting the cathode fluid (obtained from the electrophoresis) to repeated electrophoresis it is possible to sep quantitatively the anhydrides from the amino acids of a mixt. As the cathode, Hg, Ag or Pt can be used, but not Ni because it causes losses in amino acids. From 10 to 13 ma per sq cm cathode surface is employed, and the soln in both the cathode and anode vessels is slightly acidified with 0.1 N  $\text{H}_2\text{SO}_4$ . Certain tech points in the procedure are discussed. S Morgulis

An attempt at the elucidation of the inhibiting action of oxidizing agents on proteolytic enzyme activity. The

nature of the effect of potassium bromate and similar substances on the baking property of wheat flour. *J. Højer Jørgensen. Biochem. Z.* 283, 134-45 (1935). *C. A.* 29, 5158.—It was found that yeast added to dough increases the activity of the proteases, because it brings in an activator, a pyruvate, which at least in part is glutathione.  $KBrO_3$  decreases the activity of the flour in an aqueous suspension, this effect being designated as BI (bromate inhibition). This inhibitory effect is greatly increased by the addition of yeast. This is attributed to the fact that in the presence of yeast the proteases which are inhibited by  $KBrO_3$  are being activated. If the proteases of an aqueous suspension of flour are completely or very nearly so destroyed by prolonged heating at 65°, the activity of yeast fails to increase the BI of the protease addition. The mechanism is supposed to be an inactivation by glutathione (phytylase). *S. Morgulis.*

**Hydrolysis of nucleotides by acid.** *Ritoshio Ichiikawa. J. Biochem. (Japan)* 22, 385-91 (1935).—The products of acid hydrolysis of yeast guanilic and xanthilic acids, as well as the liberation of purine bases and  $H_2PO_4$  from muscle adenylic and inosinic acid have been studied.

**Does an oxidation-reduction potential exist in the hemoglobin-methemoglobin system?** *Kochiro Saito. J. Biochem. (Japan)* 22, 395-397 (1935).—Either by titration of hemoglobin with  $K_2Cr_2O_7$  (CN) or by measuring the potential  $E$  of mixtures of hemoglobin and methemoglobin, the values for  $E$  were found to be independent of the (M/Hb) ratio. Only when the methemoglobin was titrated with  $Na_2SO_3$  was there occasionally a connection with the theoretical formula of the oxidation-reduction system. To explain these results, it is assumed that hemoglobin exists in 2 forms, as an active and an inactive product, only the former existing in reversible equilibrium in the reduction-oxidation system. Furthermore, the reaction active hemoglobin  $\rightleftharpoons$  inactive hemoglobin also forms an equilibrium. Hemoglobin prep. from blood corpuscles exists only as the inactive form. *S. Morgulis.*

**The influence of histolyses on enzymic processes.** *A. M. Uneykoff and N. S. Levantova. Med. exp. (Ukraine)* No. 1, 38-50 (1934).—The influence of hepatolysis on the glutathione content of dog blood depends on the total amt. present at the time of expt. If the total glutathione is fairly high, neither high nor low mol. fractions of hepatolysis produce any marked change, in low glutathione bloods a sharp rise is observed on introduction of hepatolysis. *Leo Naasarevich.*

**Phosphatase of the brain.** *K. Venkateshwar and N. C. Dutta. Current Sci.* 4, 424 (1935).—The alk. phosphatase from sheep brain has an optimal reaction of pH 9.6, and is activated by  $Mg^{++}$ ; the increases in activity of the enzyme 100% when  $Mg$  is added in optimal quantities (0.001 M, 0.002 M), the acid phosphatase, from the same source, which has an optimal reaction of pH 5.0 is not activated by  $Mg$  and resembles the urinary and salivary phosphatases. *Waldischmidt-Letz and Nonnenbruch. (C. A.)* 29, 5133.—They suggest that the acid phosphatase demonstrated by Bamann and Ruedel (*C. A.* 29, 4819) is due to the presence of erythrocytes in their extracts. This appears to be untenable, because the erythrocyte phosphatase is activated by  $Mg$  salts, while the acid phosphatase exd. from the brain is not so activated.

**The denaturation of egg albumin by ultraviolet radiation.** *Janet H. Clark. J. Gen. Physiol.* 19, 199-210 (1935).—The coagulation of whole egg albumin by ultraviolet radiation involves (1) the light denaturation of the albumin mol., (2) a reaction between the light-denatured mol. and water, possibly similar to heat denaturation but occurring at a lower temp., and (3) the flocculation of the denatured mols. to form a coagulum. Light denaturations is unimol., independent of temp., and occurs over a wide range of pH. The temp. coeff. of the reaction between a light-denatured mol. and water is  $10^{+4}$ . The reaction occurs rapidly at 40°, a temp. at which heat denaturation is inappreciable. *C. H. Richardson.*

**The altered effect of electrical stimulation on the metabolism of red cell suspensions.** *Erne Ponder and*

*John Macleod. J. Gen. Physiol.* 19, 265-81 (1935).—The apparent increase in rate of  $O_2$  consumption when an a. c. or induced c. current is passed through a red cell suspension (mammalian and nucleated red cells) in buffered  $CaCl_2$  soln. is not a metabolic effect. The current produces a permanent vol. decrease which has been interpreted erroneously as an increase in  $O_2$  consumption, for  $O_2$  utilization is not involved. The effects occur immediately after Pt electrodes are used, they disappear when the electrodes are platinumized, they are apparently related to obscure electrostatic phenomena. *C. H. Richardson.*

**Acetylation of tyrosine in pepsin.** *Robert Herriott. J. Gen. Physiol.* 19, 293-301 (1935).—Cryst. 60% active acetylpepsin has 7 Ac groups/mol. of pepsin, 3 of these groups are readily hydrolyzed in acid at pH 0.0 or in weak alkali at pH 10.0. The tyrosine-cryptophenyl content of this acetylpepsin is less than that of pepsin by 3 tyrosine equivalents. Hydrolysis of this acetylpepsin at pH 0.0 or 10.0 results in an increase in no. of tyrosine equivalents, hydrolysis at pH 10.0 also increases the sp. activity. Acetylation of the phenol group of acetyltyrosine by leucine under similar conditions shows a similar effect of pH on the rate. Conclusion: The Ac groups in 10% active acetylpepsin, which are responsible for the decrease in sp. enzymic activity, are 3 in no. and are attached to 3 tyrosine-phenol groups of the pepsin mol. *C. H. Richardson.*

**Biofluorescence II. The partial purification of Cyprindina luciferin.** *Rupert S. Anderson. J. Gen. Physiol.* 19, 301-51 (1935), cf. *C. A.* 27, 4201.—Powd. Cyprindina, exd. with  $EtOAc$  and  $CHCl_3$  was used in the prepn. of the luciferin ext. *Method.* The powder was exd. with boiling 0.1 M  $NaCl$  soln. contg. 10%  $EtOH$ , the filtrate being run into a chilled mixt. of 2.5 M  $HCl$  and  $BuOH$ . *Method 2.*—The solid residue was exd. with  $CHCl_3$ . *C. A.* 18, 2964, 26, 4351, involving extn. with  $MeOH$ , gave better yields and was used in the later work. The  $MeOH$  extn. is made under H for 24 hrs., with 5-10 cc.  $MeOH/g$ . Cyprindina powder. To the  $MeOH$  filtrate is added 25 cc.  $BuOH$ , the  $MeOH$  is removed from the de-aerated soln. *in vacuo* at room temp. The  $BuOH$  extn. is chilled and benzoylated with 2 cc.  $BzCl$  until the soln. contains no free luciferin. The soln. is then washed with 3 successive equal vols. of water to hydrolyze the excess  $BzCl$ . The  $BuOH$  fraction is then dissolved in 10 vol. of water. A highly colored material, apparently dissolved in the  $BuOH$  formed in the reaction, remains as a sep. phase. The inactive luciferin in this suspension is exd. with 80 cc.  $EtOAc$ , then with 3 portions of 40 cc.  $EtOAc$  each. The  $EtOAc$  fractions are cured, the  $BuOH$  fraction is removed. The residue contg. inactive luciferin in  $BuOH$  is mixed with 250 cc. 0.55 M  $HCl$  soln. and de-aerated at  $35-100^\circ$  for 1 hr., then cooled in an ice bath. The luciferin is exd. from the  $HCl$  soln. by 40 cc.  $BuOH$ , and then by four 20-cc. portions. The benzoylation and hydrolysis are repeated. The yield of luciferin is 50-65%. *C. H. Richardson.*

**Relation of oxygen tension and temperature to the rate of reduction of cytochrome.** *T. J. B. Suer. J. Gen. Physiol.* 19, 329-50 (1935).—The rate of appearance of the cytochrome C absorption band in a suspension of bakers' yeast contg. various mists. of  $O_2$ - $N_2$  was detd. at 6 temps. At each temp. there was a linear relation between the reduction time of cytochrome and the  $O_2$  tension. Absorption bands of cytochrome were seen when the  $O_2$  tension reached a crit. value  $sp.$  for each temp. This pressure is probably identical or very near to the crit.  $O_2$  tension usually found in  $Sh. O_2$  tension relationships. The  $O_2$  tension in the suspension is reduced by the respiratory activity of the yeast cells.—An equation is given with which rates of  $O_2$  consumption are related, from measurements of the reduction time of cytochrome. A rapid optical method of measuring  $O_2$  consumption is proposed for use with cytochrome-contg. microorganisms. More accurate detns. will be attempted with cytochrome and thermocouple amplification. *C. H. Richardson.*

**Carotenoid origin of cholesterol.** *W. M. D. Bryant. Chemistry & Industry* 1935, 1082.—Further evidence is adduced in support of the theory that substances having

the sterol skeleton are formed by cyclization and degradation of the polyene pigments (cf. Bryant, *C. A.* 30, 1044). In meeting Spring's objection (*Chemistry and Industry* 1935, 972) to this mode of formation, B. suggests that the Et and Me groups in the side chains of stigmastanol and ergosterol can be formed by "local synthetic action" or by ring opening of a more highly cyclized carotenoid deriv.

W. Gordon Rose

The preparation of sulfur derivatives of proteins by means of carbon disulfide. Jean Lorseleur. *Compt. rend.* 201, 966-8 (1935).—Alk. solns of gelatin, egg albumin and casein react with CS<sub>2</sub> to form S derivs of proteins. These derivs are sol in MeOH, EtOH and Me<sub>2</sub>CO. Ten g protein is dissolved in 100 cc 0.1 N soda, and 100 cc alc is added. Ten cc CS<sub>2</sub> is added to this soln in 10-15 min. The yellow soln is warmed under reflux 15 min at 40-45°, 1.51 0.05 N H<sub>2</sub>SO<sub>4</sub> is added to the cooled soln, resulting in the formation of a milky ppt similar to fibrin. Drying this ppt causes it to become insol in alc.

W. Gordon Rose

Autoclave splitting of protein by means of absolute methanol. V. S. Sadikov and V. A. Vadova. *Compt. rend. acad. sci. U. R. S. S. [N. S.]*, 3, 317 20 (1935).—Blood serum was dried at 120°. Two thousand g of this residue was heated with abs. MeOH in an autoclave 3 hrs at 180° (50-60 atm). The reaction products were completely sol in MeOH. 25.7% of protein N is converted to NH<sub>3</sub>. A cycloleucylvaline, C<sub>11</sub>H<sub>18</sub>N<sub>2</sub>O<sub>4</sub>, m 275.5°, was obtained from the water-sol fraction of the material remaining after removal of MeOH and NH<sub>3</sub>. It is sol in AcOH and CHCl<sub>3</sub>, slightly sol in ether and MeOH and insol in H<sub>2</sub>O and benzene. Four g dissolves in 100 g boiling abs. alc. After hydrolysis of 0.5 g by refluxing 36 hrs with 25% HCl, analyses of the Cu salts of the resulting amino acids were in accord with the theoretical values for leucine and valine.

W. Gordon Rose

Orienting rat feeding experiments with deuterium oxide. Signe Schmidt-Nielsen and Sigvald Schmidt-Nielsen. *Kgl. Norske Vidensk. Selskab. Forh.* 8, 27-8 (1935).—Rats receiving 10-50 mg D<sub>2</sub>O per day for 2 months grew and developed normally. Young rats were given a 20-50% D<sub>2</sub>O soln in H<sub>2</sub>O corresponding to 1.1-2.7 ml D<sub>2</sub>O per rat per day for 8 days. No abnormal effects were observed, except that the rats were unusually thirsty, this thirst persisting 5-6 days.

W. Gordon Rose

The distribution of choline. John P. Fletcher, Charles H. Best and Omood McK. *Solandt Biochem. J.* 29, 2278-84 (1935).—The total choline content of various rat tissues was estd by digesting the tissue in 18% HCl, acetylating the extd choline and assaying the resultant acetylcholine on the isolated rabbit intestine. The choline contents of 28 different tissues from white rats and of 34 dietary constituents including vitamin-rich foods and concentrates are reported.

E. W. Scott

The action of various chemical compounds on the vegetable phosphatases. Jean Courtois. *Compt. rend.* 201, 855-6 (1935), cf. *C. A.* 30, 490°.—The influence of glycerol, EtOH (90%), PO<sub>4</sub> (0.1 M), AsO<sub>4</sub> (0.02 M), NaF (0.002 M) and strychnine (0.004 M) on the reactions of takadiazase, and the phosphatases of white mustard seed and sweet almond with α- and β-glycerophosphates was studied. The influence of any compound depended on the origin of the enzyme (AsO<sub>4</sub> and PO<sub>4</sub> behaved the same to all 3). For a given enzyme, the same inhibiting agent influenced equally the hydrolysis of either of the 2 glycerophosphates.

E. W. Scott

Dicarboxylic acids as intermediate products in the biological breakdown of normal saturated fatty acids and their derivatives. P. E. Verkade and J. van der Lee. *Rec. trav. chim.* 54, 893 8 (1935).—V. and v. d. L. claim priority of their work over that of Fliscentrauer and Bertrand (*C. A.* 29, 6629°).

E. W. Scott

The destruction of certain hydrolyzing enzymes in the course of their action. Robert Bonnet and Berthe Natal. *Compt. rend.* 201, 1060-2 (1935).—Expts. on the hydrolysis of glucides by enzymes indicate that in every case there is an autodestruction of the enzyme during the progress of the reaction.

E. D. Walter

Oxidative inactivation of cozymase with Schardinger enzyme preparations. Fritz Lipmann. *Nature* 136, 913 14 (1935), cf. *C. A.* 28, 188°, 29, 8011°.—In a mixt. of a milk prepn of Schardinger enzyme and yeast macerate inhibition of glucose fermentation occurred after shaking with O for 1-1.5 hrs. Removal of the O did not restore fermentation. However, addn of a small amt. of boiled yeast macerate or purified cozymase did restore fermentation.

E. D. Walter

Chemical difference between protein linked and free nucleic acid. Gerhard Schmidt. *Science* 83, 15 (1936).—A comparative study was carried out on the effect of phosphatase on thymonucleic acid and thymonucleohistone. While free nucleic acid was nearly quantitatively dephosphorized within 24 hrs in a unimol reaction, only about 20% of the H<sub>2</sub>PO<sub>4</sub> was released from the nucleohistone under the same conditions. The nature of this chem. difference is discussed.

E. D. Walter

The aldehyde dehydrogenase (Schardinger's enzyme) of milk and the influence on it of copper and of bacterial activity. W. Ritter. *Landw. Jahrb. Schweiz* 49, 873 85 (1915).—This enzyme is definitely injured by small quantities of Cu, especially when present during low temperature pasteurization of the milk or cream. The methylene blue reduction time is increased. The growth of various organisms can counteract to a considerable degree this effect of Cu during pasteurization. The reason for this effect of bacteria is unknown, but it parallels the inhibitory influence of bacteria on the development of tallowy flavor in milk contg. Cu. H<sub>2</sub>O<sub>2</sub> is quite injurious to the enzyme. Metol, hydroquinone and the preparation of Kertesz (*C. A.* 29, 1525°) increase the methylene blue reduction time. Of the metals used in handling milk and its products, only Cu and its alloys have a marked effect on the Schardinger enzyme.

E. O. W.

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## B. METHODS AND APPARATUS

STANLEY R. BENEDICT

The estimation of the total lipides and the lipide partition in feces. Herbert C. Tidwell and L. Emmett Holt, Jr. *J. Biol. Chem.* 112, 607-131 (1936).—A method is described for determining the partition of fecal lipides which permits the estimation of the true neutral fat, unsaponifiable fat acid and soap fractions in a single sample (1.5-2 g. less if quite fatty). The lipides other than soap are first extracted with a fat solvent, weighed and then titrated with alkali to det. the free fat acid and (by difference) the so-called "neutral fat" (neutral fat plus unsaponifiable matter). The ext. is then saponified, the unsaponifiable material being ext. and weighed, all fractions can then be calculated except the soap. The residue from the 1st ext., which contains the soap, is acidified into fat acid, which is extd., weighed and calc'd. as soap. Added material can be recovered in the appropriate fraction with an error rarely exceeding 1%. A more accurate picture of the facts can be obtained by expressing the partition in abs. quantities rather than in percentages of fecal fat, which may lead to quite erroneous conclusions regarding fat splitting.

A. P. Lethrop

A new color reaction of the hexoses and polyhexoses and its application for the colorimetric determination of glucose in blood. Juan A. Sanchez. *Semana med.* (Buenos Aires) 1935, 11, 914-17.—A mixt. of 15 cc. H<sub>2</sub>SO<sub>4</sub> with 5 cc. of a soln. of not more than 0.0001 mg. glucose under the influence of the heat caused by mixing develops an intense red color. This color is caused by hexoses only, whether free or present in polysaccharides. The color is in proportion to the concn. For detn. of glucose in serum mix 1 cc. of CCl<sub>4</sub>-CO<sub>2</sub>H reagent, filter, wash and add H<sub>2</sub>O to make 5 cc. and pour on 15 cc. H<sub>2</sub>SO<sub>4</sub>. Shake and keep at 100° for 5 min. Cool and compare with a standard. Citrated blood serum cannot be used, but oxalated plasma is suitable. The standard is prep'd with glucose-free serum. Blood is incubated at 37° for 24 hrs. This destroys the sugar. After deproteinization, sugar is added in suitable quantities to make standards.

A. E. Meyer

Detg. *pu* with the glass electrode (Wolters) 2

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## C—BACTERIOLOGY

LAWRENCE H. JAMES

Carotenoids of purple bacteria. I. P. Karrer and U. Solmsen. *Helv. Chim. Acta* 18, 1506-15 (1935).—It is probable that the compn. of the carotenoid mixt. from purple bacteria depends on the nutrient medium and the strain. The following results are valid only for Gaffron's strain Z, cultured under definite conditions. The purple bacteria contained an entire series of previously unknown carotenoids which was sep'd into its components by chromatographic analysis. The alc.-washed bacterial mass was ext'd with CS<sub>2</sub> and the deep-red ext. was conc'd. and taken up in a ligron-MeOH mixt. On addn. of a small amt. of H<sub>2</sub>O the bacteriorhodospyll sep'd and the lower layer. The ligron soln. of carotenoids was washed and evap'd *in vacuo*. The product was partially sol. in petroleum ether. The insol. violet residue was crys'd. from benzene and yielded 20 mg. of glittering violet crystals of a new carotenoid, *rhodospillactin* (I), C<sub>42</sub>H<sub>54</sub>O<sub>6</sub> or C<sub>44</sub>H<sub>56</sub>O<sub>6</sub>, m. 218°, giving a blue color with SiCl<sub>4</sub> in CHCl<sub>3</sub> (absorption max. 642 mμ), absorption max. of I in CS<sub>2</sub>, CHCl<sub>3</sub>, benzene and abs. alc., 573.5-534-496, 541-507-476, 548-511-482 and 523-491-(465) mμ, resp. Absorption of the petroleum ether soln. in 8 tubes (60 cm. long, 5 cm. diam.) filled with Ca(OH)<sub>2</sub> and washing with petroleum ether gave a red-brown upper zone from which was recovered by elution with a mixt. of petroleum ether and MeOH one more benzene, black-red crystals of *rhodospill* (II), m. 159-60°, absorption max. in CS<sub>2</sub>, CHCl<sub>3</sub>, petroleum ether and abs. alc., 547-503-478, 521-486-453, 501-497-440 and 505-474-(445) mμ, resp. It is not hydrolyzed on treatment with EtONa. The second red zone consisted of a mixt. of carotenoids the bright red-brown zone was eluted, taken up in petroleum ether and evap'd and yielded, in fine needles, a third carotenoid, *rhodopurpurin* (III), m. 161-2°, absorption max. in CS<sub>2</sub>, CHCl<sub>3</sub>, petroleum ether and benzene, 550-511-479, 523-487-(458), 502-472 and 537-493 mμ, resp. Preliminary investigation of the purple bacteria showed the presence of a fourth pigment, *flavorhodin* (IV) with absorption max. in CS<sub>2</sub>, CHCl<sub>3</sub>, petroleum ether and abs. alc. at 502-472, 482-453, 467-437 and 472 mμ, resp., which are in close agreement for those found by Chagaff and Dieryck (C. A. 27, 747) for their extract from *Sarcina lutea*. Another carotenoid with absorption max. in CS<sub>2</sub>, 518-487 mμ significant of *β*-carotene was found in another previous ext. in which IV was not detected. The compn. of the carotenoid mixt. may also vary under the influence of unknown circumstances.

C. R. Adinolfi

Chaulmoogra oil and the morphological modifications of *Mycobacterium tuberculosis*. F. Baisemirali. *Boll. soc. microbiol. Ser. ital.* 7, 333.—Baisemirali's purified chaulmoogra oil has a specific action on *M. tuberculosis*, modifying its resistance to acids, and favoring the granular decomposition.

G. A. Bravo

Sulfur bacteria of the mud baths of Bormio. A. Monti. *Boll. soc. med. chir. Para.* 11 (1935), *Boll. soc. microbiol. Ser. ital.* 7, 333.—In the mucilaginous mud baths of Bormio S bacteria were found which utilize H<sub>2</sub>S produced by anaerobic microorganisms in the reduction of MgSO<sub>4</sub> and CaSO<sub>4</sub>. In these bacteria, S is contained in form of oil drops, which at the death of the micro-organisms are transformed into rhombic S. G. A. Bravo

*Corynebacterium diphtheriae*. Maria T. Cavassa. *Patologia* 27, 726-37 (1935).—Among the various media for the cultivation of *C. diphtheriae* (even with blood of rabbit, horse or sheep, instead of defibrinated ox blood) appears the best for the rapid growth of *C. diphtheriae*, but no medium could supply a very rapid diagnosis (e. g., within 12 hrs.). All media were *pu* always slightly lowered (5.0-6.8). The behavior of the various strains of bacterium toward the sugars is variable. Tryptone production is greater in the strains cultivated under aerobic conditions and at a *pu* near neutral point. The strains which show a tendency to acidify the

culture medium are unsuitable as producers of toxin

G A Bravo

Carbohydrate intolerance and intestinal flora II Bacteriologic studies of the fecal flora J B Gunnison, T L Althausen and M S Marshall *Arch Internal Med* 57, 100-106 (1936)

J B Brown

Methylene blue reduction by *Brucella abortus* and *Brucella melitensis* Bau Kien-Hun and Wang Kan. 7 *Hyg Infektionskrankh* 117, 379-402 (1937) —When the reduction of methylene blue by *Brucella abortus* and by *B. melitensis* was tested in the presence of various alcohols, sugars and amino acids, the former reacted more slowly

Rachel Brown

Hydrogen donors for *Staphylococcus aureus* cultures D Bach *Compt rend soc biol* 120, 608-10 (1935) —Methylene blue was used as the H acceptor. Of the 66 compounds tried as H donors the most active were Na lactate and the 15 common sugars rhamnose excepted. Most of the protein amino acids and the Na salts of common organic acids were inactive. Development of dehydrogenases by *S. aureus* during growth. *Ibid* 673-4. —The 12 hr cultures contained more dehydrogenase than the 48 hr cultures

L E Gilson

Changes in the pH of the culture medium during the production of staphylococcus toxin J Schwartz *Compt rend soc biol* 120, 1085-6 (1935)

L E Gilson

Fermentation of glucose by bacteria of the coli aerogenes group Delia Rosa Canepa and Carlos S de la Serna *Folia biol* 1, 238-43 (1935) —*Escherichia coli*, *Aerobacter aerogenes* and a no of intermediate forms all produce  $CO_2$ ,  $H_2$ ,  $EtOH$ , acetylmethylcarbinol, 2,3-butylen glycol and formic, acetic, lactic and succinic acids from glucose, but the relative proportions of these products formed are different for each species. This offers a possible means of identification

L E Gilson

Incubator hygiene IV The virucidal effect of formaldehyde on fowl pox virus Robert Graham and E H Barger *Poultry Sci* 15, 48-52 (1936), cf C A 28, 4447. —Fowl pox virus in a 1% aq suspension on cotton squares, on the feet and down of day-old chicks, upon being subjected to routine incubator fumigation ( $CH_3O$  released from cheesecloth in ams of 20 cc/100 cu ft) survived 30 min and often 45-50 min but was consistently noninfective after 90 min. There was no evidence that fowl pox virus rendered noninfective by  $CH_3O$  fumigation retained antigenic properties

K D Jacob

Phosphoglyceric acid in the dissimilation of glucose by *Citrobacter freundii* C H Werkman, E A Zoellner, Henry Gilman and H Reynolds *J Bact* 31, 5 (1936) —Phosphoglyceric acid was isolated as an intermediary in the dissimilation of glucose by *C. freundii*. This organism was shown to be able to convert phosphoglyceric acid into pyruvic acid. This may be the same as the mechanism of muscle glycolysis

John T Myers

Hypersensitivity to certain bacterial extracts, as demonstrated by changes in the nonfilament filament ratio of neutrophilic leucocytes Mervin H Stiles *J Bact* 31, 62-3 (1936)

John T Myers

The immunizing antigen of pneumococcus Types I and II Lloyd D Felton and Benjamin Prescott *J Bact* 31, 60 (1936), cf C A 29, 2195. —When Type I pneumococcus polysaccharides were treated with 0.1 N NaOH at 100° or  $NH_4OH$  at 4°, there was a drop in the amt of acid in an acid distillate from 7.3 to 0.8%, in the -oxyl group % from 2.9 to 0, in bisulfite addn in a 10 mg sample from 11.9 to 0.23 cc in I addn from 1.3 to 0.03, and in glucose no from 15.0% to 0. Type II polysaccharides showed similar changes

John T Myers

The existence in the *Phytomonas tumefaciens* of an endotoxin capable of causing the formation of tumors in plants André Bonvin, Max Marbe, Lydia Mesrobian and Petre Juster *Compt rend* 201, 984-6 (1935) —Elementary microanalysis of the complete antigen of *Phytomonas tumefaciens* gave the following values: C 44.0, H 7.4, N 1.7 and P 1.0%. Acid hydrolysis yields 40.8% reducing sugars and 18.1% fat acids. Injection of this antigen in the stems of *Helianthus annuus* induces the

growth of tumors in the cortex of the stem, similar to those produced by injection of the live bacillus W. G. R.

Physiological studies of several pathogenic bacteria that induce cell stimulation in plants J A Pinckard *J. Agr. Research* 50, 933-52 (1935). —Routine physiol studies on certain common bacterial media showed that each of the organisms examd gave characteristic growth reactions distinctive for each organism except the oleander and olive-knot bacteria, which showed similar cultural characters in all these media. The H-ion concn at which growth was prevented in liquid culture varied from pH 3.6 to 4.4 in the acid range and approx 9.5 to 10.5 in the alk range. Comparative utilization of several types of compds indicated that oxamide, L-tyrosine and L-cystine were the only sources of N used in which all the organisms distinctly produced similar reactions, and that the only compds of C in which similar reactions were produced were starch, pectin, phlorizin and the Na salts of formic, acetic, propionic, glycolic, malonic, succinic, tartaric and malic acids. H ion concn of the sterile liquid medium contg yeast ext., glucose and mineral salts had a marked influence on the oxidation-reduction intensity of this medium. Strong acid reactions were accompanied by relatively strong oxidizing potentials. Measurements of oxidation-reduction potential made at frequent intervals with various growing cultures in a yeast-ext medium showed that all the organisms produced relatively strong reducing potentials in undisturbed liquid cultures

W. H. Ross

Enzyme formation and polysaccharide synthesis by bacteria Evelyn A Cooper and John I. Preston *Biochem J* 29, 2267-77 (1935). —Enzyme formation and polysaccharide synthesis by bacteria related to *B. mesentericus* and bacteria pathogenic to plants were studied. *Ps. pruni*, *Ps. prunicola*, *Ps. mors-prunorum*, *Phytophthora*, *Ph. campestris* and *Ph. syringae* with sucrose as a source readily formed a polysaccharide of the fructosan type which appeared analogous to the levan formed by *B. mesentericus* and *B. subtilis*. *Ph. phaseoli*, *Ph. solanaceorum*, *Erwinia carotolora*, *Ph. tabaci* and *Ph. marginalis* occasionally formed polysaccharides. *Erwinia amylovora*, *Ph. barketti*, *Ph. cannae*, *Ph. malvacearum*, *Erwinia phytophthora*, *Ph. stewartii*, *Ph. juglandis*, *Ph. tumefaciens* and *Radiobacter* gave negative results. Mannose and arabinose inhibited polysaccharide formation from sucrose by *B. mesentericus*, *B. megatherium* and *Ps. pruni* and from sorbitol by *Acetobacter xylinum* apparently by a direct toxic effect on the cells of the organism. Glycerol, mannitol and sorbitol decreased the diastatic power of *B. mesentericus* cultures. *B. megatherium* and *Ac. xylinum* could not synthesize polysaccharides from Na acetate, malonate or succinate and these compds. had no effect on the synthesis from sucrose by *B. megatherium*. The peptone in the media could be replaced by asparagine, leucine or alanine without affecting the polysaccharide synthesis by these organisms. No evidence of dextran or pentosan formation by any plant pathogen was discovered

E. W. Scott

The action of certain sulfur compounds on the fermentation of glucose by propionic bacteria (*Propionibacterium* 11) Paulette Chair *Compt rend* 201, 857-8 (1935), cf C A 29, 6268. —By the previous technic, it was found that quantitatively cystine, cysteine and glutathione (I) imparted the same amt of activation to the fermentation. The optimum activation concn of I was found to be 1 in 25,000

E. W. Scott

Does the secretion of proteases exist in bacteria? Wacław Moysch *Compt rend* 201, 859-60 (1935). —The protease of *Serratia marcescens* was not present in fresh cultures, the amt of protease was less in agitated cultures contg more bacteria than in undisturbed cultures contg fewer cells and no protease was found in cultures killed with toluene,  $CHCl_3$  or  $Et_2O$ . Conclusion. Any protease present in bacterial culture is due to the solution of dead bacteria. Live bacteria cannot set free proteases

I. W. Scott

Vital staining of bacteria grown on media containing stains Andras Hegedus *Algyar Orvos Arch* 36,

499-401(1935).—Bacteria grown on media which contain stain concentrate the stain in their cells. 31 F

Cellular reactions to wax-like materials from acid-fast bacteria. The unsaponifiable fraction from the tubercle bacillus. Strain H-37. I. R. Sabin, E. C. Southam and R. M. Thoms. *J. Exp. Med.* 62, 75 (10/1935). The unsaponifiable fractions from the mycobacteria, though they are extremely stable, form emulsions and tend in H<sub>2</sub>O, are nevertheless remarkable stimulants of cells. They give rise to new monomers which surround these waxes and then flow into gas cells which engulf them. The property of adsorption of the waxes makes it possible to identify them within the giant cells which have phagocytized them. Within the foreign body granules the waxes are slowly disintegrated. They appear not to damage the cells and have no ill effect that they take no part in cell division. C. J. West

Certain chemical and physical factors causing bacteriolysis. A. C. H. Yen. *Trans. 14th Congr. Far East Assoc. Trop. Med.* 1, 303 (10/1935). The effect of electricity on bacterial suspensions is due to the chem. substances formed in electrolysis. Hypochlorite, hypobromite and hypiodite are highly effective since there is bacteriolysis only when these substances appear as products of electrolysis. The hydrolysis of urea accumulated in many cases of electrolysis within 1 hr. are insufficient to cause bacteriolysis on account of their low toxicity. I. S. Yen

The chemical influence of bacteria on blood pigments. Maxillole Kuntze. *Trans. 14th Congr. Far East Assoc. Trop. Med.* 1, 331 (10/1935). Hematin was produced by bacteria or water vibrio, and oxyhemoglobin will be changed easily under the complete aerobic condition to hematin and xanthomoglobin. I. S. Yen

The influence of etheral oil on the tuberculous bacilli and experimental tuberculosis. Karen Inama. *Trans. 14th Congr. Far East Assoc. Trop. Med.* 1, 323-23(1935). I. etheral oil shows sterilizing action on tuberculous bacilli as on colon bacilli. I. S. Yen

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## D—BOTANY

THOMAS G. MILLIOT

Carotenoids from the anthers of *Lilium tigrinum*. A new carotenoid. Anthraanthranin. I. Karrer and A. Oswald. *Helv. Chim. Acta* 18, 1303-1315 (1935). The anthers of 1200 flowers of *Lilium tigrinum* were vacuum dried in glass ampoules at 40° for 6 hrs. The 160 g. of dried material was extr. with petr. ether and the concd. ext. was taken up in 100 cc. of CH<sub>2</sub>Cl<sub>2</sub>. The deeply colored petr. ether ext. of phytoanthranin was dissolved in 200 cc. of petr. ether and upon with a vol. of 15 g. of Na in 300 cc. alc. Careful dist. with 100 cc. benzene for 2 days in a separatory funnel caused the carotenoids to sep. out as a red-brown ppt. between the petr. ether and alc. phases. The ppt. was filtered off, washed and freed from impurities by boiling with petr. ether. The washed ppt. was dissolved in hot 1:1 CH<sub>2</sub>Cl<sub>2</sub> and on standing in the ice-box gave 0.3 g. of colored crystals. Chromatographic sep. by absorption by CaCl<sub>2</sub> gave a small overhead zone of impurities, a long brown-red zone from which 0.1 g. of xanthanthranin (II) was afterward eluted, and a bottom zone of yellow anthraanthranin (I). The carotenoids were eluted with a mix. of benzene and 10% alc. 1 crystal, common to spindle-shaped crystals and compact clumps, m.

176° (mired in p. with paprika capsanthin, 176°). Its absorption spectra in CH<sub>2</sub> and CHCl<sub>3</sub> were in complete agreement with those of capsanthin from *Capsicum annum*. Apparently, this is the first appearance of this pigment elsewhere than in the species species (II). *Calliandra* (Calliandra) in 211° (cor.) is very similar to xanthanthranin, but contains 1 more O atom. It differs very slightly from it in m. p. and also in its absorption spectra. This previously unknown xanthanthranin may contain OH groups but the yield (25 mg.) was too small for further investigation. The CHCl<sub>3</sub> soln. gave a deep blue color with SnCl<sub>4</sub> (absorption max. at 587 mμ). When in a 1% soln. of H<sub>2</sub>O layered with concd. HCl, the acid bottom zone of the soln. takes on a blue color, changing shortly afterward to a greenish tint. C. R. Adair

Effect of artificial wounds on the ripening of some fruits. G. Leoncini. *Ann. sper. agrar. (Rome)* 17, 201(1935). 1. Plums, pears, apples and larks were wounded with a sterilized needle, then analyzed at various periods of ripening. The sugar contents increased, the free acids and tannin contents decreased, and the ripening was always accelerated. G. A. Bravo

Some aspects of the biochemistry of the olive fruit, with regard to the mannitol content. G. Leoncini. *Ann. sper. agrar. (Rome)* 17, 31-40(1935). The max. of mannitol content in the olive leaves was found in the 4 months of autumn and winter, when the plants show a less metabolic activity. The sum was found in the autumn (young period, together with a max. content of oleic acid). G. A. Bravo

Chemical researches on early- and late-ripening fruits. I. R. Nucumini and A. Zaccagnini. *Ann. sper. agrar. (Rome)* 17, 41-71(1935). —Ruits ripening in spring and summer (apricots, cherries, peaches, plums and grapes) contain less tartaric acid and more citric acid than late varieties, except grapes, apples and pears show a nearly inverse behavior. II. R. Nucumini and F. Cerri. *Ibid.* 45-61.—Male acid is always present in greater amt. in the early varieties of fruits, but the total content of the acids is greater in late-ripening fruits. III. R. Nucumini and G. Bucci. *Ibid.* 65-67.—In peaches, the ratio male acid/citric acid is 1:1, and the ripening period is 20-60 days; in apricots, the ratio is 1:1 and the period 70-80 days; in pears and apples the ratio is about 1:0 and the periods are, resp., 70-80 and 80-90 days; in grapes, the ratio male acid/citric acid/tartaric acid is 5:7:1:3, and the ripening period is 100-110 days. These facts show that male acid is utilized in plant metabolism more readily than other acids. IV. R. Nucumini, G. Marchetti and I. Bagnoli. *Ibid.* 63-71.—Complete ripening of the fruits during the total ripening period show a decrease in pectin, an increase in the 1:1-ol, mitters, an increase in the pectin substance, and an increase of the tannin up to a max., then a decrease. The total and protein N contents decrease during ripening, but the non-protein (probably amino) N content is const.

G. A. Bravo  
The ripening of the sorb apple. R. Nucumini and O. Bartoli. *Ann. sper. agrar. (Rome)* 17, 73-81(1935). —The acid contents decrease slowly during ripening of the apples. The glucose and fructose contents increase with a const. ratio. The sucrose and sorbitol contents increase during the first ripening of the fruits on the plant, but decrease when the fruits ripen on straw.

Changes in sugar content during the ripening of some varieties of fruits. G. Leoncini and F. Roggi. *Ann. sper. agrar. (Rome)* 17, 83-104(1935). —The max. sucrose content in pears was found in the period of complete ripening, while reducing sugars decrease rapidly. Watering of the plants diminishes, and dryness increases the sucrose content of the fruits. Some explanations of these phenomena are given. G. A. Bravo

Changes in some constituents of olives during the ripening period, in relation to fatty matter formation. G. Leoncini and I. Roggi. *Ann. sper. agrar. (Rome)* 17, 123-127(1935). —No analysis was found between the content of sugars in olives and the formation of fatty matter. It is supposed that reducing sugars are probably formed

by the partial hydrolysis of pectin substances. The changes observed in the behavior of fats, total ext., cellulose, sugars, etc., vary markedly in the varieties examined, but the N-contg. matter is quite const. An approx. inverse relationship exists between N-free ext. and fatty matters.

G. A. Bravo

Effect of the enrichment of the atmosphere in oxygen on the development of plants. Marin Mollard. *Compt. rend.* 201, 1160-2 (1935).—The normal growth of both the radish and *Sterigmatocystis nigra* was definitely inhibited by the presence of increased amounts of O in the atm.

Rachel Brown

The glucide metabolism of *Sterigmatocystis nigra* is a function of the nitrogen source in the culture medium. Robert Bonnet and Raymond Jacquot. *Compt. rend.* 201, 1213-15 (1935), cf. C. A. 29, 5146\*.—The formation of citric and oxalic (I) acids was detd. for the growth of *Sterigmatocystis nigra* in sugar media contg. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> or KNO<sub>3</sub>. The detns. were made at the time the mycelia were young, adult and partially autolyzed. In the presence of gluconic acid, xylose, arabinose, levulose, galactose, sucrose, maltose and inulin, but not lactose I was formed if KNO<sub>3</sub> was the source of N. I is a by-product due to a secondary phenomenon of growth.

Rachel Brown

Phosphorus metabolism in the leaves of some plants with constant foliage during the course of turning yellow. Emile Michel Durand. *Compt. rend.* 201, 1215-17 (1935).—The phenomena of migration which precede the falling of the leaves of Algerian ivy, cherry laurel (I) and aucuba bring about reduction in the total P sol in alc., lipid P, total P sol in dil. acid, mineral P, sol org. P and insol. residual P. In the green leaves phytin is present, the amt. depending upon the species, but with the exception of I it is absent in the yellow leaves.

Rachel Brown

The reaction of the medium and the activity of ordinary vs. performed mycelia of *Aspergillus*. Virgilio Bolcato. *Ann. chim. applicata* 25, 423-32 (1935).—See C. A. 30, 745\*.

A. W. Conti

Granule building cell substances enter through the living plasma lemma. (Observations on epidermal cells of *Allium cepa*.) Otto Bank and Karel B. Estefák. *Protoplasma* 24, 404-8 (1935).—The taking up of color (methylene blue, methyl violet, crystal violet, neutral red, methyl green) brings about the entrance through the plasma lemma of the granule-building substances from the protoplasts of the epidermal cells of *Allium cepa*, following plasmolysis with neutral salts. The vitality of the protoplasts is not injured, and the plasma lemma is apparently unharmed. The colloidal cell substances, diffused from the protoplasts and forming granules with the dyes, change their form characteristically in warm solutions (30-50°) or hot solutions of the nitrates of different ions (K, Na, NH<sub>4</sub>), these changes do not take place in cold solutions or in solutions of other salts of the same ions. A wound stimulus and plasmolysis with AgNO<sub>3</sub> act similarly to the dyes.

F. L. Dunlap

Biochemical study of the internal mechanism of photoperiodism. A. E. Murneek. Mo. Agr. Expt. Sta., *Bull.* 340 (Ann. Rept. 1933), 63-4 (1934).—As short-day soybean plants approached the reproductive stage there was a rapid accumulation of N in the stems, particularly in the nodes and internodes. All detd. forms of N were found including coagulable, proteose, basic, NH<sub>4</sub>, amide, humin and amino, but not NO<sub>3</sub>. The greatest differences were evident in the relative concns. of amide, humin, amino and NO<sub>3</sub> fractions. In the long-day plants there was very little NO<sub>3</sub> in the tips of the plants. In the presence of sufficient quantities of labile carbohydrates, NO<sub>3</sub> was used rapidly for the synthesis of proteins, and these in turn for the development of vegetative organs. In reproductive plants there was a rapid movement of N to the flowers and fruits and an accumulation of the carbohydrates, particularly starch, in the stems. Both carotene and xanthophyll were in greater concn. in short-day than in long-day plants.

C. R. Fellers

The hydrogen ion concentration relations of vine leaf

Edith S. Salacz. *Magyar Ámpelológusok* 9, 37-47 (1935).—Vine leaves were disintegrated by hand, moistened with CO<sub>2</sub> free distilled water, mixed to a pulp with purified Danube sand and the turbid liquor was filtered. The pH values of the solutions were detd. according to Gillespie in the presence of Sørensen indicators. American vine types gave values from 3.6 to 4.6, Hungarian ones also from 3.6 to 4.6 but showing less variation. The pH values for 5 vine types were detd. every 10 days from July 8 to Oct. 29. A Furmint vine leaf seemed to be more alk. than an Oporto one, which is said to be more sensitive to *Peronospora*. No connection between pH values and *Peronospora* sensitivity of vine leaves could be proved.

S. S. de Finály

Acidity of vine leaf and *Peronospora*. István Káhn. *Magyar Ámpelológusok* 9, 47-57 (1935).—Colorimetric detn. of pH values of vine leaves showed that direct sunshine increases the pH values of leaves by 0.3 (av.). No connection between chlorosis and pH values of leaves could be detd. The occurrence of *Erysiphe vitis* seemed to have an acidifying influence. Some systematic distribution of pH values of leaves of the same branch could be detected. pH values over 4.1 may give some protection against *Peronospora*. The immunity of some vine types against *Peronospora* is without doubt caused by some other factor than pH value.

S. S. de Finály

Osmotic pressure determinations on vines. Imre Szolnoki. *Magyar Ámpelológusok* 9, 60-7 (1935).—The osmotic pressure of vine leaves was detd. by the plasmolytic method, with 3.53% KNO<sub>3</sub> solutions. The European vine type showed 16.8, and the American 12.6 atm. This explains why the xerophytic leaves of European vines remained fresh 4-5 days when put in water, while the American ones dried in 2 days.

S. S. de Finály

The interrelations of catalase, respiration, after-ripening and germination in some dormant seeds of the Polygonaceae. Elizabeth R. Ransom. *Am. J. Botany* 22, 815-25 (1935).—The seeds of *Rumex crispus* and the 6 species of *Polygonum* studied were dormant at maturity. The optimum temp. for after-ripening of the moistened seeds was 3-6°. When fruits of *P. scandens* were kept for a time in a satd. atm. at 30°, dormancy became more pronounced and the time required for after-ripening at 3-6° was increased. The catalase activity of satd. fruits and seeds was greatest at 3-9° and lowest at 30°, while the rate of respiration was lowest at 3-9° and highest at 30°.

L. E. Gilson

Vitamin A activity of the thallus of a species of *Mucor*. W. H. Schopfer and A. Jung. *Compt. rend. soc. biol.* 120, 1093-5 (1935), cf. C. A. 29, 2198\*.—*Phycomyces blakesleanus* was grown as previously described. The vitamin A content of the dry thallus, detd. by feeding expts., was equiv. to 30 mg. % of carotene. Colorimetric methods indicated a higher carotene content. L. E. Gilson.

The capsules of *Mycotorula albicans* and other yeast-like fungi. Pablo Negrom. *Folia biol.* 1, 235-6 (1935).—The isolation of a sp. polysaccharide from the capsules of *M. albicans* is described. The polysaccharide is sol. in water. It acts as a hapten and does not cause the formation of an antibody when injected into animals. It gives a complement-fixation reaction with serum of rabbits immunized with the whole microorganism. It gives a precipitin reaction with some immune sera and not with others. Probably the capsules of other species of yeast-like fungi contain sp. polysaccharides.

L. E. Gilson

The adsorption and elution of cucumber mosaic virus. B. N. Uppal. *Indian J. Agr. Sci.* 4, 650-62 (1934).—The virus is active in the pH range 5-9. It is readily adsorbed by fuller's earth in quantity as small as 0.125 g./100 cc. of the virus ext. Adsorption does not necessarily inactivate the virus unless the acidity of the medium is greater than pH 5. The virus can be eluted by changing the pH of the suspension to 6.0-6.7. Although the virus cannot be eluted with NH<sub>4</sub> salts such treatment does not inactivate the virus, a change in the reaction of the suspension that has been treated with NH<sub>4</sub> to pH 6.7 frees the virus *in vitro* in an active state.

K. D. Jacob

Yield of citric acid from sugar as a basis for estimating the mechanism of its formation from the latter. V. S. Butkevich and M. S. Gusevaya. *Compt. rend. acad. sci. U. R. S. S.* 18, 3, 405-8 (1935).—An active strain of *Aspergillus niger* was grown on 100 cc of nutrient contg sugar, 20g,  $\text{KNO}_3$  0.35g,  $\text{K}_2\text{HPO}_4$  0.04g,  $\text{MgSO}_4$  7.74g, 0.04g,  $\text{ZnSO}_4$  7.74g, 0.04g, and  $\text{FeCl}_3$  0.0015g. Sugar (20%) was introduced under the film. The soils were then brought to pH 3 and 0.1%  $\text{Mg}(\text{NO}_3)_2$  and  $\text{KNO}_3$  added. The fungus was kept at 30° for 8 days on the nutrient and sugar. Both sugar and citric acid were detected by two different methods. The yield of citric acid exceeded that obtainable for the mechanism in which it is formed from sugar by way of al. fermentation. In expts with sucrose the yields approach the max. if sugar were entirely changed to citric acid. A scheme is proposed in which aldol condensation takes place in the sugar mol, forming a 5-membered ring. This is followed by oxidative breaking of the ring to give citric acid. Various other schemes proposed are discussed. S. P. Jeffrey.

The isolation of pectic substances from wood. Ernest Anderson. *J. Biol. Chem.* 112, 531-9 (1936).—The compn. of certain pectic substances isolated from the cambium layer and the sap wood of black locust approximates closely certain of the pectinic acids and of certain other substances the polygalacturonic acid obtained from *Citrus pectus*. The presence of d. galacturonic acid has been definitely established in the Ca pectate from both cambium and sap-wood. Methyl pentose sugars are apparently absent. "It appears that the pectic materials in wood are deposited in the middle lamella and the primary cell wall in the cambium at cell development and remain even in the old wood. Later in the growth of the wood other materials appear to be deposited on the pectic materials and protect them from the action of pectic solvents. The H<sub>2</sub>O-sol. pectic material seems to be composed of a Ca salt with lignin part may be combined with cellulose or with lignin. While no detn. was made of the amt. of pectic material present, the sap-wood apparently contains less than 3% and the cambium less than 15%." A. P. Lotzow.

The storage and physiology of tropical fruits. C. W. Wardlaw and E. R. Leonard. *Trop. Agr. (Trinidad)* 12, 415-10 (1935). (cf. C. A. 24, 5359).—Results are presented of expts with bananas, tomatoes, avocados, limes, grapefruits, papaws and other fruits. The rate of respiration and the concn. of gases in the internal cavities of papaws were measured repeatedly during utilization of the flower to decay of the fully matured fruit. The results show that many conclusions based in the past upon measurements of the external respiration of fruits alone will have to be modified. While the curves for rate of respiration and concn. of  $\text{CO}_2$  in the internal gases run approx. parallel until the seeds mature and the white flesh begins to color, they do not thereafter. This is due to changes in the permeability of the skin and flesh and consequently to changes in the rate of diffusion of gases. The internal concn. of  $\text{CO}_2$  begins to rise when the seeds are mature, although the external rise begins only when the fruit begins to color externally and reaches the characteristic when the fruit is fully ripe. While the metabolism of the fruit as measured by external respiration appears to diminish at this point, the rapid accumulation of  $\text{CO}_2$  in the internal cavities from this time on shows that this is not necessarily true.

Physiology of "limb chlorosis" of the lupine. Helmut Schmitz. *Der. deut. botan. Ges.* 53, 897-10 (1935).—The onset of lime chlorosis of the lupine depends on the alkali, of the medium directly in contact with the roots. Since the lupine can control this to a certain extent by root excretions the susceptibility to chlorosis depends upon the intensity and quality of these excretions. L. P. M.

Effect of the external medium on plant metabolism I. The metabolism of nitrogenous substances in a woody plant grown in a warm and humid atmosphere. Raoul Combes. *Rev. p. botan.* 46, 513-27 (1934).—A no. of 5-yr.-old beech trees were transferred in Feb. into a greenhouse kept at 23-25° and at a high humidity and compared

at intervals with respect to the ratio protein N/sol. N with similar trees remaining outside. The curve for the buds and the leaves developed therefrom of the trees left outside showed a marked rise from May 12 to June 10, followed by a rapid fall. This rise was due to a decrease in sol. N, at the time of the increase in proteins incident to the developing leaves (Apr. 7-24) the ratio remained unchanged. The corresponding curve for the trees grown in the warm atmosphere showed a smaller maxima in the period from Apr. 29 to Nov. 3. II. The absorption of mineral matter and the elaboration of organic matter in a herbaceous plant grown in light of different intensities. J. Panchaud. *Ibid.* 549-601.—*Raphanus sativus* grew best in full daylight. The water contents of the plants grown under conditions in which they received  $1/4$ ,  $1/2$ , and  $1/3$  of full solar radiation gradually decreased during the course of the development of the plants, while those receiving  $1/4$  and full solar radiation increased in water content up to the time of flowering, after which they also decreased. The ash contents of the plants were higher with decreased illumination. III. The respiration of leaf tissues formed at different light intensities. A. M. Bronger. *Ibid.* 641-54.—Leaves of *R. sativus* which had developed in only  $1/4$  or  $1/2$  full solar radiation respired much more slowly than leaves from plants which had received more light. The highest rates for the  $\text{CO}_2$  output of the leaves grown under the most unfavorable light conditions were obtained at the beginning of growth after germination and again at the time of flowering. The R. Q. remained close to unity regardless of light intensity during development. IV. The metabolism of nitrogenous compounds in a herbaceous plant grown at different light intensities. Léon Eschoute. *Ibid.* 47, 70-86 (1935).—The ratio insol. P/sol. P (in 10%  $\text{CCl}_3\text{CO}_2\text{H}$ ) increased rapidly during germination, after which it decreased up to the time of flowering and then rose again (cf. *R. sativus*). The percentage of P was highest at the end of germination and decreased thereafter up to nearly the end of the growth period, when it showed a slight increase. In the early stages of development the plants grown in stronger light had the highest P contents, at later stages more P was present in the plants receiving less illumination. In the course of flowering and fruiting P migrated from the tubers to the aerial parts of the plant. V. The metabolism of nitrogenous substances in a herbaceous plant developed in different light intensities. Marie Moreau. *Ibid.* 523-51.—The percentage of protein, which was very high in the seeds (*R. sativus*), decreased from the beginning of germination to the time of flowering, after which a slow increase took place. At the early stages of development the plants grown in the different light intensities all contained about the same percentage of protein, but as growth progressed the plants under the most favorable light conditions stored up nonnitrogenous substances and thus contained a lower percentage of protein. Sol. N increased from the time of germination to the time of flowering, when it started to decrease.

1. Lawrence P. Miller. The formation of chlorophyll in roots and the reduction of silver salts by chloroplasts. R. J. Cauther. *Rev. p. botan.* 47, 401-24, (1934-35).—Under certain conditions the roots of a large no. of plants retranslocated become green when exposed to light. The presence of sugar in the medium or other conditions which increase the quantity of sugar in the roots are favorable for the production of the green color. Only living chloroplasts reduce  $\text{AgNO}_3$ . The reduction is considerably augmented by light but is not due to the reducing action of the direct products of assimilation.

Production of flavin enzyme systems in germinating plants. Hans v. Euler and Ole Dahl. *Biochem. Z.* 282, 255-61 (1935).—Total flavins and the nonoxidizable fraction of the flavins were detd. in plant extracts. Barley contains about 3-7 times as much total flavin as oat during the 1st 5 days of germination, and in the former the synthesis is practically completed on the 2nd day; whereas in germinating oats it increases steadily and continuously. Ungerum detd. peas contain much flavin; it is present almost entirely



as bound flavin. In the exerts from the germinated seeds the bound flavin makes up approx.  $\frac{1}{2}$  of the total.

S Morgulis

Types of swelling of various kinds of fibers and their relation to the problem of fiber structure. K. Griffioen. *Plant (A) E. Z. Wiss. Biol.* 24, 584-601 (1935).—Expts. on the swelling of various kinds of fibers showed that NaOH causes regular swelling in fibers without any "balls" or bladder-like swellings, whether the fibers were from monocots, dicots or gymnosperms. Salts also cause a regular swelling.  $H_2SO_4$  and Schweizer's reagent give "ball formations" in monocots and gymnosperms as well as in the wood fibers of *Betula* and *Fagus*, but in the bast fibers of dicots these reagents cause the fibers to swell evenly. The spheroidal (irregular) swelling depends on the width of the fiber (i.e., on the amt. of cellulose) and obeys the laws of surface tension. For all these reasons the "transverse membrane" theory of Lédike is not tenable. It is possible that longitudinal membranes exist but only at the margins of the various layers and not at the borders of the individual lamellae. It is very questionable whether the lamellae are surrounded by special membranes. In swelling studies, too much confidence must not be placed in lines of cleavage, splits and fractures. No reagents could be found which would attack only main valence chains (chem. forces) or only cohesion compds. (van der Waals forces). All cause swelling and hydrolysis, which action is dominant depends on various factors such as the concn. of the reagent, the structure of the fibers, method of handling, etc. Polaroscope studies showed that neither the cellulose nor the small particles (as seen by Farr when treated with concd. HCl) are homogeneous, on the other hand, they represent heterogeneous mixts. O. R.

The assimilation of oxalic acid by *Aspergillus niger*. Denis Bach and Jean Fourmer. *Compt. rend.* 201, 982-4 (1935).—Nine to 30 mg. oxalic acid is assimilated by 200 mg. *Aspergillus niger* in 8 hrs. at  $pH$  2.03-4.57 from a complete glucose nutritive medium containing 200 mg. oxalic acid. The wt. of the mycelium is doubled or tripled in 24 hrs. in this medium. When the nutrient medium contains no glucose, the wt. of the mycelium does not increase, but oxalic acid is assimilated if the  $pH$  is below 5.8. W. Gordon Rose

The participation of potassium in production of material by the higher plants. I. The action of potassium and photosynthesis. K. Maunwald and A. Frank. *Z. Pflanzenphysiol., Dargang Bodenk.* 41, 8-25 (1935).—A review and discussion. Seventy-five references. C. J. S.

The assimilation of calcium by potato plants. K. Heller, K. Peh and J. Pruska. *Z. Pflanzenphysiol., Dargang Bodenk.* 41, 26-7 (1935).—In connection with the investigation on  $K_2O$ . C. A. 29, 1492, similar expts. with Cs showed that about 2% of the Cs offered in Richter's nutrient soln. concn. 0-16 mg. Cs/l. was absorbed by the plants. The error of the mean yields in these expts. was so large that the effect of the smaller additions of Cs is uncertain, the larger additions were certainly toxic. Only the roots of plants in the latter case showed any tendency to accumulate  $CaO$  in preference to  $K_2O$ . C. J. S.

The chlorosis of hydrangea (*Hydrangea hortensis*) in relation to iron. Werner Scholz. *Z. Pflanzenphysiol., Dargang Bodenk.* 41, 129-34 (1935).—Shoots of hydrangea were grown in porcelain dishes containing 4 kg. HCl-extd. quartz sand, with 0.21 g.  $CaCO_3$  and in half the dishes 0.3 g.  $FeSO_4 \cdot 7H_2O$ , all watered with a soln. of fertilizer chemicals. Additional dishes with earth-peat mixts., some with 100 g. pptd.  $CaCO_3$ , were included. Observations on growth, compn. of leaves and incidence of chlorosis led to the following conclusions. The commonest cause of chlorosis in hydrangea is excess  $CaO$  in the soil, which makes Fe unavailable to the plant. The Fe of chlorotic plants is immobile, old leaves may contain much Fe, while new growth is deficient and chlorotic. Chlorotic leaves are neither especially high in  $CaO$  nor strikingly low in Fe. A special addition of an Fe salt to soil in which chlorosis occurs may prevent the latter without being of particular benefit to the plant in other ways—its growth is still unsatisfactory. Further expts. should be undertaken to

investigate the functional disturbances in root and leaf cells and the  $P_2O_5$  economy of plants grown under conditions favoring chlorosis. Approx. 40 references.

C. J. Schollenberger

The relation between vitamin A and the plastids. Ph. Joyet-Lavergne. *Compt. rend.* 201, 1042-4 (1935); cf. C. A. 29, 1872, 6253. Vitamin A is localized in the mitochondria of the plastids as shown by the  $SbCl_5$  reaction on the leaves of *Elaeagnus canadensis* and *Isis germanica*. The former conclusion, that vitamin A is an essential constituent of chondrome, is verified. E. D. Walter

Characterization of hydroxylamine in autolyzed green leaves. Maurice Lemoigne, Pierre Monguillon and Robert Desvieux. *Compt. rend.* 201, 1077-10 (1935); cf. C. A. 29, 6918. The plant juice, after clarification with  $Pb$  subacetate and destruction of the nitrates, is oxidized by I in  $AcOH$  soln. The formation of  $HNO_2$  under these conditions characterizes  $NH_4OH$ . E. D. Walter

Toxins of wilting. O. K. Elpidina. *Compt. rend. acad. sci. U. R. S. S. (N. S.)*, 3, 320-4 (1935); cf. C. A. 27, 2979.—Expts. with *Fusarium* preps. on tomato and sweet clover plants indicate that the wilting of plants attacked by fungi is caused by  $NH_3$ , resulting from the N metabolism of the parasitic organism. E. D. Walter

A new type of plant lipochrome. I. M. Heilbron, B. Lythgoe and R. F. Mupers. *Nature* 136, 983 (1935); cf. C. A. 29, 6276. A new pigment, mioxanthin, was extd. from *Rhodospira rubra*, one of the blue-green algae. The carotenoid crystallizes from  $Et_2O$ -MeOH in deep Cu-colored needles, m. 117-18°. It has a single absorption band at 458-460 m $\mu$  in CS<sub>2</sub> and resembles in this respect the crustacean pigment astaxanthin. It has no acidic properties. Failure to yield a Na salt precludes the possibility of its being hematochrome. E. D. Walter

Plant pigments and reproduction. R. H. Roberts and Norman Livingston. *Science* 82, 595 (1935).—In leaves of Wealthy apple, beets, tobacco and *Datura* there was somewhat less chlorophyll, carotene and xanthophyll in the fruiting than in the vegetative condition. J. J. Willaman

The curly-top-resistant beet variety. A. W. Skuderna, Charles Price, J. O. Culbertson and C. E. Cornman. *Facts About Sugar* 31, 17 (1935).—The new variety, No. 1, bred by the U. S. Dept. of Agr., far surpasses all com. brands in disease resistance, and compares favorably with them in yield, sugar content and purity. F. W. Zerlan

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## E—NUTRITION

PHILIP B. HAWK

The reed (*Phragmites communis*) as a source of vitamin C. S. Balakrishnan. *Compt. rend. acad. sci. U. R. S. S. (N. S.)*, 3, 270 (1935).—The leaves of the common reed contain 0.2% of ascorbic acid. (1) A daily min. dose of 0.5 mg. of I obtained from aq. expts. of the reed maintained guinea pigs kept on a scorbutic diet free from scurvy for 60 days. Large doses proved nontoxic to human subjects. The inoculation of the ext. with lactic fermentation bacilli under strictly anaerobic conditions augmented the reductive power of the ext. from 2- to 4-fold. Since similar results were obtained with skim milk the procedure may have profitable use in the food industry. C. R. A.

Growth-promotion effects of l- and d-araboflavins (6,7-dimethyl-9-(l- or d-1-araboflavin)-alloxan). I. L. Kautsky and M. N. Steinberg. *Hist. Chim. Ada Biol.* 1336-37 (1935), cf. C. A. 29, 6237. —Wt. curves for rats on a B-free diet to which daily doses of 10, 20, 20-40  $\gamma$  of l-araboflavins were added failed to show a const. rate of growth after 40 days. In contrast to these findings, curves for a daily dose of 10  $\gamma$  of the optical antipode, d-araboflavins (II), show a const. rise in wt. of 0.8 g. per day over a period of 50 days. Synthetic l-noboflavins gave a daily rise of 1.3 g. for a daily dose of 10  $\gamma$  and the d-isomer produced a daily rise of 0.1 g. with a daily addn. of 3  $\gamma$  to the diet. It is remarkable that the biologically active form of araboflavins is that optical form in which arabinose does not occur naturally and that II differs from lactoflavin in the spatial disposition of 1 of the OH groups in the sugar residue. C. R. Addinall.

Crystalline derivatives of vitamin E. Preliminary report. Riang Ha Kim. *Sci. Papers Inst. Phys. Chem. Research* (Tokyo) 28, 74 (1935). —Treatment of 30 g. of vitamin E concentrate, by 210-35°, obtained from rice germ oil by the method of Hamano (cf. C. A. 29, 2545), gave 8.5 g. of white crystals which were dissolved in MeCO and fractionated by treatment with MeCO, MeOH, EtOH, AcOH and AcOH. A difficultly sol. fraction (0.5 g.), m. 148°, was assumed to be inactive and was not further investigated. A second fraction (1.5 g.) C<sub>18</sub>H<sub>35</sub>O<sub>5</sub>, m. 156°, was isolated and on hydrolysis with alc. KOH gave a physiologically active vitamin E. Rats kept on a vitamin E free diet (lard 7, cod liver oil 3, Et<sub>2</sub>O 65 and dextrin 95%) for 130 days were given an oral dose of 0.5 mg. This single dose increased the wt. of 2 rats gave birth to 40 young. A 3rd fraction, in 158°, was isolated from the mother liquor. The residual material was recryst. from MeCO and EtOH and yielded 0.7 g. of crystals, m. 134°, from which free vitamin E was regenerated. The residual amorph. of the 4 crystal fractions was saponified with Me alc. KOH at room temp. and the liberated material was converted into the ester of  $\beta$ -anthracenecarboxylic acid. The yellow powder (0.8 g.) obtained gave Witky's and Lieberman's reactions but the color changed quickly to green and showed fluorescence. Photographic plates of the crystals are given. C. R. Addinall.

Biologic behavior of bran and potato protein in metabolism. A. Bickel, R. Sander and J. Schilling. *Munch. med. Wochschr.* 82, 1482-51 (1935). —The feces N increases on bran and potato diets in comparison with a corresponding casein diet. The urinary excretion of N as a G. N. is higher than when an equimol. amt. of casein N is given. Eleanor W. J. Butz.

Effect of feeding dogs with vegetable "proto-acid". I. L. Kautsky. *Trudy Leningradsk. Bol'sh. Bol'sh. Obshch. Organizm. No. 8*, 13-17 (1935), cf. C. A. 29, 6237. —Young dogs, 9 weeks old, were placed on a diet in which the animal protein was replaced by the "proto-acid" of peas. The exp't lasted 45 days, during which the pups received daily 30-40 g. of the "proto-acid". The gain in wt. of these pups was identical with that of the controls. X-ray diagrams failed to reveal any signs of rickets. A morphological and analysis of the control and exp't pups, performed at the end of the exp't., yielded identical results. H. Cohen.

Water interchange. IX. Variation of diuresis and urine pH in animals with acid and alkaline diet. M. Saviano. *Atti accad. Lincei* 21, 715-20 (1935), cf. C. A. 29, 2210. —A dog kept on an acid diet (bread, meat and water) shows a pH of 5.4-5.5, and after diuresis (injection of excess water) the pH drops to 6.0. On an alk. diet (within 3 hrs. of feeding, and then drops to 7.5. On alk. diet the pH increases to 7.7-8.1. Similar results were obtained with rabbits. Poly-pyuria with an acid diet reduces the acidity of the urine, while with an alk. diet it becomes more acid, providing the exp'ts. are not prolonged as to disturb the acid-base equil. of the system, by exhausting the mineral reserves in the system. A. W. Coonten.

Development of Jensen sarcoma under special conditions of diet. V. I. Iamian. *Atti accad. Lincei* 21, 721-5 (1935). —White rats fed on (1) an ordinary mixed diet, (2) an exclusive diet of corn, corrected for salt and vitamin deficiency, and (3) an oats diet, corrected as in 2, and then all inoculated with Jensen sarcoma tumor showed no difference in the development of the tumor. Another set in which the vitamin-deficient diet was used showed a slower rate of tumor development. A. W. Coonten.

Fat metabolism. VII.  $\beta$ -Oxidation of normal saturated dicarboxylic acids administered orally. P. V. Yrlande, J. van der Lee and A. J. S. van Alphen. *Z. physiol. Chem.* 237, 189-90 (1935), cf. C. A. 29, 5429. —The excretion of sebatic (C<sub>16</sub>), suberic (C<sub>18</sub>) and adipic (C<sub>10</sub>) acids after ingestion of tetracapsin, and of nonadecarboxylic (C<sub>19</sub>), azelaic (C<sub>9</sub>) and pimelic (C<sub>7</sub>) acids after ingestion of trandecylin, may represent  $\omega$ -oxidation before and after one or two  $\beta$ -oxidations, or it may indicate successive  $\beta$ -oxidations after an initial  $\omega$ -oxidation. The latter process has now been demonstrated by recovery of suberic and adipic acids after feeding  $\beta$ -sebacic acid, and of azelaic and pimelic acids after feeding nonadecarboxylic acid to dogs. A. W. Coonten.

Vitamin-carrier studies. XI. Preserved spinach as vitamin C carrier. N. E. Sheplevska and T. L. Izumrudova. *Z. Unters. Lebensm.* 70, 274-6 (1935), cf. C. A. 29, 7401. —Certain samples of preserved spinach contained 200 vitamin C units per kg., while other samples were practically free from this vitamin. XII. The onion as a vitamin C carrier. I. The vitamin C content in onions kept for a short time. N. E. Sheplevska. *Ibid.* 277-8. —11teen cc. of onion juice is adequate to protect guinea pigs from the onset of scurvy and the dosage may be less than this amt. for protection. In the autumn, 1 l. of onion juice contains 67 antiscorbutic units. Hence the onion has weak antiscorbutic properties. 2. Vitamin C content of onions which have been kept for some time. N. E. Sheplevska and T. L. Izumrudova. *Ibid.* 278-9. —One l. of juice obtained from onions which have been kept for some time contained less than 67 antiscorbutic units. XIII. The vitamin C content of dried onions. S. N. Matko. *Ibid.* 279-80. —Feeding of cooked ordinary onions and of leeks to exp't. animals in amts. up to 8 g. in the case of the former and 5 g. in the case of the latter showed no antiscorbutic action. XIV. The antiscorbutic properties of the rutabaga. *Ibid.* 280-2.

The juice of the yellow-meated rutabaga which has been kept through the autumn contains not less than 570 vitamin C units per l. and its min. prophylactic dose lies between 0.35 and 1.75 cc. If these rutabagas are kept through the winter, the vitamin C content per l. is less than 570 and the min. prophylactic dose is greater than 1.75 cc. XV. The antiscorbutic properties of sulfured white and white cabbage. *Ibid.* 283-4. —Dried, sulfured white and completely cooked cabbage which has been kept 2 months after drying and for 3 months during the course of these exp'ts. contained in 1 kg. about 160 vitamin C units and its min. prophylactic dose was in the neighborhood of 6 g. XVI. The action of certain extraction agents on the antiscorbutic activity of fir needles. The infusions. N. I. Orlov. *Ibid.* 285-8. —The min. therapeutic dose of the original fir-needle infusion (1 l.) for guinea pigs was between 1 and 3 cc. This infusion was prep'd from the fir needles by treatment with a 0.2% HCl soln. A treatment of this infusion with Et<sub>2</sub>O lowered the antiscorbutic activity notably and its min. therapeutic dose was between 3 and 6 cc. Treatment with CHCl<sub>3</sub> did not lower the antiscorbutic activity, as much as the min. therapeutic dose of such an infusion was about 3 cc. F. L. Dunlap.

A year's exclusive meat diet and seven years later. Clarence W. Leach. *Am. J. Digestive Diseases Nutrition* 2, 478-58 (1935). —A summary of clinical and lab. findings on 2 Arctic explorers living under av. conditions in a New York climate.

The relation of vitamin A to anophthalmos in pigs. Fred Hale. *Am. J. Ophthalmol.* 19, 1087-91 (1935). In the course of exp'ts. relating to maternal vitamin A

deficiency, 42 pigs (4 litters) were born blind and showed such other defects as cleft palate, cleft lip, accessory ears, etc. Complete genetic tests indicate that maternal deficiency of vitamin A will result in a variety of defects in the offspring. Edward Eagle

The Harben lectures, 1935 Problems of nutrition and growth I The digestion of starch P Armand-Delille *J. State Med.* 43 683 95(1935) II The role of the vitamins *Ibid.* 696 708 III Light and heliotherapy in nutrition *Ibid.* 709-19 Edward Eagle

Miscellaneous studies on poultry grit Roy H. White Md Agr Expt Sta, Bull No 377, 313-35(1935) — Limestone of low Mg content is adequate to supply all the Ca needed for egg-shell making in laying hens Crushed oyster shells serve in the dual role of Ca carrier and grit for laying hens Excess grit or limestone passes from the body in the excrement The grinding of food particles is only a secondary function of grit in chickens Furnishing mineral for egg-shell formation and the sepn of food particles to facilitate the churning action of the gizzard are primary functions C R Fellers

Ten year report of studies in child development and parent education Ethel B Waring N Y (Cornell) Agr Expt Sta, Bull No 638, 69 pp (1935) — A condensation of 67 different research studies or surveys on related subjects Project 26 is a dietary study of nursery school children by Olive R. Jones (*Ibid.* 31 3) J found the av. caloric intake for the noon meal was only 371 cal Protein constituted 13%, fat 39% and carbohydrate 48% of the total cal in the food intake of pre school children Ca, P and Fe intakes were, resp. 0.80, 0.91 and 0.0065 g daily When the meals were begun with a serving of fruit juice the voluntary intake of other food was considerably increased C R Fellers

Ordinary dried blood versus soluble blood flour for calves W L Krauss and C F Monroe Ohio Agr Expt Sta, Bull No 548 (53rd Ann Rept), 57(1935) — A month's feeding trial showed no advantage for the spray dried sol. product in the dry feed system of raising calves C R Fellers

Sources of vitamin A for chicks I Comparison of carotene and vitamin A as found in cod-liver oil R M Belhke and P R Record Ohio Agr Expt Sta, Bull No 548 (53rd Ann Rept), 73(1935) — Approx. 100% of pure carotene per 100 g of basal ration (white corn, wheat middlings, caecm, irradiated yeast, bone meal, CaCO<sub>3</sub> and NaCl) was required to meet the basal requirements of chicks for vitamin A for the 1st 8 weeks Vitamin A as found in cod liver oil was equal to carotene when fed on the same biol. rat unit basis On the basal ration alone, none of the chicks survived longer than 6 weeks C R Fellers

Influence of feed of the cow upon the vitamin G content of the milk C H Hunt and A E Perkins Ohio Agr Expt Sta, Bull No 548 (53rd Ann Rept), 74(1935) — Cows on a low protein ration also low in vitamin G, consisting of timothy hay and beet pulp, produced milk of lower vitamin G potency than cows fed an ordinary winter ration contg. a good grade of alfalfa hay The vitamin G content of milk from cows on pasture varied directly with that of the pasture grasses C R Fellers

Nutrition of turkeys J E Hunter, D R Marble and H C Kneidel Pa Agr Expt Sta, Bull No 320 (45th Ann Rept), 8(1935), cf C A 30, 1095 — When vitamin D is supplied in an oil contg. 108 U S P units per g, the requirement of growing turkeys for this supplement is approx. 68 U S P units per 100 g of rations Corn gluten meal in combination with animal protein feeds produces a superior flesh in young turkeys When a combination of meat, fish and milk protein is used to supply the animal protein in a 24% protein starting mash, 50% of any 2 of the 3 animal protein products may be replaced with protein from soybean oil meal or corn gluten meal, when the loss of Ca and P is compensated C R Fellers

Relation of the color and carotene content of butter fat to its vitamin A potency Ray Treichler, Mary A Grimes and G S. Fraps Tex. Agr. Expt Sta, Bull No 513,

3-34(1935) — The carotene content was estd from the absorption of light by the melted butter fat at 470 and 480 mμ as measured with a spectrophotometer. The absorption was calcd to carotene by means of factors obtained from measurements of the absorption of light by known amts of purified carotene dissolved in butter fat. Both the carotene and the vitamin A decreased during the period of feeding of the cows that had been on pasture before the period of lactation began The carotene of butter fat from a cow fed on white corn decreased from 9.18 p p m at the beginning to 0.06 p p m in 28 weeks Most of the decrease occurred during the 1st 6 weeks The carotene content of butter fat of cows on yellow corn decreased from 10.00 at the beginning to 0.29 p p m in 28 weeks The cows on yellow corn produced butter fat contg. more carotene and vitamin A than the cows on white corn The cow on a daily ration of 3 lb of alfalfa leaf meal in addn. to yellow corn produced butter fat which decreased from 12.1 p p m to 1.78 p p m of carotene in 32 weeks Cows depleted of vitamin A and placed upon Sudan grass pasture produced butter fat in which the carotene content increased during the entire period of the expt from 0.53 to 8.03 p p m of carotene The carotene content of the butter fat of the cows on pasture continued to increase after the vitamin A potency had reached a max. of 50 units per g Sorghum silage fed in liberal amts did not produce butter fat high in vitamin A or in carotene The carotene content of butter fat depends upon the feed, it was high in the butter fats of the cow receiving a high amt of carotene If it is assumed that 0.6 p p m of carotene is equal to 1 unit of vitamin A potency, in the case of the cow fed on white corn the percentage of vitamin A potency due to carotene decreased from 38% at the beginning of the expt to 1% near the end In that of the cow receiving yellow corn, it decreased in one case from 47% at the beginning to 9% at the end In that of the cow receiving daily 3 lb of alfalfa leaf meal in addn. to yellow corn, the percentage of vitamin A potency due to carotene varied from 30 to 55. The high vitamin A potency ascribed to carotene in the butter fat of the cow receiving daily 6 lb of alfalfa leaf meal again raised the question as to whether all of the coloring matter is really carotene and has vitamin A potency The decrease in the carotene and vitamin A contents of butter fat during the lactation period of the cow is due to deficiency of vitamin A potency in the feed and not to the stage of lactation The relation between the carotene content and the vitamin A potency of butter fat depends upon the quantities of vitamin A and carotene stored by the cow at the beginning of the lactation period, upon the length of time which the feed deficient in vitamin A and carotene has been fed, and upon the extent of such deficiency The data resulting from the spectrophotometric analysis of the butter fats from 2 cows were converted into monochromatic eqns and the color was expressed as dominant wave length, colorimetric purity and luminosity A decrease in carotene content was accompanied by a decrease in purity and an increase in luminosity, reflecting the loss of yellow color. The butter fat of the goat on green pasture is low in carotene but high in vitamin A potency Goats seem to have a greater power to transform carotene into vitamin A than have cows Butter high in vitamin A is likely to be highly colored, but all colored butter is not high in vitamin A Light colored butter is likely to be low in vitamin A The chances are high that butter that is naturally very yellow will contain more vitamin A potency than light-colored butter Bibliography C R Fellers

Studies of the values of different grades of milk in infant feeding J A Newlander and C H Jones Vt Agr Expt Sta, Bull No 389, 5-40(1935) — By expts on pigs, the relative feeding values of fresh, evapd, powd and remade milks were detd Fresh milk proved best insofar as gains in wt, dry matter wt, vigor and body compn were concerned next were evapd milk and 1 of the samples of powd milk Marked changes in body compn occurred with age increase Pigs at birth contain about 80% H<sub>2</sub>O Protein constitutes approx. 60% of their dry matter while ash and fat contents average about

17% each. The ratio of fat to protein is 1.36. As age advances the dry matter and fat percentages increase decidedly until at weaning time total dry matter is 60% and fat 15.6% greater than at birth. The percentages of fat and protein are approx. equal. C. R. Teller.

The effect of feeding deaminated versus untreated cod-liver oils on growth, egg production and mortality of poultry. H. S. Gutteridge. *Can. Dept. Agr. Pub.* 475, Tech. Bull. 1, 29 pp. (1935).—See C. A. 29, 809-9.

G. G.

Biological and biochemical investigation of the ascorbic acid content of plant tissues containing chlorophyll or not. Lucien Rasmussen, A. Gotsend and L. P. Leblond. *Bull. Soc. chim. Ind. 17*, 1649-76 (1935).—See C. A. 30, 751.

Crystalline vitamin B. Experimental and clinical observations. M. Vorkau, R. R. Williams and R. J. Waterman. *J. Am. Med. Assoc.* 105, 1540-5 (1935).—Evidence shows frequent deficiency of vitamin B in the human dietary. In a study of 100 cases of clinical neuritis in which vitamin B crystals, 30 mg. daily, were administered orally, 41 were rendered symptom free, 49 were benefited and 8 showed no improvement. In 8 cases of gastro-intestinal hypomotility and atony, 6 were cured by ingestion of vitamin B. P. P. Griffiths.

Studies on crystalline vitamin B. Observations on diabetes. Martin G. Volinac, Robert B. Williams and Robert J. Waterman. *Am. J. Digestive Diseases Nutrition* 2, 541-57 (1935).—A split deficiency of vitamin B changes a disturbance of the carbohydrate metabolism characterized by a rise in the blood sugar and in the glycogen content of liver and muscle. Diabetes mellitus is suggestive of a nutritional disturbance and there is reason to believe that a deficiency of vitamin B may be a factor in the production and clinical course of this condition. In a series of 11 cases of diabetes mellitus to whom an av. of 10 mg. of vitamin B, was administered daily for 24 consecutive days, 6 showed an increased carbohydrate utilization. Edward F. Eagle.

Diminution of the iodine number of the fats of guinea-pig liver and adrenals in antismoking C. L. De Caro and M. Chaim. *Bull. soc. sci. nat. sper.* 10, 235 (1935).

Helen Lee Gruehl

Deficiency osteoporosis. Joseph Lebowich. *Arch. Path.* 20, 742-51 (1935).—A case of deficiency osteoporosis is described in a 61-year-old white woman. Clinically, deficiency osteoporosis appears to be due to lack of vitamin D. The changes at the chondro-osseous junction in deficiency osteoporosis are similar to those in rickets. Osteon is markedly increased in amt. and thickness as is also in rickets. The last 2 changes are considered pathognomonic of rickets. Hence there is some histologic evidence that deficiency osteoporosis and rickets may be the same entity.

Effect of the plane of nutrition on the course of animal typanosomiasis. M. H. French and H. I. Hornby. *Typanosomiasis Territory, Ann. Entom. Soc. Am.* 1024, 46-50.—The course of *T. congolensis* disease from infection to crisis is unaltered, or at any rate is not favorably affected, by planes of nutrition higher than those of bare maintenance. Digestibility experiments, sheep and goats, with rations of hay and concentrates, showed that even the amt. of anemia and loss of wt. of affected animals can be attributed to impaired digestion. Analytical data are given. J. D. Jacob.

The lime requirements of sheep. M. C. Franklyn. *New Zealand J. Agr.* 51, 27-36 (1935).—In 127 cases of milk fever in ewes the Ca content of the blood serum amounted to 2-8 mg./100 cc. as compared with about 10 mg. in the blood serum of healthy animals. In the majority of cases the Ca did not exceed 6 mg./100 cc. Several ewes were kept on a diet particularly low in Ca for 2-3 years without showing symptoms usually associated with milk fever. It appears that a low Ca content in the diet is not the sole cause of milk fever in farm animals, although it may contribute to the incidence of the disease. The alarm danger of sheep in N. Zealand is not caused by Ca deficiency in the diet. In goats with mature wethers

receiving high-Ca diets the storage of Ca was not with certain animals and neg. with others. At present it is impossible to draw up a definite set of Ca requirements for sheep under various dietary conditions.

The hemoglobin content of the blood of laying hens on practical poultry rations. M. O. Scutlar, C. A. Ivey, J. B. Hurt and J. G. Haign. *Poultry Sci.* 15, 9-14 (1936).—A practical laying ration of 15% protein and 1.5 mg. Cu per hen per day supported heavy egg production without concurrent lowering of the hemoglobin content of the blood. Feeding of added Fe and Cu as FeSO<sub>4</sub> and CuSO<sub>4</sub> failed to increase either the hemoglobin content of the blood or the egg production. There was no evidence of anemia in healthy, noninfected laying hens receiving the practical ration. There was evidence that during heavy laying 20% of the food Cu and 10% of the food Fe may be lost in the eggs. The Fe and Cu contents of some poultry feeds and supplements were for yellow corn 36.4, 2.8; wheat 50.0, 7.2; alfalfa 10.0, 2.6; and oyster shell 62.0 and less than 0.2 mg./kg., resp. K. D. Jacob.

Growth of chick embryos from hens fed different protein levels. Robert Proulx, and R. B. Thompson. *Poultry Sci.* 15, 18-24 (1936).—Neither the growth nor the total N content of the embryos was significantly affected by varying the protein content of the hen ration from 12.0 to 24.7%.

K. D. Jacob

The vitamin D content and hemoglobin building properties of dehydrated kelp for chicks. M. Wain Miller and Gordon J. Pearce. *Poultry Sci.* 15, 15-18 (1936).—A sample of dehydrated kelp contained little or no vitamin D and did not increase the hemoglobin content of the blood of chicks on a normal ration. Extracts supplemented with kelp and cod liver oil were not superior to the same supplemented with cod liver oil alone. The kelp contained 14.0-7.44, ash 35.44, protein 8.57, Ca 1.60, P 0.33, Cu 0.00094, Fe 0.00091 and B 0.15%.

K. D. Jacob

The phosphorus requirements of growing chickens, with a demonstration of the value of controlled experimental feeding. W. T. Watkins and H. H. Mitchell. *Poultry Sci.* 15, 32-41 (1936).—In general, it appears that the greater the consumption of a diet uninhibited in some particular, the more poorly nourished will be the animal with reference to the function affected by the unbalanced ration. Chickens in the self-maintaining phase of growth require more than 0.26% of P in the ration but no more than 0.5% for fast growth and calcification of the bone. K. D. Jacob.

Availability of calcium salts for bone formation and rickets prevention in chicks. H. J. Dolan, C. A. Ivey, J. B. Hart and J. C. Haign. *Poultry Sci.* 15, 42-7 (1936).—The Ca in limestone, gypsum, rock phosphate, Ca gluconate, "Dicalpho," steamed bone meal and dolomite appeared to be equally available for bone formation in chicks that were fed a basal exclusively ration supplemented with varying levels of cod-liver oil. The ash content of the fat-free bone was higher when the supplement contained P in conjunction with Ca. With the levels of cod-liver oil added to the ration were lost, Ca intake produced better growth and bone formation. Cod liver oil did not produce Ca chloride. When extremely insol. Ca salts were fed their availability was proportional to their soly. in 0.5% HCl, increasing the cod-liver oil had no effect on the availability of the Ca in such combs. K. D. Jacob.

A new factor, not vitamin G, necessary for hatchability. R. B. Neulter, T. C. Rye, R. R. Ellis and Harry W. Faust. *Poultry Sci.* 15, 17-20 (1936).—A basal feed mix composed of ground yellow corn 62.0%, pure wheat bran 9.25%, rolled oats 18.8 and alfalfa leaf meal 5.8% contains at least enough vitamin G to meet the requirements of chickens for hatchability but lacks some other factor necessary for high hatchability, which is relatively abundant in dried pork-liver meal and green grass, and is present to some extent in a mix. of desiccated meat meal, fish meal and alfalfa buttermilk. Dried whey is not a good source of this factor. K. D. Jacob.

Vitamin A storage by chickens. Arthur D. Holme,

Francis Tripp and Percy A. Campbell *Poultry Sci.* 15, 71-8(1936).—The av. vitamin A content of livers from 8-week-old chicks that received 0.5% of yardine oil as a supplement to their ration was about 4 times greater than that of livers from chicks that received 0.25% of oil, a significant difference due to the higher level of oil was also evident in the livers of 12-week-old chicks. Regardless of the amt. of oil fed, the vitamin A content of livers from 12-week-old chicks was larger than that of the livers from comparable 8-week-old chicks. The no. of eggs produced by 9-month-old pullets increased with the amt. of oil contained in the rations, none of the pullets showed signs of vitamin A deficiency. High-producing flocks fed rations rich in vitamin A maintained an adequate body store of vitamin A throughout the reproduction period. Thirty-one references. K. D. Jacob

The nutritive value of proteins for maintenance. Samuel Morris and Norman C. Wright *J. Dairy Research* 6, 283-302(1935).—When minimal quantities of protein are fed in the maintenance ration of steers, a deficiency of lysine will lead to a marked increase in the urinary N output. A deficiency of cystine had no effect. The catabolism of body tissue is shown by the high creatine excretion and  $\frac{1}{2}$ N ratio of the excess S and N excreted. This is thought to be the result of excessive deamination with the production of mild acidosis. As regards urinary partition of N, there is some indication that a lysine-deficient diet causes a decreased percentage excretion of urea N and an increase of ammonia N. Biol. values were calculated with maize-germ meal, wheat gluten, rye flour and blood meal as the sources of protein in the ration. The only marked difference in biol. value was in the case of wheat gluten, which is very deficient in lysine. An approx. value for the lysine requirements for maintenance has been calculated, i.e., 0.80 g. of lysine N per 1000 lb. live wt. A. H. Johnson

The B content of foods. A. G. van Veen *Genescheud Tydschr. Nederland. Indie* 75, 2030-61(1935).—It was found, from the study of different rations used in the tropics, that, if a normally working inhabitant of these countries with an av. wt. of 50 kg. and a daily calory supply of about 2500 assimilates less than 150 international B units a day he runs a great risk of getting beriberi. If the supply is greater than 200 units these chances are practically nil. Ricebirds and rats were in the expts. J. C. Jurgens

Present status of the vitamin B complex. C. A. Elvehjem *Am. J. Pub. Health* 25, 1334-9(1935).—The vitamin B complex consists of at least 5 factors, B<sub>1</sub>, B<sub>2</sub>, B<sub>3</sub>, B<sub>6</sub> and B<sub>12</sub>. A brief survey with 27 references. J. A. Kennedy

Vitamin content of important foods in the child's diet. Carl R. Teller *Am. J. Pub. Health* 25, 1340-5(1935).—A short summary of research findings at the Massachusetts State College. The data deal largely with vitamins C and D in milk, fruits and vegetables, with some information on other vitamins. Nineteen references. J. A. K.

Effect of ketogenic diet on the blood sugar and the respiratory quotient of children. Fritz B. Talbot and Yelma Bates *Am. J. Diseases Children* 50, 827-39(1935).—The R. Q. is reduced in children by the use of ketogenic diets, although not to as great an extent as in adults. The blood sugar is reduced, usually to 70-80 mg. %. The basal heat production is not affected by the diet. E. R. Main

Digestion of milk and of modified milk in vitro. Dorothy Letter and Frederic W. Schlutz *Am. J. Diseases Children* 50, 1101-6(1935).—Unreated milk, lactic acid milk, evapd and powd. milks are digested rapidly and completely at pH 4-5 by artificial gastric juice which contains rennin. In the absence of rennin digestion occurs only in the range of pH 1.8-3.0. I. R. Main

Digestion of milk and of modified milk in vivo. Dorothy Letter and Frederic W. Schlutz *Am. J. Diseases Children* 50, 1107-12(1935).—The time required to produce the highest concn. of sol. N in the gastric contents after ingestion of untreated milk is 1.5 hrs., buttermilk and un-treated milk with added rennin 0.5 hr., evapd. milk with

or without added rennin, lactic acid milk and protein milk 0.5-1 hr. and powd. milk with or without added rennin 0.5-1.5 hrs. E. R. Main

Prevention of rickets with a cod liver oil concentrate in milk. M. G. Peierman and Lily Epstein *Am. J. Diseases Children* 50, 1152-8(1935).—The administration of 223 units of vitamin D per day in the form of a cod-liver-oil concentrate incorporated in evapd. milk provided adequate protection against rickets in infants of the susceptible age period. The infants received in addn. 30 cc. of ripe pineapple juice which gave adequate protection against scurvy. E. R. Main

A further study of the antirachitic factor in human milk. I. A. Sabri and M. M. Fakri *Arch. Disease Childhood* 10, 377-80(1935); cf. C. A. 27, 1921.—Rickets in Egyptian infants appears to be due to an insufficient supply of provitamin D in the mother's milk as well as to the prevailing deficiency in ultraviolet radiation. L. R. M.

The calcium, phosphorus and nitrogen retention of rats on soybean-egg powder and whole milk powder diets. I. Reid *Chinese J. Physiol.* 9, 307-14(1935).—In balance expts. with growing rats further evidence was obtained that soybean-egg powder (cf. C. A. 28, 3709, 29, 4052) contains adequate amts. of vitamin D, Ca and P for normal bone development. The N retention and digestibility were greater than when whole milk powder was used. L. A. Maynard

A preliminary report on the nitrogen metabolism of preschool children. Lau Chen Kung and Wen-Yuen Fang *Chinese J. Physiol.* 9, 375-82(1935).—In expts. with 2 four-year-old children eating a freely chosen, mixed diet contg. soybean milk, the av. daily N intake ranged from 0.33 to 0.56 g. per kg. and the retention from 0.03 to 0.034 g. per kg. In expts. with 2 three-year-old children given a controlled mixed diet with either soybean milk or cow milk the N retention was too variable to reveal any differences between the 2 milks. With intakes of 0.47 to 0.53 g. per kg. retention ranged from 0.46 to 0.14 g. per kg. L. A. Maynard

Teeth and diet. Celestino Guglielmi *Semana med. (Buenos Aires)* 1935, II, 1643-4.—The importance of vitamin D, Ca and P is discussed. A. E. Meyer

Influence of calcium on carbohydrate metabolism. Taichi Harada *Bull. Chem. Soc. Japan* 10, 431-60(1935).—The little studied subject of the effect of Ca in carbohydrate metabolism has been investigated with albino rabbits. Ca lactate was used because it was found to be the most easily assimilated of the common Ca salts. The rabbits, wt. approx. 2 kg. each, were allowed to fast 24 hrs. after which the sugar in the blood and urine was detd. A soln. of 15 g. of glucose was injected into the peritoneal cavity with or without 0.5 g. Ca lactate and (or) 0.2 cc. adrenaline ( $\frac{1}{100}$ ). The blood sugar was again detd. after 0.5-hr. and at 1-hr. intervals for 4 hrs. and finally after 24 hrs. The sugar in the urine for the 24 hr. period was also detd. Adrenaline alone or glucose or a combination of the 2 all raised the blood sugar for several hrs. and increased the sugar in the urine. Ca lactate greatly retarded this increase in both blood and urine sugar when injected with glucose or glucose and adrenaline but had little effect when injected with adrenaline alone. Conclusion. Diabetes is probably attended by imperfect Ca metabolism and this helps to account for the excess blood and urine sugar. John F. Milberry

Studies relating vitamin C deficiency to rheumatic fever and rheumatoid arthritis, experimental clinical and general considerations. II. Rheumatoid (atrophic) arthritis. James F. Rinehart *Ann. Internal Med.* 9, 671-8(1935).—Subacute or chronic vitamin C deficiency in the guinea pig produces an arthropathy with many similarities to rheumatoid arthritis. Hence it may be a factor in the etiology of the disease. John T. Myers

Chemical relationship of blood cholesterol with a summary of our present knowledge of cholesterol metabolism. Lewis M. Hurst and Hazal M. Hunt *Ann. Internal Med.* 9, 717-27(1935).—Diets of exclusive animal food-stuffs high in fat will increase blood cholesterol and may

be a factor in arteriosclerosis. Hypercholesterolemia is frequent in xanthomas, hence a cholesterol-free diet may be useful. In diabetics, more than 400 mg % indicates the imminence of serious complications. Hyperthyroidism causes a drop and hypothyroidism an increase in blood cholesterol. It is low in hypoparathyroidism, and high if there are cholesterol deposits in the body. J. T. M.

Data leading to the supposition of a general vitamin deficiency in certain regions of Spain. L. Carrasco Cadenas *Arch med exp* 37, 501 (1934). *Enferm soc hig aliment* 23, 367. In various regions of Spain veridical thalassa and hemeralopia are encountered, indicating an insufficiency of vitamins in the diet. Similarly, deterioration of teeth and rickets are extremely prevalent in children. A. Papneau-Couture.

The vitamin potency of foodstuffs in Spain. E. Carrasco Cadenas and Calmen Olmedo *Arch med exp* 37, 618 (1934). *Enferm soc hig aliment* 23, 367. Expts showed that fresh tomato juice contains 60-100 carotene units of vitamin A per g., less than 2 units of vitamin B per 2 g., and sufficient vitamin C per 15 g. to retard the appearance of otherwise fatal scurvy. A dose of 0.5 to 1 g. of Andalusian olive oil contains just sufficient vitamin A to prevent xerophthalmia, viz. 8-10 units per g. and 3 antirachitic units per cc. Egg yolk contains no antirachitic vitamin B. A. P. C.

Carotenoids and the visual cycle. Geo. Wald. *J Gen Physiol* 19, 251-77 (1935). The cone-disk pigment and choroid layer of the eye of *Rana pipiens* or *R. esculenta* contain about 17 xanthophyll and about 4  $\mu$  vitamin A. During light adaptation, xanthophyll falls 10-20%. Light-adapted retinas contain about 0.2-0.3  $\mu$  A. Dark-adapted retinas only a trace. Retinene, a hitherto undetected carotenoid, is liberated from visual purple by destruction with  $\text{CHCl}_3$  and by bleaching to visual yellow by light. It reacts with  $\text{SnCl}_4$  to yield a deep blue color, with a single band at about 664 m $\mu$ . Free retinene is removed from isolated retinas by treatment to visual purple and by decomposition, contain several times as much A as retinas from light-adapted frogs. The visual purple system expends A and is dependent upon the diet for its replacement. Visual purple behaves as a conjugated proton with retinene in the proretinetic group. Vitamin A is a precursor and a decomposition product of visual purple, the visual processes constitute a cycle. C. H. Richardson.

The nutritive value of the proteins of corn-gluten meal, husseed meal and soybean meal. Kenneth L. Turk, F. B. Morrison and L. A. Maynard. *J Agr Research* 51, 401-12 (1933). Cf. C. A. 28, 5359. Metabolism studies were conducted with 3 growing weanling rats to det. the digestibility, storage and bio. value of the proteins of soybean-oil, corn-gluten and husseed meals. Each of the feeds was added to a low N ration in such amounts as to furnish a protein level of 10% with approx. 1% additional being furnished by the other ingredients of the ration. All rations were equalized in energy content. The av. coeffs. of apparent digestibility for proteins, the av. percentage of protein intake stored, and the av. of the bio. values of the protein were, resp. 67 b, 23.8 and 72.8% for soybean-oil meal, 66.3, 26.6 and 67.7% for corn-gluten meal, and 63.3, 25.7 and 67.7% for husseed meal. These data show the superiority of the proteins of soybean-oil meal over those furnished by husseed meal and corn-gluten meal. They indicate that it is possible to measure differences in quality of protein using sheep and the N-balance type of experimentation. W. H. Ross.

The importance of cystine for the growth of fur rabbits. A. I. Deravlev. *Sovetskoye Dolo* 1935, Nos. 4-6, 14-17. The addition of 1-10 g. of S daily to the feed of rabbits had no effect. Addition of 0.5 to 10 g. cystine daily increased the wt. and quality of the fur. B. V. Shvartsbereg.

Chemical nature of vitamins. F. G. V. Percec. *Pharm. J.* 193, 651-2; *Chemist and Druggist* 123, 730-7 (1935).—A survey of the present chem. knowledge of

vitamins, with special reference to ascorbic acid (vitamin C). S. Waldbert.

The experimental production of ascorbic acid in dogs by means of a blackening-producing diet. Tom D. Speer and Alexander S. Dowling. *Am J Physiol* 114, 52-62 (1935). Cf. C. A. 27, 4565. The basic diet consisted of cornmeal 400 g., cottonseed 50 g., casein (freed from vitamin B) 95 g., coarsened oil 30 cc., cod-liver oil 15 cc. and salt mixt 25 g. This was supplemented with 2 cc. of a 50% soln. of ferric  $\text{NH}_4$  citrate each day. The loss of wt. and development of anemia, dermatitis and blackening can be prevented by the admin. of large supplements of vitamin C and can be remedied by the admin. of small amounts of yeast. E. D. Walter.

Absorption of grass and butter carotenes on aluminas. A. E. Gillam and M. S. El Ridi. *Nature* 136, 914-15 (1935). Cf. C. A. 29, 5529. When pure  $\beta$ -carotene from grass is twice adsorbed on aluminas, it changes partially into a substance similar to  $\alpha$ -carotene. The process is reversible. On reabsorption the substance reverts in part to the original  $\beta$  form. Cryst.  $\alpha$ -carotene is also changed under these conditions. It should be recognized that changes can be brought about by the process of chromatographic absorption itself. E. D. Walter.

Ascorbic acid (vitamin C) and phytoascorbomata. László Havas. *Nature* 136, 984 (1935). Cf. C. A. 29, 8054. 8051. Injection of a viscous sol. of ascorbic acid into tomato plants increased the size of tumors produced in these plants by *Phytophthora tumefaciens*. The av. wt. of the tumors of the plants treated with ascorbic acid was about 27% greater than that of the control plants. The total amt. of ascorbic acid injected during the 14-day period was about 27 mg. per plant. E. D. Walter.

Differentiation of the antidermatitis factor. Albert C. Hogan and Luther R. Richardson. *Science* 83, 17-18 (1935). Cf. C. A. 28, 7309. Rats become denuded on tinitin, and are healed by flavins. Dermatitis is healed by wheat-germ oil, but growth does not occur unless flavins are added. Wheat-germ oil has no effect on denuding, and flavins have no effect on dermatitis. Vitamin B carriers, eliminated through plate glass for 10 hrs. at a distance of 10 in. with a 1500 w. Mazda bulb, heal dermatitis but flavins in adm. must be added to support normal growth. Apparently the denuded condition and dermatitis have little or no relation to each other. E. D. Walter.

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## I—PHYSIOLOGY

HOMER W. SMITH

**Variations in the lactose content of milk.** W. R. Brown, W. E. Peterson and R. A. Gortner. *J. Dairy Sci.* 19, 81-92 (1936).—A similarity was found between the lactose content of the morning and evening milk of individual cows, when the samples were taken at the regular milking periods. The lactose content showed considerable variation between successive hourly samples. Hourly variations were found in the blood sugar of milking cows. Simultaneously collected samples of blood and milk show little or no positive correlation in sugar content. In general, samples of milk collected 1 hr. later than the blood samples showed greater tendency to higher correlation in sugar content than do simultaneously collected samples. It is suggested that improved comparative studies might be obtained by first stripping the gland, taking the blood sample, then taking the milk sample by stripping 1 hr. later. Philip D. Adams.

**Effect of gangliectomy on mineral composition of the bone.** Alfredo Terranum. *Patologica* 27, 777-80 (1935).—In the bones of the posterior extremities of the dog, after lumbar gangliectomy, there was noted a slight decrease in the total ash (as from 63.52 to 63.41%), a decrease in the Ca content (from 26.80 to 24.11%), an increase in the Mg content (from 0.299 to 0.334%) and a slight increase in the P content (from 10.24 to 10.30%). All the data are based on the wt. of degreased dry bones. G. A. Bravo.

**The glycolytic power of the human blood.** N. Satam. *Fa biologia* 27, 78-84 (1935).—The expts. made in man by means of drugs (Synergine Sandoz, atropine) acting on the vegetative nervous system do not support the idea that this system would have a direct influence on glycolysis in vitro. G. A. Bravo.

**Carbon monoxide intoxication and its relationship to fatigue.** Ugo Bassi and Carlo Sorresina. *Rass med applicata lavoro* 6, 249-268 (1935).—Fatigued guinea pigs showed less resistance to the action of illuminating gas (15% CO) than quiet animals. When the toxic action of CO is prevalent, there is an increase, and when the fatigue is prevalent there is a decrease of hemoglobin and of the globular value. Fatigued and intoxicated animals decreased in wt., but those only intoxicated showed a slight increase. G. A. Bravo.

**Radiation of heat from the human body.** V. James D. Hardy and Carl Muehlenheim. *J. Clin. Investigation* 15, 14-19 (1936).—About 95% of infrared rays are absorbed within 2 mm. of the surface. The therapeutic effect is confined to the surface. The absorption spectrum of human wet skin is essentially that of water. Upon drying other absorption bands appear. J. B. Brown.

**The effects of inhalation of helium mixed with oxygen on the mechanics of respiration.** Alvan L. Barach and Morris Eckman. *J. Clin. Investigation* 15, 47-61 (1936).—Helium decreased the effort of the respiratory musculature and as a phys. function lowered intrapleural pressure. J. B. Brown.

**A test for abnormally large amounts of parathyroid hormone in the blood.** Bengt Hamilton and Walter J. Highman. *J. Clin. Investigation* 15, 99-100 (1936).—cf. C. A. 27, 117.—Serum from a patient is injected intramuscularly into the legs of a rabbit which has fasted for 12 hrs. Immediately afterward the rabbit is given 0.276 g. CaCl<sub>2</sub> in 10 cc. water by stomach tube. This dosage is repeated at 1, 3 and 5 hrs. Serum Ca detns. are made and the results are interpreted based on the Ca rise. J. B. Brown.

**Filterable serum calcium in late pregnant and parturient women and in the newborn.** Marie Andersch and Fred W. Oberst. *J. Clin. Investigation* 15, 131-3 (1936).—As values for filterable serum Ca are constant in nonpregnant, late pregnant and parturient women and in the newborn, reported variations are due primarily to changes in the total Ca. J. B. Brown.

**Liver function as tested by the lipemic curve after**

**intravenous fat administration.** Arthur Nachlas, G. Lyman Duff, Herbert C. Tidwell and L. Emmett Holt, Jr. *J. Clin. Investigation* 15, 143-51 (1936).—Administration of CCl<sub>4</sub> to dogs causes difficulty in removal of fat from the blood after intravenous injection. This is probably due to liver damage caused by CCl<sub>4</sub>. The lipemic curve after fat injection may be of value in the study of liver function. J. B. Brown.

**The lipides of human blood.** Eldon M. Boyd and H. J. Tweddell. *Trans. Roy. Soc. Can.* 29, Sect. V, 113-21 (1935); cf. C. A. 29, 748<sup>9</sup>.—Blood samples were taken from 9 human subjects every 3 hrs. during the day and 4 hrs. during the night and analyzed for total lipide (I), total fat acids (II), phospholipides (III), total cholesterol, ester cholesterol (IV), free cholesterol (V) and neutral fats (VI). III and V remained relatively constant during the 24 hrs. on a normal balanced diet. IV began to rise slightly during the p. m., reaching a max. at 4 a. m. VI was lowest at 4 a. m. and increased to a max. plateau at 8 a. m. to 5 p. m. The curve for II closely paralleled that for I. From the changes recorded it is concluded that variations in IV and VI occur chiefly in the red blood cells. Rachel Brown.

**The estrogenic activity of 1-oxo-1,2,3,4-tetrahydropheanthrene, an addition to our communication on 1-oxo-1,2,3,4-tetrahydropheanthrol 7.** Adolf Butenandt and Gerhard Schramm. *Ber.* 68B, 2303 (1935).—cf. C. A. 30, 1375<sup>7</sup>. Cook, Dodds and Hewett, *C. A.* 27, 1449, 3525.—1-Oxo-1,2,3,4-tetrahydropheanthrene is estrogenically active for castrated female mice in a single 70 mg. dose, but inactive if divided into 6 portions and administered in 3 days. W. F. Bruce.

**Gases in the blood during muscle work.** I. N. N. Blokhin. *J. Physiol. (U. S. S. R.)* 19, 1258-61 (1935).—Work which requires increased breathing, bordering on asphyxiation, markedly lowers the amt. of CO<sub>2</sub> in the blood, both in the arteries and in the veins, the muscles intensively detain O<sub>2</sub> and give up CO<sub>2</sub> to the blood. H. Cohen.

**The toxicity of the air exhaled by man.** M. I. Gramentzki and I. I. Sivertsev. *J. Physiol. (U. S. S. R.)* 19, 1265-70 (1935).—The action of exhaled air on an isolated frog heart was studied by an improved Straub pneumatic registration method. In most of the expts., the replacement of the ordinary room air by exhaled air caused a weakening in the heart activity. The causative agent is not merely the CO<sub>2</sub> of the exhaled air, some other volatile substances, probably of basic nature, are responsible. The air of old and sick persons is more toxic than that of young and healthy individuals. H. C.

**General character of the unconditioned reflex activity of the two parotid glands.** Ya. P. Sklyarov. *Med. eksptl. (Ukraine)* No. 2, 66-73 (1935).—The activity of the glands depended on the localization of the stimulant (food) in the mouth, when placed on the right side the right gland worked more intensively, when the stimulant was on the left side the left gland secreted more. Apparently the excitation spreads along one side of the reflex arc with very little crossing. S. A. Corson.

**The aqueous and mineral fraction of saliva during continuous secretion.** P. Ya. Mukhina. *Med. eksptl. (Ukraine)* No. 2, 74-89 (1935).—In acute expts. on dogs the spinal cord was cut, producing a continuous secretion from the submaxillary glands. Every 12-21 min. warm Ringer soln. was introduced intravenously to compensate for the loss of fluid. The quantity of saliva decreased regularly after 3-5 hrs. This was due to a decrease in the nervous stimulation and not to exhaustion of the aq. function of the gland because bleeding (thus stimulating the central nervous system), injection of pilocarpine (0.1 cc. of a 1% soln. per 5 kg. of body weight) or elec. stimulation of the cut nerve again increased salivation. This aq. function was practically inexhaustible because repeated stimulation of the nerve sometimes produced a greater salivation than the first stimulation. Moreover, the gland which was continuously active did not respond less intensely to pilocarpine than the gland which was inactive throughout the expt. (because of cut nerve).

The latter did give at first a greater percentage of solid residue which decreased subsequently. The rate of secretion of both sublingual glands was usually similar and had no relation to the blood pressure. The decrease in the solid residue with time (this was apparently independent of the rate of secretion) was mostly at the expense of the org. components, the salts remaining practically const.

**Influence of the water vapor content of an odorous gas on the olfactory sensation** H. Wierdeman *Arch. neerland. physiol.* 20, 591-5 (1935).—Fifty four of the observations showed considerable differences in the intensity of the smell of isoamyl acetate when brought to the nose through the aid of dry air as compared with humid air. Dry air gave sensibly greater results. T. L. B.

**The effect of high concentrations of light negative at morphine ions on the growth and activity of the embryo** L. P. Herrington and Karl L. Smith *J. Ind. Hyg.* 17, 253-8 (1935).—A comparison of the weight curves and the mean values for hemoglobin through the growth period of the group with those of a comparable control group revealed no significant differences. The mean activity of the expl. group was significantly greater than that of the control group. Fifty albino rats were exposed to an atm. contg.  $1.2 \times 10^6$  neg. ions per cc.

The ions had a mobility in the order of 1 cm. per sec. during the age period from 21 to 700 days. A. L. E.

**Cytology of the adrenal** F. J. McKenzie and Laura J. Naiman *Mod. Agr. Expt. Sta. Bull.* No. 349 (4th ser. Rept. 1933), 13 (1934).—Changes were found in the fat and mitochondria content of cortical cells during the estral cycle. In the zona glomerulosa these changes involved a variation in the number and size of the fat droplets. The changes in fat content were continuous throughout the cycle showing an increase in proestrus and early estrus and reaching a height in early metestrus. This was followed by a decrease throughout diestrus. Changes in the fat content of the cells of the zona fasciculata and reticularis closely paralleled those of the zona glomerulosa. C. R. Teller.

**Effect of alkalinization of drinking water on the pH of the jugular blood of feeder cattle** Paul Gerlaugh, C. H. Hunt and B. H. Edgington *Ohio Agr. Expt. Sta. Bull.* No. 548 (3rd Ann. Rept.), 78 (1935).—1 or 2 cattle receiving approx. 3 gal. daily of water contg. 1%  $\text{NaHCO}_3$ , the pH of the jugular blood was 7.68 compared to a value of 7.54 for the controls. In another shipment of cattle these resp. values for pH were 8.01 and 7.84. There was no difference in the occurrence of disease in the untreated and alkalinized cattle. R. Teller.

**Basal metabolism of women over 35 years of age** J. Hingham McKay and Mary Brown Patton *Ohio Agr. Expt. Sta. Bull.* No. 548 (3rd Ann. Rept.), 82 (1935).—The basal metabolism of women above 35 years of age remains at a fairly uniform level until the age of 50 is reached, after which the basal metabolic index declines to a definitely lower figure per unit of wt. or surface area. The heat production at ages 25-50 averaged 21.1 cal. per kg. per 24 hrs. At age 50-60 this value averaged 19.5, at age 61-18 yrs., the value was 25.4 cal. C. R. Teller.

**Osteostrophy and hormone influence** Josef Marx *Osteostroph. Rev.* 12, 1-4 (1935).—Expts. on guinea pigs with "Thymicola" (cf. Labopharma at Berlin) and "Verhen" (of Richter at Budapest) proved that not only the secondary thyroid gland but also the thymus and spleen take part in the control of Ca metabolism. An increased calcification caused by an artificial hyperfunction of secondary thyroid could not be prevented by simultaneous administration of thymus and spleen preps. Curing of osteostrophy by means of such hormones seems to be successful. S. S. de Finis.

**Butyric acid content of normal urine** Lashby King *Bull. soc. chim. biol.* 17, 1540-5 (1935); cf. C. d. 29, 7057.— $\text{PrCoH}$  does not occur in normal human urine. About 4-8 mg. wet butyric acid is excreted in the urine in 24 hrs. Butyric acid of feces *Ibid.* 1546-8.—The micro-

num and the feces of healthy young infants contain no  $\text{PrCoH}$ . Feces of adults contain 40-130 mg. per 100 g. fresh material. L. F. Gibson.

**A characteristic of the normal physiological state** The bromine index of the urine N. Bessendorf, A. Vallette and R. Sauerz *Bull. soc. chim. biol.* 17, 1573-98 (1935). The Br index or Br no. of the urine is the no. of mg. of free Br fixed by 1 cc. of urine under specified conditions. The method is described. The Br no. for the urine of healthy infants aged 1 month is 0.5-1.0, age 6 months, 0.9-1.1, 1 yr., 1.2 and 2 yrs., 1.2-1.4. In pathological states it may exceed 7. The Br no. for adults is 0.8-4.7, av. about 2.6. It is increased in fat rich diuretics and in rickets. It is not dependent on the mineral salts, or the total C of the urine. It depends partly on the phenols and indazole derivs., and probably the indole derivs. of the urine, hence it is related to the protein metabolism. In avitaminosis C the Br no. increases, indicating a possible connection between vitamin C and protein metabolism. L. F. Gibson.

**Doses of folliculin required to produce intersexes and limits of stage of intervention** E. Eneine Wolff and Albert Gangelinger *Compt. rend. soc. biol.* 120, 114-16 (1935). A drop of oil soln. of folliculin benzoate contg. 2500-5000 mouse units introduced into the abdominal sac of a chick embryo on the 4th or 5th day of incubation converts it to an intersexual form. Structure of the genital glands of different types of intersexes obtained by injecting folliculin into male chick embryos *Ibid.* 116-18.—Development of the male intersexes obtained by injecting folliculin into the embryo. Morphological changes in the genital organs after hatching. Histological changes in the genital organs after hatching. E. Eneine Wolff *Ibid.* 904-5.—Action of male hormone (androsterone) injected into the chick embryo. Production of intersexes *Ibid.* 1312-14.—Discussion of results obtained with male hormone *Ibid.* 1314-16.

**Sexual inversion of the ovarian euchaize in the chick embryo [by injection of sex hormones]** Vera Danachakov. *Compt. rend. soc. biol.* 120, 277-9 (1935).—Mechanism of the action of folliculin injected into a male chick embryo *Ibid.* 950-3.

**Azotemia and chloremia in vitro** J. Loslever *Compt. rend. soc. biol.* 120, 605-7 (1935).—The addition of N compounds to blood *in vitro* modifies the Cl content of the corpuscles in a sense which depends on the permeability of the corpuscles for the compounds added. Thus, urea, which readily enters the cells, causes a decrease in cell Cl, while glycerol, which does not enter, causes an increase. The effect in either case equalizes osmotic pressure. Azotemia and chloremia during hystolysis *Ibid.* 675-8.—In tissue destruction *in vivo* each of the various waste N compounds entering the blood stream affects the corpuscle Cl in one or the other of the above-mentioned ways.

**Changes in the epithelium of the bladders of mice during urine retention after injections of estrone** E. Laroze *Compt. rend. soc. biol.* 120, 813-5 (1935).—Estrone was repeatedly injected into young mice. Retention of the urine occurred in some of the mice after several injections. In such cases the epithelial cells of the bladder showed a marked proliferation. Probably the result of the prolonged contact with this hormone in the bladder contents. L. E. Gibson.

**Presence in the urine of certain women of a substance antagonistic to estrous hormone** D. Duran and P. Dragon *Compt. rend. soc. biol.* 120, 852-3 (1935).—When the urine of a certain woman, age 30, with symptoms of ovarian insufficiency, was injected into young female mice either before or during the same time that injections of urine from pregnant women were made the Aschheim reaction was neg. in every case. The presence of an antagonistic substance is postulated.

**Adsorption of creatine and creatinine by red corpuscles.** K. Kael *Compt. rend. soc. biol.* 120, 916-17 (1935).—Washed beef erythrocytes did not adsorb creatine from



dal solns in Locke soln Under the same conditions they adsorb considerable creatinine, the proportion increasing with the temp throughout the range 7-44°. Effect of  $pH$  on the adsorption of creatinine by red blood corpuscles. *Ibid* 918-20.—The  $pH$  range 6.4-8.3 was used. Max. adsorption occurred at 7.3. L E Gilson.

*Folliculuria during nursing* Giuseppe Tata *Rass. clin terap sci affini* 34, 265-70 (1935), cf C A 29, 7428.—During the period of amenorrhoea nursing women showed very low or no folliculuria while with return of menstruation the titer rose to the normal values of the sexual cycle. Helen Lee Gruel.

Recent advances in science agricultural physiology Arthur Walton *Science Progress* 30, 510-13 (1935).—A review devoted chiefly to endocrine studies. J S H.

The chemistry and physiology of the androsterone group Kurt Tscherning *Angew Chem* 49, 11-16 (1936).—The following subjects are discussed: (1) the natural representative of the androsterone group, (2) the specificity of the androsterone action, (a) the capon comb test, (b) the vesicular gland test, (3) the relation between the androsterone group and the estrone group. Data and twenty-five references are given. Karl Kammermeyer.

Chemical studies on the adrenal cortex I. Fractionation studies on hormone concentrates J J Pfaffner, Oskar Wintersterner and Harry M Vars *J Biol Chem* 111, 585-97 (1935). II. Isolation of several physiologically inactive crystalline compounds from active extracts Oskar Wintersterner and J J Pfaffner *Ibid* 599-612, cf. C A 28, 6814.—The permittive purified fraction (11.9 g) from 100 kg of beef adrenal gland assaying 15 dog units per mg was subjected to a  $C_2H_4$  distribution procedure and a  $C_2H_4$  concentrate of 450 mg assaying 250 dog units per mg was obtained. This was further fractionated and a final active fraction of 153 mg assaying 400 dog units per mg isolated. It was a pale yellow sirup which would not crystallize. Evidence was obtained of the presence of an  $\alpha, \beta$ -unsatd ketone and of hydroxyl groups in the hormone. In the process of fractionation of the hormone concentrates 5 physiol inactive cryst compds were isolated. Four were  $N$ -free and the 5th,  $C_{17}H_{24}O_2N_2$ , is in all probability leucylproline anhydride. One of the compds is probably identical with the substance regarded by Kendall, et al *Proc Staff Meetings Mayo Clin* 10, 245 (1935) as the life-maintenance hormone. The discrepancy between the findings of these investigators and the authors' results regarding the physiol activity of this compd is discussed. Preliminary analytical characterization of the compds is given but larger quantities are necessary to establish with certainty their compn.

The biological activity of theelin Roland C Meyer, Lloyd C Miller and Geo F Cartland *J Biol Chem* 112, 597-604 (1936).—Theelin has little biol activity as regards the adult spayed rat but much when detd in the immature rat. The quant relationship between theelin and theelin differs with different methods of assay. When administered in oil and measured by the vaginal smear method international standard theelin is approx 90 times more active than theelin and 250 times more active when injected in aq 10% alc contg 0.5%  $Na_2CO_3$ . Theelin is 4 times as active as standard theelin when detd by the opening of the vagina in immature rats.

The influence of feeding amino acids and other compounds on the excretion of creatine and creatinine. Meyer Bodansky with the technical assistance of Virginia B Duff *J Biol Chem* 112, 615-24 (1936).—Arginine, aspartic acid, glutamic acid, cystine, histidine, tyrosine, choline, betaine, sarcosine and the salts of guanidine administered in comparatively large doses by stomach tube produced no effect on the excretion of creatine or creatinine in young adult rats. The output of creatine was definitely increased and that of creatinine was not influenced by glycine. Alanine produced similar though less striking or const changes. Increased elimination of creatine following ingestion of guanidine-acetic acid suggests its probable conversion into creatine but at best this is

only presumptive evidence of extra creatine formation. Creatine given in doses varying from 35 to 120 mg was recovered in the urine within 48 hrs in amts varying from 30 to 80%. The oxidation product, creatone, produced no significant change in either the creatine or creatinine output. A P Lorthrop.

The composition and action upon calcium metabolism of Ah-chiao (donkey skin glue) and commercial gelatin T G Ni *Chinese J Physiol* 9, 329-38 (1935).—Donkey skin glue, derived mainly from collagen, resembles gelatin in contg the essential amino acids, lysine, arginine and histidine. Though contg only very small amts of Ca both the glue and gelatin markedly increased Ca retention as shown by limited balance expts with 2 dogs. The possibility of a Ca-carrying action by glycine is suggested. L A Maynard.

The hematopoietic action of Ah-chiao (donkey skin glue). T G Ni *Chinese J Physiol* 9, 383-94 (1935).—In dogs the drug possesses a hematopoietic action comparable to gelatin, which is not dependent upon the Fe and Cu contents. L A Maynard.

Blood indican in normal conditions Luigi Pinelli *Biochim lerap sper* 22, 563-7 (1935).—The indican in the blood of normal persons varies between 0.20 and 0.72 mg per 1000 cc. It is found exclusively in the plasma, not in the corpuscles. A E Meyer.

Ketogenesis in liver tissue and its autoregulation. S Leites and A I Odinov. *Biochem Z* 282, 345-61 (1935).—Rabbit liver pulp incubated for 24 hrs produces, apparently through autooxidation,  $\beta$ -hydroxybutyric and acetoacetic acids. This ketogenic process is much more pronounced in livers from rabbits which had fasted 48 hrs. The optimum conditions for acetoacetic acid formation in liver from nonfasting rabbits is  $pH$  5.6, and for  $\beta$ -hydroxybutyric acid formation  $pH$  7.0, but in liver from fasting rabbits the optimum  $pH$  is 5.6 for both. The amt. of ketogenesis in 24 hrs depends to a degree upon the original acetone body content of the liver, the lower the preformed content the more intense is the production of new acetone bodies. Likewise the ketogenesis from added butyric acid depends upon the content of the liver in preformed acetone bodies, with a low preformed level  $\beta$ -hydroxybutyric acid is formed, but with a high level the amt. of  $\beta$ -hydroxybutyric acid after incubation for 24 hrs may actually diminish. Added to the liver of fasting rabbits, butyric acid stimulates more intensive ketogenesis than in livers of nonfasting rabbits, and only when the preformed ketogenesis is very extensive does it actually exert a depressing effect. Neither  $AcONa$  nor pyruvic acid has any influence on the ketogenesis in the incubated liver pulp. S Morgulis.

Influence of the cortical hormone on the glycogen content of the liver Herbert A Hochfeld *Biochem Z* 282, 392-8 (1935).—The liver glycogen of the rat on a pure fat diet is somewhat higher than on a normal diet, if the glycogen is detd following one fast day. Likewise in rats, after one day of fasting, the liver glycogen is greater if the animals, 3 days previously, have been fed pure fat and injected with cortical hormone than if they have been fed a mixed diet and treated with the hormone. The glycogen content of the liver is much higher if the cortical treatment is given also during the fast day. This seems to indicate sugar formation from the fat depots under cortical hormone influence. S Morgulis.

Transformation of fat to sugar. Walter Haarmann *Biochem Z* 282, 406-18 (1935).—The lactic acid formation in surviving organs is increased by the addn of butyric and of  $\beta$ -hydroxybutyric acids. This increase is not covered by the loss of carbohydrate, therefore, the fat acids must serve as the source of the lactic acid formed, and thus of sugar since lactic acid is converted to sugar. S Morgulis.

Comparative studies on lactic acid formation and carbohydrate loss in tissues under aerobic and anaerobic conditions. Walter Haarmann and Hans Brunk. *Biochem Z* 282, 419-33 (1935).—Considerable differences were observed in the lactic acid formation and carbohydrate diminution in various organs and animals in the presence or absence

of  $O_2$ .  $O_2$  does not cause a rise in lactic acid in the heart muscle (beef, dog) or in skeletal muscles of beef or brain tissue of dog. In  $O_2$ , on the addition of glucose or glycogen, the lactic acid is unchanged. In the muscles of rabbits, however, the lactic acid formation on the addition of glucose or glycogen is less in  $O_2$  than in its absence. In brain tissue (dog) lactic acid is formed from glucose both aerobically and anaerobically, just like in tumor tissue. The decrease in carboxylate in the absence of  $O_2$  is usually much greater than the corresponding increase in lactic acid. The carbohydrate of beef muscle decreases less aerobically than anaerobically, but in rabbit or dog muscle this difference is not observed. In the beef heart the glucose diminishes much more under aerobic conditions than under anaerobic, but in the dog heart the reverse is the case. In the brain glucose disappears more rapidly aerobically than anaerobically. Glycogenolysis in beef muscle is greater under aerobic than under anaerobic conditions, but in rabbit muscles or in beef heart there is no such difference.  $O_2$  does not inhibit lactic acid formation from pyruvic acid, methylglyoxal, fumaric or succinic acid. Succinic acid is not transferred to lactic acid at all under anaerobic conditions.

S Morgulis

Disappearance of lactic acid in tissues under aerobic and anaerobic conditions. Walter Haarmann and Hans Brunk *Biochem. Z.* 282, 434-441 (1935).—Lactate added to tissues disappears equally well both under aerobic and anaerobic conditions.

S Morgulis

Calcium metabolism in the first phase of coagulation. III. Mechanism of thrombin formation. H. Fessenden *Biochem. Z.* 283, 111 (1935), of *C. A.* 29, 8793. The reaction of  $Ca^{++}$  with prothrombin, which initiates the thrombin formation, obeys the mass-action law. The inhibiting effect of  $Ca^{++}$  prothrombin excess is attributable to the formation of other inactive substances besides thrombin. Under the influence of thromboplastin more  $Ca^{++}$  complex is formed (thrombin- $Ca$ ) depending upon the thrombin present in the serum. The reaction likewise follows the law of mass action. Thus, prothrombin +  $Ca^{++}$   $\rightleftharpoons$  thrombin + thromboplastin  $\rightleftharpoons$  thrombin, or thrombin/(thrombin/prothrombin)  $\times$   $Ca^{++}$   $\rightleftharpoons$  thrombin. Normal dog serum contains the optimum amt. of  $Ca^{++}$  for the prothrombin content, but an excess of prothrombinogen for the amt. of thromboplastin.

S Morgulis

Cleavage, oxidation and energy metabolism in the dog. IV. Absorption of fructose by the organs of the dog in vivo. M. Wieruchowski and H. Fessenden *Biochem. Z.* 283, 30-44 (1935); of *C. A.* 30, 10904.—Fructose administered by continuous intravenous injection is absorbed with remarkable constancy by the various organs, in the following order: liver 21 mg. per g. per hr., of the portal vein 3.5 mg. and the motor system 0.4 mg. (for muscles alone 0.8 mg.), the head organs 0.39 mg., and all other organs 2 mg. The formation and absorption of glucose pass the usual cycle observed in the fasting state while the fructose is intensively metabolized. The liver produces 6.1 mg. glucose per g. and per hr. which is taken up as follows: head organs 1.3 mg. and portal vein organs 1.0 mg., motor system 0.02 mg. and the other organs 0.32 mg. per g. and per hr. One-fifth of the fructose absorbed by the liver is discharged into the blood as lactic acid. On the other hand, in the organs of the head and of the portal vein the lactic acid is discharged into the blood is independent of the fructose absorption and is related to the glucose absorption, just as in the fasting state. Only in the motor system part of the lactic acid may be derived from the fructose. The administration of inositol,  $C_6H_{12}O_6Na$  or  $AClH$  has no effect on the glucose formation in the liver.

S Morgulis

The principal path of lactic acid formation in muscles. O. Meyrhold and V. Kessling *Biochem. Z.* 283, 83-111 (1935).—In addition to the 2 mols. lactic acid resulting from the interaction of  $\alpha$ -glycerophosphate and pyruvic acid, with the primary phosphorylation product of hexose, whereby 1 mol. inosophosphate is oxidized to phospho-

glyceric acid and 1 mol. pyruvic acid is reduced to lactic acid. The second process is much more rapid. The glucolysis of the muscle, like the fermentation process, can be split into partial reactions either by means of  $NaP$  or  $CH_3CO_2H$ , the pyruvic acid being the biol. equiv. of the  $AClH$  and lactic acid of the  $alc$ . The phosphorylation and dephosphorylation proceed stoichiometrically through the adenylic acid system. The glucolysis, as fermentation in that hexosediphosphate reacts in muscle ext. with pyruvic acid reducing it to lactic acid and becoming oxidized to phosphoglyceric acid. This reaction does not involve recarbonation, so that it may progress even when the adenylic acid system has been very much attenuated. The function of methylglyoxal as an intermediate phase is entirely denied.

S Morgulis

Synthesis of creatine phosphate in organ extracts and in living spermatozoa. Isabella Török, *Biochem. Z.* 283, 129-33 (1935).—The transformation of phosphopyruvic acid to phosphocreatine proceeds best, next to muscle ext., in expts from testes. This is apparently a function of the live sperm, because considerable synthesis is secured also with spermatozoa washed in serum.

S M

The part played by the myogen volume in the muscle fiber volume. Wilhelmine Haumann and Hans H. Weber *Biochem. Z.* 283, 146-52 (1935).—Lactate added to about 35% of the vol. cut mammalian muscle, but in uncut single fibers it probably does not take up quite 20% of the vol.

S Morgulis

Influence of thyroparathyroidectomy and of bile acids on the bile acid excretion and the  $pH$  of fasting bile. Shunsaku Tsujioka *J. Biochem. (Japan)* 22, 307-74 (1935).—Before parathyroidectomy 0-71 cc. bile was secreted in 6 hrs. of sp. gr. 1010-1025,  $pH$  6.81-7.02 and a taurocholic acid content of 144-890 mg., or 0.213-0.329% concn. Two or 3 days after thyroparathyroidectomy the bile excretion decreased on the av. 48.4% with a 39.2% smaller taurocholic acid content (the relative concn. increased 32%) and a  $pH$  7.03-8.42. On feeding taurocholic acid, 3-5 days after the operation, the secretion of bile increased on the av. 21% and that of taurocholic acid 37.8%, while the  $pH$  decreased to 6.70-7.88. Thyroparathyroidectomy thus leads to a thickening of the bile and greater alkali, which is a reflection of the general alkalosis.

S Morgulis

The composition of the nitrogenous components of dog stomach wall. Kozo Ikeyama *J. Biochem. (Japan)* 22, 337-407 (1935).—The fundus, cardiac and pyloric of dog stomach were analyzed. The muscular and mucous layers were analyzed separately. The material was cut into small pieces and preserved in 0.4% alc, then it was extd. 3 days in an alc-ether mixt. and filtered off. The total in these sep. portions varied from 0.14 to 0.15 g. The various amino acid fractions were detd. in the  $HCl$  hydrolyzate and the results of this analysis are tabulated. Likewise hydrolysis was carried out with  $H_2SO_4$ . The original alc. ext. was analyzed for volume of which the highest content was found in the fundus muscles, then in the pylorus (muscle and mucous layers).

S M

The mechanism of origin of alimentary hyperglycemia. Yoshikatsu Fukui *Biochem. (Japan)* 22, 447-50 (1935).—The stimulation theory of alimentary hyperglycemia assumes that the resorption of sugar from the small intestine is not quick enough to account for the rapid rise in glucemia. However, it has been shown that the absorption actually proceeds rapidly and, furthermore, levulose administered by mouth very quickly appears in the peripheral blood. The claim of the adherents of this theory that mere contact of the sugar soln. with the intestinal mucosa acts as a releasing stimulus is likewise denied as not fitting the facts. On the basis of this theory also the sugar level in the hepatic vein should be higher than in the portal vein, thus, however, is not the case. Were the alimentary hyperglycemia due to a sugar mobilization from the liver it might be expected to be dependent upon the liver glycogen store, but in fasting the hyperglycemia response is even greater than in the nourished state, and definite alimentary hyperglycemia occurs in other condi-

tions, where the glycogen is low, whereas the mobilization hyperglycemia (pique, adrenaline, pilocarpine) is definitely dependent on the glycogen depots. Another argument for this theory is the inhibition of the alimentary hyperglycemia by ergotamine or atropine. This is experimentally corroborated, but it is shown that these drugs actually slow up the absorption of the glucose from the intestines. On the contrary, phenobarbital narcosis which paralyzes the vegetative nerve centers, has no effect at all on the alimentary hyperglycemia. The very rapid rise in blood glucose does not contradict the absorption theory because the absorption is really very rapid. In favor of this theory is also the fact that there is no noticeable difference in the glucemia level of the hepatic and portal blood, thus showing that sugar is mobilized from the liver. The inhibitory effect of the vegetative nervous system poisons has been shown to depend upon the inhibition of the absorption of sugar from the intestine. Sectioning of the splanchnics and of the vagus causes a flattening of the glucemia curve of the peripheral blood in alimentary hyperglycemia. Sectioning of the splanchnics also flattens the sugar level in the portal vein, but sectioning the vagus, or both the splanchnic and vagus, causes actually a rise in the level. These effects are attributed to effects on the sugar absorption from the intestines. The fact that levulose administration causes chiefly a rise in the blood glucose loses its significance for the stimulation theory, since it fails to produce any difference in the sugar content of the hepatic portal and the sugar level in the hepatic vein. The increase in glucose after the feeding of glucose must be attributed to its conversion to glucose in the liver. The diseased liver has this ability in a more limited degree, which indirectly speaks for such a conversion. S Morgulis

Internal exchange in the glandular tissues A M Uteviski, E I Kovtun and K M Shleifer *Med expil* (Ukraine) No 1, 32-7(1934)—The glucolysis in the glandular tissues is different from that in the muscles. Glucose is a better generator of lactic acid than glycogen. The formation of lactic acid is increased by the presence of pyrotartaric acid salts. The presence of alanine also stimulates the generators of lactic acid. L N

Liver and glutathione Léon Binet and G Weller *Compt rend* 201, 992-3(1935)—Detrit of total glutathione (I) in arterial, portal and hepatic blood by the method described (C A 28, 4095<sup>7</sup>, 29, 6933<sup>9</sup>) demonstrates that I is taken from the liver by blood during periods of fasting, but is absorbed from blood by the liver during periods of digestion. W Gordon Rose

The diffusible calcium in the serum of laying and non-laying hens Milton W Taylor and Walter C Russell *J Agr Research* 51, 663-7(1935)—By the use of collodion membranes, the diffusible Ca of the serum of non-laying hens was found to be  $5.4 \pm 0.42$  and that of the same hens in the laying condition  $5.3 \pm 0.17$  mg per 100 cc of serum. No difference was observed in the level of diffusible Ca when the birds changed from a non-laying to a laying condition or vice versa. The nondiffusible Ca in the serum rose from a value of  $6.4 \pm 0.53$  mg per 100 cc for non-laying birds to a value of  $16.1 \pm 1.17$  mg per 100 cc for the same birds in the laying condition. W H Ross

Purified growth hormone from beef anterior pituitary II Dialyzable growth hormone E Dugemans and J Freud *Acta Brevia Neerland Physiol, Pharmacol, Microbiol* 5, 109-11(1935), cf C A 29, 6294<sup>7</sup>—Preps capable of promoting growth in hypophysectomized rats and of low N content were obtained from the dialyzate of crude or purified exts of the anterior pituitary. E W S

The physicochemical state of the adrenalinic hormone in the blood David Broun and H Scheiner *Compt rend* 201, 794-5(1935), cf C A 29, 8084<sup>7</sup>—Fresh suprarenal glands were suspended in serum, distd H<sub>2</sub>O and H<sub>2</sub>O acidified to pH 3.5-4.0. Injections of these solns produced hypertension. The serum soln lost this property upon ultrafiltration. The others did not. Addn of CaCl<sub>2</sub>, distd H<sub>2</sub>O or acid (to give pH 4.0-4.5) to the

serum soln released the adrenaline (I). Conclusions I in the blood is fixed on the nonultrafilterable constituents of the serum from which it is released by dial, addn of acid or of Ca ion. E W Scott

Nitrogen metabolism in protein starvation Gilberte Mourot *Compt rend* 201, 1044-6(1935); cf C A 29, 1872<sup>1</sup>—A discussion. E. D. Walter

The physicochemical state of acetylcholine in the blood David Broun and Hermann Scheiner *Compt rend* 201, 1046-8(1935)—Acetylcholine appears to exist in the tissues or in the blood as a physiologically inactive complex. The nervous influx liberates acetylcholine from this complex in proportion to the intensity of the stimulus. E D Walter

Chemistry of estrogenic substances J W Cook, E C Dodds and F L Warren *Nature* 136, 912-13 (1935), cf C A 29, 5899<sup>7</sup>—Subcutaneous injections of 300 mg of Na benzylidene pyruvate, in 3 cc of H<sub>2</sub>O, into ovariectomized rats did not produce any estrogenic activity. Subcutaneous injections of 8 mg of either Na benzylidene pyruvate or Na furylidene pyruvate into mice, at 24-hr intervals for 3 days, did not produce any estrus response. E D Walter

The occurrence of creatinuria in adult Chinese males Lu Gwei Djen and B S Platt *Trans 9th Congr Far East Assoc Trop Med* 1, 379-86(1931)—The percentage occurrence of creatinuria for the following age groups is: 21-32 yrs, 90.2%, 18-30 yrs, 60.9%, 23-58 yrs, 30.0%; 21-58 yrs, 52.3%. There is no difference due to sex. I S Yun

Factors affecting milk and butter fat secretion (Whetham, Hammond) 12 Sex hormones (Ruzicka, et al) 10 Androsterone and related sterols (Marker, et al) 10

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## G—PATHOLOGY

H GIDEON WELLS

Cholesterol content of whole blood in patients with arterial hypertension A H Elliot and F R Nuzum *Arch Internal Med* 57, 63-72(1936)—Uncomplicated arterial hypertension is not accompanied by hypercholes-

terolemia. Further, the cholesterol level is not increased when vascular degeneration or renal impairment accompanies the hypertension. Higher values for cholesterol are more frequently found in underweight persons than in obese.

**Adrenal insufficiency.** Jules Stahl, Dana W. Atchley and Robert F. Loeb. *J. Clin. Investigation* 15, 41-49 (1936).—The decrease in Na content and increase in blood urea in adrenal insufficiency are not interdependent although they sometimes occur simultaneously. Withdrawal of NaCl from the diet of an adrenalectomized dog maintained on cortical extract results in increase in urea and decrease in Na, as did also the withdrawal of extract. If an universally potent test was employed, no change in Na and urea was observed on a low-salt diet. Withdrawal of salt or cortical extract decreased renal function.  $\text{NH}_4\text{Cl}$  excretion was decreased by decrease in dosage of extract. Striking improvement in well-being was observed following large doses of extract before consistent changes in blood Na, K or urea were observed. Likewise, heavy dosage inhibited appearance of decreased renal function. Standardization of cortical extract based on its effect on blood urea in the adrenalectomized dog is unreliable, unless rigid control of the salt content of the diet is maintained. J. B. Brown.

The effect of drugs in the production of agranulocytosis with particular reference to amadopyrine hypersensitivity. Wm. Dameshek and Abraham Colburn. *J. Clin. Investigation* 15, 53-57 (1936).

**Plasma lipids in chronic hemorrhagic nephritis.** Irvine H. Pace, Euben Kirk and Donald D. Van Slyke. *J. Clin. Investigation* 15, 101-110 (1936).—Of 41, 30, 140.—Total lipids in nephritic as well as normal plasma can be estimated as 1.3 times the total C, with an error of less than 1%. Details of the different lipide fractions by the gravimetric method (C. A. 29, 6149) were made on 32 patients with chronic hemorrhagic nephritis, the degree of renal damage being slight to almost complete. In the chronic active stage (urea clearance over 20% normal) there is a tendency to lipemia, the plasma lipides being near or above the upper limits of normal, 1.0-2.0 g./100 cc. Near the terminal stage the lipemia decreases and toward the end may fall below normal. Free cholesterol, cholesterol esters, phosphatides and neutral fat fractions rise and fall together, the percentage of the total of each deviating essentially from the normal. The high N/P ratio (3-15) in the terminal lipides is evidence that the N was present in the form of petroleum ether-sol substances other than phosphatides. The severity of the lipemia does not parallel the plasma protein deficit. Plasma lipides in essential hypertension. *Ibid.* 109-112.—Of 16 cases of uncomplicated essential hypertension in none was the concentration of total lipides or any of the lipide fractions outside the usual range of normal values. There seems to be no basis for associating the arterial changes in essential hypertension with hypercholesterolemia or elevation of the cholesterol/phosphatide ratio. In 2 cases of malignant sclerosis the lipide content was in or above the normal range. J. B. Brown.

The problem of an antidiuretic substance in the blood of patients with eclampsia and other hypertensive diseases with observations on spinal fluid. Geo. Levitt. *J. Clin. Investigation* 15, 135-141 (1936).—The methods employed were unable to detect an increased amount of diuretic substance (presence of the posterior pituitary lobe) in the blood of patients with eclampsia and related disturbances.

**Vitamins, hormones and disease.** T. Swan Harding. *Am. Med.* 41, 835-9 (1935).

**Serum proteins in cancer.** Wladislaw Kopaewsky. *Comp. rend.* 201, 1263-5 (1935).—Of C. A. 29, 4979.—In cancer the total of the serum globulin and albumin decreases while the myxoprotein may increase as much as 100%. There is a gradual parallelism between the total of the hydrophobic proteins and the rapidity of the lactation of the serum. Not only the amount but the phys. state of the hydrophobic colloids affects the acceleration of the lactation of serum in cancer. R. B.

Phosphoric esters in normal and malignant tissues

E. L. Outhouse. *Trans. Roy. Soc. Can.* 29, Sect. V, 77-84 (1935).—The total, free and org. P of the  $\text{CaCl}_2\text{COOH}$  extract of tumors was determined together with the fractionation of the org. P into Ba-sol and insol and Pb-sol and insol fractions. The org. P was lower in benign than in malignant tumors, the highest value for the former being 14 mg. % and the lowest for the latter 2.7 mg. %. The mean value for malignant tumors was 5.0 mg. %. No hexose phosphates were present. A phosphoric ester was isolated from the alc-insol. Ba fraction of the Pb-precipitable phosphates. The formula  $\text{C}_4\text{H}_{10}\text{O}_8\text{P}_2$  suggests that it may be either hexoseamorphosphate or the phosphoric ester of an amphoteric diol. The phosphoric ester of thioamide was isolated from the Ba fractions pptd. in 70% alc at pH 10. Rachel Brown.

**Pneumococcus immunity after gold treatment.** W. A. Collier. *Z. Hyg. Infektionskrankh.* 117, 476-60 (1935).—Of C. A. 29, 7484.—Intraperitoneal or pulmonary treatment of mice with killed pneumococci confers less immunity than when the mice are treated simultaneously with the Au prep. Auto-Detoxin (Type 70). In the latter case dead or living pneumococci can be used. Pulmonary immunization appears to be more effective than intraperitoneal immunization against a pulmonary infection but less so against an intraperitoneal infection.

**Cerebrouse cysts in the frontal skull cysts.** Carl Trepp and Bruno Lokardt. *Z. Psychiat.* 128, 318-42 (1936).—The contents of a frontal cyst of 300 cc. vol. showed no spectroscopic evidence of blood pigments and only traces of Fe, but gave a strong reaction for sugar. Such high sugar values are not observed in reactions for cholesterol and P values neg., protein was pos. The main constituent of the dry residue was a cerebrouse mist. Contg. carbon and keratin in about equal proportions. For the type of cyst the name *cerebrouse* is proposed.

**The structure of cystine calculi.** Endre Solod. *Oron. Mediap* 79, 1196-7 (1935).—Of C. A. 29, 7451.—X-ray photographs of cystine calculi show that they consist of 3 layers, the center being a phosphate material of small but well-developed crystals. Then there is a semi amorphous phosphate layer of fine structure; it does not show the same symmetry in all directions. The surface cystine layer covers both of the others. Thus shows the fine cyst. phosphate was formed first.

**Physicochemical properties of tissues with respect to the normal or pathological state of the organism.** XXI Effects of darkness and light on the development of fat cancer. Fred Viles, A. de Coulon and J. A. J. J. *And. ph.* *And.* 12, 235-77 (1935).—Of C. A. 29, 6644.—Strong illumination (with ordinary incandescent lamps) seemed to accelerate the development of papilloma and cancer in mice treated with fat.

**Alotemia and increase in alkali reserve in animals.** René Huvenne, R. Truhaut and C. Samet. *Comp. rend. soc. med.* 120, 715-18 (1935).—A woman with complete anemia due to compression of the ureters by a tumor was given enough  $\text{NaHCO}_3$  daily to keep the alkali reserve normal or a little above. She lived 20 days. Blood urea gradually increased to 0.5%. L. E. Gilson.

**Nature of the tuberculous antibodies.** Albert Meyer and André Pic. *Comp. rend. soc. med.* 120, 772-4 (1935).—There are 2 antibodies. One is specific for the lipides of the tubercle bacillus and the other for the protein. They were prep. from each other by the method of D'Alessandro and Soňa (C. A. 29, 5116). Tuberculous antibodies. *Ibid.* 940-5.—Serums from animals treated with tubercle bacilli contain the 2 antibodies mentioned above. They do not contain any antibody capable of reacting with both lipide and protein. L. E. Gilson.

**Oxalic acid content of the blood in cancer.** E. Aysaun, F. H. Colombes and A. Montaur. *Comp. rend. soc. med.* 120, 934-5 (1935).—In 64 of 90 cases examined the blood oxalic acid was within the normal range. In the others it was somewhat above normal. No correlation

between oxalemia and the stage of the disease was found

L I Gilson  
Sensitivity of Rous sarcoma virus to the photodynamic virulicide activity of gonacrine and methylene blue P Haber *Compt rend soc biol* 120, 964-6 (1935) — Neither compd had any action on the virus *in vitro* under strong artificial illumination Twelve references

L I Gilson  
[Decrease in] the tuberculous antibody content of the serum during acute and chronic fatty degeneration of the liver in rabbits intoxicated with chloroform or phosphorus in oil injected subcutaneously F Pezzangora *Compt rend soc biol* 120, 977-80 (1935)

L I Gilson  
Aninsulin P Barral *Compt rend soc biol* 120, 993-4 (1935) — HCHO was added to insulin and the mixt incubated 40 days at 60° The prepn had no hypoglycemic action It acted as an antigen when injected into rabbits Complement deviation reaction for aninsulin *Ibid* 995-8

L I Gilson  
Nature of diphenyl toxin and powdered purified anatoxin Seigo Hosoya, Koshiro Kagabe, Teisunosuke Tanaka and Akiyoshi Momma *Compt rend soc biol* 120, 1030-2 (1935), cf C A 29, 4074

L E G.  
Role of the fourth component of alexin in the mechanism of a Wassermann reaction positive for the spinal fluid but negative for the blood serum Otto G Bier *Compt rend soc biol* 120, 1113-16 (1935), cf C A 29, 6300

L E Gilson  
Exanthema from picric acid and quinine bisulfate Passive transfer of hypersensitivity by the Prausnitz-Kuestner method Alberto Midana and Filippo Franchi *Minerva med* 1935, II, 641-6 — In 1 case each of picric acid and quinine hypersensitivity a pos Bloch-Jaeger reaction was obtained in the allergic individual and the sensitivity was locally transferred to the skin of normal persons by the Prausnitz-Kuestner method H L G

Lipides in the liver of the cat during bile stasis and biliary decompression A Cantarow, H L Stewart and M M Lieber *Arch Pathol* 20, 535-47 (1935) — Studies were made of the quantity and distribution of stainable lipid and doubly refractive material in the liver in 39 cats with uncomplicated total bile stasis of from 10 hrs to 42 days' duration and in 21 cats following biliary decompression of from 1 hr to 21 days' duration The quantity of stainable lipid decreased markedly in both hepatic and Kupffer cells during stasis and was practically absent in the majority of animals after the 7th day A return of large quantities of stainable lipid was noted in animals several days after relief from prolonged total stasis Doubly refractive material was present in the Kupffer cells in large quantities through the entire period of total stasis, differing markedly from the stainable lipid in this respect Although necrotic lesions frequently contained large quantities of fat, observations during the early and late stages of stasis indicate that the stainable lipids distributed indiscriminately throughout the lobule are not necessarily dependent on regressive changes in the affected cells Stainable lipid was not observed in the epithelium of the bile ducts, except in the later period of stasis at a time when the ducts were undergoing destruction as a result of organization of areas of hyaline necrosis It is suggested that the consistently large amount of doubly refractive material in the Kupffer cells and the relatively small quantity in the hepatic cells during stasis are dependent on a delay in the transfer of this material from the former to the latter under the experimental conditions H F H

V H Moon  
Mechanism of acute inflammation *Arch Pathol* 20, 561-70 (1935) — The local vascular and cellular phenomena of acute inflammation result from the liberation of substances from injured cells One such substance, which apparently is some combination of histamine, produces the vascular reactions resulting in congestion, capillary dilatation and permeability, edema and local elevation of temp Systemic leucocytosis results from the injection of histamine phosphate into animals Histamine released from extensive areas of injured tissue is probably a factor in producing the resulting leucocytosis A substance released from injured cells attracts leucocytes to the

area of injury. This same substance may also be effective in systemic leucocytosis Apparently this substance is not histamine There is evidence that increase in the metabolic rate and in the rapidity of circulation follows the injection of histamine This may be a factor in the systemic reactions which accompany extensive inflammation. Harriet F Holmes

E F Hirsch *Arch Pathol* 20, 665-82 (1935) — The radial inclusions of giant cells observed in tubercle-like granulation tissues are crystal forms of fats solid at body temp, such as palmitin or stearin, sepd from an oil system contg cholesterol or substances with the phys properties of cholesterol The formation of these crystals in a liquid fat system is according to the usual laws governing crystals, and the factors accomplishing the supersatn of the system are mainly the abstraction of the liquid portion faster than the combustion of the dissolved solid fat. Certain chem changes take place in the compn of the crystals in the tissues so that they become insol in fat solvents Further changes or addns in the tissues produce the elastin-staining qualities Harriet F Holmes

Analysis of Type I pneumococcus specific precipitate Herbert O Calvery, *J Biol Chem* 112, 167-9 (1935). — Analysis has been made of the purified ppt. from type I pneumococcus specific hapten and antisera and the following av values (N fractions expressed in percentage of total N) for Type I and Type II ppts, resp (*Proc Soc Exptl Biol Med* 32, 1099 (1935)) were obtained ash 0.27, 0.17, moisture 4.85, 6.1, total N 15.85, 16.0, amide N 4.5, 3.7, humin N 0.6, 0.6, amino N 75.2, 75.0, S 1.25, 1.3, tyrosine 5.5, 5.5, tryptophan 2.05, 2.2, cystine 2.55, 3.1, arginine 4.95, 5.5, histidine 0.95, 1.1, lysine 5.37, 4.8, aspartic acid 4.6, 4.4 and glutamic acid 6.55, 6.3 Attention is called to the differences between the values for cystine which are beyond exptl error. In general the values agree with those reported for serum globulins A F. Lohrop

The effect of cysteine on tetanus toxin Philip B Cowles *Yale J Biol and Med* 8, 265-8 (1936) — Cysteine in the presence of O<sub>2</sub> is able to detoxify tetanus toxin; the resulting product is a toxoid which can stimulate antitoxin production and is able to unite with antitoxin The reaction does not appear to be of the same nature as is that of cysteine with Cu G H W. Lucas

Post hemorrhagic uremia L. Meyler *Acta Med Scand* 87, 313-25 (1935) — Both experimentally and clinically, extrarenal uremia occurs after severe hemorrhage. By maintaining the water supply with subcutaneous fluid administration it is possible to prevent the onset of uremia in the exptl animals The advisability of large quantities of water together with sufficient food in cases of large losses of blood is discussed S Morgulis

Distribution of isoagglutinins in blood serum fractionated through electrophoresis Anders Grönwall *Biochem Z* 282, 257-62 (1935) — Human serum of type O was thoroughly fractionated by electrophoresis, and in each fraction the agglutinin titer, total N and pptn by (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> were detd The isoagglutinins are always found in one of two fractions pptd by 30% (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, both the α- and β forms being present there. S M

Hypersenulism associated with congenital atresia of the pancreatic duct Walther Benoit. *Endocrinol* 16, 313-27 (1935) S Morgulis

The lipid content of immune aggregates Frank L Horsfall, Jr and Kenneth Goodner *J Biol* 31, 69 (1936) — Ppts formed by the interaction of antipneumococcus horse or rabbit serums with Type I pneumococcus capsular polysaccharide contain lipids characteristic of the species origin of the antibody. John T. Myers

The serological flocculation rate in the region of considerable antibody excess Sanford B Hooker and Wm C Boyd *J Gen Physiol* 19, 373-8 (1935) — Antigen antibody reactions in test tubes can be divided into 2 phases (1) combination, (2) secondary aggregation resulting in flocculation or agglutination Strong concns of the reactants make the second phase immediately visible, under these conditions the first phase must be almost

instantaneous. The second phase can be explored by a current technique if suitable dials are used. II and II have observed in several simple precipitin-antigen systems that in regions of considerable antibody excess flocculation time varies inversely and in the same ratio as the change in concentration. A physicochemical interpretation of this observation is given based upon the theory of van Schmelouhowski (*C A* 11, 3140) for the velocity of colloidal flocculation.

C. H. Richardson  
The immunological specificity of the euglobulin and pseudoglobulin fractions of horse and human serum. *Trans. Harris and Harry Lacle. J. Gen. Physiol.* 10, 393-96 (1935).—Euglobulin was isolated from horse and human serum by pptn. with  $(\text{NH}_4)_2\text{SO}_4$  (1 : 1 satn) and pptn. on subsequent dialysis. Pseudoglobulin from the same source was isolated by pptn. with  $(\text{NH}_4)_2\text{SO}_4$  ( $1/2$  :  $1/2$  satn) and then taken, the fraction which remained in soln. on subsequent dialysis. The serum of rabbits injected with either of these anti-embryal pptn. with both. However, two antigenically distinct albumins (globulins I and II) were found in the serum. They were largely averted with so-called euglobulin and pseudoglobulin, resp. Ordinary methods of pptn. do not completely remove the other antigen from a globulin fraction. Antisera to these euglobulin and pseudoglobulin fractions therefore contain antibodies to both antigens. Each protein soln. pptn. all the antibody sp. for its antigen and in addn. pptn. a portion only of the antibody sp. for the other antigen. This immunological specificity is an inherent property of the two globulins present in the serum. It is not destroyed by an antigen induced by their pptn. and purification nor by lipoids assoc. with the protein. C. H. Richardson

The connecting links between immunology and chemistry. L. E. den Dooren de Jong. *Chem. Weekblad* 32, 294-601 (1935).—discusses W. Gordon, E. C. R. E. C.

The proteins of the cancerous cell. Charles Achard and Maurice Dietrich. *Compt. rend.* 201, 731-61 (1935).—Exts. of beef and equine tumors were fractionated into 2 fractions. One, dispersible in  $\text{H}_2\text{O}$ , contained a globulin and products of cellular metabolism, i. e., peptides. The other, not dispersible in  $\text{H}_2\text{O}$ , contained a myxoprotein and a phosphoprotein contg. 0.35% P. The albumin isolated was similar to serum albumin except it was of much lower mol. wt. This property may be due to the repulsive of protein synthesis in cancerous tissues. E. W. Scott

Immunological studies on lymph. I. The complement content of lymph. J. Murakami. *Japen. J. Microbiol.* 1, 2, 136-47 (1935).—M. found complement in normal rabbit lymph. I. S. Yun

Relationship of vitamins to diphtheria toxin and antitoxin. M. Aino. *Japen. J. Microbiol.* 1, 2, 138-52 (1935).—M. investigated the relationship between the resistance to diphtheria toxin and the production of diphtheria antitoxin in guinea pigs which were fed with vitamins A, B, C or D. He found that the resistance to diphtheria toxin was strong in guinea pigs fed vitamin C. I. S. Yun

Pathological creatinuria. Lu Gwei Dien. *Trans. 9th Cong. Far East. Assoc. Trop. Med.* 1, 160-176 (1934).—Creatinuria was found in pseudopyretic, muscular dystrophy, amyotonia congenita and generalized octetis chronic cases, so B. maintains that there is undoubtedly some connection between the activity of the endocrine system and creatine metabolism. C. S. Yun

Eppinger, Hans, Kamnitz, Hans, Popper, Hans, et al. Die akute Entzündung der Permeabilität. Pathologie. Vienna: J. Springer, 208 pp. M. 25

Hartner, P. F. Pathologie desactive. Paris: Masson & Cie, 162 pp. F. 25

Pandolfi, Rosario. Comportamento della eliminazione urinaria e delle diverse frazioni di siero negli individui normali ed in quelli affetti da tumore maligno accompagato o no da metastasi cancerose. Rome, Zamperini & Lorenzini, 7 pp.

Shelley, Horace M.: An Epitome of the Laboratory Diagnosis and Treatment of Tropical Diseases. London: John Dale, Sons & Danclose, Ltd. 51 pp. 2s. 6d

Vignes, Henri, Lauret, G., Olivier, Jean, and Olivier-Palud, P. Maladies des femmes enceintes. T. 11. Affections du foie, du pancréas, maladies de la nutrition, paral. abdominales. Peritoneum. Paris: Masson & Cie, 338 pp. F. 25

## H—PHARMACOLOGY

A. V. RICHARDS

Chemical studies of acute poisoning from mercury dichloride. Torald Sollmann and Nora E. Schreiber. *Arch. Internal Med.* 57, 46-62 (1936).—Elimination of  $\text{HgCl}_2$  was studied in 4 cases of  $\text{HgCl}_2$  poisoning which were fatal. Considerable amt. of  $\text{Hg}$  were removed only by early emesis. Not over 1-3-25% of the  $\text{Hg}$  taken was removed by gastric lavage and colonic irrigations. Urinary elimination was negligible, there being anuria in the fatal cases.  $\text{Hg}$  concn. in vomitus, feces and urine ran level after the 1st, 2nd or 3rd day. In 3 autopsies concn. of  $\text{Hg}$  was highest in the kidney (3-8 mg. 100 g. moist organ), liver was 2nd with  $1/2$  - 1 the kidney, colon, spleen, intestines  $1/2$ , skeletal muscle and lungs  $1/2$ , brain  $1/2$ , blood  $1/2$ , gastric fluid lower than blood, bile variable between the liver and blood. Three times as much  $\text{Hg}$  was found in the liver as was found in both kidneys. J. B. Brown

Chlorophyll and regeneration of blood. Effect of administration of chlorophyll derivatives to patients with chronic hypochromic anemia. Arthur J. Fetek. *Arch. Internal Med.* 57, 72-84 (1936).—The administration of chlorophyll products after a period of medication with Fe was followed by a significant increase in the concn. of hemoglobin. The body can apparently use pyrrrole substances for the building of hemoglobin. J. B. Brown

Potassium permanganate poisoning. Stanley G. Williston and Mark L. Newman. *Biol. Med.* 1, 58-9. A review with report of a fatal case following administration of 29 g. of  $\text{KMnO}_4$  through the urethral canal. J. B. Brown

Pantolaine in spinal anesthesia. David C. Bull and Caldwell B. Twiss. *Ann. Surgery* 103, 27-34 (1936).—Rachel Brown

The toxicity of bromoform (Gutierrezia macrocephala) for sheep, cattle and goats. Frank P. Matthews. *J. Am. Vet. Med. Assoc.* 41, 55-61 (1936).—Rachel Brown

Initial impressions of atropine-pilocarpin in the treatment of malaria in Uganda. A. Forbes Brown. *J. Trop. Med. Hyg.* 38, 501-4 (1935).—Atropine treatment of malaria seems to be quicker and more efficacious than quinine treatment. Rachel Brown

The calcium content of the blood during experimental poisoning with sodium fluoride. T. A. Stutesel. *J. Physiol. (U. S. S. R.)* 19, 1259-64 (1935).—Rabbits and dogs were fed for several months a diet which contained a daily dose of 0.02 g. of  $\text{NaF}$  per g. of body wt. The amt. of the blood Ca of the animals remained unchanged. H. Cohen

Resorption, distribution and elimination of fluorides during the poisoning of an animal with sodium fluoride. S. D. Gadvalina and T. A. Stutesel. *J. Physiol. (U. S. S. R.)* 19, 1245-56 (1935).—About 90% of the fluorides fed to dogs was retained in the organism, and 10% was excreted with the urine and feces. When  $\text{NaF}$  was injected intravenously, the elimination was effected not only through the kidneys, but also through the intestines. The F content of the blood of dogs poisoned by the oral administration of  $\text{NaF}$  did not change during the 1st 4-5 months. Later on, the blood F content increased somewhat. The F content of tissues of rabbits poisoned by  $\text{NaF}$  increased to about twice the normal value in the course of 3-5 months, the F content of the bone increased 5 times during the same period. H. Cohen

The breakdown of mesalene and mesalene-like substances in the organism. K. H. Johnson and Johannes Müller. *Z. physiol. Chem.* 235, 11-22 (1936).—Rabbits are 70 times as tolerant to mesalene,  $\text{C}_6\text{H}_5\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$ , the chief alkaloid of pyrethrum, as human

beings The most striking effect in rabbits and dogs is a strong retention of urine, lasting as long as 48 hrs After feeding 400 mg of the HCl salt to rabbits some 40-50% was excreted as 3,4,5-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H (I). This acid, however, is relatively nontoxic, and a dose of 800 mg fed to a rabbit was excreted to the extent of 60% without retention of urine or evidence of physiol. changes. Closely related substances, e. g., 3,4,5-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>CHO and 3,4,5-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH (II) were decidedly more toxic, the former being excreted as 3,4,5-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>CO<sub>2</sub>H and the latter as a nitrogenous substance contg MeO. In human beings, on the other hand, mescaline was not excreted as I, and I itself was excreted to 75% without causing drunkenness or urine retention. An excretory product of mescaline was an oil which on hydrogenation took up 3 H<sub>2</sub> and formed a satd. crystalline substance of the formula C<sub>11</sub>H<sub>17</sub>O<sub>3</sub>N, contg 1 MeO and incapable of forming a picrate, chloroaurate or chloroplatinate, in contrast to the hydrogenation product of mescaline. Mescaline has a more powerful action on normal persons than on schizophrenics, whereas the reverse is true of the 2,3,4 isomer I, m 121°, was prep'd by coupling of 3,4,5-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>COCl with CH<sub>3</sub>N<sub>3</sub> to trimethoxy- $\alpha$ -diazoacetophenone, m 103°, conversion of the latter by NH<sub>4</sub>OH + AgNO<sub>3</sub> to trimethoxyphenylacetamide, m 125°, and hydrolysis of the amide with KOH in MeOH. II, an oil, was obtained by treatment of mescaline with HNO<sub>3</sub> and purified by sapon of the *p*-nitrobenzoyl deriv., m 100°.

A. W. Dux

The breakdown of dimethylamine and dimethylamine oxide in the animal body. Fritz Horn. *Z. physiol. Chem.* 238, 84-90 (1936).—After subcutaneous injection in dogs PhNMe<sub>2</sub>, unlike NMe<sub>3</sub> and NEt<sub>3</sub>, is not eliminated as the oxide. Both PhNMe<sub>2</sub> and PhMe<sub>2</sub>NO are broken down at least as far as O<sub>2</sub>H<sub>2</sub>NCH<sub>2</sub>OH. Poisoning by PhNMe<sub>2</sub> leads to formation of methemoglobin.

A. W. Dux

Evaluation of the relative toxic effects of large doses of calciferol and the crystalline antirachitic preparation substance L. J. van Niekerk. *Arch. nederl. physiol.* 20, 559-61 (1935).—Conclusion. The ratio of toxic to antirachitic activity of the substance L is identical with that of calciferol. The min. toxic dose of calciferol was found to be the same as that de'd by Askev (*C. A.* 26, 3284) and Windaus (*C. A.* 26, 2217).

F. L. D.

The relation between the chemical constitution and pharmacological action of phenylhydrazine derivatives. W. F. v. Oettingen and W. Deichmann-Greubler. *J. Ind. Hyg. Toxicol.* 18, 1-15 (1936).—PhNHNH<sub>2</sub> and 11 derivs. of it were studied with regard to their toxicity on subcutaneous injection, their effect on the skin, their anemogenic action, their effect on the blood pigment, and their effect on the blood pressure and respiration at elevated temps. Mice and rats were used as exptl. animals. Comparative min. fatal doses are recorded. Toxicity depended largely on soly in water. Substitution of 1 H of the hydrazine group reduced toxicity. Surface applications indicated that PhNHNH<sub>2</sub> was most irritating, probably owing to the C=S group. A. L. F.

Pathological studies on the organic effects of various hydrazine derivatives. W. C. Hueper. *J. Ind. Hyg. Toxicol.* 18, 17-36 (1936).—Rats injected with lethal or slightly sublethal doses of benzylphenylhydrazine,  $\alpha$ -ethyl  $\beta$ -propylacrolein phenylhydrazine, butylphenylhydrazine and formylphenylhydrazine developed lesions in the liver, heart, pancreas, spleen, kidney, testis and brain in addn. to hemorrhage, edema, hyperemia and infarcts. The acrolein deriv. was most toxic. A. L. F.

Urine sulfate determinations as a measure of benzene exposure. W. P. Yant, II H. Schrenk, R. R. Sayers, A. A. Horvath and W. H. Reinhardt. *J. Ind. Hyg. Toxicol.* 18, 69-88 (1936).—Seventy-nine dogs were subjected to a variety of conditions of exposure to benzene vapor in air. Analysis of urine specimens showed that a rapid and marked decrease occurred in the percentage of inorg. sulfates of the total sulfates in the urine. It is believed to be due to the oxidation of benzene to phenol or phenolic derivs. which are conjugated in the liver

with sulfate ions to form ethereal sulfates. The change in inorg. sulfates is proportional to the severity of benzene exposure. This procedure may be an invaluable aid in the control and prevention of chronic benzene poisoning.

A. L. Elder

Nicotinized drinking water as a preventive against ascarid and coccidial invasions of chickens. R. E. Rebrasser. Ohio Agr. Expt. Sta., *Bull. No. 548* (53rd Ann. Rept.), 77 (1935), cf. *C. A.* 28, 3789<sup>1</sup>.—No appreciable difference was observed from the use of nicotinized drinking water in the prevention of either ascarid or coccidial invasions in chicks. Over 700 chicks were used. The concn. of nicotine varied from 1 teaspoonful of 40% soln. of nicotine sulfate to 5-50 gal. of water.

C. R. Fellers

Clinical experiences with sulfarsenol. Ferenc Foldvác. *Orvosi Hetilap* 79, 1300-9 (1935). S. S. de F.

The pharmacology of myrrh, krameria and enodictyon. David I. Macht and Hilah F. Bryan. *Am. J. Pharm.* 107, 500-11 (1935).—The local effect of these 3 vegetable astringents was studied on the mucous membranes of the mouth, tongue and pharynx and compared with that of tannic acid alone. Tannic acid markedly delayed the absorption through the mucous membrane of the powerful alkaloid nicotine subsequently applied, while the vegetable astringents did not. This was true of the mucous membranes of the pharynx, of the tongue and of the cheeks. Expts. on surviving intestinal segments in oxygenated Locke soln. revealed that tannic acid alone produces a slight inhibition of the amplitude and the rate of rhythmic contractions without injuring the prep'n. The 3 vegetable astringents, however, produced paralysis and death of the intestinal muscle, as indicated by its failure to respond to subsequent treatment with such powerful stimulants as pilocarpine, etc. Conclusion. In respect to their local effect on the intestines, myrrh, krameria and enodictyon act differently from pure tannic acid solns. This difference must be ascribed to the presence of volatile oils or some other constituent of the 3 vegetable astringents.

W. G. Gaessler

Repeated injections of a gold derivative of a sulfur compound. Tolerance and localization. A. Leulier and G. Bérnard. *Compt. rend. soc. biol.* 120, 651-4 (1935).—Sr aurothiopropanol sulfonate, prep'd from Na alloxhydrin and SrCH<sub>3</sub>, is a white salt contg 41-2% Au and sol in about 100 parts water at 20°. Repeated intramuscular injections of an oil suspension are well tolerated by guinea pigs. Growth is not interfered with. One guinea pig received a total equiv. to 1 g Au in 9 months. Au was found in all tissues exam'd but was much more abundant in the kidneys than in any other organ.

L. E. Gilson

Pharmacology of some hydroxyphenoxethylalkylamines. D. Bovet, A. Simon and J. Druey. *Compt. rend. soc. biol.* 120, 690-3 (1935).—A preliminary note on 9 comp'ds. In general those of the type HOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> (R = Me or Et) are adrenergic and non-nicotinic in their action and those of the type HOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> are nicotinic and nonadrenaline.

L. E. Gilson

The continuous injection of insulin into dogs. Minimum dose when injected into a peripheral vein. A. Baudouin, E. Azrad and J. Lewin. *Compt. rend. soc. biol.* 120, 769-71 (1935).—When injected into a leg vein of dogs (with or without chloralose anesthesia) the min. dose of insulin causing an appreciable decrease in glucemia was about 0.05 unit/kg/hr. Minimum dose when injected into an artery or a mesenteric vein. *Ibid.* 805-6. In both cases the min. dose affecting glucemia was about 0.01 unit/kg/hr.

L. E. Gilson

Effects of zinc salts on blood. G. Protrowski. *Compt. rend. soc. biol.* 120, 830-2 (1935).—ZnSO<sub>4</sub> retards the clotting of blood *in vitro* but not *in vivo* (rabbits). Changes produced in the corpuscles are described. L. E. Gilson.

Analyses of bloods treated with moranyl or liquoide (to prevent coagulation). R. Nattan-Larrier and P. Tchermakofsky. *Compt. rend. soc. biol.* 120, 857-9 (1935).—Neither anticoagulant affected the pH, alkali

reserin or urea, cholesterol, glucose, Ca or K content of the blood. L. E. Gilson

Effect of continuous intravenous injection of adrenaline on the ketone bodies of the urine. A. Baidoun, H. Binard, J. Levin and J. Sallet. *Compt rend soc Biol* 120, 840-2 (1935), cf. C. A. 30, 1120. A small increase in urinary ketone bodies was produced in dogs.

Changes in serum phosphatase after ingestion of chloform. H. Metzger, P. Barthelme and M. Urban. *Compt rend soc Biol* 120, 206-7 (1935). Dogs were given 2 cc. of  $\text{CHCl}_3$  dissolved with olive oil by stomach tube daily or 2-3 times a week for several weeks. During the treatment serum phosphatase fluctuated between 5 and 10 times the normal value. It slowly returned to normal when treatment was stopped. Bile secretion was normal throughout. No jaundice occurred.

Effect of leucithin on cardiac activity. J. Karsak. *Compt rend soc Biol* 120, 920-2 (1935), cf. C. A. 29, 511. Leucithin, 0.1-0.5%, added to the Ringer's solution used for perfusing the isolated frog heart increases the frequency and amplitude of the contractions. It acts directly on the myocardium and has no effect on the activity of the cardiac nervous. Influence of leucithin and other compounds decreases the tension on the arteries of vagomimetic substances. *Ibid* 922-4. The isolated frog heart was perfused with Ringer's solution and the response to acetylcholine died before and after the addition of very small quantities. No effect of leucithin, Na taurocholate and capryl etc. Each compound decreased the action of the acetylcholine, the effectiveness decreasing in the order given.

Action of gonococci on the virus of lymphogranuloma. R. Schoen and L. Rempe. *Compt rend soc Biol* 120, 959-60 (1935). Gonorrhea kills the virus *in vitro* but is ineffective in live infected guinea pigs. L. E. Gilson

Distribution of chloride in the tissues of rabbits after intravenous injection of sodium chloride. T. Svedoloff and René S. March. *Compt rend soc Biol* 120, 941-2 (1935).

Influence of colchicine on the cardiac inhibiting effects of faradization of the pneumogastric nerve. Raymond-Haust. *Compt rend soc Biol* 120, 951-3 (1935). In dogs large doses of colchicine slightly decreased the sensitivity to the electric stimulations. L. E. Gilson

Mechanism of the curative and preventive action of rubulose and similar compounds in experimental streptococcus infection in mice. C. Lévaditi and A. Yusef. *Compt rend soc Biol* 120, 1077-9 (1935), cf. C. A. 29, 7140. The compounds prevent the encapsulation of the streptococcus and neutralize the leucocidin and hemolysin which it secretes.

Permeability of the placenta to medicinal and toxic substances. Quinine. M. T. Régner. *Compt rend soc Biol* 120, 1089 (1935). Quinine passes from the mother into the fetus. L. E. Gilson

Comparison of the toxicity of some local anesthetics on amphibian heart. Leonardo Donatelli. *Arch farmacol* 1936, 4, 482-96 (1935). Tests on frog heart *in situ* showed increased toxicity in the order given for identical amounts of procaine, stovaine, cocaine, parocaine and perocaine.

Synthetic animal malaria. W. O. Kermack. *Science Progress* 30, 467-9 (1935). Plasmodium (a quinine drug) is not suitable for the general treatment of malaria and is rather toxic. Aetabin (an acridine derivative) is almost as effective as quinine and is superior to the latter in reducing the size of relapses. J. S. H.

The efficacy of carbon tetrachloride in roundworm (Ascaridia hexastri) control. J. T. Ackert and G. L. Graham. *Poultry Sci* 14, 225-31 (1935). A dosage of 4 cc.  $\text{CCl}_4$  per kg. of body wt. gave complete removal of the worms from chickens 10 weeks old and had practically no toxic effect on the chickens. A dosage of 10 cc./kg. caused a 25% mortality of the chickens. The egg production of pullets was materially reduced for a

period of 7-10 days after administration of  $\text{CCl}_4$  at the rate of 4 cc./kg.

Absorption and excretion of aetabin. N. D. Kehat. *Records Malaria Survey India* 5, 393-404 (1935). Aetabin is given orally to human beings in three 0.1-g doses at intervals of 3 hrs. It appears in the urine 75-90 min. after the first dose, but if the intervals are reduced to 1 hr. it appears in about 60 min. Aetabin may continue to be excreted from the body up to 60 days after the administration of a total of 0.3 g. of the drug at intervals or in a single dose. The duration of the drug in the urine appears to be influenced by the extent of its excretion. At least 50-70% of the administered dose is excreted in the urine. The slow excretion of the drug may be due to its retention in the body tissues either in a complex form or colloidal combination, when the concentration in the fluids decreases, the absorbed drug diffuses slowly from the tissues and is finally excreted by the kidneys. The protection afforded by aetabin against malaria seems to be due to its prolonged retention in the body tissues. There seems to be little or no advantage in administering aetabin either intravenously or intramuscularly.

K. D. Jacob

The influence of food in the stomach on the absorption and excretion of aetabin. N. D. Kehat. *Records Malaria Survey India* 5, 405-11 (1935). Food delays absorption and lessens the fraction of the dose of aetabin which is excreted in the urine, not only during the first 24 hrs., but also during the following 3 days. Equiv. doses of the hydrochloride and the muscarine were given orally. More of the latter was excreted in 3 days than of the former. A protein-rich diet seemed to retard the excretion of aetabin.

K. D. Jacob

The influence of ovary lyrate on egg production in hens. V. Link and S. Volkovskiy. *Arch internat Hyg* 1935, 1 (S. S. R.) 1935, No. 3, 86-98 (in English 98). Lays on 4500 laying hens showed that injection of ovary lyrate had a positive effect on egg production. Although the egg production decreased during the molting period it was still 15-20% above the controls. The suggestion that ovary lyrate is a sex agent, exerting an organotropic influence upon the organs from which it has been prepared, was not confirmed. Ovary lyrate exerts a stimulating action upon all functions in the sense of a "common protein effect." There is an increase in the amt. of hemoglobin and erythrocytes, and a more intense functioning of the gastric organs. Studies on the chemical composition of ovary lyrate showed that the greater effect of the injections depends to a certain extent on the amt. of N introduced.

S. A. Karjalainen

Biochemical aspects of anesthesia. Ivan Maxwell. *J. Med. J. Australia* 2, 841-5 (1935). Review.

G. H. W. Lucas

Sources and pharmacology of impurities in anesthetics. Byron L. Stanton. *Med. J. Australia* 2, 845-9 (1935). A review.

G. H. W. Lucas

The total calcium and phosphorus in rats treated with small doses of parathion. Marlene Pura. *Arch sci* 1935, 55, 659-63 (1934). The total ash of rats treated with small doses of parathion hormone shows no noticeable changes. The P content is considerably increased, Ca shows only an insignificant change.

A. E. M.

Quinquerant arsenicals in the treatment of congenital aphthous. Raúl Cibulski Aguirre and Jorge de las Carreras. *Revista med. (Buenos Aires)* 1935, 12, 1973-83.

A. E. Meyer

Influence of low molecular animal organ hydrolyzates on the reticuloendothelium and on phagocytosis. A. L. Leroy and M. Laptewa-Popova. *Acta Med. Scand* 87, 347-57 (1935). Acidosis resulting from injection of  $\text{NH}_4\text{Cl}$  invariably lowers the ability of the reticuloendothelial system to absorb Congo red and decreases the phagocytic ability of neutrophils and monocytes. Simultaneous administration, however, of organ hydrolyzates (consisting chiefly of polypeptides and amino acids) has the opposite effect.

S. H. Mordis

Phosphate metabolism and the influence of thyroidin.



Lydia Pasternak and Irvine H. Page *Biochem Z* 282, 282-92 (1935).—Phosphatides injected intraperitoneally into mice are completely metabolized. Furthermore, under the influence of thyroxine the phosphatide content of rat muscles may be nearly doubled. This is not due to damage of the phosphatide metabolism, since phosphatide injected intraperitoneally into mice treated with thyroxine is completely metabolized just as in the untreated mice. The increase in the phosphatide content in the muscles is not due to an infiltration from other locations, inasmuch as the total phosphatide content of the animal is about 40% higher. Thyroxine feeding also causes a rise in the cholesterol content of the entire organism and not of the muscles alone. Since the increase in phosphatide content is abs., not merely relative, it is actually formed under the influence of the thyroxine.

S. Morgulis

Comment on Traube's critique of our theory of narcosis  
Kurt H. Meyer and H. Hemmi *Biochem Z* 282, 444 (1935), cf. *C. A.* 29, 8143.—Reply I. Traube *Ibid* 445-6.—Conclusion Kurt H. Meyer *Ibid* 447.

S. Morgulis

Histological changes in the adrenal cortex of the rabbit after prolonged insulin treatment. F. Schenk and H. Langecker *Endokrinol* 16, 305-11 (1935).—Treatment with small doses of insulin extending over 4-12 weeks causes an enlargement of the adrenals with loss of differentiation of the various regions.

S. Morgulis

Effect of bile acids on calcium metabolism. XII. Thyroparathyroid tetany and the state of the calcium in blood of dogs receiving bile acids. Tadao Hoshijima *J. Biochem. (Japan)* 22, 375-83 (1935).—The daily administration of cholic acid to thyroparathyroidectomized dogs either decreases the severity or entirely abolishes the attacks of tetany, it greatly prolongs the life of the operated animals but does not affect the hypocalcemia.

S. M.

Intravenous manganese in the treatment of psoriasis. John Barr *J. Med. Soc. New Jersey* 32, 376-9 (1935).—Intravenous injection of 20 mg.  $MnCl_2 \cdot 4H_2O$  + 9.5 mg.  $CaCl_2$  in 5 cc. of normal saline solution produced marked improvement in 68% and some improvement in an additional 21% of 93 patients. Review of clinical literature on Mn.

James C. Munch

Action of Soviet ephedrine on the blood vessels. N. G. Polyakov and Stankevich *Khim. Farm. Prom.* 1934, No. 6, 28.—No difference was found in comparing domestic and imported samples.

L. Nasarevich

Pharmacology of convolvul. Ya. Kh. Nolle *Khim. Farm. Prom.* 1934, No. 6, 35, 7, cf. *C. A.* 29, 1885.—Convolvul is a powerful stimulant for the central nervous system, producing convulsions and death due to respiratory failure. Convolvul is a good local anesthetic just as lasting as cocaine.

L. Nasarevich

Testing of bile stimulants. V. V. Zverev *Khim. Farm. Prom.* 1935, No. 2, 128-8 (1935).—Rabbits tested by Stransky's method showed that high bile stimulation is produced by hexamethylenetetramine and decholin.

L. Nasarevich

The sensitivity of the organism to drugs in acid and alkaline conditions. E. S. Rosovska and A. I. Cherkas *Med. eksptl. (Ukraine)* No. 1, 50-61 (1934).—Sodium salicylate (20% soln.) was introduced subcutaneously, 0.2 g. per kg., and the salicylic acid detd. in the blood and urine at intervals during 48 hrs. During the administration of mixed food the max. salicylic acid content in the blood is reached in 1-2 hrs., then it drops and in 24 hrs. the blood is free from it. In urine the salicylates are eliminated in 24 hrs. in 50% of cases. The total amt. eliminated is 12-20% of that injected. During the administration of acid foods the rise in concn. is much slower and the max. is reached later than in the previous case. The elimination is similar. In case of alk. foods the max. concn. is reached in 1 hr., in 4-5 hrs. only traces of salicylates are found, and the total elimination is 35-70%.

L. Nasarevich

The cardiac activity in the fetal rat. E. L. Cory *J. Exptl. Zool.* 72, 127-45 (1935).—Lactic acid soln. (0.1%) applied directly to rat fetuses (3.5-5.0 g. wt.) or injected

into the maternal circulation produced an irregularity of heart rate similar to that in asphyxia. Alk. solns. ( $NH_4OH$ ,  $NaHCO_3$ , in Locke soln.) had no effect on the heart rate. The fetal rat heart does not react to adrenaline or to adrenergic secretion.

C. H. Richardson

Röntgenographic findings in lead poisoning. A. van der Plaats-Keyzer *Mededeel. Dienst Volksgezondheid Nederland - Indië* 24, 127-9 (1935).—Lead poisoning causes an increased absorption of Röntgen rays at points where bone growth is fastest, this is attributed to deposition of Ca, rather than a concn. of Pb at these points, since an amt. of Pb 10 times that found in the joint of a patient that died of Pb poisoning cast a barely visible shadow when a soln. of a Pb salt was absorbed on a filter paper, and a Röntgen picture made of this paper around the bone of an apc. Pb poisoning is believed to be congenital, since it was found in a baby 36 days old. Eight Röntgen pictures are reproduced.

W. Gordon Rose

The effect of 2,4-dinitrophenol upon calcium, creatine and creatinine excretion in the rat. Leonard I. Pugsley *Biochem. J.* 29, 2247-50 (1935).—The feeding of 2,4-dinitrophenol in daily doses of 25 mg. to rats resulted in increased Ca excretion (fecal), marked creatinuria and increased creatinine excretion.

E. W. Scott

A quantitative study of the phenomena of synergism. Potentiation of hypnotic action in mice. Laia Olszyska *Compt. rend.* 201, 796-7 (1935).—By injecting together inactive doses of EtOH and ethylbutylbarbiturate, sleep of 2-78-min. duration was produced, depending upon the proportions used. A mixt. of effective doses of the 2 hypnotics produced sleep of only slightly longer duration than that produced by each compd. separately.

E. W. Scott

A new true sympathicolytic: the rauwolfine of Koepfli. Raymond Hamet. *Compt. rend.* 201, 1050-3 (1935).—An alkaloid, rauwolfine, was extd. by Koepfli from the *Tabernaemontana ventricosa*, which reversed the action of adrenaline in dogs.

E. D. Walter

Contractive effect of coumarin. A. de Moura Campos *Rev. flora med.* 1, 639-46 (1935), cf. *C. A.* 29, 7502.—The intensity and duration of the contractive effect of coumarin on the muscles of the frog are proportional to the concn. of the drug. Action of coumarin on chronaxie of the heart. *Ibid* 647-57.—Fluext of coumarin (Silva Araujo) depresses the tonicity, chronicity and bathmotropism of the frog heart, the bathmotropic property is the last to recover. Action of coumarin on the arteries. *Ibid* 658-61.—Fluext of coumarin acts as a vasodilator, the effect is, in general, proportional to the dose. Action of coumarin on the smooth muscles of the digestive apparatus. *Ibid* 662-70.—Fluext of coumarin and ext. of tonka bean increase the tonicity and often the contraction of the stomach, duodenum and small intestine of the frog, inhibit the cecum, increase the tonicity of the crop muscles and intestinal contraction of quiescent birds, and inhibit the *quiescent* birds, and inhibit the *quiescent* birds, and inhibit the *quiescent* birds.

O. W. Wilcox

The effect of arsenobenzene preparations on mice infected with *Trypanosoma cruzi*. T. Minaguchi and Z. Rin *Japan Z. Mikrobiol. Path.* 29, 1495-1502 (1935).—Neither arsenobenzene sodium nor neoarsobenzene have preventive or healing actions on *Trypanosoma cruzi* infections.

I. S. Yun

The influence of acridine derivatives on blood picture and its relation to sterilizing action. Yanosuke Hirakawa *J. Med. Coll. Kyoto* 5, 338-49 (1935).—The relationship between blood picture in animals and sterilizing action *in vitro* was investigated. Administration of 0.5% rivanol, 0.5% trypaflavin, 0.5% panseptin or 0.5% trypasol increased the no. of white blood cells. Sterilizing action *in vitro* was observed. Trypaflavin was the most powerful then panseptin, trypasol and rivanol.

I. S. Yun

Factors related to increased absorption produced by the Chinese drug ah-chiao. T. G. Ni *Trans. Sth. Congr. Far East Assoc. Trop. Med.* 1, 648-57 (1934).—The Ca content in blood serum and cholic acid in intestinal contents were increased in animals fed ah-chiao.

I. S. Yun

Effect of morphine injection on the blood cells in normal individuals and in opium addicts. C. L. Cheng and W. C. Ma. *Trans. 5th Congr. Far East Assoc. Trop. Med.* 1, 659-73 (1934).—Change found in the blood of morphine-injected rats, morphine-injected normal individuals and in human opium addicts were polikocytosis, reticulocytosis, leucocytosis, mononucleosis and thrombocytopenia. I. S. Yun

Heffter, Arthur, and Heubner, Wolfgang. *Handbuch der experimentellen Pharmacologie*. Bd. III. 11. 4. Seltene Erdmetalle, Molybdän und Wolfram. Wismut. Berlin. J. Springer. Pp. 2189-2730. M. 64. Cf. C. A. 29, 1519.

Stein, Robert O. Haarkrankheiten und kosmetische Hautleiden mit besonderen Berücksichtigungen der Therapie. Vienna. J. Springer. 215 pp. M. 12.10, cloth, M. 13.10.

## I—ZOOLOGY

R. A. GORTNER

Chemical and physical characters of silkworms fed with mulberry and with *Melastoma aurantiaca* leaves. Guido Colombo. *Boll. ufficiale staz. sper. seta* 5, 52 (1935).—Silkworms fed with mulberry and with *Melastoma aurantiaca* leaves gave cocoons with very similar characters, the decrease in wt. by degumming the silk was, resp., 19.50 and 19.29%, and the av. wts. of the cocoons were 0.40 and 0.34 g., resp. G. A. Bravo

The phosphatide content of the flies of *Bombyx mori*. Bianca Jona. *Boll. ufficiale staz. sper. seta* 5, 50.8 (1935).—The lecithin content in the  $\text{Et}_2\text{O}$  and  $\text{EtOH}$  exts. of the air-dried butterflies was 1.04-1.36%. G. A. Bravo

Embryo chemistry of the cephalopods. Tatsunori Kawanishi. *Z. physiol. Chem.* 238, 91-5 (1936).—The eggs from 1000 squids collected on the Japanese coast were analyzed at 2 stages of incubation. As development progressed the embryos increased while the jelly decreased in wt. Likewise the mineral constituents ( $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{SO}_3$ ,  $\text{Cl}$ ) increased in the embryos with corresponding decrease in the jelly, and the same was true of total N, amino N and N precipitable by phosphotungstic acid. During the first 5 days amylase, esterase, tributyrinase, nuclease, glycerophosphatase and urase were demonstrated. After 16-18 days incubation amylase, nuclease and urase showed greater activity, and arginase, naphthalenesulfatase and protease became demonstrable. The enzyme actions in cephalopod eggs are similar to those in avian, reptilian and amphibian eggs. A. W. D.

Carbohydrate and fat metabolism in the incubation of the *Hynobius* egg. Makoto Takamatsu. *Z. physiol. Chem.* 238, 96-8 (1936), cf. C. A. 30, 122. During 3 successive incubation periods of 2 weeks each the eggs of *Hynobius* showed a progressive loss of glycogen and fat, while the total cholesterol remained practically constant. A. W. D.

The chemistry of *By. larvae*. Masaji Tomita and Teki Kume. *Z. physiol. Chem.* 238, 101-4 (1936).—Analyses are reported of larvae of the meat fly, *Sarcophaga carnaria*. The main constituent of the ash was  $\text{NaH}_2\text{PO}_4$ , as shown by 39.41%  $\text{Na}_2\text{O}$  and 22.65%  $\text{P}_2\text{O}_5$ . Amino acid depts. showed histidine 0.149, arginine 0.543, lysine 0.438, proline 1.545, phenylalanine 2.248 and glutamic acid 0.885%. The presence of a large no. of enzymes was demonstrated. A. W. D.

Silica of the siluroderms. E. Kahane. *Bull. sci. chim.* 17, 1554-8 (1935).—The dry tissues of *Onchidium asurum*, *Parapercnia goodeanae* and *Onchidium asurum* contained 0.17-0.97%  $\text{SiO}_2$ , partly as mineral silicates. L. F. Gilson

Vitamin C in invertebrates. A. Grouard and R. Rattmann. *Compt. rend. sci. Biol.* 120, 743-53 (1935).—Values are given in mg. per g. fresh tissue: earthworm intestine 0.1, muscle 0.010; sea urchin, digestive tract 0.2, genital organs 0.4; mollusks, 6 species, various tissues 0.02-0.3; various crustaceans, muscles 0.02-0.6, other organs 0.05-0.5. L. E. Gilson

Metabolism studies on the egg of the frog (*Rana fusca*). IV. Glycogen content of the egg from segmentation to hatching. Jean Brachet and Joseph Needham. *Arch. Biol.* 46, 821-35 (1935), cf. C. A. 28, 7369; 29, 3406. —There was no diminution in egg glycogen during the first 24 hrs. of development. With the beginning of gastrulation glycogen diminishes, the proportion of glycogen increasing to almost 100% at the tadpole stage. Helen Lee Gruehl

Content of glycogen in tapeworms (cestodes). I. A. Smorodintzev and K. V. Bebeshin. *Compt. rend. acad. sci. U. R. S. S. [N. S.]* 3, 413-14 (1935).—Glycogen depts. were made on *Taenia saginata*, *T. solium* and *Diphyllobothrium latum*. The freshly expelled worms were washed, heated in 60%  $\text{NaOH}$ , 2 parts  $\text{EtOH}$  was added, then the material washed with abs. alc. and finally  $\text{Et}_2\text{O}$ . After hydrolysis with  $\text{HCl}$  the sugar was detd. with 3. I. ehling's soln. *T. saginata* contains 60% of glycogen in the dry residue, which is twice the protein and 5 times the lipid content. The other 2 contain 1/3 the amt. of the protein, but still a greater amt. than the lipids. C. E. P. Jeffreys

Artificial mutations under the combined influence of x rays and salts of heavy metals in *Drosophila melanogaster*. N. N. Medvedev. *Bull. Inst. Genetics (U. S. S. R.)* 1935, No. 10, 211-22.—The action of x-rays on *Drosophila* cultured on a medium contg. 1%  $\text{Pb}(\text{OAc})_2$  yielded a higher frequency of mutations than the action of x-rays alone, the ratio of mutations in the 2 series being  $1.98 = 0.580\%$ . S. A. Karjala

The effect of high-frequency alternating potentials on the production of mutations in *Drosophila melanogaster*. Yul Kerkis. *Bull. Inst. Genetics (U. S. S. R.)* 1935, No. 10, 223-6 (in English 226).—The mutational effect of h.f.-frequency alternating potentials on flies placed between the plates of a condenser was studied. The wave lengths, time of exposure, and the percentage of lethals and semilethals obtained were: 12.8 m., 70 sec., no lethals, 0.58% semilethals; 2-3 m., 32 sec., 0.23 and 0.94%, resp.; 6 m., 45 sec., 0.70 and 0.76%, resp. The control showed no lethals and 0.21% semilethals. The effects are too small to be reliable. Little tendency toward sterility in males was observed with the sublethal doses. S. A. Karjala

The influence of sex on the lipides of certain marine mollusks. J. Timon-David and G. Ceresola. *Compt. rend.* 201, 833-4 (1935).—The females of the 3 classes of mollusks studied, *Eledone moschata*, *Murex brardaris* and *Mytilus galloprovincialis*, were distinguished from the males by a greater fat content and a greater unsat. in their fats. L. W. Scott

Acid formation in frozen and thawed *Arbacia punctulata* eggs and its possible bearing on the problem of activation. John Runnström. *Biol. Bull.* 69, 345-50 (1935); cf. C. A. 27, 2300.—Eggs of *Arbacia punctulata* were frozen at  $-80^\circ$  in  $\text{CO}_2$  snow and ether. After a period of time the eggs were thawed at room temp. During the thawing considerable acid is formed, as measured by the Warburg manometric method.  $\text{CH}_3\text{ICOO}_2\text{Na}$ ,  $\text{NaF}$  and  $\text{CuCl}_2$  in concn. of 0.03 M, 0.06 M and 0.004%, resp., had no inhibiting effect on the acid formation. Addn. of hexosemonophosphate does not enhance the acid formation. Pyruvate has no influence on the amt. of acid formed. Apparently this acid formation has nothing to do with lactic acid formation. Influence of iodacetate on activation and development of the eggs of *Arbacia punctulata*. *Ibid.* 69, 351-5; cf. Ellis, C. A. 28, 3453.— $\text{CH}_3\text{ICOO}^-$  does not inhibit the fertilization of the *Arbacia* egg. This enzyme or other activities in which SH groups are involved do not play an essential role in the fertilization process.  $\text{CH}_3\text{ICOO}^-$  (0.03 M) is harmful to the development of fertilized eggs. The addn. of pyruvate or lactate restores respiration but does not inhibit the injury to the fertilized egg. A carbohydrate breakdown is probably a necessary condition for the morphological differentiation of the anterior part of the larva. E. D. Walter

Change in rate of respiratory metabolism in a teleost

fish induced by acclimatization to high and low temperature Nelson A Wells *Biol Bull* 69, 361-7 (1933) — O metabolism of *Gillichthys mirabilis* ("mud suckers") acclimatized to high and low temps was studied. Fishes acclimatized to a high temp have a much lower rate of metabolism at a common intermediate temp than do those acclimatized to a low temp. The magnitude of this difference is a function of the difference between the acclimatization temps. E D Walter

Some relations between respiratory metabolism in fishes and susceptibility to certain anesthetics and lethal agents F B Sumner and N A Wells *Biol Bull* 69, 364-78 (1935), cf. preceding abstr. — Two species of teleost fishes, *Fundulus parvipinnis* and *Gillichthys mirabilis* were kept at widely different temps. When tested at these temps, they were found to differ markedly in the rate of their respiratory movements. These rates were higher at higher temps than at lower ones. Susceptibility to urethan was also higher at the higher temps. When fishes acclimatized to high and low temps were transferred to a medium temp, those from the warmer temp displayed a lower respiratory rate and a lower susceptibility to anesthetics (urethan, chloroform and ether) and lethal agents (NaCN, KCN, asphyxiation and excessive temp) than those from the colder water. E D Walter

Insect metabolism at temperatures below zero I V. Kozhanchikov *Compt rend acad sci U. R. S. S.* [N. S.] 3, 373-6 (1935) — An app. is described for detn. of O consumption of insects at low temps. In the larvae of *Pyrausta nubilalis* a decrease in temp from 6° to 0° caused a 2.25 fold decrease of O consumption, while a decrease in temp. from 0° to -6° caused a 1.38 fold decrease of O consumption. Larvae of *Loxostege sticticalis* showed no decrease of O consumption in a temp decrease from 0° to -6°. The R Q at 0° (0.90-1.00) shows the important role of carbohydrates, while the R Q at -6° (0.71-0.82) and at 20° (0.68-0.79) shows the prevalent role of fats or proteins in larval metabolism. E D W.

The visual purple system in marine fishes George Wald *Nature* 136, 913 (1935), cf. C. A. 28, 6473\* — The chemistry of the visual purple system of the sea robin (*Prionotus carolinus*), the black sea bass (*Centropristis striatus*) and the porgy or scup (*Stenotomus chrysops*) is identical with that of frogs. In the retina the following cycle occurs: visual purple → retinein → vitamin A → visual purple. F D Walcr

Nature of crystals found in ameba R H Luce and A W Pohl *Science* 82, 595-6 (1935) Evidence adduced by crystal angles, in p and chem tests indicates that the crystals are possibly Ca chlorophosphate.

J. J. Willaman

## 12—FOODS

F C BLANCHARD and H A LEPPER

The freezing of foodstuffs R Heiss *Angew Chem* 49, 17-21 (1936) — A math. evaluation of the most favorable freezing conditions is presented. Freezing and cold-storage expts. were carried out with meat and milk under varying conditions. Conclusions: (1) Meat (a) Freezing is to take place as soon as possible after the kill, as the pH value influences freezing changes, and oxidative changes are accelerated by decreasing pH values. (b) Freezing is to be carried out as fast as possible, the freezing temp. should not be higher than -17° to -20°, the former view that the freezing changes become particularly great if freezing is carried below a certain temp. was found to be wrong and the crit. interval was found to be between -4° and -9°. (c) If long storage periods are to be used, the storage temp. must not be higher than -17° to -20°, and in order to obtain as few freezing changes as possible it is necessary both to freeze fast at low temp. and store at the lowest possible temp. (2) Milk. In contrast to meat, the freezing changes play only a subordinate role compared with changes due to storage, the temp. interval of -10° to -12° is to be avoided and storage is to take place in air- and vapor-tight containers after preliminary removal of the dissolved O<sub>2</sub>. Exptl. data and many references are given. Karl Kammermeyer

Determination of available and total carbon dioxide in baking powders and self rising flours F W Edwards, F B Parkes and H R Nanj *Analyst* 60, 814-17 (1935) — To det. the available CO<sub>2</sub> the sample is weighed into a test tube which is provided with a 2-hole stopper carrying a tube for the introduction of water and another for the escape of the gas. The tube is suspended in a larger bottle-shaped vessel which is likewise provided with a 2-hole stopper carrying the same tube that leads to the test tube and another which can be connected to a suction pump. At the start the sample is in the inner test tube and the outer vessel contains a measured vol. of standard Ba(OH)<sub>2</sub> soln. The vessel is evacuated and thereupon water is allowed to run into the test tube and the CO<sub>2</sub> liberated is drawn through the Ba(OH)<sub>2</sub> soln. by the suction. Finally the excess Ba(OH)<sub>2</sub> is titrated with 0.1 N oxalic acid to an end point with phenolphthalein. In a fresh sample, the total CO<sub>2</sub> is detd. similarly but with acid instead of water to accomplish the decompn. of the alkali carbonate. The results of 13 analyses of a sample contg. 41.52% CO<sub>2</sub> ranged from 39.96 to 41.52%. W T. H.

The composition of crude fiber A G. Norman *J Agr Sci* 25, 529-40 (1935) — Detailed analyses were made on various agricultural products, on the crude-fiber fraction from these materials and also on the residue at the end of 1.25% acid treatment before final 1.25% NaOH extn. The crude-fiber detn. is misleading because the fraction obtained bears no definite relationship to the structural constituents of the material. The cellulose is partially attacked and lignin extensively removed. Considerable variation is found in the lignin content of crude-fiber fractions. Highly lignified materials do not necessarily yield a crude fiber high in lignin. Since the presence of lignin exercises a direct effect on the digestibility of the material, any empirical method should include all the lignin, and alk. treatment must be avoided. Acid hydrolysis may be a possible alternative method if a correction for protein be made in the case of materials high in N and a detn. of the lignin content made. I C F.

A method for determining *Escherichia coli* in milk, buttermilk and cheese M Lerner *Lact* 15, 831-53 (1935) — The indole method is recommended because it gives clear results, is easy to carry out and enables one to judge the sanitary quality of the products tested even in those cases where acidity and reductase tests have given favorable results. A H Johnson

Manufacture of noodles from green beans Chia Wei Ou and Pin Yen Wu *Golden Sea Research Inst. Chem. Ind. (China) Bull* No. 15, 56 pp (March, 1935) (in Chinese) — The production of noodles from green beans (*Phaseolus aureus* Roxb.), which has been for a long time an important agricultural industry in China, is subjected to scientific study. These beans contain starch, cellulose, hemicelluloses and proteins, the starch being enclosed in cellulose. The beans are first soaked in water, crushed and ground with water. The mixt. is then treated with "acid liquor" obtained by fermentation of these beans. The enzymic action of this liquor causes a portion of the protein material to dissolve and thus enables the starch to be isolated. After sepn., washing and drying, a small portion of the purified starch is mixed with hot water to form a paste, then raw starch (from green beans) is stirred in to form a very thick liquid which is allowed to drip through a perforated ladle to form noodles. After cooking in boiling water the noodles are soaked in cold water, treated again with the "acid liquor," dried and preserved. The "acid liquor" used in these treatments

can be used over again, or it can be used as pig or cattle feed, or as fertilizer.

C. L. Tseng  
Methods of analysis of sodium glutamate condiments  
Ting-Yih Chow *Chenistry* (China) 2, 693-8(1935).—  
The following details in the analysis of mono-Na *d*-glutamate condiments are outlined: (1) moisture, detd by drying at 100° to const wt., (2) H<sub>2</sub>O of crystn, detd by heating at 125-8° (after removal of moisture) to const wt (usually about 1 H<sub>2</sub>O to 1 mol of the salt), (3) Na *d*-glutamate content, detd by (a) Kjeldahl method (total N), (b) Van Slyke's method (amino N), (c) Sørensen's method (adding HCHO and then titrating with 0.1 N NaOH), (d) ignition to Na<sub>2</sub>CO<sub>3</sub> and titration with 0.25 N HCl, or (e) isolation as *d*-glutamic acid HCl, the last method being considered the best, (4) NaCl, detd by titration with AgNO<sub>3</sub>, (5) H-ion concn, detd by means of the quinhydrone electrode, (6) sulfate radical, detd by pptn as BaSO<sub>4</sub>, (7) Millon's reaction, (8) size of crystals, measured by means of a micrometer, (9) heavy metals.

C. L. Tseng  
Ascorbic acid (vitamin C) content of some Indian plant materials M. Damodaran and M. Srinivasan *Proc Indian Acad. Sci.* B27, 377-80(1935), cf *C. A.* 29, 6317.  
—As a preliminary to a study of ascorbic acid, D and S have tested various Indian plants to find a source for a supply of the acid. The acid content of various parts of the plants was detd by the Tullmans-Harris method (cf *C. A.* 23, 3277), that is, by the reduction of 2,4-dichlorophenolindophenol by plant exts in 5% CCl<sub>3</sub>CO<sub>2</sub>H and by titration with I<sub>2</sub>. The leaves of *Sesbania grandiflora* and the leaves and pods of *Moringa pterygosperma* contain about 2 mg ascorbic acid per g of fresh material. These, especially the latter, are exceptionally free from interfering substances and are suggested as a source for ascorbic acid. The Indian gooseberry gives the highest titration values (3-4.7 mg/g pulp) but apparently this is due in part to interfering substances. The juice of this gooseberry and of the cashew apple exhibit great stability and may contain some mechanism that protects ascorbic acid from oxidation. Among the citrus fruits the *Santur* orange (0.4-0.63 mg./cc of juice) and the pumelo (0.50-0.62 mg./cc.) were the richest in ascorbic acid. The ascorbic acid contents of the other plants tested were tabulated.

John D. Milbery

The state of water in tissues Bound water of colloids  
T. Moran *Dept. Sci. Ind. Research, Rept. Food Investigation Board* 1934, 25-6(1935).—In detg the state of water in tissues by the "pressure" method (Lloyd and M., *C. A.* 28, 3957), the activity of the water (*a*) remaining in the colloid under a pressure *P* is given by the equation  $(\ln a/1P)T = V/RT$ , in which *V* is the molal vol of the water in the system. At activities of water down to about 0.7 the method gives results almost identical with the freezing method both for gelatin and for egg albumin. At high pressure, corresponding to low activities of water, a pseudo-plateau is reached in the curve for the hydration-activity of water quite unlike the vapor-pressure isotherm for either gelatin or albumin. It is probable that at low H<sub>2</sub>O contents a considerable resistance is offered by the framework or collection of micelles, and this resistance reaches a limiting value when the micelles of hydrated colloid are all touching, at which point the water is all intramolecular in type. Such an explanation fits in with the results of Moran (*C. A.* 26, 4744), Sørensen (*Compt. rend. lab. Carlsberg* 12, 164(1917)) and A. S. Turby and Lomax (*J. Soc. Chem. Ind.* 53, 979(1934)). Adsorption of salts by proteins  
T. Moran. *Ibid.* 26-7.—An attempt to measure the adsorption of salts by proteins by making use of the fact that at high pressures none of the water bound to the protein is free to dissolve salts indicated a max. adsorption of Na-ion by isoelec. gelatin of 0.031 g., or 1.35 mg. ions, per g. of gelatin. The actual adsorption will depend upon the initial ratio of NaCl to gelatin. Bound water of muscle T. Moran. *Ibid.* 27.—A reasonably exact figure for the firmly bound water of a protein or tissue can probably be obtained by detg the H<sub>2</sub>O content of the protein in equi. with a pressure of approx. 10,000-40,000 lb. per sq. in., the figures obtained by this method being of

the same order as those obtained by other methods.  
The eutectic of muscle T. Moran. *Ibid.* 27-8.—  
Direct measurement of the eutectic of muscle (or muscle-plasma) by a cond. method indicated that it is approx. -37.5°. The uptake of water by gelatin. J. Brooks and T. Moran. *Ibid.* 28-0.—Preliminary expts. carried out to det. whether there is any marked change in the relative proportions of the isotopes of water in the liquid or gel phase during the swelling of gelatin indicated that greater accuracy in the expts. would be necessary to det. quantitatively any "isotopic discrimination," but the results (based on *d* measurements accurate to within 20 p. p. m.) were sufficient to show that, if present, it is small. Bound water or hydration of egg albumin and hemoglobin in saturated solutions of sodium and potassium chlorides G. S. Adair and T. Moran. *Ibid.* 29-30.—  
The hydration of egg albumin and of hemoglobin in NaCl and in KCl was detd by (a) gravimetric analysis of the satd. soln. in terms of protein, salt and water, (b) measurements of *d*, and (c) *n* measurements. For both proteins the hydration in both solvents and at 1° and 20° was found to be approx. 0.13 g. per g. of dry protein.

A. Papineau-Couture

Graphical or numerical evaluation of farnograms?  
I. Kopetz *Muhlenlab.* 5, 97-102(1935).—The "Hungarian" and "Viennese" methods of evaluating farnograms, both of which involve the use of a planimeter, are described, and the advantages of the latter are pointed out.

Clinton L. Brooke

The aleurone layer and its utilization I. Morphological comparisons of the aleurone cells of wheat and rye L. Grunstedt, I. Fromm and F. Bauer. *Muhlenlab.* 5, 81-90(1935).—The aleurone cells of Austrian wheats have walls averaging 4.5  $\mu$  in thickness. In Austrian ryes the walls average 4.5-5.5  $\mu$  in thickness. The transverse walls of the aleurone cells are somewhat thinner than the longitudinal walls. The thickness of the aleurone cell wall is principally dependent on the variety, but is also influenced by climatic conditions. The possible influence of cell-wall thickness upon the utilization of the proteins is discussed.

Clinton L. Brooke

Improving the baking quality of German wheat by conditioning Ernst Berliner. *Muhlenlab.* 5, 81-2(1935); cf. *C. A.* 30, 1683.—Wheat with weak gluten can be improved by conditioning, but no further improvement is effected in wheat with strong gluten. Low gassing power is never increased, but high gassing power may be lowered. The danger of weakening the gluten through proteolytic action during conditioning has been exaggerated. There is no proof that proteolytic activity can be inhibited and diastatic activity stimulated by suitable treatment. Excessive diastatic activity can be reduced by heat treatment, but diastatic activity cannot be increased, even by tempering for several days, unless the moisture content of the wheat exceeds 25%.

Clinton L. Brooke

The baking quality of wheat flour Hugo Kuhl. *Muhlenlab.* 5, 89-94(1935), cf. *C. A.* 29, 75083.—A review with 16 references.

Clinton L. Brooke

Recent entomological investigations Hardness of rice F. A. Squire. *Agr. J. British Guiana* 6, 84-8(1935).—When rice was stored at 90% relative humidity the samples gained in wt. and decreased in hardness; the reverse was true of samples stored at 50% relative humidity. The hardness of rice was permanently increased by parboiling.

K. D. Jacob

Chemical factors influencing the quality of wheat and flour F. B. Working. *Kan. Agr. Expt. Sta., Rept.* 1932-4, 51-2(1934); cf. *C. A.* 29, 2343.—In the centrifugal method of detg. flour absorption, a 30-min. period of soaking was more satisfactory than the 5-min. period previously used. The larger errors were limited to varieties unsuited for bread production. In baking where malt wheat flour was added, sugar production or strictly diastatic activity was of little importance. The chief improvement was due to the proteolytic enzymes and other undetd. factors. Even the purest diastase contains considerable quantities of proteoses and other substances. Oxidizing agents in the amts. normally used in flour

bleaching and dough prepn greatly increased the amt of water sol phosphate in the flour. Some evidence was found that lecithin was partly transformed into cephalin by the oxidizing agents. C R Fellers

Biennial reviews of the progress of dairy science. Section B. Bacteriology and mycology applied to dairying. A T R Mattick, L R Hixcox and J G Davis. *J Dairy Research* 6, 422-53 (1935).—A review is given in which technic is discussed under the headings of the plate count and the methylene blue reduction test. Milk production and pasteurization are discussed in relation to cleaners and disinfectants, mastitis, colon aerogenes organisms and milk faults. The lactic acid and other bacteria of significance in dairying are considered in relation to biology, respiration, fermentation, protein metabolism, fat metabolism and fermented milks. Recent work on cheese is discussed under the headings of pasteurization, starters, ripening processes and faults. Butter is reviewed in relation to starters, churn sterilization and keeping quality during storage. Eleven references are given on technic, 69 on milk production, 55 on pasteurization, 74 on lactic acid bacteria, 62 on cheese and 25 on butter. A H Johnson

Economic methods of equipping the [milk testing] laboratory. F F A Smith. *Proc Intern Assoc Milk Dealers, Lab Sect 29th Ann Convention*, Oct, 1935, 3-19. E. H.

Judgment of the quality of milk from a bacteriological standpoint. W Majowski. *Chem Hekblad* 32, 713-15 (1935).—Polemico with Filippio (C A 30, 1448). M claims that the redutase test alone is not a criterion for the quality of milk. J C Jurjens

Germinical property of milk. B. W. Hammer. *IA Agr Expt Sta, Rept Agr Research* 1934, 85 (1934).—Inhibition of acid development by butter cultures is probably caused by a filtrable form of life gaining entrance to the cultures from the air, although there is a possibility that the slow acid production is due to some sudden change taking place in the bacterial cultures which results in the production of an inhibitory substance. The restraining action of the bacteria free filtrates of butter cultures was destroyed by heating to 60° for 10 min. C R Fellers

Mastitis streptococci in bulk milk. I J Pullinger. *J Dairy Research* 6, 369-82 (1935).—A technic for examg bulk milk by cultural and microscopical means, for evidence of mastitis among cows is described. To demonstrate a slight contamination with hemolytic streptococci, cultural exam of gravity cream is advocated, while if much significance is to be attached to the actual count, whole milk should be cultured. Microscopical exam of gravity cream or of centrifuge deposit for streptococci or leucocytes is a useful addnl test and may give information that is not obtainable by cultural methods, but it does not serve as a substitute for cultural exam. The data indicated the high wile spread incidence of chronic streptococcus mastitis. A H Johnson

The sequence of bacterial and chemical changes occurring in acidified milk. L A Burky, C P Sanders and J F Cone. *J Bact* 31, 188 (1936).—The leucocyte count may reach millions, and streptococci may be present for a considerable time before phys and chem changes occur in the milk. John T Myers

The effect of process of manufacture on the vitamin G content of dried skim milk. H J Davis and L C Norris. *J Dairy Sci* 19, 110 (1936).—There was no significant destruction of the growth promoting component of the vitamin G complex contained in skim milk when dried by the Merrell-Soule or the Gray-Jensen spray process or the open roller process. No measurable destruction occurred as a result of holding liquid skim milk in storage tanks at 170° F for 18 to 21 hrs previous to drying, or of slightly scorching the milk powder during drying, or of neutralizing sour skim milk previous to drying. At a pH of approx 9.5 (titratable alkalinity of 0.15%) a slight destruction appeared to occur. P D A

The surface tension of cow milk. G Belle. *Lat* 16, 13-20 (1936).—The surface tension of 70 mixed milks of the Casablanc region varied between 55.1 and 48.9 dynes

at 18°. The av surface tension was about 50.4 dynes. The surface tension of milk was highest immediately after milking and decreased about 3 dynes after 2-3 hrs. On further holding for 24 hrs, the surface tension continued to decrease slightly. At 20° the milk had about the same surface tension as at 18°. However, when the milk was cooled to 10° and to 0°, a lowering of surface tension resulted. A run surface tension was obtained at 0° which was about 3 dynes lower than that obtained at 18°. A H Johnson

A test for the freshness and the coagulating intensity of milk. E Pijanowski. *Lat* 16, 1-13 (1936).—The general basis of the test is that milk will form a curd due to the decompn of the Ca-casein complex when the acidity reaches a certain point (on the av, 27° Soxhlet-Henkel). If the quantity of acid required for curdling a sample of milk is less than that calcd, it may be assumed that the capacity to form this curd prematurely is due in part to coagulating enzymes that have been produced by microorganisms in the milk, e g, *Bacillus subtilis* and others. The higher the quantity of acid required for curdling, the higher may the degree of freshness of the milk be considered. A formula is given for calcg the degree of freshness of milk. Correction values are given for converting values obtained at several temps to 15°, to which temp the degree of freshness is calcd. 0.1 N H<sub>2</sub>SO<sub>4</sub>, HCl, lactic and formic acids were used in detg the degree of freshness, H<sub>2</sub>SO<sub>4</sub> was the most satisfactory. In detg the degree of freshness, the acid is allowed to flow very slowly into milk at the same temp. The flask contg 30 cc of milk is carefully and constantly agitated until curdling occurs. A H Johnson

A comparison of the methods of determining the fat content of milk. I Stankevics. *Acta Univ Latvensis, Lauksaimniecības Fakultat Ser. I*, 551-75 (in German 575-6) (1931).—Comparative data obtained by various methods with various types of samples are presented in numerous tables. Just as satisfactory results can be obtained with normal milk by the methods of Morsin and of Høyberg as by the H<sub>2</sub>SO<sub>4</sub> method of Gerber. Samples of milk which have been preserved with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and formalin cannot be analyzed by the first 2 methods, as the percentage of fat so obtained is too low. These 2 methods are likewise unsuitable for milk samples which have been kept a long time without preservatives, as the results obtained show considerable variation when compared with those by the methods of Gottlieb-Röse and Gerber. Milk samples which have become somewhat acid can be analyzed with good results by the methods of H and M. Both fresh and old milk can be satisfactorily analyzed by the method of H if the time is increased about 6 min. With the method of M the fat does not sep in the allotted time. In order to prevent sapon of the fat in the butyrometer, the percentage of fat must be read after not more than 30 min in the method of H and not more than 1 hr in the method of M. From the standpoint of simplicity, cheapness and sufficient precision for practical purposes, as good results are obtained by the method of H as by that of Gerber. M G Moore

Lipoid phosphorus and the phosphatides of cow milk. J E Lobstein and M Flatter. *Lat* 15, 946-54 (1935).—By the use of several analytical procedures, the phosphatides in cow milk were found to be about 300 mg per l. The procedures used were such as to eliminate the inclusion of protein and inorg phosphates in the exts; this probably accounts for the low results obtained in this work as compared with the higher results obtained by other investigators. For the purification of lipoid P, the method used was to ppt the casein with AcOH, to remove the fat from the coagulum with ether and to ext the phosphatides from the residual coagulum with alc. The alc ext was then subjected to fractional pptn with acetone and MgCl<sub>2</sub>. In this manner about 150 mg of phosphatides per l of milk was obtained. This procedure yielded about 50% of the phosphatides known to be in the milk and gave a product contg about 20% impurities. The phosphatides of milk form a colloidal complex with the proteins or are united with the proteins by loose chem ties to give

combinations in sol. in ether but capable of dissolving in alc and acetone. A. H. Johnson

**Soft-curd character induced in milk by intense sonic vibration.** Leslie A. Chambers *J Dairy Sci* 19, 29-47 (1936).—The curd reducing effectiveness of oscillators operating at 1100, 1200, 2100, 3000, 610 and 360 cycles was studied in relation to original curd tension, temp., velocity of flow, acoustic energy output and variations in the mech. features of the app. The percentage reduction in curd tension was greatest in hard-curd (60 g and more) milks, the final values approaching a const. level in the soft-curd range irrespective of the original curd tension. No reduction occurred in milk treated below 18° and very little at temps below the m. p. of butter fat. The 360-cycle oscillator was most efficient at a milk flow of 250 gal per hr. With a sound output of about 900 w, more than 50% reduction was obtained. The degree of reduction in curd tension bore a linear relationship to power input up to 2000 w. This represents about 18 w per 1% decrease in tension. Differences in frequency were found to be negligible within the range explored. A direct relation was found to exist between degree of fat dispersion and curd-tension reduction. Increasing no. of fat particles weakened the curd matrix and provided increased adsorptive area on which protein was fixed. Since but a small proportion of the total fat in milk need be finely subdivided to reduce the curd tension, a method was devised for producing soft-curd milk by vibration without impairing the final cream vol. Philip D. Adams

**The electrical decalcification of milk.** J. Pien and J. Bause *Lait* 16, 20-6 (1936).—The elec. decalcification of milk by passing it between Al electrodes has the disadvantage of producing much foam and a ppt. of casein on the electrodes and on the floor of the decalcifying vessel. The amt. of casein pptd. depends on the degree of decalcification of the milk. On reduction of the acidity from 24° to 18° Dornic, 3 g. of casein per l. of milk was pptd. 1 at and solids-not-fat were also removed from the milk as a result of elec. decalcification, but the quantities removed were not as great as the quantities of casein removed. The lactic acid which disappears as a result of elec. decalcification is not destroyed but is found in the pptd. curd and neutralized by basic salts which are formed at the electrodes. Milk which has been neutralized electrically does not show any decrease in lactate ion (as it is still present bound in the protein and as neutral lactate). Milk which has never been allowed to sour can be rendered alk. owing to the basic salts which are liberated as a result of the elec. decalcification procedure. Mild treatment by the decalcification procedure does not have any effect on the flavor or odor of the milk except as the flavor of acidity is concerned, while strong treatment gives rise to off-fishy odors which cannot be removed by aeration. The off-flavors and -odors are increased by subsequent pasteurization. Elec. decalcification has no effect on the bacterial content of the milk. Electrically neutralized milks were not considered suitable for sale. A. H. Johnson

**The age-thickening of sweetened condensed milk.** III Effect of reaction and changes in the electrical conductivity during manufacture and aging. V. C. Stebnitz and H. H. Sommer *J Dairy Sci* 19, 55-65 (1936).—Increasing the acidity of raw milk causes the sweetened condensed milk to thicken more rapidly during storage, while decreasing the acidity makes a more stable product. With unstable milk a change of 0.1 pH exerts a marked effect; with stable milk the effect is not so great. During the storage of sweetened condensed milk at 37° there is at first a slight decrease in pH and then a gradual rise of about 0.2 pH after which it remains fairly const. during the aging period regardless of rate of thickening. During the spring of the year the unstable milk may be stabilized by adding 60 to 125 g. of NaHCO<sub>3</sub> per 1000 lb. of raw milk, being more effective when added to the raw milk than when added to the finished product. Increase in acidity during forewarming cannot be considered an important factor. Changes in elec. cond. cannot be correlated with the stability toward age-thickening. Philip D. Adams

**Rancid flavor in milk.** Earl Weaver, F. L. Fouts and Ruth Reder *Oklahoma Agr. Expt. Sta. Rept.* 1932-4, 149-50 (1934).—Preliminary data indicate there is a direct correlation between rancid fresh milk and high fat content. The latter often occurs as an accompaniment of cystic ovaries and breeding abnormalities. Aborted cows often produced rancid milk. Rancid milk was found as often in early as in late lactation. C. R. Fellers

**Effect of cottonseed meal upon the dairy products.** K. I. Keith, A. H. Kuhlman, Earl Weaver and W. D. Gallup *Oklahoma Agr. Expt. Sta. Rept.* 1932-4, 164-6 (1934).—There was no significant difference in the fat content of the milk from cows fed cottonseed meal and the controls. The cottonseed meal butter was somewhat lacking in flavor and color and had an objectionable gummy body. The cream took an abnormally long time to churn. C. R. Fellers

**Detergents employed in the dairy industry.** G. Ginn. *Lait* 15, 848-70 (1935). The various alkalies employed as detergents in the dairy industry are discussed and the mechanism of their action is reviewed. A. H. Johnson

**The use of tribasic sodium phosphate in cleaning dairy equipment.** L. A. Rogers and Fred C. Evans *J. Bact.* 31, 87-8 (1936).—A 5% soln. of Na<sub>3</sub>PO<sub>4</sub> is an excellent detergent and has a distinct bactericidal action. The corrosive action can be eliminated by the addition of 3% of Na<sub>2</sub>CO<sub>3</sub> to the phosphate crystals. John T. Myers

**Crystalline structure of different ice creams.** W. H. E. Reid *Mo. Agr. Expt. Sta. Bull.* 340 (Ann. Rept. 1933), 41 2 (1933), 41 C 28, 737-9. As the butterfat content was increased from 8 to 15%, there was an increasingly fine texture. Additional increments of fat made the texture "chalky". There was a direct relation between serum solids (varied from 8 to 15%) and fineness in texture. Increase in sugar content from 8 to 14% gave a finer crystal structure. The addition of nuts, cocoa, fruits, etc., increased coarseness. Heat shocking and rehardening of ice cream samples decidedly improved the texture by a fusion of ice crystals. Photomicrographs showed that when the mix was altered, the structure of the ice cream was decidedly modified as related to no., shape and size of the ice crystals and air cells comprising the resultant ice cream. C. R. Fellers

**Effect of heat on the colloidal, physical and chemical changes occurring in milk.** E. R. Garrison and Hans Jenny *Mo. Agr. Expt. Sta. Bull.* 340 (Ann. Rept. 1933), 40 1 (1933).—In a refined electrolysis method, the temp. of the milk in the cell had a pronounced effect on rate of dialysis. All experiments were conducted at 30°. The dial. of the milk with distd. water increased the rate at which cations were removed during the 1st 30 min., but afterward the effect was not measurable. The rate of anion release was not affected by dial. The amt. of cations removed during 5 min. intervals was not proportional to the dial. factor and electrolysis was of no value in detg. the watering of milk. Aging the milk at 2° for 96 hrs. had no effect on the rate of cation removal. Souring affected the rate of release of the anions more than the cations. There was no difference in Holstein and Jersey milks or in raw and pasteurized milks. C. R. Fellers

**Kind of acids in butter and the distribution of these acids between the water and the fat phases of the butter.** B. W. Bird *Idaho Agr. Expt. Sta. Rept. Agr. Research* 1934, 88—A method for detg. the wt., the av. mol. wt. and the I value of the acids sol. in the fat phase of the butter was developed. The method involves the titration of the acids with alc. KOH in fat, petroleum ether and abs. alc. solns.; the removal of soaps from the fat-petroleum ether soln. with 50% alc.; the liberation of the acids with 1.4 H<sub>2</sub>SO<sub>4</sub>, the extn. of these acids with petroleum ether; the removal of the solvent; the weighing of the acids and the detn. of their I values. *A semimicro Hanus I method* was developed as follows: Add 2 cc. of CHCl<sub>3</sub> and 6 cc. of Hanus I soln. (from a specially constructed automatic pipet) to 0.01 to 0.1 g. fat or fat acids in an E. flask. Let the reaction proceed for 30 min. Add 2 cc. of 15% KI soln. and immediately 30 cc. of recently boiled and

cooled distd water Titrate the excess I with 0.025 N  $\text{Na}_2\text{S}_2\text{O}_3$  soln C R Fellers

Causes of the tallowy flavor in strawberry ice cream E W Bird Ia Agr Lxpt. Sta, *Rept Agr Research* 1934, 90-1—There was no correlation between the oxidase values of the ice cream and the development of flavor defects There was no consistent change in fat contents from the beginning to the end of the storage period Fe played no part in the development of the defect Samples prepd with condensed milk showed a marked tendency for more rapid and more pronounced flavor defect than did samples prepd with dry skim milk There was a marked correlation between Cu content and flavor defect Conclusion The factors causing the flavor defect are the presence of Cu and the oxidation of the small amts of fat in skim condensed milk or in whole condensed milk C R Fellers

The correlation between the spoilage of butter and the presence of fat splitting and casein digesting bacteria C N Stark and B J Scheib *J Bact* 31, 87(1936)—In the absence of other spoilage factors there seems to be a direct correlation between the no. of fat-splitting and casein digesting bacteria and the keeping qualities of butter John T Myers

Bacteriological analysis of butter from the region of Brest Biology of the typhoid bacillus in butter J Brisou *Lait* 15, 804-8(1935), cf *C A* 29, 8043—About 5 g. of butter is first emulsified in 15 cc of sterile bile Five cc of the bile butter emulsion is then added to a flask contg 50 cc of physiol salt soln, 50 cc of 6% peptone soln and 2 cc of malachite green (1.200) The flask and contents are then incubated at 37° for 24-36 hrs (or a shorter period if the medium decolorizes too rapidly) After incubation, the material is inoculated on freshly prepd Pb acetate-agar plates After growth on this medium the colonies are identified in the usual way The vitality of the organisms in butter was studied and considered to be related to the  $pH$  and not to the free acidity At  $pH$  values below 6 the vitality was reduced A H Johnson

Factors affecting milk and butter fat secretion I Variations in fat weight, fat percentage and the amount of fat in the milk required to make a given weight of butter Elizabeth O Whetham and John Hammond *J Dairy Research* 6, 320 39(1935)—The amt of fat in the milk required to make 1 lb. of butter has been taken to indicate the size of the fat globules in the milk and hence the type of "cream line" produced With a const milk yield the fat percentage varies between breeds, but not to the same extent as it does when no account is taken of the differences in av yield of the different breeds The same generalization applies to the fat butter ratio, i. e., size of fat globules The effect of equal increases of milk yield on the butter fat ratio is much greater between breeds than it is within any one breed It is suggested that the size of the fat globule is detd by two factors (a) the rate of butter-fat formation by the cell, which varies with breed and stage of lactation and (b) the rate of milk secretion, which affects the size of the globules by the rate at which they are washed out of the cell Regression lines of fat yield, fat percentage and fat butter ratio on milk yield have been plotted under different circumstances affecting milk yield—between breeds, within breeds, with age and period of lactation The changes in milk yield during the lactation period may be due for the most part to changes in the rate of secretion of the cells, and changes due to age are caused chiefly by changes in the no. of cells secreting In accordance with this idea, it follows that when the yield of milk is increased by increased rate of secretion of the gland cell, the percentage of fat in the milk will decrease (period of lactation), but when the yield of milk is increased by the no. of cells secreting, the fat percentage in the milk will remain unchanged (age) This may explain why sires can effect an improvement or otherwise in milk yield in conjunction with or independently of fat yield II The color of butter fat *Ibid* 341-51—The colors of the butter from individual cows were measured on a color scale of

yellow shades on cellulose strips and the results analyzed statistically The mean values and the variability curves of the ranges of butter color in the different breeds of dairy cattle are given Circumstances which may affect these values are given The color of butter from milk of the various breeds decreased in the following order South Devon, Guernsey, Jersey, North Devon, Friesian, Short-horn, Ayrshire, Dexter, Red Poll and Kerry The color of the butter fat was not related to its vitamin A potency 2 Thus goat butter of a chalky white color may have just as great a vitamin A activity as yellow butter The two most important factors affecting butter color are the genetic characters of the cow and the method of feeding A slight increase in the shade of color of the fat occurs under most of the various conditions which give rise to increased milk yield This may be due to increased intake of coloring matter (assocd with greater food intake) in proportion to butter fat produced The color of the fat is high in the 1st few weeks of lactation and falls gradually as more fat is required from the body in addn to that from the food After about 180 days' lactation, the color rises again as the butter yield falls and more of the fat is supplied by the food intake It is suggested that butter fat derived from body fat will be paler than that from food fat when the latter is sufficiently provided with plant pigments A H Johnson

Soil reaction and suitability of milk for cheese making J P Hussmann *Milchwirtschaft Zentr* 64, 438-42 (1935)—It is known that feed rich in nutrients produces a milk of high mineral content and that such a milk is highly suitable for making cheese Acid soils produce poor feed Hence acid soil may be the ultimate reason for poor cheese It has observed such correlation E O Whittier

The function of pepsin and rennet in the ripening of Cheddar cheese I R Sherwood *J Dairy Research* 6, 407-21(1935), cf *C A* 29, 7513—The use of pepsin resulted in cheeses in which the extent of protein degradation was considerably smaller than in control rennet cheeses The type of N partition was influenced to some extent by the proteolytic action characteristic of the particular enzyme employed The use of an increased proportion of rennet led to an anticipated greater extent of protein breakdown, the type of N partition not being appreciably affected No direct correlation was observed between the com quality of these cheeses and the extent or type of protein breakdown which occurred The fact that an increase in the proportions of rennet or the replacement of rennet by pepsin influenced so greatly the extent of the protein breakdown taking place during the ripening process and yet neither brought about dissimilarity in the form of the curve for sol N and nonprotein N nor altered appreciably the type of N partition affords evidence for the role in attacking cheese protein played by the proteolytic enzymes contained in these milk coagulants It is suggested that a rough guide for the comparative protein breakdown that will occur in cheeses is given by the measurement of the degradation brought about in milk by the resp coagulating enzymes, although a marked increase in the content of rennet or pepsin alters the type of protein breakdown in milk in the direction of more complete degradation With cheese, secondary reactions (due probably to bacterial action) among the nonprotein constituents lead to relative increases in subpeptone N With most of the samples of rennet examd, it was found that previous heating of the soln to 34° at  $pH$  7.25 for 10 min, and to 70° at  $pH$  5 for 2 min, to destroy the rennin or pepsin exerted but little influence on the proteolytic activity With a few samples of rennet, the extent of the breakdown induced in milk was diminished and a small alteration occurred in the type of proteolysis Similar heating of 5% pepsin soln was shown to have no effect on the proteolytic action of the enzymes upon milk Since the coagulating power of pepsin was reduced enormously ( $1/10$  to  $1/100$ ) by the heating, it appeared that coagulating action and proteolytic activity are due to sep enzymes Heat treatment at  $pH$  5 and 7.25 was accordingly not effective in destroying rennin

and pepsin. Accordingly, the measurements of the N partition in cheeses manuf'd. by the use of such heat-treated prep'n. without det'n. of the comparative proteolytic action of the samples on milk do not furnish reliable evidence of the role of rennin or pepsin in cheese making.

A. H. Johnson

**Effect of various phases in the manufacture of casein by the natural sour method on its physical and chemical properties.** D. R. Thorpe, H. C. Hansen, R. S. Snyder, R. F. Wood and R. L. Olmstead. *Ida Agr. Expt. Sta., Bull. 212, 3 10(1935)*. Approx. 220 sample lots of casein were manuf'd. and exam'd. chemically for odor, color, soly, yield, moisture, fat, N, pH, ash, cond., total and free acidity and viscosity. Thoroughness of washing was the most important factor of all the various changes made from the standard method. Either increasing or decreasing the acidity of the milk from 0.64% at the time of coagulation resulted in a lower color and odor score. A fat content of the skim milk above 0.1% and insufficient pressure on the green curd adversely affected both color and odor. Both a setting temp. higher than 40° and a cooking temp. higher than 49° decreased the soly. due to the formation of a tough curd. The viscosity of the casein increased with increased temp. in prep'n. Increasing the original acidity of the skim milk resulted in a higher viscosity but decreased the odor score and increased the acidity of the casein. Yield of casein was det'd. by completeness of pptn., loss of curd in draining and washing, loss of impurities in washing and pressing and loss of moisture in drying. Low ash content depended on the use of an acidity of 0.64% at time of coagulation of the skim milk to obtain complete pptn. and thorough washing. At cooking temps. above 49° the curd was tough, rubbery and difficult to wash. High fat content in the finished casein was undesirable. Washing increased the N content of the casein. If the N content falls below 11%, the quality should be questioned. Ash content is also a fair criterion of purity. The only important consideration found in producing casein of low acid content was thorough washing of the curd. Of the various tests studied those which gave the best index of quality were color, odor, soly, viscosity, N, pH and cond. A uniform high-quality casein was produced by the following standard method of manuf.: Use skim milk contg. 0.01% fat or less and a titratable acidity of 0.18% or less. Use sufficient whey starter to raise the acidity of the skim milk to 0.3% at a temp. of not below 21° and with an acidity of the starter not below 1.15 or above 1.35%. Set at a temp. of 40° until an acidity of 0.64 has developed. Coagulate by heating to 10° with const. stirring. Drain off the whey and break the curd into pieces about the size of an egg. Wash the curd with cold water, using 1/4 as much water as original skim milk. Allow to stand in cold water for 15 min. with frequent stirring. Drain the wash water from the curd. Press continuously at not less than 500 lb. pressure for 16 hrs. Dry at 49° for 8 to 10 hrs. in a drying tunnel equipped with accurate temp. controls and good air circulation. C. R. Fellers

**Viscosity of the white and of the yolk.** A. J. M. Smith. *Dept. Sci. Ind. Research, Rept. Food Investigation Board 1934, 53 6(1935)*, cf. C 30, 175\*.—Relative values for the viscosity of yolk at different H<sub>2</sub>O contents were obtained by measuring the time for 30 g. of yolk to pass through a capillary 30 cm. long by 1.04 bore under a head of 45 cm. of Hg. The H<sub>2</sub>O contents were det'd. by drying 4 hrs. at 112°. Addn. of 1 part H<sub>2</sub>O for 10 of initial yolk reduces the viscosity to 10% of its original value, addn. of 2 parts H<sub>2</sub>O for 10 of initial yolk reduces it to 1/10. Comparison of the data with the uptake of water by the yolk from the white during storage (C 25, 5459) indicates that the uptake of water may be the chief cause of the decrease in viscosity of the yolks of stored eggs. If an attempt is made to measure the relative viscosity of thick white by a capillary-tube method, successive shearings produce a progressive change in the direction of greater fluidity, until an approx. steady value is reached, which is of the same order as the value obtained for thin white. On forcing thick white through a small orifice under a

moderate pressure-head (e.g., 30 cm. of Hg), the structure is largely destroyed; if forced through rapidly (e.g., under a head of 71 cm.), the structure is to a great extent retained, even after several repetitions. When the complete white is dried to const. wt. over H<sub>2</sub>O, and then reconstituted by adding to the dry material an amt. of H<sub>2</sub>O equal to that lost, the thick and thin parts reappear in approx. their initial proportions, and the whole white appears similar to that of a fresh egg, but the viscosity is much reduced. A similar result is obtained when the white is frozen (at -5°) and thawed again. The changes in the fluidity of whites and yolks were measured in samples stored for 6 months or more at 0° in air, pure CO<sub>2</sub>, pure N, and mixts. of CO<sub>2</sub>, O and N, and in air after waxing the shells. The fluidity of the yolk increased steadily in every case, but most in the eggs stored in N, where the development of anaerobic bacteria was serious; the change was least in the eggs stored in CO<sub>2</sub>, and, from the standpoint of fluidity of the yolk, 20% CO<sub>2</sub> was as effective as 100% N. The viscosity of complete white decreased very little in eggs stored in air. Waxed eggs showed a greater increase in fluidity than unwaxed eggs stored in air at the same temp. and relative humidity. A. Papineau-Conture

**Yolk color of eggs produced by hens fed various feeds and pigments.** R. B. Thompson and W. P. Allbright. *Okl. Agr. Expt. Sta., Rept. 1932-3, 114-17(1934)*.—The character or amt. of green feed included in the hens' ration had no influence on the color of the egg yolks before storage. Both alfalfa meal and yellow corn meal gave yolks of very uniform moderately yellow color after storage. Hens fed Sudan II dye in capsules produced deep orange eggs. C. R. Fellers

**Effect on egg quality of five different vegetable proteins.** R. B. Thompson. *Okl. Agr. Expt. Sta., Rept. 1932-4, 117 18(1934)*.—Hens having access to wild onions produced eggs entirely normal in color and flavor both before and after storage. Hens which had access to manure heaps, stagnant water or decomposed materials produced eggs of very poor storage life. Cottonseed meal, when included in the ration, gave eggs yolks of an olive color. Alfalfa-fed hens produced eggs of good yolk quality both before and after storage. Smutty wheat had a very bad effect on egg quality. C. R. Fellers

**Changes in the quality of the proteins of meat as determined by digestion with pepsin.** I. A. Smorodintzev and I. N. Liskovskaya. *Bull. soc. chim. biol. 17, 1519-53 (1935)*, cf. C 30, 515\*, 778\*.—Lean beef reaches its max. digestibility in 96-120 hrs. after killing when stored at 1-3°, or in 6-24 hrs. when kept at 36°. L. E. G.

**Changes in the solubility of the calcium compounds during autolysis of muscle tissue.** I. A. Smorodintzev and I. N. Liskovskaya. *Bull. soc. chim. biol. 17, 1814-21 (1935)*.—Lean beef from the hind quarters of cows 5-7 yrs. old contained 4.2-6.7 mg. % Ca. One hr. after killing 30% of the Ca was ext'd. by water, after storage for 24 hrs. at 1-4° 57% was ext'd., and after 72-120 hrs. 62%. The increase in the soly. of the Ca parallels the formation of lactic acid in the muscle. The soly. of the inorg. P showed practically no change during the first 72 hrs. L. E. Gilson

**Vitamin B and B<sub>12</sub>(G) content of South Dakota tailless lamb (muscle, liver, pancreas, thymus) and vitamin C content of liver.** I. dith. Peterson. *S. Dak. Agr. Expt. Sta., Ann. Rept. 1931, 31-5(1931)*.—The amt. of lean lamb muscle required to give a gain of 25 g. in 8 weeks (Sherman-Chase unit of vitamin B) was less than 1 g.; liver, less than 0.5 g.; tongue, 1 g.; brain, less than 1 g.; kidney, less than 1 g.; heart, 0.5 g.; pancreas, 1 g. The lamb was richer in vitamin B than beef. The work is being continued. C. R. Fellers

**Note on the effect of "condition" on the color of body fat in rabbits.** R. Hurrell. *J. Agr. Sci. 25, 541-4(1935)*.—Fat color is of importance in beef produced by English markets because of discrimination against deep yellow fat. Data were collected to ascertain the extent of difference in fat color between lean and fat animals. A tentative conclusion is that in fattening a proportion of the ingested pigment is laid down in the fat but that when such fat is



used for body maintenance the pigment is not wholly reabsorbed but coned in the fatty tissue, thus deepens the color. The results suggest an explanation for the fact that fat color in old cows and steers which have undergone seasonal fluctuation in condition is usually a darker tint than in young heifers and steers which have fattened steadily, and that animals once fat and killed in poor condition are usually darker in color than fat ones.

I C Feustel

Effect of interior temperatures of beef muscle upon the press fluid and cooking losses. Alice M Child and J. Agnes Fogarty. *J Agr Research* 51, 655-62 (1935).—The ratio of press fluid to dry matter is greater in muscle heated to 58° than in that heated to 75°. The moisture content of press fluid varies directly and the total N content varies indirectly with the interior temp of the muscle. The coagulable N in press fluid varies inversely with the interior temp, but there is comparatively little difference in the noncoagulable N in press fluid from muscles heated to 58° and to 75°. An inverse relationship exists between the percentage of press fluid and the total cooking losses in muscle heated to 75°. No relationship of this kind is indicated in muscle heated to 58°.

W H Ross

Effect of hydrogenated lard, storage lard and heated lard on the destruction of vitamin A in foods. V E Nelson, P Mable Nelson and Belle Lowe. *Ida Agr Expt Sta*, *Rept Agr Research* 1934, 81 (1934), cf *C A* 29, 2614.—Unheated fats when mixed with butterfat did not cause destruction of the vitamin A content of the latter. On the other hand, heated fats did cause definite destruction of vitamin A. Three cow brands of oleomargarine similarly did not cause loss of vitamin A when mixed with butterfat. It is believed all these brands contained coconut fat which acted as a protective agent. When lard was heated to 80° instead of 120° and mixed with butterfat there was little loss of vitamin A and a retardation of rancidity.

C R Fellers

Methods for determining pentoses as furfural in citrus fruits. Walton B Sinclair and E T Bartholomew. *Am J Botany* 22, 829-42 (1935).—In the expts described, practically theoretical yields were obtained from known samples of furfural by the bromate method. Theoretical yields of pentoses as furfural were not obtained from *d* xylose and *L*-arabinose. Steam distn did not give higher yields than ordinary distn. In testing citrus fruit juice and tissues, other substances distd over with the furfural and subsequently reacted with the Br, this causes an error with the bromate method. These substances were extd from the tissues with 95% EtOH and Et<sub>2</sub>O, after which comparable results were obtained by the phloroglucinol and bromate methods, the latter giving slightly higher values. Mature Valencia orange peel distd with 12% HCl yielded 15.86% (based on dry wt.) of furfural by the modified bromate method and 14.98 by the phloroglucinol method. The corresponding values for lemon peel were 17.45 and 16.55. Navel and Valencia orange juice sac tissues yielded 12-14%.

L E Gilson

Aguate (Persea gratissima, Gaertn.) Ernesto Parodi. *Italia agricola* No 11 (1935). *Agr coloniale* 29, 578.—This subtropical plant can be cultivated in Italy, in the regions with temperate winter temps. The best fertilizer is ammonia N 5, P<sub>2</sub>O<sub>5</sub> 7 and K<sub>2</sub>O 2%. The av compn of the fresh fruits is H<sub>2</sub>O 68.51, proteins 1.59, fats 21.54, carbohydrates 7.14 and ashes 1.22%, 450 g of aguate pulp may give an av of 1036 cal.

G A Bravo

Acid and pH variations in Ananas comosus Merr in relation to swells caused by Clostridium spp. C H Spiegelberg. *J Bact* 31, 85 (1936).—Com swells may result from a too high pH (above 4.4) in cans during and after processing of pineapples.

John T Myers

Culinary quality in white potatoes. J S Cobb. *Am Potato J* 12, 335-46 (1935).—The results of cooking tests and chem analyses indicated that good cooking quality (a mealy, flaky, white fleshed cooked potato) is closely associated with high starch and dry-matter contents and low N content of the tuber. There was no relation between the

fertilizer treatment of the plant and the cooking quality of the tuber. There was a definite tendency for baked potatoes to give higher quality scores than boiled ones of the same lot, apparently because of the higher proportion of starch to water in the baked potatoes. There was a 25% loss of wt due to loss of water during baking while boiling gave no loss nor gain of wt. Twenty three references.

K D Jacob

Sugar feeding-stuff experiments. S Koch. *Centr. Zuckerind* 43, 890-1, 951 (1935).—The feeding values of plain beet pulp, Steffens pulp and dried cosettes were tested. With milk cows the results with Steffens pulp and dried cosettes were equal, but definitely superior to plain pulp. With pigs Steffens pulp and dried cosettes could advantageously be substituted for about 50% of the usual barley ration and very appreciably reduce the cost of production of 1 kg of meat.

F R Bachler

Influence of an increase of excess of base in a food ration on the acid base equilibrium, condition of health and yield of milk cows. E Brouwer. *Biedermann's Zentr. B Tierernahr* 7, 463-95 (1935), cf *C A* 29, 3001.—Experiments were conducted on 2 groups of 13 cows each which were fed a practical ration, but with an increase in excess of base, produced by the addn of carbonates and bicarbonates, to det if there was a favorable or unfavorable influence on the health, the milk and fat production and on the composition of the butter fat.

The "base" mixt was composed of Na and K bicarbonate and magnesite (plus some CaHPO<sub>4</sub>). The mutual ratios of the elements Na, K, Ca, Mg and P in the ration were practically unaltered by the addn of the mixt so that only the pure function was studied. In this and in former expts with ordinary Netherlands rations, composed of grass hay, Netherlands grass silage and concentrates, the urine was found to have  $pH = 7.5-8.0$ , total CO<sub>2</sub> = 200 vol %, organic acids = 100 mg equivalents per l and NH<sub>3</sub> usually lower than 5 mg equivalents per l.

On the same ration (without chalk) the total CO<sub>2</sub> content of the blood plasma was 61.3 = 3.8 vol %. In increasing, in one of the groups, the quantity of the base mixt, to nearly 900 g per cow per day, the base excess was increased by more than 7.7 g equivalents, so that the total base excess in the ration was 12.6 g equivalents per day per cow. The urine  $pH$  rose from an av of 8.10 in the control group to 8.27 in the exptl group and the CO<sub>2</sub> content from 236 to 555 vol %, while the org acid content fell from 123 to 52 mg equivalents per l.

This last-mentioned fact was not caused by a considerable reduction in the daily production of org. acid, but wholly or at least for the most part by a considerable increase of the quantity of urine. In feeding rations giving rise to either alk or acid end products of metabolism, the secretion of org acids per day does not, in B's opinion, considerably increase or decrease.

The total CO<sub>2</sub> of the blood plasma increased only to a small extent, namely from 61.4 to 67.9 vol %. The live wt and the health of the 2 groups were practically the same. There were only small differences in the daily milk output, fat and fat free dry substance. Neither was there any influence on the fat percentage, percentage of fat free dry substance of the milk, or on the I no or Reichert-Meissl value of the butter fat.

These expts show that a healthy cow, with sound kidneys, can tolerate, at least for some time, a very large base excess without unfavorable consequences. It may also be concluded that it is not to be feared that on a mineral acid silage the addn of too large an amt of chalk or soda might produce alkalosis. One hundred eighteen references.

F L Dunlap

Comparison of the chemical analyses of carpet grass and Bermuda grass. E C Cling, J P LaMaster and J H Mitchell. *S Car Agr. Expt Sta*, 47th Ann Rept 51-3 (1934).—From Apr 5 to Oct 25, 8 clippings of grass plots were made, weighed and analyzed. There is a higher dry-matter content and higher protein in Bermuda grass than in carpet grass. Carbohydrates are higher in the latter. The ash content of Bermuda grass averaged 6.5% as compared with 7.9% for the carpet grass, the resp percentages of crude protein were 10 and 14.7.

C R Fellers

The botanical and chemical compositions and the feeding values of hays imported from Italy and France (into Switzerland). H. Gutknecht. *Landw. Jahrb. Schweiz* 49, 789-804 (1935).—Sixteen samples of Italian and 13 of French origin were examined. The Italian samples were quite similar to av. Swiss hays, but the French samples in general contained less digestible protein, less utilizable starch (Kellner coeff.) and less  $P_2O_5$ . F. O. Whittier.

Nutritive value of lucerne. IV. The leaf-stem ratio. H. E. Woodman and R. E. Evans. *J. Agr. Sci.* 25, 578-597 (1935), cf. C. A. 29, 5890.—Sep. analyses of the leaf and stem fractions of lucerne cuts showed that at all stages of growth the leaves are richer in protein and poorer in fiber than the stems. Nearly 50% of the dry matter of the stems in the flower stage consists of fiber. Leaves display a much higher degree of uniformity in protein and fiber content and are also very definitely richer in ether ext. and slightly richer in N-free extractives than the stems. Both leaves and stems show a decrease in  $K_2O$  content with advancing maturity although the stems continue to be richer than the leaves. The stems suffer considerable reduction in ash content with increasing growth. The flowers tend to follow the leaves in org. compn. and the stems in mineral content. I. C. Feustel.

The chemical treatment of straw by means of sodium hydroxide solution. N. D. Pryanishnikov and E. A. Nesterova. *Problems of Animal Husbandry* (U. S. S. R.) 1935, No. 4-5, 209-17.—The nutritive value of straw is greatly increased by 8 extras with 1.5% NaOH soln. The percentages of org. matter, N-free material, and crude cellulose before treatment were 48.71%, 43.90% and 58.28%, resp. After treatment the values were 67.70%, 52.15% and 82.27%, resp. The starch equiv. (Kellner) changed from 10.22 to 32.77. S. A. Karjala.

Feed value of whey. E. C. Damrow. *Natl. Butter and Cheese J.* 26, No. 22, 24 (1935). A. H. Johnson.

Detn. of N in foodstuffs (Shirokov, Volovinskaya) 7. Cottonseed and its products [nutritive values of meal] (Gallup) 27. Variations in the lactose content of milk (Brown, et al.) 11F. Colloid chemistry of rice starch and cooking of rice (Sakurada, et al.) 2. Aldchydase of milk (Ritter) 11A. Effect of  $KBrO_3$ , etc., on the baking property of wheat flour (Jørgensen) 11A. Storage and physiology of tropical fruits (Wardlaw, Leonard) 11D. Water-proofed paper for wrapping candy (U. S. pat. 2,029,300) 23. App. for gelatin extn. (U. S. pat. 2,024,835) 1. Humidifiers for bakers' ovens (Brit. pat. 436,599) 1. Protein degradation products (Brit. pat. 436,591) 16. Dicalcium phosphate [for use with foods] (U. S. pat. 2,029,967) 18.

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Preserving wheat germ. Peter J. Donk and Alexander R. MacDonald (to Cerobrix Ltd.) Brit. 436,765, Oct. 17, 1935. To stabilize the germ against rancidity and improve its keeping qualities, it is ground to about the fineness of flour, NaCl being mixed therewith prior or subsequent to or during the grinding to absorb moisture and inhibit too rapid diastatic action of yeast in baking, and an inert non fatty filler, e. g., potato, rice or corn flour or rice polishings, is added, the product being dried before or after addn. of the filler by heating without free access of air or in a current of  $CO_2$ , N or other inert gas.

Germinial substance suitable for use in foods from seed kernels. Moritz Hienstock. U. S. 2,028,132, Jan. 21, 1935. For recovering germinal substance from a mixt. obtained on mechanically degerminating seed kernels such as those of *Ceratonia siliqua* and related plants of the families of the Caesalpiniaceae and Mimosaceae, the main portion of the endosperm is removed from the mixt. and the remaining mixt. contg. the germinal substance and other constituents of the kernels is introduced into a liquid medium such as  $C_2H_5Cl$  and  $CaH_2$  which dissolves fatty substances and has a sp. gr. under working conditions intermediate the sp. grs. of the germinal substance and of the associated substances and which is indifferent to gluten-forming proteins of the germinal substance, so that the mixt. seps into 2 layers, the upper of which contains the germinal substance, the temp. is reduced to give required sp. gr. and the layers are sepd. Cf. C. A. 30, 1142.

Food for diabetics. Karl Wille and Eugen Fritsch. Brit. 435,978, Oct. 2, 1935, F. 788,864, Oct. 18, 1935.

Baked goods poor in carbohydrates are prepd. by treating wheat or rye germ, freed from oil, for a day at about 30° and then for several days at room temp., with yeast and lactic acid, mixing the product with fresh germ and fermenting again for several hrs. with yeast, adding further germ and yeast and fermenting again and finally working up to baked goods.

Bakers' yeast. Alfred Schultz (to Standard Brands Inc.) U. S. 2,029,592, Feb. 4. A high-protein stock yeast having a protein content of over 50% on the dry basis is added to a yeast nutrient soln. such as one prepd. with use of molasses,  $NH_4$  phosphate or phosphoric acid and  $(NH_4)_2SO_4$ , and the P nutriment is restricted to 1.25-2.0% (preferably about 1.6%), expressed as  $NH_4H_2PO_4$ , and based on the wt. of the molasses, in order to produce a yeast, by propagation with aeration, which contains less than 50% protein on a dry basis and which has high baking quality and keeping quality.

Bakers' yeast of low protein content. George W. Kirby and Charles N. Frey (to Standard Brands Inc.) U. S. 2,029,572, Feb. 4. A high-protein type of stock yeast (having a protein content of over 50% on a dry basis) is added, in a proportion of at least 30% of the total sugar, as molasses, to a nutrient soln. contg. yeast-assimilable sugar (such as that of molasses) and a yeast-assimilable inorg. N compd. such as  $NH_4$  sulfate and phosphate, yeast propagation is effected with aeration while

limiting the yeast-assimilable inorg. N nutrient to about 4 to 12%, expressed as  $\text{NH}_4$  sulfate and based upon the total sugar calcd. as molasses, so that there is produced a high yield of yeast of good baking and keeping qualities having a protein content substantially less than that of the stock yeast added. Aq.  $\text{NH}_3$  may be added during the process to regulate the  $\text{pH}$  and supply a portion of the N for the yeast growth (suitably instead of  $\text{NH}_4$  sulfate as a N source for the yeast).

**Starch preparations** Hermann Luthje Brit. 437,028, Oct. 3, 1935. A baking aid or bread improver is prep'd by blending a neutral cold-setting starch that contains no chemicals with so much raw untreated starch, e. g., potato starch, that the  $\text{H}_2\text{O}$ -binding capacity of the product does not exceed the ratio 1:8.

**Flour** Otto H. Jock Brit. 436,030, Oct. 3, 1935. To improve the baking qualities and so to enable an increased percentage of European wheat to be used in the flour mixt. prep'd for dough-making, a vegetable phosphate-vegetable oil mixt. contg. about 35% oil is mixed with flour in the proportion of 1 of mixt. to 4 of flour to produce an intermediate product which is subsequently mixed with the bulk of the flour. The phosphatides are obtained as lecithin by the extrn. of soybeans.

**Fermented milk foods** Marcelle Mignard (see Devaux) Fr. 788,801, Oct. 18, 1935. Foods made from fermented milk, e. g., yogurt, have their nutritive and digestive properties improved by adding during the fermentation a soln. obtained from a culture of active bacilli after the bacilli are killed.

**Sterilizing milk** E. I. du Pont de Nemours & Co. Inc. Fr. 784,208, Oct. 25, 1935. Milk or products made therefrom or contg. milk are sterilized by adding at a temp. below 50°, preferably in the cold, 0.0075-0.004% by wt. of  $\text{H}_2\text{O}_2$  before or after heating to a temp. below 61°. For a high concn. of microbes the heating may be carried to 63°.

**Stabilizing compound for chocolate mixtures** David E. Lunn (to International Patents Development Co.) Can. 354,430, Nov. 20, 1935. As a stabilizing agent, powd. starch is intimately mixed with a small quantity of finely ground agar-agar in quantity from 1 to 10% by wt. of the starch on the dry basis. The mixt. has substantially uniform distribution.

**Stable chocolate syrup** David E. Lunn (to International Patents Development Co.) Can. 354,431, Nov. 20, 1935. The ingredients of the syrup are incorporated in a protective colloidal suspension contg. swelled starch and agar-agar in quantity 1.0-10.0% by wt. of the starch on the dry basis. The starch prevents the chocolate fibers and fats from sepp. The agar-agar prevents the starch particles from being ppt'd from the suspension.

**Stable chocolate syrup** David E. Lunn (to International Patents Development Co.) Can. 354,432, Nov. 20, 1935.—The syrup contains 0.7-1.2% by wt. of the finished syrup of swelled starch and agar-agar, in which the proportion of agar-agar to starch is 1.0-10.0% by wt. of the starch on the dry basis. Cf. preceding abstr.

**Cream cooling and aerating device** Edward Franklin and Thorsten Nelson U. S. 2,029,021, Jan. 29, Various structural details.

**Freezing ice cream with solid carbon dioxide** Clarence I. Justheim U. S. 2,029,025, Jan. 28. A mixt. formed of powdered gelatin, powdered egg, powdered skim milk, cream, sugar and flavoring is stirred with powdered solid  $\text{CO}_2$ .

**Butter, cream, fats, etc. "Elact"** Gesellschaft für elektrische Apparate G. m. b. H. Brit. 436,372, Oct. 4, 1935. This corresponds to Fr. 770,814 (C. A. 29, 832), the skimmed or unskimmed milk or liquid dairy residues used for the washing are first treated electrically by the process of Brit. 433,576 (C. A. 30, 522).

**Margarine** Harburger Oelwerke Bruckman & Merckel (Frans. Blomem, inventor) Ger. 629,988, Oct. 31, 1935 (Cl. 554, 1,031). MEL or a synthetic nutritive medium is fermented with lactic acid bacteria and is added to margarine. EtOH or its derivs., such as alcoholates or esters, is then added. In an example, ruff acidified by bacteria is added to margarine. EtOH is then added, and the mass is mixed and churned. Cf. C. A. 29, 4831.

**Pectin** W. W. Cowgill (to Sargol, Inc.) Brit. 437,683, Nov. 4, 1935. See U. S. 1,973,113 (C. A. 28, 6879). Any adherence-retarding agent may be used in place of the glycerol, e. g., olive oil, lemon oil, cod-liver oil, refined mineral oil, waxes, paraffins, sugars.

**Gelatin** Carl Freudenberg G. m. b. H. Fr. 789,602, Nov. 4, 1935. Gelatin is improved by subjecting it to a swelling by means of a noma swelling agent, e. g., glycerol, and if necessary to a heat treatment, then incorporating in the gelatin softening agents insol. in water, such as castor oil or fish oils, after having eliminated to a large measure or completely the swelling agent.

**Preserving animal tissues such as cured meats, etc.** Sidney Musber. U. S. 2,029,249, Jan. 29. The surface of meats, fish, poultry or the like is dusted with an antioxidative material such as pulverized oats, which may be mixed with salt.

**Meat preserving salt** P. Genu and D. Blanckert. Belg. 479,602, April 30, 1935.  $\text{KNO}_3$  is mixed with  $\text{NH}_4\text{Cl}$ , and Na biphosphate and salt are added to the mixt.

**Preparing artificial sausage casings for shipment and handling** Bernard H. Schenk (to Viking Corp.) U. S. 2,028,691, Jan. 21. Various details.

**Preserving fruit** Alexander Gerner-Rasmussen. Brit. 435,618, Sept. 24, 1935. Divided on and added to 435,587 (C. A. 30, 1143). The process of 435,587 is modified by adding to the coating liquid a small amt., e. g., up to 2%, of gum sandarac.

**Preserving fruits and vegetables** Will H. Ewell U. S. 2,029,970, Jan. 29. Apples, pears, apricots, peaches, etc., are sliced and the slices are immersed in aq.  $\text{CO}_2$  soln., removed, drained and packed (suitably in boxes lined with oiled paper) and may be placed in cold storage until desired for use.

**Preservation of foodstuffs** J. Foulon. Belg. 406,902, Jan. 31, 1935. The foodstuffs are placed in tightly closing steel cells in which a small piece of S is burned just before the cells are closed. The  $\text{SO}_2$  thus formed is subsequently replaced by an atm. of  $\text{CO}_2$  under slight pressure. The process is especially applicable to eggs.

**Preserving food, fabrics, etc.** Wilhelm J. H. Hinrichs. Brit. 435,394, Sept. 16, 1935. See Ger. 610,018 (C. A. 29, 4067).

**Coffee substitutes** Eduard Jalowetz and Max Hamburg. Brit. 436,116, Oct. 4, 1935. See Fr. 784,172 (C. A. 30, 1869).

**Coffee substitute** Franz Pracht. Fr. 789,618, Nov. 4, 1935. Simple or complex albuminous material of vegetable or animal origin, preferably contg. P, is dissolved into its elementary components in the presence of water, under pressure and at temps. of 100-220° and above, decomposition of the amines or aminophosphoric acid being avoided; then the ext. is evap'd to dryness under vacuum and roasted, if necessary after decomposition of the albumins and in the absence of air. Decomposition to fatty acids,  $\text{NH}_3$ , mercaptans, etc., during roasting is avoided.

**Apparatus for making yogurt under controlled temperature** Soc. générale de constructions électriques et mécaniques (Alsthom) Fr. 789,792, Oct. 16, 1935.

**Sterilizing and canning foods** Charles O. Ball (to American Can Co.) U. S. 2,029,303, Feb. 4. App. and various operative details are described.

## 15—CHEMICAL INDUSTRY AND MISCELLANEOUS INDUSTRIAL PRODUCTS

(PLASTICS, RESINOIDS, INSULATORS, ADHESIVES, ETC.)

BARIAN &amp; MINI

Chemical foreign trade in 1935 Otto Wilson *Ind Eng Chem* 28, 304 8(1936) P 11

Progress in the German chemical industry in 1935 Bruno Waeser *Ind Chemist* 12, 10 11(1936) L 11

Perkin Medal awarded to Warren K Lewis for his work in chemical engineering Anon *Ind Eng Chem* 28, 256(1936) — Biographical and portrait of L. and list of Perkin medalists Cf following abstr G G

Application of physical data to high pressure processes W K Lewis *Ind Eng Chem* 28, 257 62(1936) — Six charts and various methods of computation are given whereby pressure vs temp relations of higher hydrocarbons (3 or more C atoms) can be calculated, such calculations require no knowledge of the crit vol, only the crit pressure and temp of the substance. These same hydrocarbon curves can be used to approximate the properties of other compounds, even though widely different in type Also in *Chemistry & Industry* 1936, 123 30

Industrial medicine and industrial toxicology G H Gehrmann *Trans Am Inst Chem Engrs* 31, No 4, 712 27(1935) C L Mantell

Natural and industrial gases A Yu Shchukov *Trans V I Mendeleev Congr Theoret Applied Chem* 1932 2, Pt 1, 730-7(1935) A review of problems connected with the utilization of natural and industrial gases in the U. S. S. R. with special reference to products of chlorination of ethylene I E Stefanovsky

Hygroscopticity of salts and salt mixtures in relation to industrial technique G Robert *Industrie chimique* 22, 887-95(1935) A review of the hygroscopticity of salt solutions dealing with crit hygrometric state and capacity for the rate of absorption of H<sub>2</sub>O vapor A P C

The recovery of solvent vapors by adsorption The Acetone process P L Lucas *Rubber Age (N Y)* 39, 199-203(1936) — An illustrated description C C Davis

Syntheses of solvents and plasticizers Yu S Zait'kind *Trans V I Mendeleev Congr Theoret Applied Chem* 1932 2, Pt 1, 720-30(1935) A review on the synthesis of methylcyclohexane, cellosolve, ethyl ether, camphor, phthalates, phoshyoric acid esters, etc I E Stefanovsky

The present status, from the chemical point of view, of synthetic resins I Jacobs *Rev gen caoutchouc* 12, No. 117, 3-10(1935) — A review and discussion C C Davis

The preparation of oil soluble resins from polyhydric alcohol and polybasic acid Jen Chuan Wee *J Chinese Chem Soc* 3, 321-4(1935) Oil sol resins are prep'd (1) by heating castor oil soap with 2,3-dichloropropanol and then cooking the product with phthalic anhydride and (2) by heating together raw castor oil and glycerol in the presence of NaOH as catalyst, and then adding phthalic anhydride and heating again. The resin prep'd by the 2nd method is suitable for varnish blending Wu H Adolph

The synthesis of oil-soluble artificial phenol resins of the type of albertol S N Ushakov and V M Zait'kov *Narodnui Komissariat Tsvetel Prom S S R, Leningrad, Plastmassy* 1, 233 46(1936) — Albertol can be prep'd from 60 parts of 40% CH<sub>2</sub>O soln, 100 parts of cresol and 10 parts of abietic acid heated together for 5 hrs, and then esterified with 8.5 parts of 28% glycerol for 7.8 hrs, at 240-60°. These amts can be varied in wide limits without greatly affecting the product, but if more than 125 parts of CH<sub>2</sub>O soln is used, the product is insol in oils. Albertol can also be prep'd by heating 100 parts of iditol with 200 parts of natural resin at 200° for 21-5 hrs. Addn of 1.00H to CaH, or 11.0H of soln of albertol raises the acid no H M Leikster

The use of furfural in the production of synthetic resins,

insulating forms and plastic compounds G S Petrov and A Ya Hasilov *Narodnui Komissariat Tsvetel Prom S S R, Leningrad, Plastmassy* 1, 245 13(1935) — Condensation of PhOH and furfural with further condensation of this product with CH<sub>2</sub>O, gives resins suitable for a wide variety of uses. Standards for tech. furfural are suggested H M Leikster

The conditions for the condensation of different portions of shale tars with formaldehyde to form products similar to rezol S N Ushakov and G S Brodskii, *Narodnui Komissariat Tsvetel Prom S S R, Leningrad, Plastmassy* 1, 241 30(1935) — The PhOH portion from shale oil, b 170-320°, gives satisfactory resins with CH<sub>2</sub>O when NaOH or K<sub>2</sub>CO<sub>3</sub> is used as catalyst. Residual high boiling, neutral oil in the resin acts as a plasticizer, but tends to decompose at high temp, and pressure H M Leikster

Testing and evaluation of thermal insulation Walter Schuler *Gas u Wasserfach* 78, 160 42(1935) — Methods of testing thermal insulation, using the Schmidt heat-flow meter, are discussed. Theoretical and actually det'd heat transmission data and calcs for most economical thickness of covering are given for steam pipe insulating materials such as kieselguhr, magnesia and in insulations built up from metal foil and glass wool. Metal-foil insulation requires careful installation and the use of a protective sheet iron cover, and is of use where light weight and freedom from dust are essential. Glass-wool insulation can be protected by a gypsum cement coating, it can be applied in insulating large gas and steam pipes where other insulations would be too heavy, and for other special purposes. Of the coverings tested, the kieselguhr is believed to be best adapted for ordinary steam pipe insulation R W Ryan

Adhesives for "Rubberoid" roofing material for high and low temperatures A V Ikhonin *Stroitel Material* 1935, No 8, 10 31 — Oil-bitumen adhesives must have different softening points according to the latitude of the place where they are used and to the angle of inclination of the roofing. Directions as to their prep'n for different conditions are given. R. K. Stefanovsky

The mechanism of the condensation of phenol with formaldehyde A Vanshel't, T Irlukova, A Itenberg, M Babits and A. Igel *Narodnui Komissariat Tsvetel Prom S S R, Leningrad, Plastmassy* 1, 181-20(1935); cf. C. A. 29, 1178 — PhOH and CH<sub>2</sub>O can be steam-dist'd from the polymerized residue of their condensation, even in the presence of 1% H<sub>2</sub>PO<sub>4</sub> and det'd. by standard methods, each in the presence of the other. The reaction rate of their condensation shows that at 60° in the presence of H<sub>2</sub>PO<sub>4</sub> equimol. amts. react. At higher temps, or in more acid media, a larger amt. of PhOH reacts, an indication of a secondary reaction. In alk. media PhOH and CH<sub>2</sub>O react in the proportions 2:3. These facts agree with the mechanism of Raschig (C. A. 7, 887), but do not agree with that of Baekel and Bender (C. A. 19, 1352). H. M. Leikster

The condensation of phenol and cresol with acetaldehyde to form plastic compounds and insulating materials. G S Petrov and A. A. Pichugin, *Narodnui Komissariat Tsvetel Prom S S R, Leningrad, Plastmassy* 1, 216 61(1935) — The prep'n. of many forms of PhOH-CH<sub>2</sub>O resins shows that they are as useful as PhOH-CH<sub>2</sub>O resins. H. M. Leikster

Recent investigations on emulsions, with special emphasis on the mechanics of formation, Wu, Chiyton, *Int. Chemist* 12, 15-10(1936); cf. C. A. 29, 4478.

The recovery of solvents in the manufacture of joint diaphragms Hans Diegmann, *Rev. gen. caoutchouc* 12, No 117, 24 6(1935). — A discussion. C. C. Davis

Effects of metals on fly sprays David G. Hoey,

*Soap* (Sanitary Products Sect.) 12, No 1, 105, 107 1  
(1936)—H states that the presence of perfume contg. terpenes or reducing substances caused a deterioration of pyrethrum (pyrs) and toxicity. Samples of standardized pyrethrum fly sprays in flint glass bottles were treated with 1% by wt of confectioner's glaze, flux, tinsplate and combinations of these, and stored for 10 months in darkness. All samples were shaken at intervals and later analyzed by the Seal method for pyr I and II (cf C A, 28, 4531<sup>1</sup>) and tested on house flies by the Peet-Grady method (cf C A 22, 4731). Confectioner's glaze or combinations of it with other materials caused considerable changes in color and about one-third loss of pyrs and toxicity. H found some metal contaminants contaminated with more glaze than used in his tests. Flux caused a small loss, tinsplate scarcely any. The effect of small strips of Cu, Zn, Sn, Al, Pb and Fe on the fly sprays was also tried in a 2 weeks' storage test. Cu and Pb both lost some wt and caused 8-13% losses in pyrs. There was little change with the other materials but these are being tested for longer periods. Two references.  
Henry H Richardson

Gas heating of platens for plastic work (Hems) 21  
Sheet vulcanized fiber (U. S. pat 2,028,932) 23 Plastic compns (Brit pat 435,713) 20 Asphalt-asbestos mixt for heat insulation (U. S. pat 2,029,289) 20 App for coating wires with rubber (U. S. pat 2,029,435) 30 Rubber devices [for production of insulators] (Fr. pat 788,840) 30 Filtering materials [for gas masks] (Fr. pat 789,336) 1 Condensation products of aromatic hydroxy compds with alkenols (U. S. 2,029,539) 10

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Purifying gases I G Farbemnd A-G (Hans Bahr 2 and Wilhelm Wenzel, inventors). Ger 620,932, Oct 30, 1935 (Cl 26d 9 04) Weak gaseous acids are removed from gases by washing with org bases and regenerating the bases by heating Bases contg at least 2 N atoms in the mol are used, the general formula being  $A^1A^2N-B-NA^1A^2$ , B being an aliphatic hydrocarbon residue and at least one of the A groups being an alkyl, aryl,  $NH_2$  or substituted  $NH_2$  group Thus, a gas stream contg  $H_2S$  and  $CO_2$  is washed with a 10% soln of dihydroxyethylenediamine in oil to remove the  $CO_2$  Other washing bases are monohydroxy methyl diethylenetriamine, and monomethyl monohydroxy ethyl diethylenetriamine

Recovering acidic gases Gerald J Horvitz Brit 436,220, Oct 3, 1935 Weakly acidic gases that yield dibasic or polybasic acids, e g,  $CO_2$ ,  $SO_2$ ,  $H_2S$ , are sepd and recovered from gaseous mixts by scrubbing with a soln of a double alkali metal  $NH_4$  salt of a tribasic or polybasic acid, e g,  $Na(NH_4)_2PO_4$ ,  $Na(NH_4)_2AsO_4$ ,  $Na_2(NH_4)_2P_2O_7$ ,  $Na_2(NH_4)_2P_2O_8$ ,  $Na(NH_4)_2C_2H_3O_7$ , to form an acid salt of the acid gas, e g,  $NH_4HCO_3$ , and a double alkali metal  $NH_4$  acid salt, e g,  $NaNH_4HPO_4$ , the resulting liquor being heated to recover the acid gas and regenerate the scrubbing soln App is described In 436,369, Oct 3, 1935, weakly acidic gases, e g,  $CO_2$ ,  $HCN$ , are removed and recovered from gas mixts by treating with  $NH_3$  in the gaseous phase, dissolving the  $NH_3$ , acid gas and  $NH_3$  salts formed in  $H_2O$ , neutralizing the soln with a soln of  $NaH_2PO_4$  or other salt of a comparatively strong acid still having more than 1 replaceable H atom, the acid gas being thereby expelled and, e g,  $NaNH_4HPO_4$  being formed, which is heated to regenerate  $NaH_2PO_4$ ,  $NH_3$  and residual acid gas The gases together with  $H_2O$ -vapor at least equal in vol to the  $NH_3$  are returned to the acid gas absorber and the regenerated  $NaH_2PO_4$  soln is concd, the cooled soln being recycled, and the  $H_2O$  driven off being condensed and returned to the absorber. App is described.

Purifying ammoniacal cuprous solutions containing carbon oxides Axel Christensen (to Chemical Construction Corp.). U. S. 2,029,411, Feb 4. A soln such as one which has been used for gas purification is heated to expel  $CO$  and  $CO_2$  together with some  $NH_3$ , the mixt. of gases is scrubbed while still hot in counterflow with cold water to absorb the  $NH_3$ , while escape of  $CO$  and  $CO_2$  is permitted through the scrubbing water, the scrubbing water is heated to expel the  $NH_3$ , and the  $NH_3$  is reabsorbed in the ammoniacal cuprous soln, gases and vapors from the heated scrubbing water which are not absorbed by the ammoniacal cuprous soln. being returned to the gases and vapors undergoing scrubbing with water App is described

Alginate Arthur H Gruart Fr 789,302, Oct 31, 1935. Marine algae are practically completely demineralized by luvitation with an acid such as  $H_2SO_4$ , and the product is converted to a crude alkali alginate, from which cellulosic material is sepd Alginic acid is pptd by treatment with a dil. acid and sepd. and is then converted to the desired alginate The alginates are preserved against fermentation by the addn. of  $CH_3O$ .

Heat recovery from steam and vapors from paper-mill digesters, etc. Dion K. Dean (to Foster Wheeler Corp.) U. S. 2,029,360, Feb 4. App is described for producing low-pressure steam from the recovered heat

Thermal insulator. Edward R Powell (to Johns-Manville Corp.). Can 354,494, Dec. 24, 1935. A suspension of mineral fibers is mixed in a gaseous atm with finely divided binding material, such as linseed oil, Na silicate and asphaltic fluxes and deposited in successive

increments on a moving support. A current of air is passed through the binder to harden it.

Thermal insulator. M. I. Pekshibaev and A. N. Atlashkin. Russ. 35,991, April 30, 1934. Wood pulp in water is mixed with powd brown clay, poured onto a gauze and pressed The product is dried at 80-100°.

Heat insulation Franz W Seving, Abel Bergqvist and Karl F. Olsson (to Kooperativa Forbundet Forening u. p. a.) U. S. 2,029,679, Feb 4 See Brit. 432,019 (C. A. 30, 538<sup>9</sup>)

Heat-insulating material suitable for partitions, refrigerators, etc Nathaniel M Elias U. S. 2,029,311, Feb 4 A mass of cellular insulating material such as one contg "mineral wool," etc., having a sp gr. less than 1.0 is associated with metal such as Al or an Al alloy serving as a binder and forming a coating or lining to give the product a finished surface

Dielectric materials The Telegraph Condenser Co Ltd and Francis C Stephan Brit 435,323, Sept. 1<sup>9</sup>, 1935 A material of high dielec const comprises a mixt. of  $PhNO_2$ , a wax or waxes and an ingredient comprising 1 of the following chlorinated rubber, the mixed halogenated compds described in Brit 410,008 (C. A. 28, 6218<sup>9</sup>), polymerized styrene or vinyl halides and esters, cellulose esters or ethers A preferred compn comprises  $PhNO_2$ , 70, carnauba wax 14 and chlorinated rubber 5 parts Paper for elec condensers may be impregnated with the compn

Dielectric materials and impregnating agents for electric insulators I G Farbemnd A-G Fr 789,691, Nov. 4, 1935 Nonliquid chlorinated phenanthrenes are used

Insulated electric conductor Rudolph A Schatzel (to General Cable Corp.) U. S. 2,029,540, Feb 4 A conductor such as a metal wire is wrapped with a plurality of layers of helically wound paper tape the adjacent layers of which are sepd by thin layers of viscous sealing material such as a rubber-oil-rosin mixt and a continuous relatively thick layer of non-hardening, oxidized, moisture-resistant, plastic compn such as stearin pitch and asbestos and an outer braid covering are placed over the paper layers. Cf. C. A. 29, 5950<sup>7</sup>.

Fire-resistant covered electrical-conducting wire. Myron E. Delaney (to Halowax Corp.). U. S. 2,028,716, Jan. 21. Covering material on a wire such as a fabric insulation is impregnated with a halogenated cyclic compd, such as tri- and tetra-chloro-naphthalenes together with a plasticizer such as chlorinated diphenyl and an inert pore-filling substance such as fuller's earth or finely divided asbestos.

Oil-insulated electrical transformers, etc. Frank M. Clark (to General Elec. Co.). U. S. 2,028,589, Jan. 21. A mineral insulating oil in a casing such as that of a transformer is protected from undue deterioration by restricting passage of light to it to wave lengths in excess of 600 millimicrons, as by use of an amber or red glass

Molding electrical insulating materials from plastic masses and fibrous fillers. A. I. Goldshtein Russ 37,752, July 31, 1935 Mechanical features

Apparatus for storing gases under pressure or vacuum for heat-insulation purposes. Carl G. Munters. Brit 435,997, Sept. 30, 1935. Addn to 415,457 (C. A. 29, 856<sup>9</sup>).

Apparatus for making sheet insulating material from "rock wool," etc Oliver F. Mottweiler. U. S. 2,029,081, Jan. 23 Structural, mech and operative details

Electric cable. Thomas R. Scott and Thomas E. D. Menzies (to International Standard Elec Corp.) U. S. 2,029,088, Jan. 28. An elec power cable has a fluid impregnated dielectric such as oil-impregnated material the changes in volume of which, with changing temps, are compensated by placing in the cable a device such as a bimetallic strip of Al and brass having a negative temp coeff. of cubical expansion

Rubber-insulated cables held in a casing containing water under pressure Robert J Wiseman (to Okonite Co.) U. S. 2,028,178, Jan. 21. Various structural de-

tails are described of a cable adapted for use with high-tension currents

**Insulating cables** Gabriel Fodor Brit 435,060, Oct 2, 1935 Cables having MgO insulation are produced by applying to the single or stranded conductor Mg in any convenient form, e.g., tube, tape, surrounding the assembly with a covering or braiding of textile or other fibrous material and converting the Mg into MgO in any suitable known manner, e.g., in an autoclave by treatment with superheated steam at not over 400°

**Insulating cables**, etc. I. G. Farbenindustrie A.-G. Brit 435,557, Sept 23, 1935 Wires or other longitudinally extended elec. conductors are insulated by winding with sheets or bands of polyvinyl chloride, which may have been further chlorinated, the sheets or bands being free from residues of solvent, and subjecting the wound conductors to heat treatment sufficient to cause fusing of the overlapping edges of the sheets or bands

**Insulating cables** Pirelli-General Cable Works Ltd. and Robert F. Horley Brit 435,451, Sept 18, 1935. The metallic conductors of elec. cables are provided with waterproof paper insulation consisting of paper tapes that have been previously dried and then vacuum impregnated with a flexible waterproof insulating compd. of such high m.p. as not to melt at the working temp. of the cable. The compd. should not melt below 60° and may consist of bitumen, wax, petroleum jelly or a mixt. thereof, with or without oil and (or) resin, e.g., medium hard bitumen 80, paraffin 20 or hard petroleum jelly 75 and gum rosin 25%

**Adhesive** K. P. Sammel Russ 39,276, Aug 31, 1934 A mixt. of fine sawdust, gelatin, cellulose and  $\text{Ca}(\text{ClO})_2$  is heated

**Adhesive suitable for attaching masks to surfaces to be lacquered** Ralph A. Wilson (one-half to George A. Chrillon) U.S. 2,029,008, Jan 28, 1935 Rubber cement 30, latex 5, a heavy mineral oil such as a lubricating oil 5 and a volatile solvent such as gasoline 55% are used together with a deodorant such as Me salicylate 5%

**Use of thermoplastic adhesives in shoe manufacture** Charles F. Pym (to United Shoe Machinery Corp.) U.S. 2,028,245, Jan 21, 1935 Various operative details are described relating to the use of a thermoplastic adhesive for fastening uppers to shoe soles. App. is described

**Linoleum cement** Imperial Chemical Industries Ltd. and Edwin B. Robinson Brit 435,992, Sept 27, 1935 Drying oils treated as in Brit 422,941 (C.A. 29, 4610\*) or fish oils treated similarly are subjected to an air-oxidation process with addn. of driers and the treated oil is fused with resins, gums or other usual addns. In examples, linseed or Japanese sardine oil bodied by heating in  $\text{CO}_2$ , or linseed oil bodied by blowing air through the heated oil, is passed through a continuous still as in 422,941 and then mixed with PbO. The oil is oxidized by flowing it over an inclined tray in an oven heated to 40–45°, through which a stream of air is passed. The oxidized oil is fused with rosin and kauri gum to give a cement which may be mixed with wood meal and pigment to give a linoleum mixt.

**Cements**, etc. I. G. Farbenindustrie A.-G. Brit 435,867, Oct 18, 1935 In the process of Fr. 723,008 (C.A. 26, 4142), the neutral metal oxides, sulfochlorides or neutral salts, are replaced by esters having a neutral action toward water and formed by the combination of aromatic-aliphatic alcs. and mineral acids, e.g., phenylchloroform, benzylidene chloride, *p*-xylylene chloride, dichloromethyl *m*-xylene or dibenzyl sulfate. Emollients, such as benzyl alc., as well as various fillers may also be added

**Cementing surfaces** I. G. Farbenindustrie A.-G. Brit 435,941, Sept 12, 1935 Adhesion of 2 surfaces is effected by applying separately to the surfaces or to 1 of them an aldehyde-urea condensation product and a substance exerting a hardening influence thereon, e.g.,  $\text{HCl}$ ,  $\text{AlCl}_3$ ,  $\text{H}_2\text{PO}_4$ ,  $\text{NaHSO}_4$ ,  $\text{NH}_4\text{H}_2\text{PO}_4$ ,  $\text{AlCl}_3$  and  $\text{NH}_4\text{Cl}$ . Starch, potato flour, fillers, etc., may be incorporated in the adhesives. The cementing is completed by pressure with or without heating

**Phenol-aldehyde condensation products** Howroyd,

McArthur and Co. Ltd. and Robin H. Croad Brit 436,308, Oct 9, 1935 Addn. to 411,828 (C.A. 28, 7044\*) Oil sol. resins are prep'd by condensing 2 mol proportions of a phenolic substance with 1 of  $\text{CH}_2\text{O}$  in the presence of  $(\text{AcO})_2\text{Zn}$ , basic Al acetate or  $(\text{AcO})_2\text{Mg}$  as catalysts and then heating the product with an oil. The acetates may be formed *in situ*. Among examples, cresylic acid or xylenol is heated with Al and the product condensed with  $\text{CH}_2\text{O}$  and  $\text{AcOH}$ , with or without the addn. of  $(\text{AcO})_2\text{Zn}$ , and then heated with castor oil

**Condensing phenols with wood fiber** S. N. Ushakov Russ. 37,782, July 31, 1931 The condensation is effected by first dissolving a phenolic aldehyde resin of a "Novolok" type in phenol, or by adding it in the condensation reaction with wood fiber. Cf. C.A. 29, 2257\*

**Condensation products of thiourea, urea and formaldehyde suitable for making molded articles** Kurt Ripper U.S. 2,029,836, Feb 4, 1936 Urea, thiourea and  $\text{CH}_2\text{O}$  in the mol proportions of 1, 1 and 1.5, resp., are reacted together in an acid soln. of a  $pH$  of 3.0 by heating the mixt. until there is effected the formation of a hydrophobic mixed condensation product which seps from a sample of the reaction mixt. on cooling

**Reaction product of thiourea with formaldehyde suitable for injection-molding or die-casting** Kurt Ripper U.S. 2,029,891, Feb 4, 1936 Thiourea is reacted with less than 2 mol proportions of formaldehyde without the addition of external heat, in an aq. medium having a  $pH$  of about 3 to 5 to prevent formation of insol. thiourea methylene compds., the resulting liquid product is mixed with a fibrous material such as paper strips and the mixt. is dried to a point where it is practically dry but still contains enough moisture to be flowable under pressure

**Hydrophobic urea thiourea formaldehyde reaction products suitable for use in molding compositions**, etc. Kurt Ripper U.S. 2,029,893, Feb 4, 1936 A mixt. of urea with thiourea is reacted with less than 2 mol proportions of formaldehyde without addition of external heat, in an aq. medium having a  $pH$  of about 3, the amount of thiourea used being sufficient to prevent formation of white insol. formaldehyde urea products. The resulting product may be used in coating, impregnating or molding compns.

**Polymerization products suitable for molded articles, coatings**, etc. Wallace H. Carothers, Arnold M. Collins and James E. Kirby (to E. I. du Pont de Nemours & Co.) U.S. 2,029,410, Feb 4, 1936 Chloro 2-butadiene-1,3 is polymerized in the presence of a film forming material which may include natural or synthetic resins, cellulose derivatives or polymerizable materials having at least 2 C atoms in an open-chain joined by more than one bond such as China wood oil. Numerous examples are given

**Synthetic resins** Ya. I. Chulkov Russ. 38,310, Aug 31, 1934 Phenols, thiophenols, their homologs or waste products obtained in the carbonization of coal sapropel, peat or shale are treated with solns. of Na or K sulfites or  $\text{NaOH}$ ,  $\text{KOH}$  and  $\text{Ca}(\text{OH})_2$ , then with excess of  $\text{Cl}_2$  and finally are filtered, dried and worked up in the usual manner

**Synthetic resins** Theodor Sutter (to Soc. pour l'ind. chim. & Bâle) U.S. 2,029,954, Feb 4, 1936 An aldehydic substance such as furfural or formaldehyde is reacted (suitably by heating together) with a polynuclear aromatic amine base the nuclei of which are linked together by less than 2 mols of methylene groups for each 2 mols of aromatic radical and which contains at least as many amine groups as linked nuclei, such as aminobenzylaniline to produce a product for making molded articles, etc. Numerous examples are given

**Synthetic resins** Beckacite Kunstharzfabrik G.m.b.H. Ir. 789,643, Nov 4, 1935 Resinous plastifiers are made from aliphatic acids of short chain not containing more than 4 C atoms between the  $\text{COOH}$  groups, e.g., succinic and maleic acids, polyatomic alcs. such as glycerols and oils having an I index below 100°, e.g., nondrying oils, the amt. of oil in the formula being 50–85% by wt. Thus, castor oil is heated with glycerol to 233° in the presence of a small amt. of litharge, maleic acid and more glycerol are added, and the mixt. is heated to

170-190° until a liquid resin is obtained. Other examples are given.

**Urea resins.** *Bakelite Corp.* Fr. 789,533, Oct. 30, 1935. Urea is condensed with an aldehyde (1.5-2.5 mol of aldehyde per mol of urea) in the presence of 1-5 g. for each mol of urea of a compd. contg. N chosen from the group comprising hydroxylated amines, monosubstituted amines and diamines and aromatic amines. The reaction is stopped when re-sulfonation is complete by adding a neutralizing agent forming an inactive addn. product with the compd. contg. N. Suitable N compounds are  $\text{MeNH}_2$ ,  $\text{AmNH}_2$ ,  $\text{C}_2\text{H}_5(\text{NH})_2$ ,  $\text{NH}_2\text{OH}$ ,  $\text{PhNH}_2$ , and phenylhydrazine.

**Vinyl resins.** *Rohm & Haas A-G* Fr. 789,166, Oct. 21, 1935. Stable dispersions capable of being highly concd. of vinyl resins insol. in water are prep'd by introducing hydrophobic groups ( $\text{COONa}$ ,  $\text{COOK}$  and  $\text{COONH}_4$ ) into the polymer mol. Thus acrylate ester is polymerized in an emulsion in the presence of Na acrylate to form a polyacrylic ester which contains a  $\text{COONa}$  group. Examples are given.

**Resins for varnishes and plastic masses.** *B. V. Makarov and P. A. Kremlyuk* Russ. 37,785, July 11, 1934. The chitin-contg. material obtained by alkali treatment of crab shells is treated, directly or after the action of lime, with benzyl chloride in the presence of alkali.

**Varnishes, agglutinants, etc.** *Isoline Tenger* (to Johann Tenger) Brit. 435,270, Sept. 16, 1935. See Fr. 770,201 (C. A. 29, 5309).

**Insoluble and insoluble resins and varnishes.** *G. S. Petrov* Russ. 37,815, July 11, 1934. High molecular sulfonic acids obtained in sulfonating mineral oils or distillates from shale, peat, etc., are condensed with aldehydes in the presence of aromatic amines in an acid, neutral or alk. medium.

**Artificial resins from polyhydric alcohols and polybasic acid anhydrides.** *Gilbert F. Hoffmann* (to Pittsburgh Plate Glass Co.) U. S. 2,028,908, Jan. 28. A hard sol. resin suitable for use in lacquers, etc., is produced by heating together a polyhydric alc. such as glycerol and an anhydride of a dicarboxylic acid such as phthalic anhydride to form a resinous product capable of further esterification, incorporating an anhydride of a "monobasic" aliphatic acid such as  $\text{Ac}_2\text{O}$  and heating the mixt.

**Plastic masses.** *B. V. Mikorov* Russ. 37,847, July 11, 1934. Sawdust, corn stalks, etc. are heated above 250° in a current of inert gas or superheated steam before being mixed with synthetic resin.

**Plastic masses from lignin.** *A. M. Nastukov, M. P. Polyakov and Kh. G. Finkov* Russ. 38,322, Aug. 31, 1934. Lignin obtained by incomplete hydrolysis of lignocellulose with acids is mixed with 6-10% of plasticizer, such as latex or benzylalcohol.

**Plastic resins from petroleum residuums.** *Urie B. Bray* (to Union Oil Co. of Calif.) U. S. 2,021,288, Feb. 4. A cracked residuum is sep'd into fractions by means of a solvent having a low solvent power for the asphaltic materials present in the residuum, asphaltic materials are sep'd. from the soln., and the dissolved cracked materials are ext'd. with a selective solvent for the more aromatic substances, the soln. of these latter is sep'd. and dist'd. to produce as a distillate a plastic resin of sp. gr. about 1.084-1.126, m. approx. 35-65° and of ductility in excess of 100 cm. at a temp. of 25°. Various details and modifications of procedure are described.

**Synthetic resin-like products.** *W. A. Noves and Gilbert F. Hoffmann* (to Pittsburgh Plate Glass Co.) U. S. 2,028,914, Jan. 28. An ester, capable of further esterification and formed from a polyhydric alc. such as glycerol, and an aliphatic "monobasic" acid, such as monoacetic, is further reacted with an anhydride of an org. "dicarboxylic" acid such as phthalic anhydride.

**Dispersion of resins.** *John A. Kenney* (to Barrett Co.) Can. 354,904, Dec. 24, 1935. An unsaponifiable resin of the paracoumarone type is dispersed by mixing in solid comminuted form with a soln. contg. Na silicate that has a ratio of at least 3 parts by wt. of  $\text{SiO}_2$  to 1 part of Na-O. The mixt. is then agitated.

**Dispersion of resins.** *John A. Kenney* (to Barrett Co.) Can. 354,905, Dec. 24, 1935. An unsaponifiable resin, such as paracoumarone or paraindene, is dispersed, e. g., by dissolving 50 parts of paracoumarone resin, m. 110°, in 50 parts by wt. toluene, whipping into 200 parts by wt. of  $\text{H}_2\text{O}$  contg. 1 part by wt. of oleic acid and 5 parts by wt. of Na silicate soln. having a sp. gr. of about 42.5°. Baumé. This mixt. may be carried out at atm. pressure. The dispersion is not inflammable. Other solvents may be used, such as  $\text{CCl}_4$ , petroleum derivs., liquid org. esters and ethers or terpene derivs. The product is used for sizing of papers.

**Composite hollow articles.** *Lauchlin M. Currie* (to Canadian National Carbon Co. Ltd.) Can. 354,915, Dec. 24, 1935. This relates to the manuf. of articles composed wholly or in part of vinyl resins. 1 g. vinyl acetate and vinyl chloride are conjointly polymerized in the proportions of 80% by wt. of vinyl chloride, in the presence of acetone and 1% by wt. of  $\text{Br}_2\text{O}_5$  as a catalyst at 40°. After removal of unpolymerized material and catalytic residues, this is compounded with 3% by wt. of carnauba wax and about 2% by wt. of alk. Ca stearate. This is made into a preform and placed within a matrix, and subjected to fluid pressure applied internally until it assumes the matrix form, and then subjected to heat to make it heat stable.

**Solvent esters.** *Carbide & Carbon Chemicals Corp.* Fr. 788,550, Oct. 18, 1935. 1-esters formed by the reaction of aromatic monocarboxylic acid such as salicylic acid and a monoalkyl ether of glycol are used as solvents and plasticizers for cellulose derivs. and natural and synthetic gums and resins and for increasing the flexibility of films. Examples are given of the prep'n of *butyryl-ethyl-139-437* and *butyryl-ethyl-139-437*, by 175°.

**Use of glycerol dipropionate and dipropionate-monoacetate as plasticizers for cellulose esters such as cellulose acetate.** *David C. Hull* (to Eastman Kodak Co.) U. S. 2,021,925, Feb. 4.

**Putty.** *Yu. S. Bakst* Russ. 37,781, July 31, 1934. A putty is prep'd from oil, lime and the residue left after treating glycerol with  $\text{H}_2\text{SO}_4$ .

**Moistureproofed molded article.** *Robert G. Lemmerman* (to Ferro Engineering Co.) Can. 355,194, Jan. 7, 1936. An aggregate, such as fine silica sand, is mixed with 5-6% of a sol. silicate. E. g., in a 1000 lb. batch the wts. may be aggregate 849, water glass 120,  $\text{H}_2\text{O}$  25 and  $\text{Al}_2(\text{SO}_4)_3$  1 lb. This is molded and dried at 212° in an atm. free from  $\text{CO}$  and  $\text{CO}_2$ .

**Transparent articles.** *L. Bronzi* Belg. 407,197, Feb. 28, 1935. A transparent mass obtained by urea-formaldehyde polymerization is dried in an autoclave and is hardened superficially by treating with a dehydrating agent such as dry  $\text{CH}_3\text{O}$  gas.

**Molded products.** *Irancous Cédre*. Fr. 788,704, Oct. 16, 1935. To a mixt. of sand-tine agglomerated by a binder such as glue or gelatin, is added  $\text{MgO}$  to prevent the molded product becoming brittle on drying, and lactic acid to prevent putrefaction and too rapid drying. The mold is smeared with a mixt. of stearin and lighting oil.

**Press for molding plastic materials such as synthetic resin mixtures.** *Rudolph J. Kauls* (to General I. Lee, Co. Ltd.) U. S. 2,021,026, Jan. 28. Various structural, mech. and operative details.

**Laminated sheets.** *John A. Kenney* (to Barrett Co.) Can. 354,906, Dec. 24, 1935. A dispersion of solid comminuted resin of the coumarone-indene type, m. above 100°, in an alc. soln. of Na silicate is mixed with fibrous pulp in proportions of about 25% resin to the dry wt. of the fiber. The resin is ppt'd. on the fiber by adding sufficient alum to the pulp to produce a pH value of about 4.6. The pulp is formed into a sheet, dried and pressed at a temp. above 150° with the sheets to form a laminated product.

**Impregnated sheet material.** *E. I. du Pont de Nemours & Co.* Brit. 435,728, Sept. 26, 1935. A pulp of cellulose fibers is treated with a mercerizing soln. to cause only superficial mercerization of the fibers and then formed



into an absorbent felt which is impregnated with a bituminous material or a mixt thereof and drying oil. A finishing coat, e. g., pigmented rubber, pyroxylin, which may be preceded by a suitable sizing coat, may be applied.

**Compound sheet materials** Edward W. Aldridge Brit 435,396, Sept 16, 1935 Laminated, decorative, glass-surfaced sheets consist of a sheet of glass decorated by paints, enamels, etc., on its interior surface united to a rear sheet of glass, decorated or not, or of asbestos cement by means of an adhesive consisting of coned latex mixed with powd.,  $H_2O$ -reactive substances, e. g., anhyd.  $CaSO_4$ , Portland cement, and, if desired, vulcanizing agents, gums or resins, bitumen, pitch, etc. An intermediate non brittle layer, e. g., cardboard, cloth, may be provided.

**Compound sheet materials** Diedrich Haack and Richard Schmidt Brit 435,088, Sept 13, 1935 A gas-proof and fireproof material is made by uniting a layer of rubber or rubber mix with an outer layer consisting essentially of Kieselguhr. The kieselguhr may be treated with Na silicate and degreasing chemicals, e. g.,  $H_2SO_4$ ,  $NH_4OH$ ,  $KOH$ , to form a flaky mass which may be rolled onto the rubber or spun and made into a fabric which is vulcanized to the rubber. This fabric may incorporate another material, e. g., cotton, and may be impregnated with, e. g.,  $Al_2(SO_4)_3$ , to render it fireproof and non-hygroscopic.

**Paper metal sheets** Aluminiumwerk Tscheln G m b H and Carl Craemer Fr 788,987, Oct 21, 1935 Metal sheets and paper are united by a mixt of  $\alpha$  adhesives and  $\alpha$  dispersions of polymerized compds., particularly polyvinyl esters. Examples contain latex, casein and polyvinyl esters, or gum arabic, glycerol and acrylic acid esters.

**Decorative sheet material suitable for floor coverings** James J. Jackson (to Sandura Co.) U S 2,028,781, Jan 28 A liquid backing compn. such as an oil or lacquer compn. is applied to one side of a porous sheet material such as a felt in a regulated amount to effect penetration into the body of the sheet while avoiding filling the spaces between the fibers within the body of the sheet, and thereafter decorative coloring material is applied to the opposite side of the porous sheet to effect penetration of the coloring material well down into the body of the sheet. U S 2,028,782 relates to impregnation of porous fibrous sheet material with a molten saturant formed of ester gum, China wood oil and chlorinated naphthalene.

**Castling belts for various transparent sheet materials** I douard M. Kratz and Erich Gebauer-Luelnegg (to Marbo Products Corp.) U S 2,028,936, Jan 28 A laminated fabric web carries a pigmented layer of a cellulose ester compn. on which are superposed a plurality of layers of soft plasticized clear nitrocellulose compn. to form a smooth, brilliant top surface, and an inert barrier film is used on the surface to protect the under coatings from the action of solvents in the film forming soln. to be cast on the belt. Various examples are given.

**Uniting layers of material such as jar cap liners by use of an adhesive** John T. Heckel and Ralph S. Walker (to Gold Dust Corp.) U S 2,029,422, Feb 4 Various structural, mech. and operative details.

**Glued plywood and veneered products** W. F. Leicester U S 2,023,287, Jan 21 Various operative details of assembly, glue treatment, drying and hot pressing.

**Laminated sound insulated panels suitable for dash panels of automobiles, etc.** Meredith S. Randall (to Woodall Industries Inc.) U S 2,028,950, Jan 28 Various structural details of composite panels which may be formed of laminations such as metal and asphalt impregnated fibrous material.

**Laminated sound insulated panels suitable for dash panels of automobiles, etc.** Herbert J. Woodall and Meredith S. Randall (to Woodall Industries Inc.) U S 2,028,962, Jan 28 Structural details of panels which may be formed of laminations of sheet metal, "Masonite," etc. Cf. C A 29, 4105 and preceding abstr.

**Wetting agents, etc.** Henkel & Cie G m b H Fr

1 789,578, Oct 31, 1935 Org. compds. contg. at least 1 lipophile group and at least 1 group causing solv. in water (the lipophile group being joined to the group causing solv. in water by means of a chain of C atoms interrupted by at least 2 groups of amides of acids), are used as agents having capillary activity, alone or with other cleaning, etc., agents. Examples are given of the use of  $\beta$ -((3-lauroyl-lammonobenzene)sulfonylmethylamino)ethanesulfonic acid and  $\beta$ -((3-oleylammonobenzoyl)methylamino)ethanesulfonic acid.

**Detergents** Henkel & Cie G m b H Brit 436,213, Sept 27, 1935 These are made by mixing  $H_2O$ -sol. pyrophosphates with water glass or other non-oxidizing alk.,  $H_2O$ -sol. inorg. alk. metal salts, other than alkali carbonates, known as cleansing agents. Abrasives may be added.

**Detergents** Julius P. Hansel Brit 436,332, Oct 9, 1935 These are composed of 1 mol.  $Na_2PO_4$  to 1.5 mols. of alkali metal salts of inorg. or low aliphatic acids less strongly dissolved in  $H_2O$  than is  $H_2PO_4$ , e. g., acetates, borates, carbonates, molybdates, silicates, arsenates.

**Detergents** Denis Lacy-Hulbert Brit. 436,866, Oct 16, 1935 These consist of soap and sulfonated lauryl and oleyl alcs. or salts or esters thereof in such proportion that the soap is more than 25% of the whole.

**Sulfonated derivatives of carboxylic esters.** Benjamin R. Harris U S 2,029,168, Jan 28 Materials such as oleostearin, lard, partially hydrogenated cottonseed or peanut oil, cocoa butter or coconut oil, contg. carbon-to-carbon double bonds, are sulfated (suitably with concd.  $H_2SO_4$  under suitably controlled conditions) to produce normally solid products which may be used as wetting, penetrating, detergent, foaming or lubricating agents, etc., in the textile, paper, pharmaceutical, soap, cosmetic, dyeing, food or other industries.

**Nonadecyl oxygen compounds** Carbide & Carbon Chemicals Corp. Fr 789,406, Oct 29, 1935 A compd. of the formula  $BuCH_2CH(CHCOCH_2CH_2(OH)CH_2CH_2Bu$ , is prep'd by condensing 5-ethyl-3-nonen-2-one and ethylhexaldehyde. This compd. easily loses its elements of water to form an unsat'd nonadecyl ketone, by 169-73°, of the probable formula  $BuCH_2CH(CHCOCH_2CH_2CHCH_2Bu$ , which is hydrogenated to a bis(ethylheptyl)-ketone of the probable formula  $(BuCH_2CH_2CH_2CH_2)_2CO$ , and a secondary nonadecyl alc., by 175-7°, indicated as 5,11-diethyl-8-pentadecanol. The alc. and its sulfate ester have valuable wetting, emulsifying and frothing properties.

**Quaternary ammonium salts** Soc. pour l'ind. chim. à Bale Fr 788,898, Oct 18, 1935 Quaternary  $NH_4$  salts are prep'd by causing compds. of the formula  $(X)_nRCOR'$  (X is active halogen, n is a whole no. below 7, R is an aliphatic, cycloaliphatic or cycloaliphatic aliphatic radical having at least 8 C atoms, R' is OH, OR', or  $NH_4R''$ , R'' being any org. radical having less than 6 C atoms, R' and R'' are H or any org. radicals) to react with tertiary amines which can only react with one of the mobile halogen atoms. The products are wetting, cleansing and emulsifying agents. Thus, Et  $\alpha$ -bromostearate,  $\alpha$ -bromolaurate and  $\alpha$ -bromopalmitate and the anilides of the acids are converted to the quaternary pyridinium salts.

**Sulfonic acids** Richard Huttenlocher and Richard Hess U S 2,029,073, Jan 23 In the production of a sulfonic acid such as an emulsifying and wetting agent, a compd. contg. an alkyl-disulfonic acid radical such as methane-disulfonic acid is reacted with an org. compd. contg. an alcoholic hydroxyl group such as hexadecanol. Various examples are given.

**Sulfonic acid soap** Otto Diepenbruck (to Shell Development Co.) Can 354,565, Dec 3, 1935 Alkali metal sulfonates obtained from mineral oils which ordinarily form turbid mixts. with hot  $H_2O$  are made to produce clear solns. by addn. of 5-25% of a highly refined cylinder oil or spindle oil.

**Sulfonated oil** Philip Kaplan (to Richards Chemical Works Ltd.) Can 354,961, Dec 21, 1935 In sulfonation of oils a low temp. is maintained by immersing sol

CO<sub>2</sub> in the mist. The oil is then washed, the H<sub>2</sub>O sep'd., and the product neutralized at not higher than about 15°.

**Disk for sound recording.** V. A. Zaikin, V. A. Trovostolov and L. P. Abramovich. Russ. 37,979, July 31, 1934. A wax disk is cooled to about 4° and sprayed with a dil. soln. of NH<sub>4</sub>OH of the same temp. to neutralize the acid reaction of the wax and to remove air from the sound path. Ag is then ppt'd on the disk in the usual manner.

**Sound records.** Samuel Whyte (to Electric and Musical Industries Ltd.) U. S. 2,028,854, Jan. 28. A sound-record material comprises a phenol condensation product having incorporated with it a small proportion of a soft water-sol soap. Cf. C. A. 29, 3750.

**Gramophone records.** Guy Hillhouse (to Trusloud Ltd.) U. S. 2,029,102, Jan. 28. Superposed thin flexible transparent disks of celluloid are placed together with an intermediate co-extensive sheet of fibrous material such as paper in a heated press in which they are pressed together to cause the surfaces of the celluloid disks in contact with the paper to be extruded into the pores of the paper to effect a permanent union of the materials.

**Phonograph records.** Hal T. Beans, George H. Walden, Jr., and Louis P. Hammett. U. S. 2,029,012, Jan. 28. A record material is prep'd by heating a mixt. of resorcinol, alc., and *p*-nitroaniline at 70° to form a soln., adding 40% formaldehyde and further heating at about 70° until the ppt. formed is dissolved and a viscous liquid is formed, adding alc., cooling the soln. to 30° and adding Turkey-red oil.

**Sound and like records.** N. V. Philips' Gloeilampenfabriek. Brit. 435,515, Sept. 24, 1935. In mixing materials on which records of sound, etc., may be made mechanically, particularly gelatin layers on which records adapted to optical reproduction may be formed, a layer on which a clearly defined, "unfrosted" track may be formed is provided by making it of gelatin produced from a soln. of *pu* less than 4 or more than 7. Thus a purified isoelec. gelatin is soaked in H<sub>2</sub>O and dissolved at 45°, the soln. is shaken with neutral Turkey-red oil, e. g., "Turkolon A," and, after being filtered, if necessary, N. HCl is added, the soln. being then poured as a thin layer onto a carrier, e. g., celluloid film, and the layer allowed to dry. Cf. C. A. 30, 977.

**Fire extinguishers.** I. G. Farbenind. A.-G. Ir. 789,327, Oct. 29, 1935. Water-sol. salts of aliphatic amines contg. an aliphatic chain of more than 10 C atoms attached to N are useful for making froth with air. Examples are dodecylmethylamine formate, benzylmethyl-(or diethyl)dodecylammonium chloride, dodecylmethylbetaine or the corresponding phenolbetaines and salts of N'-dodecylmethylthylenediamine. Cf. C. A. 29, 6336.

**Fire-extinguishing liquid.** Albert C. Malzac. Ir. 789,402, Oct. 29, 1935. The liquid contains 2 or more halogenated *or* derivatives, 1 being, e. g., MeBr and the other CCl<sub>4</sub> or trichloroethylene, alone with constituents having a vapor tension sufficiently low to permit manipulation in air at ordinary temp., and a stabilizer, e. g., PhNH<sub>2</sub>, to prevent formation of COCl<sub>2</sub> or HCl. The constituents may be emulsified in water or fireproofing salts. Cf. C. A. 29, 6941.

**Use of foaming agents and water in fire extinguishers.** Bror Falsson. U. S. 2,029,330, Feb. 4. Various details of app. and operation.

**Fire extinguisher.** J. Breny and P. Delvaux. Belg. 407,668, March 30, 1935. A pulverized alkali metal salt product is mixed with refrigerating products.

**Foam fire extinguisher.** V. N. Shymrin. Russ. 32,302, Sept. 30, 1933. Construction details. Addn. Russ. 34,320, Jan. 31, 1934.

**Leather substitute.** Albert J. Hanley (to Respro Inc.) U. S. 2,029,172, Jan. 28. A rubber compn. is applied to a textile fabric base having attached loops of thread projecting from it in an uncut state, the rubber compn. is dried and vulcanized, and during the process the uncut loops of thread are pressed into flattened condition in the applied compn. substantially parallel to the general plane of the fabric to provide a smooth fibrous surface.

**Coated fabrics.** Johnson & Johnson. Brit. 435,782,

Sep. 27, 1935. See Can. 353,366 (C. A. 29, 83614). Normally tacky adhesive tapes having a fabric or paper backing are provided on the non-adhesive side with a thin film of rubber and, superimposed thereon, a coating of a cellulose deriv. applied in a solvent medium comprising a solvent for rubber. In an example, the final coating consists of a soln. of 1% cellulose in a mixt. of PhMe and MeOH or AcOPt.

**Impermeabilizing surfaces** such as those of cellulose or its derivatives. Harold Hibbert (to Celanese Corp. of America) U. S. 2,024,770, Jan. 24. The surfaces are exposed to vapors of a substantially unpolymerized vinyl compd. such as divinyl ether or of a glycidic and the polymerizable material deposited on the surface is polymerized to form a surface coating.

**Moistureproof material.** John H. Collins (to du Pont Cellophane Co. Inc.) Can. 354,404, Nov. 26, 1935. A sheet of regenerated cellulose is coated with a compn. comprising, e. g., 5 parts of a film-forming substance, 5 parts of a moistureproofing wax such as high-melting paraffin, and 5 parts carnauba wax dissolved in a solvent. The film-forming substance is selected from natural rubber, gutta-percha, balata and synthetic rubbers. The solvent is evaporated at a temp. at least equal to the m. p. of the wax in the compn. Cf. C. A. 29, 8163.

**Impregnating fibrous materials.** United Shoe Machinery Co. de France. Fr. 789,294, Oct. 25, 1935. Bands of fibrous material, such as parts of shoes, are stiffened by dissolving a cellulose acetate having an acetyl index between 48 and 54.5, preferably 50-4, in a hot liquid (e. g., EtOH contg. 25-55% of water) which is capable of dissolving the acetate in the hot and pptg. it in the cold, impregnating the fibers with the hot soln. and cooling.

**Bituminous aqueous emulsions for impregnating roofing paper, artificial leather, paper tubes, preparing varnishes, etc.** G. D. Kreitzer. Russ. 18,109, Aug. 31, 1934. Emulsions are prep'd from molten, or finely disintegrated or dissolved bitumens or pitches or their mixts. with emulsifiers by using for stabilizer mineral oils of high viscosity or petroleum derivs. with the addn. of electrolytes such as CaCl<sub>2</sub>.

**Applying lining material** such as asphalt and wax composition to the inside of cans by spraying. John H. Murch (to American Can Co.). U. S. 2,028,798, Jan. 24. Various operative details for coating the inside of cans for holding beer, etc.

**Apparatus for lining internal walls** of, or molding, hollow bodies such as a tube, with thermoplastic material such as bitumen. Vereinigte Stahlwerke A.-G. Brit. 435,000, Sept. 12, 1935.

**Protecting pipe lines, etc.** Stuart P. Miller and Leslie T. Sutherland (to The Barrett Co.). Brit. 435,325,

Sep. 19, 1935. Objects such as pipe lines are protected by placing about them a flexible shield comprising fibrous material, e. g., felt, paper, wood veneer, impregnated with a synthetic resin that is cured before the shield is applied. Unimpregnated reinforcing material may be treated with preservatives, e. g., creosote. Pipes may be coated with bituminous enamels before application of the shield, which may be secured by adhesives, e. g., asphalt, tar, plastic cement.

**Films, etc.** Celluloid Corp. Brit. 435,784, Oct. 17, 1935. In producing films or foils by depositing a film-forming soln. on a support, evap. the volatile solvent and stripping the film, etc., from the support, the tendency of the film, etc., to curl is reduced by subjecting to the action of heat applied only to or in greater quantity to the air side, i. e., the side not in contact with the support. Suitable film-forming compns. are given and app. is described.

**Film-forming compositions** containing chlorinated rubber, etc. Leo Rosenthal and Reinhard Hebermehl (to I. G. Farbenind. A.-G.) U. S. 2,029,688, Feb. 4. Chlorinated rubber material is used together with a S-contg. reaction product made from S and toluene, xylene or ethylbenzene in the presence of AlCl<sub>3</sub> for mixing compns. suitable for coating metals and for other uses.

Soybean phosphatides. Wells W. Ginn (to Chemical

Novelties Corp.) U S 2,020,261, Jan 23 A product suitable for various uses similar to many of those of lecithin is produced by hot pressing soybeans to obtain soybean oil, scgp sludge from the expressed oil, blowing the sludge with air to remove odor, filtering it, further scgp oil by settling or centrifuging (suitably at a temp of 55-70°)

Separating albuminous substances from the castor oil palat I M Zolotnitskii Russ 37,785, July 31, 1934 Husks of the castor-oil plant are treated with hot alkalis and the albuminous substances are scpd from the alk soln by treatment with waste liquors of gelatin manuf

Decalcomania James J McNutt (to American Decal Corp.) U S reissue 10,839, Jan 23 A reissue of 1,996,519 (C A 29, 3481)

Transfer paper Louis C Antrim (to Autographic Register Co.) Can 354,229, Nov 19, 1935 A sheet of paper, such as waterleaf, is impregnated with oil and ink soln One side is coated with pyroxlin to make it nontransferable, and the other side coated with Al powder to retard the ink transfer. Cf C A 28, 75341

Fireproofing compositions Grete Kindermann (nee Huesmann) Brit 435,240, Sept 17, 1935, divided on 429,857 A compn for fireproofing fabrics consists of an aq soln of  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{H}_3\text{BO}_3$  and borax, to which is added a small amt. of a urea- $\text{CH}_2\text{O}$  condensation product

Rendering textile materials flame resistant Ernest R Hanson (to Halowax Corp.) U S 2,028,715, Jan 21 Cloth, paper or the like is treated with a material such as halogenated acenaphthene or halogenated phenanthrene which may be plasticized with a chlorinated diphenyl

Etching agent for printing plates P L Kalman Russ 37,721, July 31, 1934 A mixt of Al salts, phosphates, alkali metal salts,  $\text{HPO}_4$ , and  $\text{NH}_4\text{Cl}$  is specified

Printing plates Hylton Swan and Sigfried Higgins (to Bakelite Corp.) U S 2,028,707, Jan 21 Printing plates, the body of which may be formed of rubber, have a facing of regenerated cellulose and may have a metal backing plate, etc U S 2,028,710 relates to plates with a body of rubber and a facing of cellulose acetate 0.001-0.005 in thick U S 2,028,711 relates to plates with a body of resinous material such as a phenolic resin and a thin cellulose acetate facing U S 2,028,712 relates to printing plates with a vinyl resin facing and a body of another material such as a rubber compn

Product for intensifying combustion and improving metallurgical products M H M van den Heuvel Belg 407,586, Feb 29, 1935 The product is a mixt of oxides of Mn (or other metals which have several degrees of oxidation) with salts or bases of alkali or alk earth metals, the ratio being preferably 2.35-2.4 atoms of Mn per atom of alkali or alk earth metal

Chemical heating composition Raymond F Reed (to Kendall Co.) Can 354,263, Nov 26, 1935 A compn for use in heat pads contains an active metal, and electrolyte that forms an acid soln and a compd, insol in water but sol in the soln of the electrolyte, of a metal that is replaceable from soln by the active metal, e g, 10 g oxalic acid, 21 g  $\text{BaCl}_2$ , 25 g cupric carbonate and 600 g Fe filings

Friction material for brake lining or clutch facing Phillip D Cannon (to Johns-Manville Corp.) Brit 435,101, Sept 4, 1935 The material is made by interweaving fibrous warp and weft strands around preformed, self-sustaining, plastic strips of friction compd, binder strands being simultaneously inserted between the strips to connect together the flat faces of the woven tube, applying addnl friction compd to the outside of the tube, compressing and shaping the tube into a flat band and hardening the friction compd, e g, rubber with usual vulcanizing agents, fillers, etc, or, alternatively or in addn, a PhOH-aldehyde resin or a drying oil, e g,

linseed, China wood oil Incompletely polymerized isoprene may also be used Cf C A 30, 11561

Coloring aluminum oxide surfaces Aluminum Colors Inc Ger. 620,793, Oct. 26, 1935 (Cl 494, 4 02) The surfaces are treated at 40° with salts of weak metal bases with a pH value of 4-5-7 measured at 25°. Insol compds are formed by hydrolysis to give a colored effect Cf C A 30, 754

Spark plug materials Armen A Kasarjian U S 2,029,570, Feb 4 A mixt of finely divided conductive and non-conductive materials such as "duraluminum" and mica which are of about the same sp. gr. is used in a pocket of a spark plug so that the mixt is included in the ignition circuit and serves to improve the efficiency and life of the plug

Metallic packing Sidney L Palmer (to American Smelting and Refining Co.) U S 2,029,240, Jan. 21 A soft metal such as Pb, Sb or Pb-Sb alloy in admixt with other packing material such as graphite and oil is extruded through a die under sufficient pressure to weld the metallic components of the mixt adjacent the die walls into a form retaining skin on the surface of the material being extruded

Fingerprints Justin J McCarthy. U S 2,028,619, Jan 21 A powdered mixt. of gum acacia with a larger proportion of hydroquinone (the proportions suitably being about 1 and 8 parts, resp) is placed on a fingerprint, a scutized sheet which has been wet with an aq soln of NaOH and  $\text{Na}_2\text{SO}_4$  is placed on the powdered fingerprint, and the sheet is removed and treated with an acid fixing bath

Sanitary-pipe making machine feeders and pug mills for molding plastic substances Alfred Farmer and Bertram A Farmer Brit 435,210, Sept. 17, 1935

Puncture sealing compositions Henri Bertrand Brit 435,967, Oct 2, 1935 These comprise mixts of starch and borax, or starch, sugar and  $\text{H}_2\text{O}$ , together with mica particles and rubber and cork in pulverulent form.

Cork compositions Bond Manufacturing Corp. Brit. 436,452, Oct 10, 1935 See Fr 771,759 (C A 29, 15414)

Decolorizing substances John J Naugle Can 354,360, Nov 26, 1935 Residues obtained by carbonizing and leaching the cooking liquors from the alkali wood-pulp process are washed, dried and heated to produce a carbonaceous material consisting of approx 97-100% of pure C, and not more than 2-4% of ash

Catalytic filters for the exhaust gases of an internal combustion engine Degea A-G (Auergesellschaft) Brit 435,050, Sept 25, 1935, and 436,331, Oct 9, 1935

Protection of metal or wood surfaces Frank L. McLaughlin (to International Latex Processes, Ltd.) Can 354,285, Nov. 19, 1935 A thin coating of nontacky rubber from an aq dispersion of rubber is deposited on the surface and this is covered with paper

Use of balsam wood for ink pads for rubber stamps Leland A. Phillips U S 2,028,842, Jan 28. Balsam wood cut across the grain is used

Ink remover Peter A Geller Can 354,185, Nov. 19, 1935 A mixt of toluene 65, anisole 31, aniline 15, sassafras oil 5 part is specified

Inking ribbon Louis C Antrim (to Autographic Register Co.) Can 354,228, Nov. 19, 1935 An inking ribbon is made of woven textile fabric impregnated with an ink soln On each side of the ribbon is an Al powder coating that retards the transfer of the ink soln to the worksheet

Treating bristles W Kedzie Teller (to Western Bottle Manufacturing Co.) Can 354,575, Dec 3, 1935 Bristles for brushes, such as tooth brushes, are made more water-repellant by soaking them in a soln contg 1-3% by wt of an Al salt, such as Al acetate, then placing them in a 4% soln of any suitable soap, such as castile soap, draining and drying Cf C A 29, 59574

## 14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

A review of waterworks and sewerage activities A. E. Berry. *Eng. Contract Record* 49, 1102-4(1935)—Projected works in Ontario are outlined R. E. T.

What are the trends in the treatment of water and sewerage? Norman J. Howard *Eng. Contract Record* 49, 1105-7(1935)—A review R. E. Thompson

Pioneering in the waterworks field Morgan D. Hayes *J. Am. Water Works Assoc.* 28, 22-9(1936)—Among the earlier practices described is the method used for applying I through the Rochester, Minn., water supply D. K. French

Mechanical control in waterworks operation A. H. R. Thomas *J. Am. Water Works Assoc.* 28, 6-21(1936)—Devices are described for the control of pressure, effluent and wash water, also automatic controls for the feeding of chemicals and introduction of chlorine C. F. C. A. 29, 5937<sup>1</sup> D. K. French

The development of the Premerton, Washington, water system Cliff C. Casad *J. Am. Water Works Assoc.* 28, 97-105(1936)—Surface water from 2 creeks is used. All water is chlorinated, and when necessary ammonia is added D. K. French

Providence water treatment Elwood L. Bean *J. New Engl. Water Works Assoc.* 49, 406-18(1935); *Water Works Eng.* 89, 79-83(1936)—The treatment has consisted of the addn. of copperas, aeration, lime treatment, mixing, coagulation and sedimentation, filtration and secondary aeration, the copperas was later chlorinated and finally replaced by  $\text{Fe}_2(\text{SO}_4)_3$ , at last commercially available. The  $\text{pH}$  is raised by  $\text{Ca}(\text{OH})_2$  to about 9.6 to reduce corrosion Specifications for Ferrisul and hydrated lime are given D. K. French

Domestic water supplies of Idaho W. V. Leonard *J. Am. Water Works Assoc.* 28, 106-7(1936)—Hardness averages from 50 p. p. m. in the northern lakes to 250 p. p. m. in Snake River. Certain deep wells carry F. D. K. French

Ground-water problems south of Budapest E. R. Schmidt. *Banyász. Kohász. Lapok* 68, 369-76(1935)—Several factories in the southern environment of Budapest tried to find usable soil waters instead of the expensive drinking water of the waterworks. Test borings showed that water originating from the Pleistocene gravel was very hard (54° German hardness), that of Mediterranean layers contained 12 g. NaCl per l. and Oligocene layers had no water at all. A thermal water contg. much Ca and S probably can be found at a depth of 1000 meters. Conditions were much the same on the other bank of the Danube. The factories are now trying to use purified Danube water. S. S. de Findly

French Indo-China supply secured through American methods H. O. Beauchemin *Water Works Eng.* 89, 67-71(1935)—Twenty-four wells furnishing more than 30 m. g. d. are used as a source of supply. The lime and alum treatment is used, followed by treatment with Ca hypochlorite for sterilization purposes. The Fe content, which is often as high as 80 p. p. m., is reduced to less than 0.3 p. p. m. by the use of pressure Fe-removal units W. A. Moore

Sulfur bacteria in the "pink" waters of the Surukhani oil fields and their significance in the geochemistry of water. V. T. Malushevsk and A. A. Malanov. *Compt. rend. acad. sci. U. R. S. S. [N. S.]*, 132, 221-4(1935)—Waters from wells 1300-1700 m. deep were found to contain both purple and thio acid bacteria in addn. to *Microspira desulfuricans*. This points to the possibility of a process of oxidation of sulfide ion to sulfate, opposite to the predominating process of sulfate reduction. These waters are unusually high in sulfates M. F.

The mineral waters of Harrogate A. Woodmansey *Analyst* 61, 23-6(1936)—Artificial solns. do not have the same therapeutic value as natural waters of the same compo. The Harrogate waters are best explained by the magmatic theory of Suess. Some of them contain con-

siderable sulfide and others considerable Fe. The compo. is discussed but no analytical data are given. W. T. H.

Physicochemical analysis of the Mondovì Luriso mineral waters Presence of lithium in besimaudite, a quartziferous, porphyritic rock in the same locality. Luigi Francesconi and Renato Bruna. *Ann. chim. applicata* 25, 460-9(1935)—Analyses of the Mondovì waters in Luriso show the presence of 0.01-0.048 mg.  $\text{Li}_2\text{O}$  per l. The waters are also radioactive. The Li is derived from the quartzitic besimaudite, which contains an av. of 0.05 mg.  $\text{Li}_2\text{O/g}$  of the mineral. A. W. C.

Difference in the behavior of water of total and of permanent hardness in the presence of soap Ed. Justin Mueller *J. pharm. chim.* 22, 563-5(1935).—Calcereous water of permanent hardness gives with small quantities of soap a perfect dispersoid or emulsoid system, provided no disturbing electrolyte is present. Water of total hardness, under the same conditions, is flocculated. S. W.

The residual hardness and the excess of chemicals in the soda-lime softening process (for water). Nils Westberg. *Tek. Tid. Uppf. C, Kemt.* 65, 89-92(1935)—Methods of calcg. the amts. of  $\text{Na}_2\text{CO}_3$  and  $\text{Ca}(\text{OH})_2$  to be introduced into hard waters to effect a max. removal of  $\text{HCO}_3^-$ ,  $\text{CO}_3$ , Mg and Ca are outlined. These are based on analytical results as well as influential factors, such as the ion concns., temp. and the reaction eqn. D. Thuesen

Prechlorination with ammonia in a turbid water supply Geo. F. Gilmison. *Water Works Eng.* 89, 87, 102(1936).—The addn. of  $(\text{NH}_4)_2\text{SO}_4$  in conjunction with  $\text{Al}_2(\text{SO}_4)_3$  to the water as it entered the mixing tank, followed by the addn. of Cl as the water passed the coagulating basin, greatly reduced the bacterial count of the water entering the filters. This treatment was also effective in combating algal growth in the basin. Equal parts of Cl and  $(\text{NH}_4)_2\text{SO}_4$  were used W. A. Moore

Preparation and conditioning of aluminum sulfate solution before applying to water. Carl Leopold *J. Am. Water Works Assoc.* 28, 74-96(1936).—It is demonstrated that agitation in dissolving is more important than soln. strength in floc formation with an alum soln. High-speed mech. agitation in a cylindrical-shaped dissolving tank is recommended D. K. French

Primary treatment plant includes magnetite filter. Water S. Bemis. *Eng. News-Record* 115, 852-5(1935).—The treatment plant constructed at Goshen, Ind., as the initial step in remedying the pollution of the watershed of the Elkhart and St. Joseph Rivers consists of a comminutor, mech. grit remover, settling tank provided with a magnetite filter and rotary sludge scraper, sludge digestion tank and open sludge beds. Complete treatment can be provided with a min. of new construction. The filter consists of a trough around the perimeter of the tank contg. 3 in. of magnetite sand supported on a phosphor-bronze screen, which in turn is supported on a steel grating. The settled liquor flows upward through the filter at an av. rate of 2.1 gallons per sq. ft. per min. Cleaning is automatically effected by a mechanism mounted on a car which travels on rails over the filter, operation of which is controlled by the head of liquid in the tank. It is expected that the filter will increase the removal of suspended solids approx. 25%. The av. detention period in the tank will be 1.5 hrs. The digestion tank has a capacity of 2 cu. ft. per capita, and the drying beds an area of 1.2 sq. ft. per capita. The sludge gas will be utilized for heating the digestion tank and buildings. The cost of the treatment plant and main pumping station, which have capacities of about 7 million gallons per day, was \$105,000 R. E. Thompson

Treatment of water for domestic purposes in a Trinidad oil field A. H. Richard. *J. Inst. Petroleum Tech.* 21, 871-86(1935).—For a domestic  $\text{H}_2\text{O}$  supply in isolated oil camps coagulation is important. Alum is preferable as a coagulating agent to  $\text{FeSO}_4$ , and basic alum is still

better. The dose need not be detd. by chem. analysis. The "jar test," consisting in adding varying amts. of chemical to samples of the  $H_2O$  in glass jars, will give the desired information. Designs are shown for a chem. dosing chamber and a circular coagulation basin. E. E. C.

Use of activated carbon in the purification of water in the tropics (Madras city water supply). T. N. S. Raghavacari and P. V. Seetharama Iyer. *Proc. Indian Acad. Sci.* 2B, 237-53 (1935).—Small-scale expts. over a 2-yr. period using (a) powder and (b) granular activated C in conjunction with slow sand filtration gave the following results: (1) Both powder and granular activated C are effective in removing tastes, odors and colors; greater reduction in colloidal org. matter results with the granular form. (2) Better overall effluents result when  $1\frac{1}{2}$  in. of granulated C is sandwiched in a slow sand filter than with sand filtration alone. (3) The C is particularly effective in reducing  $H_2S$  production in the sandwiched filter even at filtration rates as low as 4 in. v. p. h. (vertical per hr.) where slow sand filtration is conducive to  $H_2S$  production. (4)  $H_2S$  production falls off with increased filtration rates, and in a sandwiched filter operating at 8-12 in. v. p. h. no  $H_2S$  could be detd. (5) Best overall results are obtained by passing the effluent from the ordinary slow sand filters through an 18-in. bed of granulated C with 36 min. contact time, final effluents are colorless, odorless and tasteless, and show approx. 75% reduction in org. matter. (6) Granular activated C used continuously for 23 months showed no deterioration in sorbing ability. G. H. Young.

The present status of water sterilization by means of the catadyn process. A. Salmony-Karsten. *Gesundh.-Ing.* 58, 736-9 (1935).—Various types of equipment used for both the contact and electrocatadyn processes are described, including a pocket electrocatadyn device for sterilizing a few cc. to 1 l. in 1 min. M. G. Moore.

Municipal water-filtration plants in Canada. A. E. Berry. *Can. Eng.* 69, No. 20, 8-9 (1935).—A list of municipal water-purification plants in various provinces of Canada gives the location, type of filter, date installed, population served and source of water supply.

Ann. Nicholson Hird.  
The water-filtration plant of Hamilton, Ontario. W. L. McFaul. *J. Am. Water Works Assoc.* 28, 57-64 (1936), cf. C. A. 30, 1475.—Alum is dissolved in flash-mixing units and mixed with the raw water. The mixing chambers are equipped with flocculators. After a 30-min. detention period the water goes to settling basins and then to the filters. Chlorine is finally added. D. K. French.

Recent taste and odor tests of paints for water tanks. Geo. L. Hall. *Eng. News-Record* 115, 639 (1935).—Brief data are given regarding addnl. tests (cf. C. A. 25, 3418) of paints made by the Maryland State Dept. of Health. The procedure employed consisted of allowing distd., tap and chlorinated water contg. 0.2 to 2.0 p. p. m. residual Cl to remain in metal vessels coated with the various paints for 15 min. and then testing the waters for taste and odor in the hot and cold. Nine paints were found satisfactory. The one paint found satisfactory in the previous tests and one found suitable in this series have been successfully used in practice. R. E. T.

Recent progress in supplying less corrosive water (soft water). Malcolm Purdie. *J. New Eng. Water Works Assoc.* 49, 419-27 (1935).—The greatest stability and absence of corrosive properties is brought about when the  $pu$  value is over 9.6, Fe less than 0.05 p. p. m., Mn absent and Al less than 0.03 p. p. m. Temp. should be low and a residual Cl of 0.02 p. p. m. should exist to inhibit bacterial activity in the mains. D. K. French.

Materials available for water pipes. R. Schmel. *Gas u. Wasserfach* 78, 971-4 (1935).—Better asphalt coatings can be secured on cast-iron pipes by preheating the pipe before dipping. Steel pipe larger than 2 in. can be coated centrifugally with a hot mixt. of petroleum asphalt with a filler such as rock dust and a softener such as paraffin wax. Pipes from 1 in. to 2 in. can be coated by rotating the pipes and using an asphalt mixt. contg. softener but no filler, but smaller pipes cannot be coated (inter-

nally) with any satisfaction, as dipping gives very uneven coatings. External coatings should be protected with heavy paper or jute wrappings. All cut pipe surfaces must be dipped in hot bitumen or otherwise protected. R. W. Ryan.

Aluminate treating for field boilers. Eugene Vago. *Petroleum World* 32, No. 8, 46, 49, 51-2 (1935), cf. C. A. 29, 7537.—A description of types of  $H_2O$  and their distinguishing tests is given. Methods of internal and external treatment are considered from a cost and operation viewpoint. Chas. Wirth, III.

Cause and prevention of turbine-blade deposits. Frederick G. Straub. *Trans. Am. Soc. Mech. Engrs.* 57, 447-54 (1935); *Combustion* 7, No. 6, 23-7 (1935).—Lab. and plant-scale expts. show that turbine-blade deposits result from contamination of the steam by boiler  $H_2O$ . Lab. studies indicate that NaOH is responsible for scale adhesion; when it is converted to  $Na_2CO_3$ , scale does not adhere, the presence of sufficient amts. of neutral salts, such as  $Na_2SO_4$ , also prevents scale adhesion. Deposition of water-soluble salts carried into the turbine by mech. entrainment is thus a major problem. For lab. expts. condensed steam from a steam heating system was passed through a deaerating heater and then pumped to an electrically heated boiler to generate steam; the steam was passed through a chamber contg. the test solns. of various dissolved salts, bubbling up through the solns. and entering an electrically heated preheater; it then passed through a suitable orifice and impinged on a single immovable turbine blade, and then through a copper-cool condenser to the condensate storage bottle. Steam pressure and superheater temps. were maintained const. by potentiometric regulators. Tests were run at 40 and 45 lb. per sq. in. abs. and 400-700°F., with a pressure of 0.8 lb. in the blade chamber. The orifice was  $\frac{1}{16}$  in. in diam. and the blade was set at a 30° angle to the line of steam flow. Conclusions: (1) The amt. of deposit is not proportional to the carry-over from the boiler, but depends upon the relationship of the various salts dissolved in the boiler  $H_2O$ , (2) salts which form a dry powder in superheated steam will not adhere to turbine blades, and the application of such salts (sulfates, chlorides and carbonates) to boiler feed water will reduce deposits by coating the particles of NaOH and allowing them to be carried on through the turbine, (3) concns. of 4-5 times the concn. of NaOH are required for effective scale prevention if  $Na_2SO_4$  is used as the added salt. Cost of treatment is less than \$500 per yr., based on expts. in a large central power plant. G. H. Young.

Sewage collection, treatment and disposal. J. Clark Keith. *Eng. Contract Record* 49, 979-80 (1935).

R. E. Thompson.  
Sewage disposal and the supply of domestic raw materials. V. Methane from sewage-disposal plants as a motor fuel. Adolf Heilmann. *Gesundh.-Ing.* 58, 764-0, 784-8 (1935), cf. C. A. 29, 5367.—General discussion. A table gives the type of disposal system and quantity of  $CH_4$  produced for 74 German cities. M. G. Moore.

Varied methods of public cleansing reviewed at International Congress. Harrison P. Eddy, Jr. *Eng. News-Record* 115, 507-10 (1935).—The proceedings of the 2nd International Congress for Public Cleansing, held in Frankfurt am Main, Germany, are reviewed.

R. E. Thompson.  
Laboratory tests and equipment for sewage works. Ralph E. Lührman. *Am. City* 51, No. 1, 17 (1936), cf. C. A. 30, 551.—Routine sewage tests are described and discussed. G. H. Young.

Factors in the design of sewage-disposal works. H. C. Whitehead and F. R. O'Shaughnessy. *Surveyor* 88, 403-7, 423-9 (1935), cf. C. A. 28, 2085.—Twelve diagrams are given showing B. O. D. as a function of the type of sewage treated. A. L. Elder.

Research with sewage filters at the Wupper Valley Buchenhofen clarification plant. Mahr and Lerner. *Gesundh.-Ing.* 59, 8-11 (1936), cf. C. A. 29, 2263.—Since during some seasons a diln. of only 1:4 is available in the receiving stream, and as the changing character of

the heavy load of industrial wastes would make the satisfactory operation of biol. treatment difficult, filtration of the sewage after it leaves the settling basins was considered. The effects of varying the grain size and depth of layer of the sand, the filtering velocity, the possible time of run between washings and the method of washing were studied with an expul. unit of 2 sq. m. filter surface. Filtration without preliminary chem. treatment was found to be quite satisfactory, but the grain size of the sand must be appreciably larger than that used for rapid filters for water treatment. Cleaning was with compressed air, then water, the vol. of water required being 1-6% the vol. of sewage filtered. No troubles due to odors or septic conditions were encountered. M. G. Moore

High lights of [sewage] plant operation in Michigan. W. F. Shephard. Mich. Eng. Expt. Sta., Bull. 64, 5-16 (1935).—General Cf C A 29, 8191. C. R. Fellers

The sewage-disposal plant of the city of Heilbronn. N. E. Seyfried. *Gesundh.-Ing.* 59, 23-6 (1936).—Details of construction (with 4 cuts) of a plant employing settling and sep. sludge digestion are given. M. G. Moore

Operating results of the North Toronto sewage works. Norman J. Howard. *Eng. Contract Record* 49, 921-2 (1935).—The bacterial count (standard agar, 37°, 24 hrs.) on the raw sewage, which is chiefly domestic in character, averaged 3,202,176 per cc. during the winter and spring months and 8,343,333 during the summer months, the max. counts being 8,800,000 and 15,000,000, resp. The effluent count averaged 24,186 during the winter and spring and 356,667 during the summer, the max. counts being 51,675 and 1,320,000 and the percentage reduction 99.2 and 95.7, resp. The av. indicated no. of *Es. coli* per cc. in the raw sewage during the cold and warm months, resp., was 165,912 and 426,250, the max. during both periods being 1,000,000, while the effluent av. was 1453 and 4187.5, resp., a reduction of 99.1 and 99.0%, resp. The storm overflow water had an av. bacterial count of 736,000, with a max. of 1,024,000, while the *Es. coli* content averaged 32,500 per cc. with a max. of 100,000. The Cl demand was considered to be that dosage which would give a residual Cl content of 0.2 p. p. m. after 10 min. contact; samples collected during the winter and spring months were treated in the lab. with a Cl dosage equiv. to the demand and subjected to bacteriol. exam. In this series the av. bacterial count on the raw sewage was 3,325,692, on the effluent 20,990 and on the chlorinated effluent 1525 per cc., an over-all reduction of 99.95%; the *Es. coli* contents averaged 203,846, 2000 and 14 per cc., resp., a reduction of 99.9%. Higher Cl dosages gave proportionally greater bacterial reductions. Similar tests on the combined storm overflow and plant effluent showed that chlorination reduced the bacterial count from 349,500 to 1645 and the *Es. coli* content from 32,500 to 5.5 per cc. The Cl demand of the raw sewage and effluent averaged 6.1 and 1.51 p. p. m., resp. The sewage temp. ranged from 40 to 51.5°F. during the winter and spring period and from 59° to 63.5° during the summer period. R. E. Thompson

Kitchener's sewage plant operation. Stanley Shupe. *Eng. Contract Record* 49, 936-7 (1935).—Industrial wastes cause difficulties. When sludge bulking due to septicity occurs, Ca hypochlorite is employed to remedy the condition. The detention periods in the aeration and clarifying tanks, resp., are 7.5 and 2.4 hrs., and the amt. of air used varies from 1.54 to 2.66 cu. ft. per gallon of sewage treated. R. E. Thompson

Sewage treatment practices. A. E. Berry. *Eng. Contract Record* 49, 933-6 (1935).—A general discussion, with particular reference to Ontario practice, includes the combating of septic conditions and sludge bulking in activated-sludge plants, the effects of trade wastes, variations in sludge content in aeration tanks, etc., standards of quality for final effluents and sludge disposal. Increased application and the use of Ca hypochlorite are the most useful aids in combating septicity. At 2 plants, actinomyces interfered with the settlement of activated sludge. In one case, application of hypochlorite corrected the condition, while in the other it disappeared without any

change in operation. The optimum amt. of sludge carried in the aeration tanks varies from less than 10 to as much as 40%, based upon the 0.5-hr. settling test. R. E. T.

The scope of various sewage-disposal systems with special reference to biochemical reduction. W. D. Turner. *Eng. Contract Record* 49, 987-9 (1935).—The bio-reduction process consists of (1) comminution, (2) addn. of bio-loam (the end product of the process), agitation for a period of 15-20 min. and settlement for 20 min., (3) addn. of ferric salts, agitation for period of 20 min. and settlement for 1 hr. and (4) further treatment of effluent by filtration or chlorination if required. The sludge is centrifuged and subjected to aerobic stabilization for a period of 10 days. The heat generated during oxidation of the sludge drives off most of the moisture, and the product is a stable loam contg. about 3% N, which can be used as a fertilizer. The process is free from odor. R. E. Thompson

Recent sewage plant developments call for fresh approach to design problems. Samuel A. Greeley. *Eng. News-Record* 115, 501-2 (1935).—Since 1930 the range of treatments has been so widened that plants can be designed more closely to meet the requirements of the waterway into which the effluent is discharged, this results in better over-all efficiency and economy. R. E. T.

Sprinkling-filter requirements revised by New Jersey. J. Lynn Mahaffey. *Eng. News-Record* 115, 753-4 (1935).—Revised regulations adopted Nov. 12, 1935, are given. The permissible amt. of sewage treated daily per acre per ft. of filter depth was increased from 300,000 to 630,000 gallons. Chem. pptn., as an adjunct to preliminary sedimentation preceding filtration in cases where the 5-day biochem. O<sub>2</sub> demand of the raw sewage exceeds 325 p. p. m. and as a supplement to final sedimentation, is recommended for consideration. Other changes are concerned with the max and min effective depth of filter bed, dosing cycle and required head on sprinkler nozzles. R. E. Thompson

Sludge handling at Mimico, Ont. W. B. Redfern. *Eng. Contract Record* 49, 939-9 (1935).—The Mimico plant is of the activated-sludge type, utilizing an Imhoff tank which was part of the original plant. The combined sludge is digested and the gas collected provides sufficient heat for the tanks and buildings. In summer, when less heat is required, the Imhoff-tank sludge is discharged directly on to glass-covered drying beds, as it dries more rapidly than digested sludge. The dried sludge is used as fertilizer around the plant and by farmers and market gardeners. R. E. Thompson

Activated carbon aids sludge digestion at Liberty, N. Y. John Lawrence and Harry Eichenauer. *Public Works* 67, No. 1, 17 (1936).—Replacing lime dosage by activated-C treatment in sludge digestion tanks increased digestion temps., raised the pH and improved sludge quality. G. H. Young

Processes occurring in the sludge-digestion space of Imhoff tanks. H. Mann. *Gesundh.-Ing.* 58, 739-40 (1935).—The gas from Imhoff tanks contains 20-5% CO<sub>2</sub> while that from sep. sludge-digestion units contains 30-55%. This is due to the fact that in the former type of unit the supernatant liquid is changing more rapidly, does not become satd. with the gas and therefore dissolves more of the sol. CO<sub>2</sub> as the gas bubbles rise through it. In a tank having a single common sludge space but 3 settling spaces, gas from the first settling space (in the direction of sewage flow) was found to contain materially less (5.6%) CO<sub>2</sub> than that from the other 2 settling spaces (about 19%). M. G. Moore

Regulation of sludge use by provincial health departments considered necessary. Wm. Storrie. *Eng. Contract Record* 49, 923 (1935).—The consensus of opinion is that there is no health hazard from use of properly digested sludge as fertilizer for garden or field crops, but opinion differs regarding the use of undigested sludge, particularly for vegetables which are eaten raw. Provincial regulation is considered advisable. R. E. Thompson

Chlorination in sewage treatment. Reginald W. Covill. *Surveyor* 88, 471 (1935).—The Cl demand of a sewage is

defined as the p p m. of  $\text{Cl}_2$  as chlorine water (10 g. of  $\text{Cl}$  per l.) required to be added to 250 cc. of sewage so as to produce less than 0.1 p p m. of  $\text{Cl}_2$ . The  $\text{Cl}_2$  demand of raw sewage ranges from 5 to 40 p p m. Excellent control of *Psychoda* flies has been obtained by the use of  $\text{Cl}_2$ . The use of 0.5-1% has been used to prevent ponding. A. L. Elder

Chlorination as applied to sewage treatment. L. H. Enslow. *Eng. Contract Record* 49, 931-2 (1935).—The use of  $\text{Cl}$  and "chlorinated  $\text{Fe}$ ," prep'd by dissolving scrap  $\text{Fe}$  in  $\text{Cl}$  water, is discussed. R. E. Thompson

Chemical precipitation at Glasgow. Alexander Hunter. *Surveyor* 88, 353-4 (1935).—Lime and  $\text{Al}$  sulfate are used as coagulants at Dalmarnock works. Forty % of the chemically treated effluent is filtered. Nearly one million tons of the crude sludge ext'd was sent to sea. A. L. E.

The revival and present status of chemical precipitation. M. N. Baker. *Eng. News-Record* 115, 748-50 (1935).—Because of the lower cost of precipitants and the development of mech. devices, there has been a revival of chem. pptn. as a method of sewage treatment since 1924, at which time the process had been largely abandoned. Thirty-four of the 3500 municipalities treating their sewage employ this method, 11 plants are under construction, and 25 have been more or less definitely projected. Tabulations are given showing the distribution of chem. pptn. plants by states and the chemicals employed. A striking feature is the general use of  $\text{Fe}$  compds. as precipitants and the subordinate role of lime.  $\text{FeCl}_3$ , prep'd in most cases from  $\text{Cl}$  and scrap  $\text{Fe}$ , is the most commonly employed chemical. R. E. Thompson

The permanganate number in the evaluation of pulp-mill waste water. H. Haupt-Bautzen. *Zellstoff u. Papier* 15, 436-8 (1935).—The org. impurities of industrial sewage water cannot be est'd by the permanganate no. alone, since impurities of different chem. character and constitution require varying amts. of permanganate. The permanganate requirements for 1 g. of the following substances are: phenol 9660, dextrin 840, albumin 644,  $\text{HOAc}$  5, sulfite waste liquor residue 2300-3000 and boric acids 3200 mg. Natural purification, due to bacterial cleavage and decomposition processes, goes on fairly rapidly for ordinary domestic sewage, while about 70% of the org. materials in pulp-mill sewage is not affected in this way. The biochem. O demand of domestic sewage water decreases rapidly under these conditions, but that of the pulp-mill waste water, very small to start with, is hardly affected. Ordinary sewage waters and pulp-mill waste waters of equal permanganate no. have very different biochem. O demands and O economies. The permanganate no. is not a satisfactory measure for the org. impurities in waters which contain slowly decomposing compds. of high mol. wt. S. I. Aronovsky

The dispersion of Anopheles larvae by the flow of streams and the effect of larvicides in preventing this. J. A. Sutton and Syed Abdul Majid. *Records Malaya Survey India* 5, 3-17 (1935).—The drifting of mosquito larvae into controlled areas of streams was greatly diminished by a continuous application of lubricating oil by means of oil-soaked sacks contg. sand or fiber and anchored in the streams above suitable booms. Paris green had a rapid kiling action on larvae in the area dusted but its effect was of comparatively short duration, when it was used in routine fashion every day or so it was relatively ineffective against drifting larvae. K. D. Jacob

Conditioning water for the textile industry (Morrison) 25 Composts and fertilizers in relation to greenkeeping. VI. Miscellaneous fertilizers [sewage sludge] (Evans) 15 Effect of the digestion of sludge on the germinating power of weed seeds (Vieh) 15 Rust damage in hot-water systems (Marx) 9 Geothermal stages and the chemistry of artesian waters (Chebotarev) 8 Filters—purifying water (Brit. pat. 435,543) 1

Blanning, Harry K., and Rich, Albert D. Boiler Feed and Boiler Water Softening. 2nd ed. Chicago: Nacker-son and Collins Co. 159 pp. \$3.

Folwell, A. Prescott. The Manual of Sewage Disposal Equipment. New York: Public Works Mag. 113 pp. \$2

Koch Pierre. L'assainissement des agglomérations, T. I. L'évacuation de l'effluent urbain. Paris: L. Eyrolles. 376 pp. P 70. Reviewed in *Bull. assoc. chim.* 52, 887 (1935)

Kolbe, Heinrich. Wirtschaftlichkeit im Dampfkessel-Betriebe. Arbeitshilfen zur wirtschaftlichen Überwachung von Dampfkessel-Anlagen. Halle. W. Knapp. 68 pp. M. 5.20. Reviewed in *Ind. Eng. Chem., News Ed.* 13, 448 (1935)

Martin, Arthur J.: The Work of the Sanitary Engineer. London: Macdonald & Evans. 472 pp. 16s. Reviewed in *Chemistry & Industry* 1935, 963, *J. Am. Water Works Assoc.* 27, 1801 (1935).

Matthews, F. J. Boiler Feed-Water Treatment. London: Hutchinson's Scientific and Tech. Publications. 256 pp. 12s. 6d. Reviewed in *Chem. Trade J.* 98, 54 (1936)

Metcalf, Leonard, and Eddy, Harrison P. American Sewerage Practice. Vol. III. Disposal of Sewage. 3rd ed. New York: McGraw-Hill Book Co. 892 pp. \$7. Cf. *C. A.* 24, 2527

Stewart, Alan W. A Manual of Practical Chemistry for Public Health Students. 3rd ed. London: The Author. 121 pp. 7s. 6d. Reviewed in *J. State Med.* 43, 744 (1937)

Smet, Denis de: Hygiène et assainissement des locaux industriels. Ventilation, chauffage, éclairage, élimination des poussières, buées, fumées, vapeurs et gaz. Brussels: Comité central industriel de Belgique. F 18.

Spring water. Gustav Offe. *Ger.* 620,990, Oct. 31, 1935 (Cl. 85b. 1 07). Mineral spring water free from living organisms and contg.  $\text{CO}_2$  is freed from  $\text{Fe}$  by treating it with  $\text{Cl}$  and filtering through a catalytic filter. Only sufficient  $\text{Cl}$  to oxidize the  $\text{Fe}$  is used. The water is passed through a mech. filter after the catalytic filter, with exclusion of air. App. is described.

Water purification. Oliver M. Urban and William R. Stemen (to Charles H. Lewis). U. S. 2,029,966, Feb. 4. For purifying water to remove dyes, etc., a reagent is used such as one prep'd from bituminous coal, lignite, cannel coal, peat or coke by treating it with  $\text{HNO}_3$ ,  $\text{HNO}_2$ , and  $\text{H}_2\text{SO}_4$  or  $\text{H}_3\text{PO}_4$  or  $\text{NO}$  or  $\text{NO}_2$ .

Water purification. Oliver M. Urban (to Charles H. Lewis). U. S. 2,029,959, Feb. 4. A reagent of the isocyanate group such as 3,5-dichlorophenyl isothiocyanate is used for eliminating amines and hydroxy compds. from water at a pH of 6.5-7.5. U. S. 2,029,960 relates to a similar use of a reagent of the acidic phenol group such as 2,4,6-trinitrophenol. U. S. 2,029,961 relates to the elimination of carboxylic acids such as propionic acid, etc., from water by use of a reagent of the acidic amine salt group such as Na 3-amino-1-propanesulfonate.

Purifying potable and polluted waters. Oliver M. Urban and William R. Stemen (to Charles H. Lewis). U. S. 2,029,962, Feb. 4. Chlorinated coal is used as a water-purifying agent and serves to remove phenols and cresols and their chloro derivs. U. S. 2,029,963 relates to the similar use of hydroxylated chlorinated coal such as that prep'd by various described methods. U. S. 2,029,964 relates to a like use of "etherated chlorinated coal" such as that prep'd by reaction of an alc. with chlorinated coal.

Water filter. S. V. Moussev. Russ. 38,051, July 31, 1934. Construction details.

Clarifying water. Marius P. Otto. Fr. 789,102, Oct. 23, 1935. The mixt. of water to be treated and the coagulating agent, e. g.,  $\text{Al}_2(\text{SO}_4)_3$ , are divided into very fine streams, this assures the intimate mixt. of the 2 elements and allows the max. amt. of impurities to be eliminated during the decantation, the yield of the filter is thus increased. Fr. 789,103. The nozzles by which compressed air is blown through the filtering mass are disposed in the mass itself at a suitable depth, this assures regularity of distribution of the air through the upper part of the filter.

ing material. Fr. 789,104. The mixt. of water and coagulating agent are caused to pass in a broken up and down path through the decantation vessel; this favors deposition.

**Zeolite water-softening apparatus** Harry M Marsh Brit. 435,589, Sept. 24, 1935 Means responsive to changes in the wt. of a const. vol. of the used brine passing through a counterbalanced bucket and normally flowing to waste during regeneration of the zeolite is used to divert part of the used brine to a tank where it is retained for use in a subsequent regeneration.

**Softening water** A E Rukovskii and N P Nase-lenko Russ 38,048, July 31, 1934 Glaucinite for use in water softening is stabilized and made suitable for a base-exchange material by first heating under ordinary or increased pressure in the presence or absence of air to 400-600°.

**Closure-members for base-exchange water softeners or filters** Catherine Jenkins Brit 435,425, Sept. 20, 1935

**Apparatus for neutralizing acidic waters** N P Sokolov Russ 38,052, July 31, 1934 Construction details are given of an app. in which CaO is used for neutralization.

**Sewage-treating apparatus** James Hopwood and Adams-Hydraulics Ltd Brit 435,862, Sept. 23, 1935 To promote coagulation and flocculation in sewage and like waste liquid, it is treated in a tank provided with 1 or more paddles supported by arms to which an oscillatory or pendulum motion is imparted.

**Spraying sewage sludge and burning it in suspension** Max H Kuhner (to Riley Stoker Corp.) U S 2,029,725, Feb. 4 App and various operative details are described.

**Simultaneous biological purification of waste waters and their sludges** R A Henry Belg 408,348, April 30, 1935 The waste waters are treated with a suspension of clay in an aq. soln. of lime, and ultimately with other inert

materials, before they are sent to the decantation tanks. The sludges are subsequently conveyed to a heated surface and treated with steam. Cf. C. A. 30, 1164°.

**Treating putrifiable organic wastes** George H. Earp-Thomas. Brit 435,380, Sept. 16, 1935 See U. S. 1,938,647 (C. A. 28, 1459°).

**Treating foul waters** Ernst Nolte, Hans J. Meyer and Erich Fromke. Brit. 435,363, Sept. 19, 1935. A small quantity of sol. phosphate or other sol. P compd. is added to industrial waste water, e. g., that contg. POH or from sugar manuf., to render effective subsequent treatment by the activated-sludge or other biol. process.

**Treating fat-containing waste waters** A de Vreese. Belg 407,624, April 30, 1935. The waste waters are treated with a Ca salt (preferably CaCl<sub>2</sub> or CaSO<sub>4</sub>) together with sufficient lime to ensure the presence throughout the treatment of an excess of Ca salt and an excess of oxidizing Ca(OH)<sub>2</sub>. Cf. C. A. 29, 3440°.

**Oxidizing putrescible organic matter in water.** Oliver M Urban (to Charles H Lewis) U S 2,029,958, Feb. 4. Diffused air is passed through water contg. org. matter to be oxidized such as that in sewage or industrial wastes in the presence of a lower salt or oxide of Cr, Mn, Os, Sn, V, Sb, Bi, Mo or Se serving as a catalyst, until at least a substantial portion of the catalyst has been oxidized through 2 units of valence.

**Treating industrial wastes such as those from distilleries, breweries, starch works, canneries, etc.** Arthur M Buswell and Clair S. Boruff U S 2,029,702, Feb. 4. In an anaerobic bacterial digestion of fermentable liquid org. wastes having a non-settleable solid content substantially in excess of 0.3% by the action of CH<sub>4</sub>-producing bacteria at a suitable *pH*, the total free and combined volatile org. acid content of the liquid undergoing treatment is maintained below 0.3%, calcd as HOAc, a high rate of gas production being thus effected. Examples with details of procedure are given.

## 15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

M S ANDERSON AND E D JACOB

**An improved instrument for turf sampling** R B Ferro. J. Board Greenkeeping Research 4, 119-20 (1935)

K D Jacob

**Some factors which influence the reclamation of water-logged and alkali lands.** O. W. Israelsen, Willard Gardner and D S Jennings Utah Agr. Expt. Sta., Bull. 250, 61-2 (1934); cf. C. A. 28, 7394°.—General and theoretical.

C R Fellers

**Minerals as bearers of the natural soil fertility.** F A van Baren. Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935 1, 184-5 (1935).—The subject is reviewed and x-ray diffraction studies on soils are correlated with data on fertility. Biotite is an important source of available K<sub>2</sub>O. Six references.

C J Schollenberger

**Soil porosity as an index of structure.** L. D. Bayer, C. M. Woodruff and J. F. Lutz. Mo Agr. Expt. Sta., Bull. 340 (Ann. Rept. 1933) 75 (1934).—Soil structure is considered as the arrangement of primary and secondary particles constituting the soil mass. Aggregation, permeability and porosity are the only indices of structure. The pore-space content of soils is made up of small capillary pores and large noncapillary pores. The former are responsible for the water-holding capacity of the soil, the latter for the air capacity. Normally, permeability is dependent on the content of noncapillary pores. Chernozem soils have a relatively low noncapillary porosity. The low hydration of the numerous small pores of a laterite is probably responsible for its permeability to water.

C R Fellers

**Physical and chemical properties of the Iredell and Davidson soils affecting erosion.** L. D. Bayer, C. M. Woodruff and J. F. Lutz. Mo Agr. Expt. Sta., Bull. 340 (Ann. Rept. 1933), 75 (1934).—A soil in good phys. condition has aggregates larger than 0.1 mm. in diam. and is rather porous. The Davidson soil had a larger and

much more porous type of aggregate, factors which contribute to its non-erosive properties. C. R. Fellers

**Effects of different soil treatments, long continued, upon bacterial activity in the soil.** W. A. Albrecht. Mo. Agr. Expt. Sta., Bull. 340 (Ann. Rept. 1933), 76-7 (1934).—On fertilized soil CaCO<sub>3</sub> did not affect nitrate formation, but on soils which had not been fertilized the addn. of lime greatly increased nitrate formation from green manures.

C. R. Fellers

**The influence of artificial and natural factors upon soil structure.** J. Apsits. Z. Pflanzenernähr., Dungung Bodenkd. 42, 1-35 (1936).—Capillary porosity is a natural property dependent upon phys. and chem. characteristics of the individual soil, and little affected by variations in cultural practice. Noncapillary porosity is more affected by the latter, and is increased to the greatest extent by plowing. The extent to which these larger voids supply O<sub>2</sub> to roots may be a critical factor in crop growth. Twenty-six references.

C. J. Schollenberger

**"Single value" soil properties.** A study of the significance of certain soil constants. VIII. Relationships between the sticky point and the nature of the exchangeable ions in the soil. J. R. H. Coutts. J. Agr. Sci. 25, 523-8 (1935); cf. C. A. 23, 5261°.—The variations in the sticky point of homionic soils, as detd. by kneading with electrolytic solns., were examd. With Na soils, a well-marked max. was found in the sticky point-electrolytic concn. curve, and the form shows parallelism with the extent of flocculation produced by varying electrolytic concns. of NaCl, Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub>, and CaCl<sub>2</sub> solns.

Irvin C. Feustel

**Technical studies of the physical and physicochemical properties and processes in soils.** D. S. Jennings, Willard Gardner and O. W. Israelsen Utah Agr. Expt. Sta., Bull. 250, 60 (1934); cf. C. A. 28, 7394°.—The minute



quantities of Cl retained by the soil colloids are measured by means of a Ag-AgCl electrode. Ca enters the soil 10-15 times as readily as does Na; hence a Ca salt even in the presence of large quantities of a Na salt will remove replaceable Na from the soil. K also enters the soil 4 or 5 times faster than Na. Ca enters the soil 1.5 to 2 times as readily as K. If the exchange complex is high in Ca, the soil is in much better phys. condition than if the exchange complex is mainly Na. Friability is greater for Ca colloids than for Na colloids. C. R. Fellers

An examination of methods for determining organic carbon and nitrogen in soils. Allan Walkley. *J. Agr. Sci.* 25, 598-609 (1935).—The details of the Drenstedt dry-combustion method for detg. C in soils are described and some simplifications suggested. The Bangor Kjeldahl method for C and N requires carefully standardized heating. Errors may arise from contamination of sandy soils by material abraded during grinding in iron or porcelain mills. For heavy alk. soils with little org. matter it is advisable to grind the soil very fine and to add water. The rapid dichromate titration method of Walkley and Black (cf. *C. A.* 28, 2823) for soil C gave satisfactory approx. results. The technic was improved and methods were devised for overcoming disturbances due to chlorides including addn. of  $\text{Ag}_2\text{SO}_4$ . The hypobromite method gave much more irregular results for C than the Bangor or dichromate methods. The latter was only slightly inferior to the Bangor method. I. C. Feustel

Measurements of acidification or alkalinization of soil suspensions with the glass electrode. E. Pfeil. *Angew. Chem.* 49, 57-9 (1936).—The measurements were carried out with the glass electrode before and after addn. of quinhidrone, and comparative measurements were made with the quinhidrone electrode. Exptl. data are reported on 27 suspensions. The measurements with the glass electrode showed considerably greater constancy with time (up to 60 min.) than those made with the quinhidrone electrode. Seven references. K. K.

An improved method for the determination of the soil reaction. W. Moore. *J. Board Greenkeeping Research* 4, 134-8 (1935).—Beginning from the bottom of the tube, graduate a test tube of the bacteriol. type, 6 in. long and  $\frac{1}{8}$  in. bore, as follows:  $\frac{1}{8}$  in. (I),  $\frac{1}{8}$  in. (II), 0.5 in. (III), 0.5 in. (IV), 2.5 in. (V) and 0.5 in. (VI). Other tubes of convenient size can be used, but all tubes in a set must be of approx. the same size. For sandy soils, loamy soils and heavy soils fill the tube with  $\text{BaSO}_4$  to mark I, II and III, resp.; add soil to mark IV, dist.  $\text{H}_2\text{O}$  to mark V and indicator soln. (bromocresol green, bromocresol purple or phenol red) to mark VI. Close the tube with a rubber stopper and shake until the contents are thoroughly mixed. Allow the tube to stand until the liquid clears, which should not take more than 1-2 min., and est. the pH from the color of the supernatant liquid. No difficulty should be experienced in reading to 0.5 pH by visual observation of the color or to 0.2 pH by comparison with color standards. For the purpose of this test, most cultivated soils can be regarded as loamy soils and most lawns as sandy soils. The amt. of  $\text{BaSO}_4$  should be increased with very heavy clay soils and soils with pH values over 8.0. K. D. Jacob

Solubilization of organic phosphorus. II. J. Alvarino and A. Bonazzi. *Proc. Assoc. Tec. Azucareros Cuba* 8, 115-18 (1934); cf. *C. A.* 29, 2233.—Fresh stable manure was mixed with ground bone (I) and left to ferment 8 months along with similar manure without bone (II). Analysis of II before and after fermentation showed 0.423 and 0.726% of  $\text{P}_2\text{O}_5$ , resp. sol. in 0.2% HCl, and 0.621 and 0.814%  $\text{P}_2\text{O}_5$  sol. in  $\text{NH}_4$  citrate, the corresponding figures for I are 3.587 and 4.834 sol. in 0.2% HCl and 1.882 and 1.817% sol. in  $\text{NH}_4$  citrate. It is evident that composting bone meal with manure does not increase the  $\text{H}_2\text{O}$ -sol. or citrate-sol.  $\text{P}_2\text{O}_5$ , whereas it does increase that sol. in dil. HCl. O. W. Willcox

Arsenic solubility in soils. W. B. Albert. *S. Car. Agr. Expt. Sta., 47th Ann. Rept.* 45-c (1934). cf. *C. A.* 28, 7395.—The use of heavy applications of Ca arsenate for boll-weevil control on cotton increased the As content of

some sandy soils sufficiently to cause As toxicity to crops. In the presence of Fe compds. in the soil, the As became insol. The use of P fertilizers increased the soly. of As in the soil. Two hundred lb. of superphosphate produced the same amt. of sol. As in the soil as 30,000 lb. of  $\text{Ca}(\text{PO}_3)_2$ . When 30,000 lb. of ferric arsenate-pptd.  $\text{Fe}(\text{OH})_3$  mixt. (1:2) was added to soil, the sol. As content of the soil was 0.8 p. m.  $\text{NaNO}_3$ , and KCl did not increase the soly. of As in the soil. C. R. Fellers

The present condition of a previously limed soil. R. H. Ganssen. *Fortschr. 11*, 365-71 (1935).—Distinct differences were found after 20-30 years on limed sandy soil plots as compared with unlimed soil. Humus showed a greater lasting effect with respect to  $\text{CaO}$  content and pH than did the underlying mineral soil. Trees on plots that had been limed and deeply plowed made a greater growth than under either unlimed or limed conditions without plowing. I. C. Feustel

Relation of lime application to availability of potash in soil. H. P. Cooper. *S. Car. Agr. Expt. Sta., 47th Ann. Rept.* 29 (1934), cf. *C. A.* 28, 7402.—Heavy applications of lime reduced the availability of the soil K. Following alfalfa, K deficiency was often observed in the cotton crop. Where the rate of application of limestone ranges from 1500 to 3000 lb. per acre there is little danger of having serious K deficiency in S. Carolina on better soil types. C. R. Fellers

Studies on the heavy limestone soils of Cuba. A. Bonazzi. *Proc. Assoc. Tec. Azucareros Cuba* 8, 47-75 (1934).—The heavy limestone soils studied are characterized by extreme impenetrability to  $\text{H}_2\text{O}$  and gases and excessive retention of  $\text{H}_2\text{O}$  once they have been wet. Residual  $\text{H}_2\text{O}$  at the permanent wilting point for sugar cane in an Oriente soil was found to be 48.7% on the basis of the oven-dry soil. The permanent wilting point and the sticky modulus of these soils coincide. The Fe minerals in these dense clays are often in the ferrous condition, an index of pronounced deaeration. O. W. Willcox

Nitrification in acid soils. M. M. Alicante. *Philippine J. Sci.* 58, 163-71 (1935).—Tests on a brown silt loam show that in soil having an acid concn. of over 0.5% the activity of nitrifying bacteria is inhibited. Nitrification in the soil increased as the acidity of the soil decreased until at an acidity of 0.009, 90.70% of the added N was converted into nitrate. During nitrification the soil acidity decreased. Tests in nutrient soln. show that nitrate production is not obtained in all cultures having an acid reaction, while a very slight degree of nitrification was observed in cultures of neutral reaction. Normal nitrate production occurred in these last when  $\text{CaCO}_3$  was added. A certain degree of nitrification can take place in an acid soil when both acid and carbonate radicals are present. The degree of nitrification depends on the amt. of carbonates present. J. R. Adams

The sulfur in seaweed, sea mud and rain waters in Brittany. Vincent, Sarazin and Hervieux. *Compt. rend. acad. agr. France* 21, 1032-4 (1935), cf. *C. A.* 29, 6689.—Along the coast, the rains, which have been enriched in sulfates and chlorides from the sea by wind-blown spray, may add sufficient S for crop requirements. Both marine plants and mud contain large quantities of S and can be used advantageously as fertilizer. The use of the mud should be preceded by sufficient liming in order to increase the speed of oxidation. J. R. Adams

The alkaline soil problem in China. Yih Hsiung. *Chemistry (China)* 2, 763-78 (1935).—A general survey of the distribution and qualities of alk. soils (or more accurately, soils rich in inorg. salts) in China, together with soil analyses and their suitability for the growth of agricultural products, is given. C. L. Tseng

Some chemical and biological changes produced in a For sandy loam by certain soil-management practices. L. S. Carter. *Soil Sci.* 40, 223-36 (1935).—Soils from 8 plots of For sandy loam were used for study. The soil was strongly acid and all plots were treated with  $\text{CaCO}_3$  before the beginning of field expts. Limestone plus complete fertilizer and lime plus phosphate and potash were the most effective in increasing the content of volatile

matter and N of the soil. Other fertilizer treatments decreased the N content. All treatments increased the  $\text{pH}$  value and the availability of P. All soil treatments increased crop yield and all increased the nos. of bacteria and fungi. The rate of nitrification of urea, several  $\text{NH}_4$  salts and cottonseed meal by one of the soils was studied. The rate varied widely for the different N compounds but in general the soils having a high content of available P had a high nitrifying power and gave increased crop yields. Production of  $\text{CO}_2$  from cellulose by the various untreated soils was decreased by the addn. of  $\text{CaCl}_2$  or  $\text{KCl}$  but was increased by  $\text{Na}_2\text{HPO}_4$  or  $\text{NaNO}_3$ . With fertilized soils the order was, in some cases, reversed. Yields of sweet clover, rye and soybeans grown in jars of the different soils in the greenhouse correlated well with crop yields under field conditions. When the crop material grown on each jar was ground and an aliquot returned to a portion of the soil from the jar,  $\text{CO}_2$  production correlated in general with crop yield.

**Soil fertility in the Sudan Gezira** N W Barritt *Empire Cotton Growing Rev.* 12, 111-16 (1935) —A review of investigations relating to aeration, respiration and nitrate formation in Gezira cotton soils. K D J

**Preliminary investigations on the phosphoric acid supply in the soils of the Bahim permanent experiments** Ahmed Mahmoud *Roy Agr. Soc. (Cairo) Bull.* No 20, Chem. Sect. 47 pp (1934) (available in English) —Comparison of the Dyer method (soly in 1% citric acid soln without neutralizing the  $\text{CaCO}_3$ ) with the method of Sigmond (soly in weak  $\text{HNO}_3$  after neutralizing  $\text{CaCO}_3$ ) showed that the latter gave much higher results for available  $\text{P}_2\text{O}_5$  but no significant difference between soils fertilized by phosphate and those not so treated. Available  $\text{P}_2\text{O}_5$  detd. by Dyer's method in 3 soils receiving no fertilizer since 1912 was 0.018, 0.016 and 0.018%, in 3 receiving nitrate only since 1912, 0.018, 0.016 and 0.020%; in 2 receiving nitrate and phosphate over the same period, 0.031 and 0.027%, and in 3 receiving nitrate, phosphate and potash since 1912, 0.032, 0.032 and 0.030%  $\text{fm}$ , sol. salts, and exchangeable bases were detd. on these same soils and mech. analyses were made on one receiving no treatment, one receiving  $\text{NaNO}_3$  and one receiving nitrate plus phosphate. The amt. of available  $\text{P}_2\text{O}_5$  soln in 1% citric acid which denotes phosphate deficiency was 0.02% or less. A soil receiving all 3 fertilizers gave the highest available  $\text{P}_2\text{O}_5$  when sampled before cotton, while one soil receiving only nitrate and one no treatment gave highest results after harvest. These differences were slight. Dyer's method can be used where the  $\text{CaCO}_3$  content is 3-5%, as in most Egyptian soils. Pot tests by the Mitscherlich method were made on various soils each receiving continuously (in the field) the same treatment from 1912 to 1930. Bahim soils, originally rich in phosphate become deficient after several years without phosphate application. These show a rapid recovery after dressing with superphosphate. Soil rich in N behave the same as unfertilized soils in respect to  $\text{P}_2\text{O}_5$ . Soil low in N recover rapidly on including berseem in the soln and allowing 4 months fallow. Application of these results to farm practice is discussed. Colin W. Whitaker

**The function of cellulose and lignin in the preservation of nitrogen in soils and in composts** Selman A Waksman and L. J. Hutchings *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 163-7 (1935) —Sand cultures with addns. of cellulose from filter paper, alk. lignin from straw, or ground straw, the usual mineral elements, and N in  $(\text{NH}_4)_2\text{HPO}_4$  or casein, and inoculated with fresh soil infusion, were incubated at 25-28° for 6 months. Org. matter and residual cellulose,  $\text{Pn}$  and N ( $\text{NH}_4$ , nitrate and total) were detd. Conclusions: The preservation of N in a sand medium depends largely upon the form of N added and the nature of the org. humus-forming substances. The function of carbohydrates in plant residues is to furnish energy for microbial activity in synthesis of cell substance, and transformation of N compounds, to protein. Lignin acts as a buffer for absorption of  $\text{NH}_4$  and direct fixation of some protein in resistant humus. Casein favored development of bacteria,  $(\text{NH}_4)_2\text{HPO}_4$  favored

filamentous fungi. Less humus was formed and more N lost from the former.

**Penetration of fertilizers** J R Cooper, Ark. Agr. Expt. Sta., *Bull.* 312, 46 (1934) —After 4 months, N from  $\text{NaNO}_3$ ,  $(\text{NH}_4)_2\text{SO}_4$ , Ammophoska and Nitrophoska was found in increased amts over check to a depth of 2 ft, the limit of the sampling. K was found in increased amts, to practically the same depth in most cases. P showed very little penetration in any form. Superphosphoska gave only a penetration of 1 in in 4 months; when used with sol. nitrates or lime, the penetration was even less. Ammophoska and Nitrophoska showed a penetration of 2 in in 4 months. On Ruston fine sandy loam at Hope, Ark., there was 1 in greater penetration for superphosphoska with  $\text{NaNO}_3$ , and with  $(\text{NH}_4)_2\text{SO}_4$  with and without lime, and 1 in less for Nitrophoska, than at Fayetteville.

**Micropopulations correlated with decomposition processes** Charles Thom *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 160-3 (1935) —Microbial decomposition of plant residues in soil is discussed in the light of recent investigations. C J Schollenberger

**Short period fluctuations in bacterial numbers in soil** H G Thornton and C B Taylor *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 175-9 (1935) —Studies on Rothamsted soils have revealed no simple relation between bacterial nos and moisture or temp. Even under const. environment and absence of protozoa there are fluctuations which cannot be explained. C J S.

**The effect of different media on soil-protozoan counts** Annie Dixon *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 179-80 (1935) —Soil-ext. agar is superior to peptone agar as a medium for the study of the soil protozoan fauna. C J Schollenberger

**Influence of the absolute reaction of the soil solution upon the growth and activity of Azotobacter** P. L. Ganev *Kan. Agr. Expt. Sta., Rept.* 1932-4, 28-30 (1934), cf. C. A. 28, 2000 —Continued studies show that "spots" in wheat fields produce greater growth of wheat contg. 1 00% more N than wheat from other parts of the fields. These nitrate spots contain few *Azotobacter*; hence the accumulation of N cannot be attributed to these organisms. Artificial "spots" can be reproduced by the addn. to the soil in the spring of urea,  $\text{CaCN}_2$  and other sol. N compounds. C R Feller

**Some observations on the Aspergillus niger method.** A M Smith *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 171-3 (1935) —Details in technique and interpretation of the biochem. test for soil fertility by *A. niger* (Nakriss, et al., C. A. 24, 5915), its advantages and disadvantages are discussd. C J Schollenberger

**Antagonistic action of soil microbes with special reference to plant hygiene.** Iwao Hino, *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 173-4 (1935) —Expts. on antagonism toward plant pathogens by soil microorganisms are reviewed, and practical applications discussed. C J Schollenberger

**The decomposition of pentosans by soil and manure microorganisms.** Jadwiga Ziemiańska, *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 167-8 (1935) —Plant residues contain 10-30% pentosans and other hemicelluloses. Their decompn. is by a specific microflora of bacteria and molds, the process may result in denitrification and loss of N, and therefore be unfavorable to the N balance in soils and manures. C J Schollenberger

**Occurrence and activities of fungi in Iowa soils.** F. B. Smith, Ia. Agr. Expt. Sta., *Rept. Agr. Research* 1934, 129 (1934); cf. C. A. 29, 3000 —*Aspergillus terreus*, *Trichoderma lignorum* and *Penicillium ranuncum* utilized xylan and cellulose but not lignin as sources of C. The presence of lignin in the culture medium decreased the amt. of  $\text{CO}_2$  produced by these fungi. *Aspergillus niger* decompd. xylan readily in a sand culture medium but to a much lesser extent in a culture soln. This organism attacked cellulose in soil cultures. A soil infusion slowly decompd. lignin in both sand and soln. cultures.

**Report on agricultural conditions in the Rupununi** C R Feller

district and Pakarama Mountains R R Foltett-Smith and A de K Frampton *Agr J British Guiana* 6, 155-84(1935) —Analyses of the soils and pasture grasses are given K D Jacob

Plant nutrition investigations H H King and A T Perkins *Kan Agr Expt Sta, Rept* 1932-4, 28(1934), cf *C A* 27, 1701 —Large amts of Fe in the soil decidedly reduced the percentage of P in the wheat plant P applications increased the amt of P absorbed by wheat plants on Fe treated soils, but such applications did not increase absorption on untreated soils  $\text{CaCO}_3$  appreciably decreased the soil acidity but had no consistent effect on the available P content of either acid or alk soils  $\text{CaCO}_3$  applied to the soils decreased the percentage of ash in the plants grown on the acid soils but increased it in plants grown on the basic soils  $\text{CaCO}_3$  decreased the percentage of N, K and P in the dry matter of lettuce and lima beans The Ca content of plants grown on basic soil was increased by the addn of superphosphate, this was not true for acid soils C R Tellers

Fertilizer reaction, soil amendments and crop production H B Mann and J J Skinner *Trans 3rd Intern Congr Soil Sci, Oxford, 1935* 1, 196-8(1935) —The trend of fertilizer use is toward materials leaving an acid residue in the soil and contg little Ca or Mg For soils naturally acid and deficient in Ca and Mg, neutralization of potential acidity of the fertilizer by the addn of dolomite is important C J Schollenberger

Humus investigations The formation of humus and the decomposition of organic matter in the soil F B Smith *la Agr Expt Sta, Rept Agr Research* 1934, 132-4 (1934), cf *C A* 29, 1201<sup>1</sup> —Significant differences were found in the concns of  $\text{CO}_2$  in the soil air under different crops In some cases plants exert a stimulating effect on soil microbial action as measured by  $\text{CO}_2$  formation Legumes produce no more  $\text{CO}_2$  than do other crops There was a slight depressing action from blue grass *Aq* exts, 1, 5, were prepd from 9 typical *la* soils and examd for colloidal matter, P, N, K, Ca, Na and other constituents There was a direct correlation between P, N and C in all 9 soils, i. e., when the N was high, C and P were also high and when 1 of the elements was low, the other 2 were also low There was an inverse relationship between  $\text{SiO}_2$  and the sesquioxides When the  $\text{SiO}_2$  was high, the sesquioxides were low and vice versa The water sol minerals were not present in the soil ext in the same proportions that exist in the soil itself Exchangeable bases were directly related to loss on ignition in Webster clay silty loam The exchangeable bases Na, K and Ca were unrelated to the amts of the same constituents found in the soils or soil exts The C/N ratio followed the same trend as the loss on ignition and the exchange capacity of the same soils The  $\text{pH}$  values of both soils and soil exts were practically the same The soil colloids, when used in various proportions as bacterial nutrients, stimulated *Azotobacter*, *Rhizobium* and *Radiorhizobium* Conclusion Colloids may play an important role as nutrients and as a habitat for the soil microflora C R Tellers

The effect of ammonium calcium balance on plant nutrition A H Lewis *Trans 3rd Intern Congr Soil Sci, Oxford, 1935* 1, 195-6(1935) —The recently increased use of concd fertilizers contg no Ca, e. g.,  $\text{NH}_4\text{H}_2\text{PO}_4$ , has caused germination trouble on acid soils Addn of a little  $\text{CaSO}_4$ , or better  $\text{CaCO}_3$ , has obviated damage Mangold and cereal seeds have sufficiently large reserves of carbohydrates not to be affected by physiol toxicity due to excess of  $\text{NH}_4^+$  C J Schollenberger

Some physiological studies with calcium cyanamide and certain of its decomposition products R M Smock *Ohio Agr Expt Sta, Bull* 555 46 pp (1935) —From 2 to 5 days following com  $\text{CaCN}_2$  treatments the  $\text{NH}_4$  contents of the roots of both tomato plants and peach trees in soil cultures increased The greater the colloidal or org contents of the growing medium, the more quickly did an increase of  $\text{NH}_4$  appear in the roots The persistence of this increase continued for 15 to 25 days Following the increase in  $\text{NH}_4$  nitrogen content by 1 or more days, there was an increased amt of nitrate It is unlikely that urea

serves as a direct source of N for plants in soil cultures following treatments with  $\text{CaCN}_2$  Tomato plants treated with  $\text{CaCN}_2$  had more  $\text{NH}_4$ , total sol, and nitrate- and  $\text{NH}_4$  free sol N than untreated plants in 2 soil types The utilization of  $\text{CaCN}_2$  is essentially an  $\text{NH}_4$  and nitrate phenomenon Injury by such decomn products as  $\text{CNNH}_2$ ,  $(\text{CNNH}_2)_2$ ,  $\text{NH}_4\text{C}(\text{NH}_2)_2$  and  $\text{H}_2\text{C}_2\text{N}_2\text{O}$  (guanlyl urea) on the peach, apple and tomato was detd Injury by  $\text{CNNH}_2$  to the tomato was accentuated by the addn of nitrate to the nutrient soln The presence of nitrate N did not increase injury due to  $(\text{CNNH}_2)_2$ , with the tomato  $\text{CNNH}_2$  is a potential source of injury to the peach, apple and tomato with com cyanamide applications on soils devoid of colloidal or org matter The  $(\text{CNNH}_2)_2$  is injurious only with extremely heavy applications or on alk soils Ca bentonite was an effective catalyst of the  $\text{CNNH}_2$  to  $\text{OC}(\text{NH}_2)_2$  change in sand-colloid mixts, but it was slightly inferior to animal charcoal in this capacity Guanidine and guanlylurea are unlikely sources of injury when com cyanamide is applied to the soil Three years' expts show com cyanamide is a satisfactory source of N for the apple tree when properly used Fifty-two references C R Tellers

Effect of fertilizers on various properties of a highly calcareous soil and on the yield and quality of crops produced D W Pittman *Utah Agr Expt Sta, Bull* 250, 17(1934), cf *C A* 28, 5164<sup>4</sup> —Treble superphosphate gave marked increased yields on sugar beets, barley and wheat while  $\text{Ca}_3(\text{PO}_4)_2$  showed no residual effect  $(\text{NH}_4)_2\text{SO}_4$  gave considerable increases in yields of wheat and barley Neither S nor  $\text{CaSO}_4$  showed any beneficial effect on crops P, nitrate and org matter increased appreciably in the surface soil as a result of a long-continued annual application of fertilizer and manure The soil reaction remained at approx 7.5 throughout the expt C R Tellers

Analysis of the factors responsible for loss of nitrogen and organic matter from dry-lands A F. Bracken and J E Greaves *Utah Agr Expt Sta, Bull* 250, 21 (1934) —When results from virgin areas were compared to adjacent cropped fields, it was found that approx 20% of the N and org matter had been lost from the tilled soil Only 1/4 of this loss has been removed by the crops, leaving the balance unaccounted for Legumes maintained the N of dry land at approx the same level as that found in uncropped soil The study is being continued in an effort to det the factor responsible for these losses in nutrients C R Tellers

Soil acidity and liming and fertilizer recommendations for various crops H P Cooper and W. R. Paden *S Car Agr Expt Sta, 47th Ann. Rept* 19-24(1934), cf *C A* 29, 5673<sup>1</sup> —The av equiv acidity of av. mixed fertilizers used in S Carolina is approx 150 lb of limestone per ton The use of such fertilizers is a potent factor in the increased soil acidities of the South Heavy liming is unnecessary and may cause serious Mn deficiencies The soils are classified as to  $\text{pH}$  C R Tellers

Fluorine, its effect as plant growth and its relation to the availability to plants of phosphorus in phosphate rocks R P Bartholomew *Soil Sci* 40, 203-17(1935) —Concs of F as high as 50 p p m had very little influence upon the germination of seeds of Sudan grass, cowpea, soybean or red clover The germination of white Dutch clover was greatly enhanced by the presence of F in the form of  $\text{CaF}_2$  and  $\text{Na}_2\text{SiF}_6$  The addn of sol fluorides up to 10 p p m in culture soln produced no consistent decrease in the amts of dry matter of cowpeas The variations were attributed to differences in the plants F in plants was found largely in the roots, only when the amts of F in the roots were relatively large was any present in the tops Considerable F was taken up by the roots of cowpeas grown in culture solns contg as little as 0.25 p p m of F in the form of  $\text{NaF}$ , while no F was taken up from solns contg as much as 0.50 p p m of F in the form of  $\text{CaF}_2$  The presence of 10 p p m of F in the form of  $\text{Na}_2\text{SiF}_6$  in the solns produced roots with as much as 1970 p p m of F The presence of chemically combined F in phosphate rocks greatly affected the availability of the

P to plants. The availability of P decreased as P content increased. The effect was most apparent in the first crop. There was a definite relation between P content of the various phosphate rocks and of basic slags and the total amt of dry matter produced. Evidence substantiated the theory that the availability of P in rock phosphate is largely a matter of the rate of soln of the rock phosphate.

M. S. Anderson

**Effect of fluorine content on availability of rock phosphates.** R. P. Bartholomew. *Ark Agr Expt Sta. Bull.* 312, 20-1 (1934), cf. *C. A.* 29, 5577. The yields of Sudan grass produced from applications of these phosphates showed a definite correlation between the percentage of F in the rock phosphate and the amt of dry matter produced in the 1st cutting. Data for the 2nd cutting were not significant. There was a significant correlation between the amt of P absorbed by the plants and the amt added in the rock phosphate. Availability of rock phosphates depends to a considerable extent on their F contents. In concns. up to 50 p. p. m. F as  $\text{CaF}_2$ , NaF or  $\text{Na}_2\text{SiF}_6$  had no effect upon the germination of Sudan grass, cowpeas, soybeans, red clover or Dutch white clover. Ten p. p. m. of F as NaF,  $\text{CaF}_2$  or  $\text{Na}_2\text{SiF}_6$  did not depress the yield of cowpeas. C. R. Teller

**The exchangeable manganese in Danish soils and its relation to plant growth.** I. Steenbjerg. *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 198-201 (1935), cf. *C. A.* 29, 7549. Gray speck disease of oats, associated with Mn deficiency, is serious on the sandy soils of Denmark, especially when they are limed to excessive  $\text{pH}$  values, which reduces the availability of Mn. Recommendations for liming are therefore based upon a detn. of  $M_n$ , "manganese value," mg Mn displaceable from 1 kg dry soil by  $M\text{g}(\text{NO}_3)_2$ , calcd. from the Mn content of successive leachings of a sample. Expts. have shown that not only  $M_n$  is important, but also  $q$ , a measure of the retention of exchangeable Mn by soil colloids.  $M_n$  is decreased by liming to high  $\text{pH}$ , excessive aeration and deficiency of moisture, and is increased by chem. reducing agents and readily decomp. org. matter, e. g., starch, and by water-logging.  $q$  is decreased by the latter factors. The heavier soils tend to high values for  $M_n$  and low for  $q$ ; hence gray-speck disease is almost unknown on them. Field and lab. studies have shown a high degree of correlation between  $M_n$ ,  $q$  and incidence of gray speck of oats on soils liable to the trouble. A quant. relation between  $M_n$  and  $q$  has been demonstrated and may be formulated for a particular soil, but the math. const. are different for other soil types. C. J. Schollenberger

**Are plants able to separate the isotopes of potassium from soils?** Oskar Eckstein. *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 186-9 (1935) (in German). Pots of sand were repeatedly cropped to oats and rye, with complete fertilization for each crop except with respect to  $\text{K}_2\text{O}$ , which was supplied by an initial heavy application of  $\text{K}_2\text{O}$  salts derived from molasses. The at. wt. of the K in the successive crops was detd. and compared with that of K from sylvinite. Owing to analytical difficulties, the values obtained, 39.15-39.18, were all too high, but the differences were insignificant, the possibility of Rh being present was excluded. Seven references.

C. J. Schollenberger

**The effect of manganese deficiency on oats, in relation to soil bacteria.** F. C. Gerretsen. *Trans. 3rd Intern. Congr. Soil Sci., Oxford, 1935* 1, 189-91 (1935). Evidence that gray speck disease of oats is not due solely to Mn deficiency is reviewed. G. believes that the disease is bacterial; plants with abnormal metabolism due to lack of available Mn are unable to synthesize org. acids sufficiently to neutralize  $\text{NH}_3$  produced by the bacteria in the roots; hence the severity of the symptoms and fatal outcome. Lack of carbohydrates in the leaves weakens them so that they bend in the characteristic manner. Vigorous plants growing under normal conditions are little affected by the bacteria. C. J. Schollenberger

**Phosphatic fertilizers, comparative trials on immediate and residual effects.** Ahmed Mahmoud, *Roy. Agr. Soc. (Cairo) Bull.* No. 21, Chem. Sect. 30 pp (1931) (available

in English).—Details are given of a comprehensive series of field expts. testing the relative merits of superphosphate (16% available  $\text{P}_2\text{O}_5$ ), concd. superphosphate (40%  $\text{P}_2\text{O}_5$ ), basic slag (10%  $\text{P}_2\text{O}_5$ ), Pphos (30% total  $\text{P}_2\text{O}_5$ , a mineral phosphate), Thiophosphate (17% total  $\text{P}_2\text{O}_5$ , a mixt. of ground rock phosphate, gypsum and S), Sulphurophosphate (16% total  $\text{P}_2\text{O}_5$ , a French product consisting of mineral phosphate and S) and bone meal (29% total  $\text{P}_2\text{O}_5$ ) on bersim, bersim fahl, cotton, wheat and maize. The soil used contained, before application of the fertilizers, 0.23%  $\text{P}_2\text{O}_5$  sol in 1% citric acid without neutralization.  $\text{CaCO}_3$  present was 2.55-2.61% and  $\text{pH}$  of the soil about 7.0. The rate of application was 32 kg  $\text{P}_2\text{O}_5$  per 4200 sq m. The 2 superphosphates were the most effective. Basic slag was the next most effective. Phosphates contg. their  $\text{P}_2\text{O}_5$  in insol. form showed no immediate effect or even a residual one over a period of 5 years. Such phosphates are not suited for Egyptian soils. Pot tests confirmed the above findings. Recommendations for phosphate application are given. Colin W. Whittaker

**Phosphate rock as filler substitute in fertilizer mixtures.** R. L. Smith. *Com. Fertilizer* 51, No. 5, 11, 16, 18, 20, 22, 44 (1935).—In pot tests with barley, turnips, soybeans and corn on Norfolk sandy loam and Cecil sandy loam soils, there were no significant differences either in crop yields, soil reaction or compn. of plant material (Ca, Mg and P contents) when ground phosphate rock and dolomite, resp., were used as fillers in the prepn. of neutral fertilizer mixts. Only a small increase in the available P content occurred when a 4-8-4 fertilizer (contg. KCl, superphosphate,  $(\text{NH}_4)_2\text{SO}_4$  and phosphate rock filler) was stored under temp., pressure and moisture conditions simulating those occurring in a fertilizer curing pile, but the available  $\text{P}_2\text{O}_5$  content increased 1.00-2.45% when the mixt. was held for 7 days at 80° under a pressure of 80 lb./sq in. The amt of phosphate rock dissolved by buffered solns. increased with the acidity of the solns. Solns. of  $\text{pH}$  4.5-6.0 dissolved appreciable amts. of phosphate rock. In pot tests on Norfolk sandy loam soil of approx.  $\text{pH}$  5.0, phosphate rock supplemented with  $\text{CaSO}_4$  produced yields of turnips and soybeans that compared favorably with those obtained by the use of superphosphate.

K. D. Jacob

**More on the question of the most suitable nutrient ratio in Nitrophoska (containing lime).** E. Blanck and H. S. Horstner. *J. Landw.* 83, 327-33 (1935).—Previously reported expts. (*C. A.* 30, 2109) were repeated on Börsinghauser soil and showed the most favorable nutrient ratio to be  $\text{N} : \text{P}_2\text{O}_5 : \text{K}_2\text{O} = 1.1 : 1.75$ . John O. Hardesty

**Zinc sulfate studies in the soil.** Owen E. Gall. *Citrus Ind.* 17, No. 1, 20-1 (1936).—When  $\text{ZnSO}_4$  was applied to different types of soils absorption of Zn ions was highest in soils of a high colloidal content, high Ca content or contg. large amts of org. matter. In pot tests with corn and cowpeas on Norfolk fine sand an application of  $\text{ZnSO}_4$  equiv. to 500 lb. Zn per acre was markedly toxic to cowpeas, but corn was not affected until the concn. reached about 700 lb. Zn per acre. The toxicity of large applications of  $\text{ZnSO}_4$  was reduced by the application of either  $\text{CaCO}_3$  or  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ . Samples of soil were taken at depths of 0-3 in. and 3-6 in. from Zn-treated citrus groves at intervals of 1 week, 11 weeks and 7 months after application of the  $\text{ZnSO}_4$ . After 1 week, during which time no rain fell, most of the Zn was recovered in the  $\text{H}_2\text{O}$ -sol. condition. 14 out of 15 in. of rain fell before the next sampling and very little  $\text{H}_2\text{O}$ -sol. Zn was found in the 1st 6 in. of soil; a portion of the Zn was converted into the replaceable condition and the rest was leached from the soil. After 7 months comparatively little Zn was found in either horizon; there was a small but definite accumulation of replaceable Zn in the 3-6 in. layer of soil.

K. D. Jacob

**The effect of the digestion of sludge on the germinating power of weed seeds.** K. Viehl. *Gesundh.-Ing.* 59, 35 (1936).—Anaerobically digested sludge possesses many advantages over fresh sludge as a fertilizer. Loss of N is much less during anaerobic digestion than during the decompn. of the fresh sludge after it is on the land. Two l. of raw sludge from the Leipzig plant was mixed with 1 l.

of digested Imhoff tank sludge, known amts. of various kinds of seeds were added and the whole was anaerobically incubated at room temps., the  $pH$  being maintained at 6.5-7.5 by the addition of small amts. of lime. Before incubation and at intervals thereafter 2.0 g. of the mixt. was spread on sterile soil and covered with a thin layer of the latter. The no. of the various kinds of plants growing was recorded. With the exception of tomato seed, which was practically unaffected after 10 weeks' digestion, the germinating power of the seeds rapidly decreased. The nos. of plants growing before incubation, after 4 weeks and after 10 weeks were: tomato 32, 25, 25, rape 38, 17, 0; *Phalaris* 43, 16, 0; millet 29, 3, 1; head lettuce 24, 0, 0; *Stellaria* 34, 4, 0; grass 4, 1, 0. M. G. Moore

Composts and fertilizers in relation to greenkeeping. VI. Miscellaneous fertilizers [poultry manure and sewage sludge]. T. W. Evans. *J. Board Greenkeeping Research* 4, 104-7 (1935), cf. *C. A.* 30, 2129. Dried sewage sludge from Bradford, England, contains mineral matter 52.7, org. matter 46.3, total N 2.51,  $P_2O_5$  0.30 and  $K_2O$  trace. Sludges from other English cities usually contain about 1% less N. The Bradford sludge owes its high N content to waste waters from the wool-scouring industry.

K. D. Jacob  
The effects of sodium chloride on some turf plants and soils. V. T. Stoutemyer and F. B. Smith. *J. Am. Soc. Agron.* 28, 16-23 (1936).  $P_2O_5$  stimulated the growth of 4 grasses and a white clover, while NaCl in the concns. used was toxic in some cases and stimulative in others. When applied with  $P_2O_5$ , the NaCl toxicity was decreased. The total exchangeable base content of the soil was decreased by the treatment with NaCl. It appears that NaCl alone or in combination with  $P_2O_5$  fertilizers may be used on sandy soils for metropolitan and seaside belt grasses.

J. R. Adams  
Minerals in pasture grasses in India. A. Viswanatha Iyer. *Indian J. Vet. Sci.* 5, 184-90 (1935).—The factors affecting the contents of P, Ca, Mg, Na, K and N in *Andropogon contortus*, *Cynodon dactylon* and *Pennisetum cenchroides* were investigated. Plants grown on highly calcareous soil that was low in available P always contained more Ca and less P than the av. value; for the species, those grown on a red laterite soil that was deficient in Ca and tended to be acidic contained the normal amt. of P but were low in Ca. Each species of grass showed a tendency toward a mineral make-up peculiar to itself. *C. dactylon* invariably showed a considerable excess of Ca over  $P_2O_5$ , while in *P. cenchroides* the tendency was for  $P_2O_5$  to exceed CaO. The N and K contents of the grasses decreased markedly with advance in maturity, P decreased to a smaller extent, while the degree of maturity had only a minor effect on the Ca and Mg. There were indications that cultivation is likely to affect the mineral content of herbage.

K. D. Jacob  
The growth of pasture in the spring, and its relation to available nitrogen supply and temperature. G. E. Blackman and A. H. Lewis. *Trans. 3rd Intern. Congr. Soil Sci.*, Oxford, 1935, 1, 291-3 (1935).—From 3 years' yields of grass cut from pasture plots fertilized with  $(NH_4)_2SO_4$  and a variety of common N carriers, it is concluded that about 25 lb. N per acre hastened the growth to the grazing stage by 3-13 days in different years. These comparisons were rather favorable to  $NH_4$  salts. The N content of the herbage indicated that N was absorbed by the roots and translocated to the leaves when the soil temp. at 4 in. was not below 5°. The herbage reached its max. N content 7-10 days after active growth began. Soil analyses indicated marked fluctuations in the  $NH_4$  and nitrate content of the upper 3 in., especially when the soil temp. was less than 5°. In general, a few days after the application of fertilizers less of the added N could be found in these forms than later. Lab. expts. along the same line indicated that the temporary disappearance of inorg. N was due to microbial fixation, and that urea accelerates the breakdown of org. N by the soil microflora. When the soil temp. at 4 in. reached 10°, growth became so rapid on all plots that the effect of fertilizer N was no longer visible. The "degree of earliness" to be expected from N fertilization

varies directly with the length of time the soil temp. is 5-10°.

C. J. Schollenberger  
Studies on paddy cultivation. V. The effect of time of application of the fertilizer. The effect of the form of phosphoric acid applied. J. C. Haugh and A. W. R. Joachim. *Trop. Agr. (Ceylon)* 83, 269-77 (1935), cf. *C. A.* 29, 2641.  $NH_4$  phosphate was applied to 0.01-acre plots of transplanted paddy at the rate of 1 cwt. per acre. The yield from the plots which received the fertilizer at the beginning of the season was the same as that from plots which received equiv. amts. in several small applications throughout the growing period. Amts. of N and P removed from the soil by the crop were approx. the same for both methods of treatment. Whether the  $P_2O_5$  was applied as  $NH_4$  phosphate or steamed bone meal made no appreciable difference in yield or in available  $P_2O_5$  in the soil. John O. Hardesty

The relative rates of uptake of ammonium and nitrate nitrogen by perennial ryegrass. A. H. Lewis. *Trans. 3rd Intern. Congr. Soil Sci.*, Oxford, 1935, 1, 204-5 (1935).—Pot expts. with grass were conducted with artificial soil composed of 94% sand and 6% Ca bentonite, with fertilizers, to avoid complications from nitrification, almost nil in this medium. The growth and compn. of grass at different stages from pots fertilized with  $(NH_4)_2SO_4$ ,  $NaNO_3$  and  $Ca(NO_3)_2$  were compared. The results indicated greatest uptake of N from  $(NH_4)_2SO_4$ ; the effect upon yields depended upon the age of the grass.  $P_2O_5$  in the herbage was highest with  $(NH_4)_2SO_4$ ; possibly this explains the superior growth. The rates of uptake of  $NH_4$  and  $P_2O_5$  are related. L. believes that direct absorption of  $NH_4^+$  by grass roots is the normal process, especially in early spring.

C. J. Schollenberger  
The influence of rainfall on the yield of cereals in relation to fertilizer treatment. W. G. Cochran. *J. Agr. Sci.* 25, 510-22 (1935).—A statistical study indicated that rainfall and other meteorological effects on the seasonal variations in yield of wheat and barley are closely associated with fertilizer treatment. The fertilizers used include farmyard manure, ammonium salts and superphosphate.

Irvin C. Feustel  
Lysimeter experiments with barley, tea and rice. S. Osugi. *Trans. 3rd Intern. Congr. Soil Sci.*, Oxford, 1935, 1, 205-7 (1935).—A brief review of results from many years' expts. Losses of N were mainly as nitrate, very little  $NH_4$ , and were greater under a summer crop than in winter. A single heavy application of fertilizer resulted in much greater loss than the same in 3 portions. The N of mineral fertilizers was lost to the greatest extent, that of animal fertilizers least. Liming increased loss of N applied as  $(NH_4)_2SO_4$ , previous composting with the fertilizer decreased loss. Losses under tea were greater than under barley or land rice, and at a max. during the warmer seasons, other similarities to previous observations were noted. There was practically no loss of N in an expt. with paddy rice.

C. J. Schollenberger  
Manganese deficiency of cereals. Plot experiments and a new hypothesis. G. W. Leeper. *Proc. Roy. Soc. Victoria* 47, Pt. II, 225-61 (May 8, 1935); cf. *C. A.* 29, 1561. Wheat plants growing on a heavily limed soil suffered from lack of available Mn. This condition was improved by (1) acidification of the soil, most conveniently by S, to a  $pH$  of at least 6.5 or (2) heavy applications of  $MnSO_4$  to the soil. The final yield of grain averaged the best on the sulfured soil. Lab. tests on various soils showed that the crucial test among soils of  $pH$  greater than 7 is to leach the soil with a soln. of quinol at  $pH$  7, with N  $(NH_4)_2SO_4$  as the reagent. Deficient soils yielded less than 15 parts of Mn per million of soil to this soln., while 2 typical healthy soils tested gave over 130 p. p. m. The theory of this test is discussed in connection with the oxidation-reduction potential of the system  $MnO_2-H^+-Mn^{++}$ . It is suggested that the plant uses  $MnO_2$  as its source of Mn, whether directly in the colloidal state or by reduction at the root-soil interface, and it is only the  $MnO_2$  dissolved by quinol at  $pH$  7 which can be used by plants, and not the more copious supply that may sometimes be dissolved with or without quinol at low  $pH$  value (1 and 2).

The amt of Mn dissolved by quinol at  $pH$  7 may be taken as an indication of whether a given soil is liable to develop a Mn deficiency after liming. If sulfluring is impractical, as, e. g., on a very calcareous soil, an "active"  $MnO_2$  should theoretically be as good as  $MnSO_4$ , especially if finely divided and worked well into the soil. The state and translocation of Mn in the soil can be studied by leaching with a series of solns buffered to a const  $pH$  and poised to steady ranges of  $pH$  by a series of ingredients such as quinol. Addnl pot tests confirmed the evidence that the failure of surface application of Mn salts is due to "positional" unavailability, which may be overcome by thorough mixing. It also makes it more reasonable to expect that large applications of  $MnSO_4$  will bring about a permanent cure on any soil, if thorough mixing is possible. In all of this work, which is described with full details, the Mn was analyzed colorimetrically as the permanganate with a Duboscq colorimeter. The color was developed by  $NaOCl$ , usually in  $H_2SO_4$ , but sometimes in  $H_3PO_4$  soln if it was necessary to avoid the formation of large amts of  $CaSO_4$ . The plant ash was treated with HF to avoid loss of Mn as insol silicate and the remaining HF removed with  $H_2SO_4$ . The  $NH_4$  acetate solns were treated by first concg them, then destroying org matter and  $NH_4$  ions with aqua regia and finally destroying chlorides with  $H_2SO_4$  or  $H_3PO_4$ . Leopold Scheffan

The influence of fertilizing on the yield and quality of flax. F Alten and G Goetze. *Ernahr Pflanze* 32, 1 14 (1936).—Culture expts were carried out in pots conig 10 kg of glass sand, 1 5% purified peat mull and a const amt. of  $P_2O_5$ . The quality of the fiber improved with increasing amts of  $K_2O$  up to 2 g in conjunction with 0 4 g. of N per pot. Increasing applications of N from 0 2 to 1 g. increased the yield but impaired the quality of the fiber. A satisfactory yield of good quality fiber was obtained from pots receiving 1 g. of N and 3 g. of  $K_2O$ . John O Hardesty

Manganese deficiency in oats at Florence [S Carolina]. W. B. Albert. S Car Agr Expt Sta, 47th Ann Rept 45(1934).—Mn deficiency is becoming more prevalent in S Carolina Coastal Plains soils. In oats there is a breaking down of the basal portion of the leaves with the tips remaining green and alive for some time after the slow discoloring and death of the basal part occurs. C R Fellers

Rice nutrition. L C Kapp. Ark. Agr Expt Sta, Bull 312, 22-4(1934); cf. C A 29, 7400\*.—When grown in nutrient solns., vegetative and root growth were retarded at Fe concns of 6 25 p p m. The toxic effect of Fe was greater in the presence of  $(NH_4)_2SO_4$  than of  $NaNO_3$ . The  $(NH_4)_2SO_4$  soln depressed root growth and produced more stubby roots than  $NaNO_3$ . Mn was not as toxic to rice as Fe. Of the various fertilizer treatments used on submerged Clarksville and Crowley soils, only N carriers increased the yield of straw and grain as well as the N content of the plant. Addns of carbonaceous materials to submerged soils decreased  $NH_4$  accumulation and decompn. of nitrogenous materials. The sol ammonium and nitrate N are lost from soils on submergence in proportion to the carbonaceous materials present. C R Fellers

The effect of potassium supply on the composition and quality of wheat. I. A. G. McCalla and E K Woodford. *Can J Research* 13, C, 339-54(1935), cf. C A 28, 13832\*, cf. C A 29, 1463\*.—Limiting the supply of K to wheat plants resulted in a decreased N content and a markedly increased Ca and Mg content of the dry matter. It retarded growth, reduced the total amt of individual nutrients absorbed per plant and resulted in an increase in the proportion of grain to total yield. It had an adverse effect on the quality of the grain as detd by gluten and baking tests. Neither Na nor Ca was effective in replacing K, but Na was better in this respect. J W Shipley

Etiology and control of seedling blights and boll rots of cotton in Arkansas. V. H. Young. Ark. Agr Expt Sta, Bull. 312, 49-50(1934); cf. C. A. 29, 2289\*.—Sore shin disease, due to damping-off fungi, and angular-

spot disease caused by *Phytophthora malvacearum* were best controlled by early planting of seed treated with Ceresan, new improved Ceresan of  $CuSO_4 \cdot H_2O$ . However, the latter had an unfavorable effect on stand of seedlings. Less and less benefit was obtained by seed treatment as the planting season advanced. C. R. Fellers

Experiments with potash fertilizer for cotton. H. P. Cooper, W B Rogers and R. W. Wallace. S Car Agr. Expt Sta, 47th Ann Rept. 16-18(1934), cf. C. A. 28, 7404\*.—When K was applied in amts less than 45 lb. per acre, the time of application had no effect on yield, however, above 45 lb per acre better results were obtained if some of the K was applied before the crop was planted. Approx 15 lb of seed cotton results from 1 lb of K added to the soil. Cotton rust is due largely to K deficiency and can be corrected by the use of K. Rust is worse in dry years. Mg deficiency is usually more prevalent than K deficiency when the rainfall is heavy. Sol. sulfates, nitrates or chlorides increase the soly of Mg in the soil and enhance the loss of Mg in drainage water. C. R. F.

Effect of manganese sulfate on the yields of Irish potatoes. H P Cooper, W D Moore and R W Wallace. S Car Agr Expt Sta, 47th Ann Rept 141-2(1934).—Soils of the trucking areas of Charleston and Beaufort are often deficient in Mn and the addn of 50-100 lb per acre of  $MnSO_4$  gave greatly increased yields of potatoes. Basic slag contains sufficient Mn to serve as a good carrier of this element. C R Fellers

Influence of fertilizer on the quality of wheat. H. F. Murphy. Okla Agr Expt Sta, Rept 1932-4, 25 (1934).—P was the principal element lacking in Kirkland sandy loam. The addn of superphosphate to this soil resulted in plumper kernels, lighter berries and a lower protein content. Baking tests showed the bread baked from the wheat was not inferior. The P increased the ash and P content of the wheat berries over that produced on unfertilized plots. C R Fellers

The response of different varieties of potatoes to different amounts of copper in a modified spray program. E O Mader and F M Blodgett. *Am Potato J*, 12, 325-34(1935), cf. C A 29, 7567\*, 30, 218\*.—The Irish Cobbler, Rural Russet and Green Mountain varieties responded to Cu treatments in the form of Bordeaux mixt to approx the same extent in final increases in yields. With a Bordeaux mixt composed of the equiv. of half as much quicklime as  $CuSO_4$ , and when most of the Cu was applied early in the season, there appeared to be no advantage in using more than a total of 60 lb  $CuSO_4$ /acre during the season. All Cu-sprayed plants retained more tubers per plant than did unsprayed plants of the same variety. All varieties showed evidence of a retardation in tuber development due to spraying. The retardation was less pronounced with the Cobblers and most pronounced with the Green Mountains; the unsprayed plants of the latter yielded more than the sprayed until after Sept. 21. K. D. Jacob

Effect of fertilizers on the handling qualities of strawberries and tomatoes. I C Haut. Okla Agr. Expt Sta, Rept 1932-4, 245-7(1934).—Chem analyses do not detect differences in compn due to normal fertilizer treatments on strawberries. Applications of 500-1000 lb per acre of fertilizers of the general compn 10-5-5, had no effect on firmness or compn. C. R. Fellers

Sugar-cane physiology. V Contents of nitrogen, phosphorus and potassium in crude chlorophyll and in the leaf skeleton and their relationships with fertilizers. C E Beauchamp, F Lazo and A Bonazzi. *Proc. Assoc Tec Azucareros Cuba* 8, 82-114(1934); cf. C. A. 28, 4102\*.—Among 10 fertilizer formulas used in growing sugar cane in a clay similar to Matanzas clay, those producing the largest growth in the field were 4-4-8, 4-4-12 and 8-8-12. These formulas produced the highest chlorophyll content, and this in turn the highest total N, P and K. As K was increased in the fertilizer it was also increased in the chlorophyll, and the growth was increased. The ratio  $K_2O$  to N + P in the chlorophyll, in percentage of skeleton, was 2 1. Increasing N in the fertilizer P and K in the chlorophyll, resulting in reduced

Analysis of the leaf skeleton bore no relationship with growth, only the compn of the chlorophyll being of significance in this connection O W. Wilcox

Copper deficiency in sugar beets D A Van Schreven Meded Inst Suikerbeteenteelt 6, 37-57(1936)—Sugar beets grown in Cu-free nutrient solns showed pronounced chlorosis of the outer leaves and were  $1/4$  as large as the controls The Sachs I-KI test showed very little starch in the leaves of the diseased plants, whereas leaves of plants grown with Cu showed abundant starch There is now no doubt that Cu is essential in photosynthesis

O W Wilcox

Heart rot disease of sugar beets E Brandenburg Centr Zuckerrind 43, 837-8(1935), Mededel Inst Suikerbeteenteelt 4, 81-102(1935)—B, preferably in the form of borax, gives protection to beets even in the following crop year Addns of 0.40 kg per hectare reduced infestation from 65.7 to 0.1%, increased the yields of roots 28%, tops 51% and sugar 40% Even better results were obtained with B-fertilizer mixts Until the question is decided whether B is a mere stimulant, or a fertilizer, or probably both, large scale field use of B is not encouraged F R Bachler

The influence of forest agriculture on the physical and chemical properties of soil Antonin Nemeš Forstwiss Centr 57, 656-68(1935)—Comparisons were made of the properties of alluvial sand soils supporting trees without cultivation and those cultivated with potatoes between rows of trees Less  $P_2O_5$ ,  $K_2O$  and CaO sol in 20% HCl was found in the cultivated profile The content of  $Na_2O$ ,  $MgO$ , N and sesquioxides was greater in the latter than in the uncultivated plot Ibid 701-8—The compn of pine needles showed a higher content of N (1.299%) in the cultivated plots than in the uncultivated plots (0.862%) Little differences were found in  $P_2O_5$  and  $K_2O$  whereas the CaO content was less in the needles from cultivated plots Conclusion The 2-year cultivation with growing of potatoes did not appreciably impoverish the soil of  $P_2O_5$  and  $K_2O$  I C Feustel

Water and mineral requirements of tree seedlings L M Turner Ark Agr Expt Sta, Bull. 312, 47-8 (1934)—Seedlings of *Pinus echinata* and *P. taeda* can grow satisfactorily in pure sand cultures with as little as  $1/2$  of the amt found satisfactory for seedling apple trees in sand cultures High N applications have a toxic effect on young pine trees The optimum water requirement for germination and growth the 1st year was 1.6 to 2 times the mean rainfall for the region C R Fellers

Leaf analysis as a means of diagnosing nutrient requirements of tropical orchard crops F Hardy, J A McDonald and G Rodriguez J Agr Sci 25, 610-27 (1935)—The compn of the leaf ash of cacao is entirely different from that of grapefruit growing on alluvial soils in Trinidad Although the lime content of the cacao soil is somewhat higher than that of the grapefruit soil the lime content of the cacao leaf ash is much less than that of grapefruit Leaf lime content bears a strict and highly significant reciprocal relationship with leaf potash content Fertilization considerably increased the content of leaf ash but results indicate greater changes in leaf compn of grapefruit compared with cacao Statistical evidence shows that a close relationship holds between leaf compn and yield of cacao The best indices of yield appear to be the leaf nutrient ratios, particularly, a low N/ $K_2O$  ratio, a high  $K_2O/P_2O_5$  ratio and a high N/ $P_2O_5$  ratio By comparing given analytical data with those of leaf material produced by "ideal" trees the particular fertilizer treatment which should confer most benefit may readily be identified I C Feustel

Fertilizers for tobacco J M Carr Ca Coastal Plain Expt Sta, 14th Ann Rept 94-8(1934), cf C A 28, 5577—Standard fertilizer mixts for the Coastal Plains areas are 8-3-5 and 8-3-8 applied at the rate of 800-1200 lb per acre P deficiency in tobacco is shown by a small abnormally green plant which never ripens properly Excessive P causes premature ripening or firing in the field Superphosphate is a satisfactory source of P Tobacco fertilizers should carry 3-4% of  $NH_3$  Horse

manure gives good results as a sole source of  $NH_3$  for tobacco When com sources are used,  $1/4$  should be from high-grade org. materials and either  $1/4$   $NaNO_3$  or  $1/4$   $NaNO_2$  and  $1/4$  urea or Ca( $NO_3$ ) $_2$  The fertilizer should contain from 5 to 8%  $K_2O$  The K recommendation includes 2%  $K_2O$  per ton from high-grade KCl and the remainder from  $K_2SO_4$  The functions of Ca, Mg and S to quality are discussed C R Fellers

Sodium arsenate as a weedicide G R W. Meady J Dept Agr Western Australia 11, 521-3(1934)—A mixt suitable for killing green timber is prep'd by dissolving 1 lb washing soda in a convenient amt of water and adding to it slowly and with const. stirring 1 lb  $As_2O_3$  made into a paste with water The mixt. is boiled until the  $As_2O_3$  is dissolved, a thin paste of 0.5 lb whiting in water is added and the mixt is dild to 4 gal The best results are obtained by applying the prep'n to dormant trees in a "frill ring" cut into the trunk as near the ground as possible A tree 4 ft in diam requires approx 1 quart of soln. K D Jacob

Insecticides and insect toxicology C H Richardson Ia Agr Expt Sta, Rept Agr. Research 1934, 96-7 (1934), cf C A 29, 26521—Paris green was most toxic, Pb arsenate second, and Ca arsenate least toxic to the larvae of the imported cabbage worm, *Pieris rapae* L. Toxicity was det'd as the dosage which kills 50% of the population (median lethal dose). Pb arsenate was somewhat more toxic to *Dalania ministra* (an apple moth) than to its close relative *D. perspicua* The toxicities of Pb arsenate,  $Zn_3P_2$  and diphenylene oxide were in the ratio of 20.7:1 *Dihydrorotenone* is more stable than rotenone, and while it possesses considerable toxicity to some insects, it is less toxic to *D. ministra* than Pb arsenate.  $Zn_3P_2$  was toxic to the differential grasshopper, *Melanoplus differentialis* Thomas, but was much less toxic to *D. perspicua* A synthetically prep'd *normcoline* comp'd was highly toxic to *Aphis rumicis*, the bean aphid C R Fellers

Seed investigations, barley-blight control R. H. Porter, E O Brown and C. M. King Ia Agr. Expt Sta, Rept Agr. Research 1934, 71-2(1934)—Barley blight, *Helminthosporium sativum*, was well controlled by the use of org. Hg compds such as Et Hg phosphate and sulfate These dusts may be used for treating the seed in 5% strength or dild with talc or gypsum to a 1% concn The seed is kept in contact with the powder for 1.5 hrs at 40° C R Fellers

The spray-residue problem in Kansas Geo A Flinger Biennial Rept Kansas State Hort Soc 42, 126-9(1932-33)—The arsenical residue on apples was higher when Pb arsenate alone was used in the entire spray schedule than when Pb arsenate was used in the early sprays and Mn arsenate, Ca arsenate,  $BaSiF_6$ ,  $Na_2AlF_6$ , nicotine tannate or oil emulsion was used in the late sprays The use of oil emulsions in the late sprays hindered the removal of arsenical residues with 1% HCl K D Jacob

A chemical method for determining the safeness to foliage of commercial calcium arsenates G W Pearce, L B Norton and P. J. Chapman N Y. Agr. Expt Sta, Tech Bull 234, 2-15(1935)—Com Ca arsenates vary in safeness to foliage when tested under the same conditions, consequently, the tendency to burn is a characteristic of the prep'n The results of gross analyses of com. prepns cannot be correlated with their safeness Water-sol As, as det'd by the official method, is not a true index to injury A tentative method was developed for detg the safeness to foliage of Ca arsenates based on the facts that (1) Water sol As det'd after carbonation of the material shows a definite relation to degree of injury (2) If the free lime normally occurring in Ca arsenates is first removed by any means, then water-sol As is a true index to injury (3) Removal of the free lime by means of  $CO_2$  under sp. conditions with the subsequent detn of water-sol As gives reproducible and concordant results Water-sol As in representative com. prepns varies from 0.20 to 11.50%  $As_2O_3$  by this procedure A safe brand should contain less than 0.75% water-sol  $As_2O_3$  A correlation between water-sol lime, "reserve alkali" and degree of injury was observed

if such detns were made after carbonation of the free lime. The water-sol. As responsible for injury is apparently derived primarily from the more sol. compds of Ca and As likely to be present rather than by decomposition of the material as a whole by  $\text{CO}_2$ . C. R. Fellers

**Seed disinfection** 1 An outline of an investigation on disinfectant dusts containing mercury W. A. R. Dillon Weston and J. R. Booser *J. Agr. Sci.* 25, 628-69 (1935). The combined result of lab. and field work suggested that there was a close relationship between compn. and fungicidal power of a seed disinfectant. In the series R-11g-X, where R is a hydrocarbon and X an acidic radical, the fungicidal power appeared to decrease with the increase in size of R. The majority of the inorg. Hg salts are of little value. No evidence of stimulation by the disinfectants was found in the case of toxic effect, but better germination and crop yields are believed to be due to control of seed-borne organisms and to the preservation of seed food reserves from attack of soil organisms. I. C. Ienstedt

**The origin of pine red rot** Georg Prehauser *Forstwiss. Centr.* 57, 649-55 (1935).—Red rot attacks the roots of pine trees when unable to penetrate a compact acid humus layer (pH 3.2-3.4) into the mineral soil beneath. Development of the roots is good in a moist litter but if the latter is composed of dry mosses the mycorrhiza are poorly developed and rotting takes place. I. C. I.

**A cooperative study in earthworm control in Rhode Island** H. I. A. North *J. Board Greenkeeping Research* 4, 101-3 (1935).—Pb arsenate, applied dry at the rate of 5-10 lb/1000 sq. ft. in mixt. with 6 quarts of screened soil, gave more than 90% control of earthworms in golf greens and fairways. Poor control was obtained with HgCl<sub>2</sub>, mowah meal and tobacco dust. K. D. Jacob

**Control of the codling moth by use of oil sprays** A. J. Ackerman, *Biennial Rept. Kansas State Hort. Soc.* 42, 17-23 (1932-33).—In expts over a period of 2 years, satisfactory control of the codling moth was not obtained by spraying the trees with Pb arsenate alone. The results were not improved by adding either lubricating oil emulsion or miscible oil emulsion to the Pb arsenate. The effectiveness of Pb arsenate sprays was considerably improved by addn. of white oil emulsion at 1% dosages in the 3 early cover sprays. The arsenical residue on apples sprayed with Pb arsenate-oil combinations was not removed satisfactorily by washing the fruit with 1.5% HCl soln. K. D. Jacob

**Arsenical compound substitutes for lead arsenate in the control of codling moth** P. G. Lamerson and Ralph L. Parker, *Biennial Rept. Kansas State Hort. Soc.* 42, 63-8 (1932-33).—Promising results were obtained with the arsenates of Ca, Zn and Mn. None of the sprays caused serious burning of fruit or foliage. Zn arsenate covered the leaves and adhered as well as did Pb arsenate but Mn arsenate seemed to be washed off more readily than Zn arsenate or Pb arsenate during heavy rainfall. Suspensions of Pb arsenate in water settled out 33% faster than did those of Zn arsenate, Mn arsenate settled out 4 times faster than Pb arsenate. K. D. Jacob

**The effect of flies and fly sprays on certain physiological processes of the dairy cow** W. M. Regan and S. B. Freeborn, *J. Dairy Sci.* 19, 11-28 (1936), cf. C. A. 20, 2555, 22, 4198, 26, 3606, 28, 5782.—Fly sprays of petroleum oils with pyrethrum or pine oil or both were tested. All had the same repellent efficiency for the first hr. but differed at subsequent intervals. Pine oil increased their efficiency in proportion to the amt. added. The loss in milk production caused by extremely heavy infestations of house and horn flies was negligible; that caused by stable flies was slightly less than 10%. Petroleum oil increased the loss in milk yield to 22%. The extreme effect was evident when high producing cows were sprayed during hot weather. Milk production was diminished and body temp. and respiration rate were elevated, but dry cows were not thus affected. Burning of the skin followed the use of oils having a viscosity lower than 40 sec. irrespective of unsulfonated residues; oils with unsulfonated

residues below 90% were dangerous when used in oils of higher viscosity than 65 sec. The application of oil impairs the ability of the skin to add in maintaining body temp. The hourly water loss through the skin of the unsprayed cow at 41° and 60% relative humidity was 113 g while for the unsprayed cow it was 223 g. This represents a loss of 46% in cooling. When 40 cc. of conc. spray was applied at an environmental temp. of 80°F, the upper crit. temp. or "pyrexial point" was lowered approx. 5°F. A water emulsion of pyrethrum and pine oils with a small amt. of petroleum was as efficient in repelling flies as petroleum sprays and was less detrimental to the cows. Philip D. Adams

**Spraying experiments for the control of fruit fly in the Stanthorpe districts** Hubert Jarvis *Queensland Agr. J.* 44, 560-3 (1935), cf. C. A. 29, 1570.—Sprays composed of (1) white oil emulsion 1 gal. and nicotine sulfate 1 pint per 80 gal. water and (2) colloidal S 1 oz. per 4 gal. water, resp., were approx. equally effective in controlling the insect. White oil emulsion alone was much less effective. K. D. Jacob

**Control of fruit fly [Chaetodacus tryoni Fogg.]** Experiments with white oil nicotine sulfate spray. J. A. Wright *Agr. Gaz. N. S. Wales* 46, 649 (1935).—Promising results were obtained by the use of a spray composed of emulsified white oil 1 gal. and nicotine sulfate 1 pint per 80 gal. water. The spray had no adverse effects on the tree and fruit. K. D. Jacob

**Experiments for the control of frog eye [of apple leaves], 1932** Wm. I. Pickett *Biennial Rept. Kansas State Hort. Soc.* 42, 60-3 (1932-33).—The disease, which is caused by the fungus *Phyllostora cydoniae* Arnaud, was best controlled by the application of summer-strength lime-S soln. as the 1st or pink spray, summer-strength lime-S plus 1.33 lb. Pb arsenate per 50 gal. as the petal-fall spray, 3.6-5.0 Bordeaux mixt. plus 1.33 lb. Pb arsenate per 50 gal. as the 1st cover spray, and 1.5-3.50 Bordeaux mixt. plus 1.5 lb. Pb arsenate as the 2nd cover spray. K. D. Jacob

**Bacterial leaf spot of peach** Geo. A. Filinger, *Biennial Rept. Kansas State Hort. Soc.* 42, 118-20 (1932-33).—The disease was best controlled by 3 applications of a spray composed of hydrated lime 4 and  $\text{ZnSO}_4$  4 lb./50 gal. Promising results were also obtained with dry-mix lime-S and Oxobordeaux (a conc. colloidal dry Bordeaux mixt.). K. D. Jacob

**Control of the red scale [of citrus] in Palestine** M. H. Sachs *Hadar* 8, 107-8, 201-4, 234-40 (1935).—A study of the distn. ranges of spray oils sold in Palestine showed that the oils are entirely too heavy for safe use on citrus trees at the concn. (about 3.5%) necessary for the control of severe attacks of red scale. Better control of the scale, with less damage to the trees was obtained with lower concns. of lighter oils. Factors affecting damage to citrus trees by oil sprays are discussed. K. D. Jacob

**The Florida wax-scale (Ceroplasma floridensis, Comst.) in Palestine** F. S. Bodenheimer, *Hadar* 8, 187-91, 193, 223-4, 227-8 (1935).—When citrus trees were sprayed with Volck white oil emulsions at 2.5, 2.0 and 1.5% concns., the av. kills of larvae were 89, 81 and 32%, resp. The sprays had comparatively little effect on vigorous adults. K. D. Jacob

**Report on experimental control of wheat rusts in 1935 [in Italy]** Cesare Sibilia *Boll. staz. patol. vegetale* 15, 481-9 (1935).—The results of expts with crude S are given. G. A. Bravo

**Petroleum summer oil sprays** Geo. A. Dean *Biennial Rept. Kansas State Hort. Soc.* 42, 20-32 (1932-33).—A review of the prepn., properties and use of the sprays. K. D. Jacob

**Manuf. of noddles from green beans [production of fertilizer] (Ou, Wu) 12.** Soil reaction and suitability of milk for cheese making (Hussmann) 12. Prepn. of  $(\text{NH}_4)_2\text{SO}_4$  (Belopolski) 18. Testing steels for their resistance to corrosion and erosion in contact with  $\text{P}_2\text{O}_5$  exts. from Vyatka phosphorites (Filipenko, Zaring) 9.



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- Irrigating and fertilizing plants. William O. Sweek (one fifth each to Wm T. McGeorge, Julius S. Benroth, James A. Saunders and Herbert H. Porter) U. S. 2,028,172, Jan 21. Water is stored at a level above that of soil to be irrigated, part of the energy of the stored water is used to generate electricity, the electricity is used to produce N oxides, the N oxides are absorbed in the water, and the charged water is applied to the soil.
- Fertilizers. Bamag-Meguain A.-G. Fr. 788,931, Oct. 21, 1935. A fertilizer which is easy to spread is obtained by leaching hot superphosphates with solid salts such as finely cryst.  $(\text{NH}_4)_2\text{SO}_4$ , and subjecting the lumps obtained to an agitation to get a granulated product.
- Fertilizer. A. J. Marin. Belg. 407,096, Jan 31, 1935. Crystd urea nitrate is mixed with  $\text{CaH}_2\text{PO}_4$  and  $\text{Ca}_3(\text{PO}_4)_2$ . It may also be mixed with Ca cyanamide.
- Fertilizer from sewage. Oliver V. Austin. U. S. 2,029,648, Feb 4. Cond. sewage is subjected to the action of steam under high pressure and at a high temp. for a few min. and then suddenly released to cause the explosive disintegration of the cell structure of the sewage.
- Stable humus products. Joost Hudig. Dutch 35,466, May 15, 1935. Insol. humus products beneficial to the soil are prep'd by mixing humic products, as peat, straw or wood waste, with readily hydrolyzable aluminosilicates (pulverized slag) and oxidizing the moist mixt. in a mixt. of steam and air at temps. below  $130^\circ\text{C}$ , e.g., at  $70^\circ\text{C}$ . Addn. of up to 10%  $\text{NH}_3$  to the vapors serves to regulate the acidity of the product and gives a valuable N content of the product. The oxidation can be promoted by addn. of Fe or Mn compds.
- Lime nitrogen. Nikodem Caro and Albert R. Frank. Ger. 620,888, Oct. 29, 1933 (Cl. 124 9). Addn. to 609,730 (C A 29, 5230). The method of 609,730 is modified by replacing the  $\text{Ca}_3(\text{PO}_4)_2$  by  $\text{CaCO}_3$ .  $\text{CaCl}_2$  is formed as an intermediate product which reacts with atm. N to form  $\text{CaCN}_2$ .
- Insecticides and fungicides. The Grasselli Chemical Co. Brit. 436,327, Sept. 30, 1935. These comprise as the active ingredient a lauryl deriv. of formula  $\text{C}_n\text{H}_{2n}\text{X}$ , where X is halogen, OR or SR, R being H or an org. radical, CN, CNO or an amino or substituted amino group in which the N of the substituted amino group does not form part of a heterocyclic ring. Suitable derivs. are lauryl alc. and its ethers and org. esters, lauryl mercaptan and its thioethers, lauryl cyanide, lauryl cyanate, laurylamine, lauryl urea, lauryl bromide, dialauryldithiocarbamates, triaurylamine, lauryl diethyldithiocarbamate and lauryl xanthate.
- Parasiticide. Max L. Tower and Harry W. Dye (to Niagara Sprayer & Chemical Co. Inc.) Can. 355,216, Jan 7, 1936. The particles of a parasiticide contg. S as the principal toxic agent are covered with a coating of the same color as the foliage to be treated.
- Calcium arsenate insecticides. Simon Klosky (to American Agricultural Chemical Co.). U. S. 2,029,264, Jan 28. Ca arsenate is associated with combined F (suitably in the form of  $\text{CaF}_2$  formed in the mixt. and amounting to not more than about 4% of the total  $\text{As}_2\text{O}_3$  content) which serves to stabilize the arsenate against deterioration by liberation of sol. As under the action of  $\text{CO}_2$ . Cf. C A 29, 3452.
- Treating plants with volatile materials such as dust-form nicotine compositions. Guy S. Hales. U. S. 2,029,166, Jan 28. Various operative details.

## 16—THE FERMENTATION INDUSTRIES

## C N FREY

- Adaptation and development of the method of partition between solvents for the determination of fermentation products. C. H. Werkman. Ia Agr. Expt. Sta., Rept. Agr. Research 1934, 63 (1931), cf. C A 28, 4167. The partition method of sepp. the lower fatty acids is based on the fact that fatty acids show marked differences in their ability to dissolve in 2 immiscible liquids such as  $\text{H}_2\text{O}$  and  $\text{Et}_4\text{O}$ . Advantage is taken of these differences by partitioning the aq. mixt. of acids with an ether such as isopropyl. From the fraction of total acid remaining in the water, a detn. of each acid can be made from a nomogram. Satisfactory detns. can be made on a mixt. of 3 acids. The method has been extended to the detn. of the alcs. such as  $\text{EtOH}$  and  $\text{BuOH}$  which oxidize to the fatty acids. Isopropyl alc., acetone and  $\text{EtOH}$  and  $\text{BuOH}$  are det'd by a modification of the partition method. C. R. Fellers.
- Hydrogenation of freshly distilled spirits. Carroll A. Hochwalt, Charles A. Thomas and Ernest C. Dyball. *Trans. Am. Inst. Chem. Engrs.* 31, No. 4, 686-97 (1935), cf. C A 30, 1174. The hydrogenation process offers a distinctly new and advantageous method for removing greenness from freshly distilled spirits, and by this removal considerably accelerates the natural aging process.
- C. L. Mantell.
- Alcoholic fermentation of sorghum grain in the solid state. Ke-Chung Chang and Tien-Fsun Kang. J.

*Chem. Eng. (China)* 2, 102-5(1935).—The old-fashioned Chinese process for the manuf. of ale liquor by fermentation of sorghum grain in the solid state can be improved by using mold and yeast cultures, by 1st cooking the grain at a pressure of 50-60 lb. per sq. in., and by allowing the mixt. to ferment at 35°. Under such optimum conditions the lab. yield is as high as 90% and the semicom. yield about 80%. The com. possibility of the improved process depends much upon the solution of engineering difficulties.

C. L. Tseng

**Manufacture of alcohol from kaoliang and corn.** Teh-Kuan Shih and Shing Chien Chow *Ind. Center (China)* 2, 227-34(1937).—The main difficulty in the manuf. of alc. from kaoliang and corn lies in the conversion of the starch into sugar. This process is best carried out as follows: (1) Select the raw material, crush, soak in  $H_2O$  overnight, and then mix with 4-5 vols  $H_2O$  and the appropriate amt. of inorg. acid (0.5% for kaoliang and 0.8% for corn) as hydrolytic catalyst; (2) cook the material under 40-50-lb. pressure for 1.5-2 hrs. to convert it into dextrin; (3) neutralize excess acid with  $Ca(OH)_2$  until it is just slightly pinkish to both litmus and methyl orange; (4) add 10% (of the wt. of raw material) of green malt or 5% green malt + 5% *Aspergillus oryzae*, and keep at about 60° for 2 hrs. to complete the conversion to sugar. Carry out the fermentation of this material with yeast.

C. L. Tseng

**Significance of the formol-protein number in judging malt.** O. Menzel *Wochschr. Brau* 52, 105-9(1935), cf. *Ibid.* 44, 490(1927).—The importance of formol N is elucidated. Results show that beer from malt with high formol N tastes flat and has poor head retention. M shows that there is no correlation between the amt. of total sol. N in wort and the amt. of formol N. The sol. of the proteins and their degradation are 2 different problems, as shown by the detailed analysis of 2 malt samples. M. shows that there is a correlation between formol no. and modification provided the protein content of the malt is within reasonable limits. The degree of modification is influenced by the protein content. The analytical results show that in case of a high formol no. the sol. N, wort-extr. N, amide N, acidity and maltose increase and the wort has a tendency to run clear. Simultaneously the difference in ext. between finely ground and coarsely ground samples and time of conversion decrease. Analysis of malt samples originating from various crops (1931-1934) are tabulated and discussed in respect to N compds. and their correlation.

S. Jozsa

**Hordein content of malts.** H. Lüers and O. Geiger. *Wochschr. Brau* 52, 193-5(1935).—Earlier investigations (Bishop, *C. A.* 22, 3729, 24, 1929) indicate that the hordein content of barleys ranging from 8 to 14.0% protein varies from 29.3 to 40.2% expressed in percentage of total protein. The hordein content of 33 malt samples was detd. by the method of Kiefferle and Lintner (Dissertation, München), full details of which are given. The hordein content expressed in percentage of the total N ranged from 21.85 to 34.2%. Although it varied with the protein content of malts irregularities were found because of various degrees of modification. Figures are suggested for values representing normally modified malts.

S. Jozsa

**Estimation of extract in barley by the method of Lüers and Müller.** R. Otto. *Wochschr. Brau* 52, 191(1935).—The use of superclastase (a product of France) is recommended by L. and M. (*C. A.* 30, 808<sup>9</sup>). O suggests trying Biofase, which is of similar character and is produced in Germany.

S. J.

**The present state of the protein question.** P. Kolbach. *Wochschr. Brau* 52, 209-14(1935).—The various protein materials in barley and their subsequent changes during malting, mashing and brewing are discussed.

S. Jozsa

**Pectin in hops.** H. Fink and J. Hartmann. *Wochschr. Brau* 52, 221(1935).—The presence of pectin was detd. by digesting hops with 10-15 times their weight of 1%  $H_2SO_4$  at 4 atm. in an autoclave. The presence of galacturonic acid was then detd. For the estn. the material was first extd. with  $EtOH$  and ether. The air-dried residual matter was boiled with 0.01 N citric acid for 24

hrs. under reflux, the filtrate concd. *in vacuo* and the pectin pptd. in the form of its Ca salt. The quantity of pectin found was about 2% of the extd. matter. The above method gave neg. results on barley and malt. The possibilities of the importance of the presence of hops are emphasized.

S. Jozsa

**The flavor of soy.** I. Kenjuro Shōji. *Bull. Inst. Phys.-Chem. Research (Tokyo)* 14, 872-7(1935).—In general, the aromatic substances of soy are composed of  $AcOH$ ,  $Me(C_2H_5)_2COOH$ ,  $C_2H_5COOH$ ,  $AmOH$ , hexyl and higher alcs., phenolic compds., aldehydes and ketones, the phenolic compds. being the most important constituents.

K. Konda

**"After-dinner" wines.** I. Astruc and A. Castel. *Ann. fals.* 28, 599-604(1935).—"After-dinner" wines ("vins de café") are wines which are fermented but a short time (18-24 hrs.) in vats like red wines, and then withdrawn from the marc and allowed to ferment to completion, yielding a product having the color of red wine, but characteristics more resembling those of white wines, which might lead to the conclusion that alc. had been added if they are judged according to the usual enological rules for red wines. Analyses of 6 such wines are tabulated and discussed.

A. Papineau-Couture

**Effect of the extract of wine on the distillation of the volatile acids of the wine.** P. Jaulmes. *Ann. fals.* 28,

590-9(1935), cf. *C. A.* 30, 808<sup>9</sup>.—The constituents of wine ext. have in practice but very little effect on the distn. of volatile acids and a quite negligible effect when the steam distn. method is used. Irreversible adsorption of the volatile acids by the colloids of wine seems to be of secondary importance and difficult to prove. Tartaric acid has always shown itself capable of liberating all the  $AcOH$  added to synthetic mixts. A study of the compn. of the volatile acids "liberated" by the action of lime on the ext. of old wines showed that these acids are formed very largely by the action of lime on the sugars present in the wine. Defecation with lime at the boiling temp. is therefore considered to be probably useless and certainly liable to introduce serious errors.

A. Papineau-Couture

**New micro method of estimating boric acid in wines.** C. Sumuleanu and Gh. Chumescu. *Ann. sci. univ. Jassy* 21, 361-8(1935).—The ash of 50 ml. beer is distd. with anhyd.  $MeOH$  and concd.  $H_2SO_4$  in a special app.; the vapors are condensed and titrated with 0.01 N  $Ba(OH)_2$ . Boric acid in certain Roumanian wines.

*Ibid.* 369-76.—Wines from 20 districts were found to contain 17.23-93.91 mg.  $H_3BO_3$  per l. New micro method for estimating total sulfuric acid in wine, applicable to all wines.

*Ibid.* 377-83.—Five ml. of wine is distd. in a special app. with 2 ml.  $H_3PO_4$ , the vapors being entrained in a current of  $H_2$ , the  $SO_2$  is absorbed in  $KOH$  soln. and detd. iodometrically.

New micro method for determining free sulfuric acid in wine, applicable to all wines.

*Ibid.* 384-9.—By use of the same app. which was used above the vapors are absorbed in 0.02 N  $I$  soln., which does not act on entrained sulfite-aldehyde, and back-titrated with thiosulfate.

Microestimation of ash and alkalinity of ash of wine.

*Ibid.* 390-2.—Ten ml. of wine is evapd., ashed and weighed, and the ash is taken up in 2-3 cc.  $H_2O$ , treated with 10 cc. 0.02 N  $H_2SO_4$  and titrated with  $Ba(OH)_2$  soln. against methyl orange.

O. W. Wilcox

**Microestimation of volatile acids in wines.** Gh. Chumescu. *Ann. sci. univ. Jassy* 21, 306-14(1935).—Five ml. of wine is steam-distd. in a modified Parnas-Wagner app.; the distillate is titrated with 0.01 N  $Ba(OH)_2$  from a buret graduated in 0.01 ml.;  $SO_2$  is detd. iodometrically after addn. of an excess of  $KOH$  to an aliquot of the distillate and acidification with  $H_2SO_4$ .

A micro method for estimating lactic acid in wines.

*Ibid.* 315-20.—One ml. of wine (decolorized if necessary with bone char) is placed in a centrifuge tube, exactly neutralized with  $Ba(OH)_2$ , treated with 0.5 ml.  $BaCl_2$  soln. and distd.  $H_2O$  to make a total vol. of 3 ml.; 7 ml. of 96% alc. is added. The soln. is sep'd. from the ppt., evapd. with 1 ml.  $K_2SO_4$  soln., the residue ignited to a white ash, treated with 5 ml. 0.02 N  $H_2SO_4$  and back-titrated with  $Ba(OH)_2$ , this gives the total acid; subtracting the previously detd. volatile

- acid gives the lactic acid. Microestimation of malic acid in wine. *Ibid* 321-5.—Malic acid in 5 ml wine is sepd as Ba malate (insol in 70% alc), which is dissolved in hot  $H_2O$  and converted into the K salt, from which malic acid is set free by 3 ml of Pmerua reagent (1 g  $\beta$  naphthol in 50 ml concd  $H_2SO_4$ ) and detd colorimetrically against a standard soln of aniline brown. Microcolorimetric determination of total tartaric acid in wine. *Ibid* 326-9.—Tartaric acid in 2 ml wine is pptd as K bitartrate by the Dubuque method (C A 26, 4907) and detd colorimetrically by the Underhill metavanadate method (C A 26, 2480). A microcolorimetric method for estimation of potassium in wine. *Ibid* 333-8.—The K in 25 ml wine is pptd as cobaltinitrite, washed with a 1% soln of  $Al_2(SO_4)_3$ , then with a 5% soln of  $Na_2PO_4$  to bring the  $NO_3$  group into soln for colorimetric detn in an acid soln. of salicyrine. The precision is within 1%. A new colorimetric method of determining nitrites. *Ibid* 330-2.—A soln contg 5-50 mg nitrite is placed in a 20 ml flask with 5 ml of a 5% soln of salicyrine (in a satd soln of Na acetate) and 5 ml of glacial  $CH_3CO_2H$ , and the flask is filled to the mark, after 15-20 min the color is compared with a graded series of solns contg salicyrine and  $NaNO_2$ . Potassium in the native wines of Roumania. *Ibid* 339-42.—Analyses of 42 wines show K content ranging 0.39-0.947 g per l. Microcolorimetric estimation of bitartrate in wine. *Ibid* 343-5.—K bitartrate is pptd from 2 ml wine with  $Et_2O$  alc mixt (2:1) and the tartaric acid is detd colorimetrically by the metavanadate method (see above). New micro method for the colorimetric determination of glycerol in wine. *Ibid* 346-51.—One ml wine, 4 ml distd  $H_2O$  and 0.5 g  $CaO$  are evapd nearly to dryness and extd with 90% alc, the alc is evapd off and the residue contg glycerol is oxidized with  $Br$  water in a sealed tube, the liquid is treated with  $Zn$  to remove  $Br$ , made up to 20 ml and filtered, 1 ml of the filtrate is treated with 1 ml of a 5% soln of pyrocatechol and 10 ml concd  $H_2SO_4$ , heated in a sealed tube in a  $H_2O$  bath for 5 min, made up to 20 ml and compared colorimetrically with a standard soln of glycerol similarly treated. Microdetermination of calcium in wines and its proportions in the native wines of Roumania. *Ibid* 352-5.—Using the Kramer-Tisdall method on 50 wines of different years, G finds Ca contents of 37-175 mg per l. Influence of the glass apparatus in the determination of small quantities of boric acid (in wine and beer). *Ibid* 356-60.—Numerous detns showed that when Pyrex or Jena glass is used for B detn in wine and beer by distn with  $H_2SO_4$ , the results are much too high, K glass is preferable. Microdetermination of boric acid in beer and its proportions in certain native beers. *Ibid* 393-7.—The beer ash is washed with  $MeOH$  into a special flask and distd as usual after addn of  $H_2SO_4$ ,  $H_2BO_3$  in Roumanian beers varies from 16-14 to 47-34 mg per l. similar analyses of Polish and Czechoslovakian beers show 2.97-7.13 mg per l. O W W.
- Report on beer analysis for March and April, 1935. *I Anker Wochschr Brau* 52, 182-3(1935), cf C A 30, 8088.—Report on 112 beer samples. S Jozsa.
- The present state of brewing technology. H Lbers. *Wochschr Brau* 52, 201-5(1935).—A general discussion. S Jozsa.
- The brewing industry—raw materials, plant methods, equipment, economies and chemical control. Gerald L. White. *Can Chem Met* 20, 3-7, 12(1936).—An illus description. W H Boynton.
- Changes in metabolism of a pure yeast culture in case of repeated pitching. E. Schuld and R. Weber. *Wochschr Brau* 52, 161-5(1935).—Various pure yeast cultures were used in lab and practical expts. The results are tabulated showing apparent ext, apparent attenuation,  $pH$ , total and volatile acidity, esters, total N, formal N and yeast crop. The results show that the physiol condition of the yeast affects but slightly the compn of beer. The greatest deviation is noted in esters, which is, however, compensated during storage. The reason for using in the brewhouse a new pure yeast culture after 5-8 pitchings is merely a safety measure to avoid possible infection and assure uniform quality of the beer. S Jozsa.
- Food yeast from wood-sugar solutions. II Hermann Fink, Richard Lechner and Eugen Heinisch. *Biochem Z* 283, 71-82(1935), cf C A 29, 8226.—Expts were carried out on the cultivation of *Torula utilis* in wood-sugar malt contg  $NH_4$  salt as the sole N source. The synthesis of protein on this artificial medium varies with the method of culturing but also with the compn of the wood-sugar malt. Best results were obtained with wood sugar prep by the Bergius method. S Morgulis.
- Margarine (Ger pat 620,988) 12.
- Gerhardt Schaefer. Beitrage zur Kenntnis der Ernährung der Heife beim Lufthefeverfahren. Dresden: Russe-Verlag. 47 pp. Reviewed in *Chimie & Industrie* 33, 1468(1935).
- Kionka, Heinrich, and Hess, Martin. Bier als Nahrungsmittel. Der Nährwert malzreicher Biere. Jena: G Fischer. 32 pp. M 220.
- Meinde, Otto. Tafeln für die Malzanalyse. Berechnet nach den Tafeln der Normalleischungskommission. 2nd ed. Nürnberg: Carl. 24 pp. M 160.
- Veken, Fr van der. Agenda du brasseur et du maltreur. Louvain C. Uystpruyst. F 850. Reviewed in *Bull assoc chim* 52, 890(1935).
- Fermentation. Julius Hanak. *Fr* 789,297, Oct 25, 1935. App for atomizing gas in liquids is described.
- Protein degradation products. Albert F. J. Friedel. *Brit* 436,591, Oct. 11, 1935. A nutrient for yeast, which may be added alone or together with oxidizing agents, e.g.,  $KBrO_3$ , or starch to bread dough or to wort for the production of alc, is produced by digesting a protein, e.g., waste yeast, leguminous seeds, with aq  $H_2PO_4$  or an acid P compd at at least  $100^\circ$ . In an example, spent yeast is heated with aq  $H_2PO_4$  in an autoclave at  $140-150^\circ$  for 1 hr.
- Apparatus for producing citric acid by fermentation. O P Protod'yakonov. *Russ* 38,129, Aug. 31, 1934. Construction details.
- Apparatus (with a rotatable malting drum) for germinating grain in malt production. Augustinus E. Jonsson. *U S* 2,029,873, Feb 4. Various structural, mech and operative details.
- Wort. Heini I. Waterman and Franciscus A. M. J. S. van Waesberghe. *Brit* 439,436, Oct 10, 1935. Wort is produced by boiling milk products, sugary substances and hops or hop substitutes, and may then be fermented by yeast to yield a beverage. In an example, skimmed milk, cane sugar, sol starch, didd malt coloring and hops are boiled with  $H_2O$ , cooled, filtered and fermented with brewer's yeast at  $10^\circ$  for 2 days and then at  $4^\circ$  for 4 days.
- Clarifying beer. Justin Erwin Pollak and Chemische Werke Marnefeld A.-G. *Brit* 435,706, Sept 19, 1935. Addn to 393,142 (C A 27, 5845). In clarifying beer as in 393,142, dry wood meal, fragments of a pitch and paraffin mixt, or two disks of pitch, about 3 mm in diam with a small central protuberance on 1 side, are introduced to the surface of the wort or beer. They sink slowly and thus clarify the liquid.
- Yeast. A M Malkov. *Russ* 37,668, July 31, 1934. Molasses is heated to boiling in the presence of Fe filings and  $H_2SO_4$ , sepd from the ppt, treated with water, superphosphate and yeast, and after fermentation has begun ( $NH_4$ ) $2SO_4$  soln is gradually added. After fermentation is complete, a current of air is introduced.

## 17—PHARMACEUTICALS, COSMETICS AND PERFUMES

W O SMITH

**Chemical composition of sweet basil oil from Virginia** F. K. Nelson and M. S. Lowman. *Am. Perfumer* 31, 69 (1935).—Oil distd. from the whole fresh flowering herb (*Ocimum basilicum* L.) gave the following results: d<sub>4</sub><sup>20</sup> 0.9123, n<sub>D</sub><sup>20</sup> 1.4875, ester value (as linyl acetate) 1.51%, alcs. (as linylalol) 65.3%, methoxyl 8.05% (as methyl-chavicol 38.15%). Small amts of cineole, eugenol and sesquiterpenes are present. A very small amt. of a d. rotatory terpene may be present. Philip D. Adams.

**Materials and manufacture of hand preparations** Maison G. de Navarre. *Am. Perfumer* 31, 71 (1935).

**Oil of parsley** Ernest S. Guenther. *Am. Perfumer* 31, 73 (1935).—Oil distd. from leaf material (without seeds) gave a yield of 0.044% and had the following consts: d<sub>4</sub><sup>20</sup> 0.911, [α]<sub>D</sub><sup>20</sup> 0°, n<sub>D</sub><sup>20</sup> 1.4623, acid value 1.4, ester value 8.9, ester value after acetylation 44.8. The oil is insol. in 90% EtOH, but sol. in 96% with opalescence. Presence or absence of seeds on leaf material changes the rotation from d- for leaf material to l- with seed material present. Philip D. Adams.

**Chaulmoogra and related oils** H. M. Langton. *Pharmaceuticals and Cosmetics* 1935, 149-50.—A brief review of early research on chaulmoogra oils and their present-day therapeutic use. Philip D. Adams.

**Calamine in pharmacy and cosmetics** J. J. Bolton. *Pharmaceuticals and Cosmetics* 1935, 153-4.—A discussion with formulas of the uses of calamine (ZnCO<sub>3</sub>) in lotions, liquid face powders, liniments and ointments, mud packs and complexion clays. Philip D. Adams.

**Cobalt in cosmetics** H. Stanley Redgrove. *Pharmaceuticals and Cosmetics* 1935, 155-6, cf. C. A. 30, 573.—A brief review of the toxicology of Co, the use of Co blue and Co green as cosmetic pigments and Co salts as hair dyes. Philip D. Adams.

**The alkaloids of Han-fang-chi** H. Hanfangchin B. Chih-Fang Hsu. *J. Chinese Chem. Soc.* 3, 375-71 (1935).—The filtrate from hanfangchin A (C. A. 29, 7579) is evapd. and treated with C<sub>6</sub>H<sub>6</sub> and the insol. material extd. with Me<sub>2</sub>CO. The resulting hexagonal crystals, when recrystd. from 95% alc., yield colorless prismatic crystals, hanfangchin B, C<sub>16</sub>H<sub>15</sub>N<sub>3</sub>O<sub>4</sub>, m. 241-2°, [α]<sub>D</sub><sup>25</sup> +272°. Hanfangchin B is a tertiary base with no phenolic hydroxyl, nitrosoamine, methylenedioxy or carbonyl group; it appears to contain 2 double bonds, 2 -NMe groups and 4 -OMe groups. Wm. H. Adolph.

**A preliminary study of cultivated Stramonium** Pien-Nien Tsou and Szu-Yi Chen. *J. Chinese Chem. Soc.* 3, 372-6 (1935).—An analysis of *Stramonium* cultivated in Shanghai shows: ash 15.18, alkaloids 0.195%.

**Note on the distribution of alkaloids in Ephedra sinica (Ma-huang)** M. Tarlé. *J. Chinese Chem. Soc.* 3, 377-80 (1935).—Chinese physicians consider that different parts of the plant have different therapeutic properties. In the young herb the total ephedrine alkaloids are distributed almost evenly between pith and outer stem wall. In the ripe plant, the pith contains considerably more than the outer wall. Wm. H. Adolph.

**Fermentation of cigar tobacco** Ya V. Fum. *Tabac-naysa Prom.* 1935, No. 4, 27-30.—Cigar tobacco, when subjected to fermentation in stacks, shows signs of insufficient fermentation in the upper layers and it is heat-damaged in places where access of air is too limited (in the center of the stack).

**The industry of essential oils and their derivatives in Italy and its recent progress** Francesco La Face. *Boll. ufficiale staz. sper. ind. essenze e deriv.* 10, 127-41 (1935).—A review.

**Artificial Agurumen oils** Alfons M. Burger. *Rieschstoff-Ind.* 10, 195-6 (1935).—The artificial bergamot, lemon, orange (Portugal), mandarin and neroli oils are discussed.

**The preparation of pharmaceutical sodium phosphate**

[Na<sub>2</sub>HPO<sub>4</sub>, 12H<sub>2</sub>O] Vincenzo Paolini. *Gazz. chim. ital.* 65, 624 (1935).—Treatises on pharmaceutical chemistry give the following reaction: Ca<sub>3</sub>(HPO<sub>4</sub>)<sub>2</sub> + 2Na<sub>2</sub>CO<sub>3</sub> → 2Na<sub>2</sub>HPO<sub>4</sub> + CaCO<sub>3</sub> + CO<sub>2</sub> + H<sub>2</sub>O, whereas Leloux and Courtois (*Traité de pharmacie chimique*, 1929, C. A. 23, 1307) give the reaction 3Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> + 4Na<sub>2</sub>CO<sub>3</sub> → 1Na<sub>2</sub>HPO<sub>4</sub> + Ca<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub> + 4H<sub>2</sub>O + 4CO<sub>2</sub>, and long ago Dextrin and Trebault (*Bull. soc. chim. Paris* 27, 499 (1877)) showed that no CaCO<sub>3</sub> is formed. Expts by P. show that the reaction is not a double decomposition, and if Na<sub>2</sub>CO<sub>3</sub> is replaced by another base, e. g., NaOH or NH<sub>4</sub>OH, Na<sub>2</sub>HPO<sub>4</sub> or (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> is formed, the medium becomes alk., and Ca<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub> is pptd. because the conditions necessary for the formation of Ca<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub> from Na<sub>2</sub>HPO<sub>4</sub> or (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> and a sol. Ca salt exist. Na<sub>2</sub>HPO<sub>4</sub> can be prepd. advantageously from Na<sub>2</sub>CO<sub>3</sub> and CaHPO<sub>4</sub>. Though the latter is only slightly sol. in cold water, in boiling water it decomposes: thus 4CaHPO<sub>4</sub> → Ca<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub> + Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>. Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> then reacts with Na<sub>2</sub>CO<sub>3</sub>. Com. CaHPO<sub>4</sub> can be obtained in grades of high purity, so that the resulting Na<sub>2</sub>HPO<sub>4</sub>, 12H<sub>2</sub>O is a pure grade.

**Anesthetics, especially cocaine and procaine, in relation to illicit traffic** C. Offerhaus and C. G. Baert. *Pharm. Weekblad* 70, 506-16, 525-39, 617-31, 675-67, 826-43, 973-97, 1125-37, 1193-1201, 1278-1311, 71, 666-75, 817-23, 1070-3, 1337-42, 1361-70, 1401-8, 72, 822-40, 861-8, 1411-35, 1413-64 (1935-1935) (English summary, 1462-4).—The history, pharmacology, fluorescence, capillarity, optical rotation and photomicrographs of a large no. of local anesthetics and their salts are discussed. The most characteristic test for cocaine is the chloroplatinate, clearly recognizable at a diln. of 1/8000. No other anesthetic gives the same type of feathery crystals. Treatment with HClO<sub>4</sub> converts cocaine into a resin from which other substances can be washed out, then the resin is dissolved in HCl and identified with PtCl<sub>4</sub>. A. W. Dox.

**Optical crystallographic study of some derivatives of barbital and phenobarbital** Martin E. Hultquist and Charles F. Poe. *Ind. Eng. Chem., Anal. Ed.* 7, 308-9 (1935).—Chem. tests are not entirely satisfactory for the identification of some barbituric acid derivs., notably those of barbital and phenobarbital, for which optical crystallographic data are given and which can be useful in the identification of these 2 medicinal substances. The following derivs. of barbital (I) and of phenobarbital (II) have been prepd. and described: bis(o-bromobenzyl)-I, thick rods, m. 140° (tube), 141° (block); m-isomer, thin plates, m. 91°, 90°; p-isomer, flat rods and plates, m. 140°, 147°; o-chloro analog, long rods, m. 127°, 125°; m-chloro isomer, thin plates, m. 102°, 100.5°; p-chloro isomer, flat rods and plates, m. 142°, 144°; p-iodo analog, rods and thick plates, m. 122°, 123°; m-nitro analog, long needles, m. 150°, 160°; p-isomer, needles and rods, m. 102°, 103°; diphenacyl-I, flat rods and plates, m. 191°, 192°; bis(p-bromophenacyl)-I, rods, m. 191.5°, 193°; bis(o-bromobenzyl)-II, short rods, m. 116°, 113°; m-isomer, thin plates, m. 130°, 130°; p-isomer, thin plates twinned, m. 117°, 118°; m-chloro analog, thin plates, m. 111°, 111°; p-isomer, 2 forms of crystals, thin plates twinned, m. 111°, 111°, and thick plates, m. 113°, 114°; p-iodo analog, thick plates and rods, m. 127-8°, 127°; m-nitro analog, flat rods and plates, m. 149.5°, 151°; p-isomer, rods and needles, m. 182.5°, 184°; bis(p-bromophenacyl)-II, rods and needles, m. 164°, 167°. In general, these compds. were obtained by refluxing equiv. amts of barbital or phenobarbital and K<sub>2</sub>CO<sub>3</sub> or Na<sub>2</sub>CO<sub>3</sub> dissolved in a little more than enough boiling H<sub>2</sub>O to make a satd. soln., and a soln. of benzyl or phenacyl halide (2 mol. quantities) in an amt. of alc. twice as large as the amt. of H<sub>2</sub>O used to dissolve the barbituric acid salt.

**Effect of hot solvents on ergot—effect of storage on the activity of ergot** R. F. Corran and F. E. Rymul. *Quart. J. Pharm. Pharmacol.* 8, 377-9 (1935).—P<sub>2</sub>O<sub>5</sub>, C<sub>2</sub>H<sub>5</sub>Cl,

$C_6H_5Cl_3$  and  $C_6H_5$  ext the major portion of the alkaloids, light petroleum does not ext them. With  $CH_2Cl_2$  and  $C_6H_5$ , quant. recovery of the alkaloids was effected, thus the alkaloids are extd and not destroyed by these solvents. W O E.

Properties of glycols—use of propylene glycol as a solvent in pharmaceutical preparations C L M Brown *Quart J Pharm Pharmacol* 8, 390-7(1935)—Attention is directed to the toxicity of  $p$ -dioxane and  $C_6H_5(OH)_2$ . In assaying  $C_6H_5(OH)_2$  (I) galenicals involving the use of  $Et_2O$  or  $CHCl_3$  for extn, the galenical should be diluted with at least  $1/4$  its vol of  $H_2O$ . Solns of certain alkaloids in I can be freely diluted with  $H_2O$  without pptn. I tends to mask the reaction of certain alkaloidal reagents, notably picric and tannic acids. Phenolphthalein can be dispensed in soln in therapeutic doses. The halogen salts of Na and K are very sol in I, as are also dyes, I would therefore be a suitable preservative, notably for syrups. It is a solvent for most volatile oils, and may function as a suitable menstruum for tinctures (especially senega) owing to its non-volatility, its solvent powers, miscibility with  $H_2O$  and  $EtOH$ , and its preservative action. I considerably retards the volatilization of  $EtNO_3$  and may conveniently replace  $EtOH$  in sweet spirits of niter. W O E.

Assay of quinine in iron and quinine citrate and quinine salts J S Toal and A J Jones *Quart J Pharm Pharmacol* 8, 401-5(1935)—Evaluation of quinine in Fe and quinine citrate by wt differs from evaluation by titration. This difference is due in part to the loss of basicity in the quinine which is effected during the manu of the scales. This loss of basicity in quinine may be brought about in the process of assay unless special precautions are taken.  $Et_2O$  and  $CHCl_3$  are not interchangeable in the assay if the final result is to be the wt of dried residue following simple evapn of the solvent. A suggestion is made to standardize the upper limit for the assay of Fe and quinine citrate by wt, and the lower limit by titration. W O E.

Flue-cured tobacco Natural aging of flue cured cigarette tobaccos L F Dixon, F R Darks, F A Wolf, J A Hall, E P Jones and P M Gross *Ind Eng Chem* 28, 180-9(1935), cf C A 29, 8230\*—The aging, under industrial conditions, of the 6 main types of flue-cured cigarette tobacco for 3 successive crops was studied for 30 months. Environmental conditions in these tobaccos were not favorable for the activity of bacteria and molds and it is believed that these agencies played a very minor part in the aging process. Little self heating occurred. Small amts of  $CO_2$ ,  $AcOH$ ,  $HCO_2H$  and  $NH_3$  were evolved during aging. Decreases in sugar, total N, water-sol N, amino N, nicotine, total acids and  $pH$  took place during the 30 month period. It is probable that the aging process is essentially chem, the main reaction being that between sugars and amino compds with the formation of melanoidins and  $CO_2$ . L E Gibson.

New medicaments F Zernik *Deut med Wochschr* 61, 178, 247, 1004, 1288, 1816(1935)—A brief description of drugs introduced in Germany during 1935. A G. Combined iodine in iodine ointment D Hennville *Analyt* 61, 27(1936)—In making I<sub>2</sub> ointments much of the I<sub>2</sub> is lost either by volatilization or by combining with the paraffin base. Some ointments contain no free I<sub>2</sub>. W T H.

Determination of elemental sulfur in sulfur ointment. H R. Fleck and A M Ward *Analyt* 61, 28-30(1936) McLauchlan and Mathews (*Analyt* 35, 610) have compared their method of detg free S with that of F and W (*Analyt* 48, 199(1922)) but it is shown here that there is the possibility of some of their values being a little too high. W T H.

Psyllium seed from Cyprus Anon *Cyprus Agr J* 29, 98-9(1934)—The samples contained  $H_2O$  8.3-11.5 and oil 4-7.0% and had swelling factors of 14.0-17.5. The seed compared favorably with com. Spanish and French seed. K D Jacob.

Paraldehyde and other preliminary hypnotics Albert H. Miller. *Anesthesia and Analgesia* 15, 14-21(1936)—A comparison of paraldehyde with barbitalure, avertin,

morphine, with and without atropine, as a preanesthetic.

G. H. W. Lucas.

The methyl index of some balsams, resins and drugs of animal origin. M M Janot and S Sabatay. *Bull. sci. pharmacol* 42, 529-32(1935)—The Me index is the quantity of Me expressed in mg developed by 1 g of substance when heated with  $HCl$ . The value for balsam of Peru varies between 19.5 and 22.8, solid balsam of Peru has an index of 31.9-32.8. The index for a no. of other substances is reported. The general use of the Me index for characterization of this group of substances is recommended. A E. Meyer.

Evaluation of mercurial antiseptics in the presence of serum Dorothea E Smith, E. J. Czarnetzky and Stuart Mudd. *J. Bact* 31, 7-8(1936)—Serum binds metaphen, merthiolate and  $HgCl_2$  so that they are ineffective for intravenous use. John T. Myers.

A practical evaluation of antiseptics for use in oral hygiene. M A Jacobson *J. Bact* 31, 9(1935)—Depending on the antiseptic dilns of 1, 2, 1, 3, 1, 4, etc (each 5 cc contg 1 cc of sterile saliva) killing 0.5 cc of a 24-hr broth culture of *Staphylococcus aureus* in less than 1 min exposure are claimed to have 2, 3 or 4 antiseptic units, resp. John T. Myers.

Bulgarian otto of roses R. Garnier and Mrs S Sabatay *Ann fals* 28, 585-9(1935), cf C. A. 28, 5927\*—Semi-com. distns were carried out on rose petals, segg the oil obtained directly and that obtained by redistn of the water, and 9 analyses of the products are tabulated and discussed. Presence of  $EtOH$  (in greater amts in the oil from the water than in the direct oil) was confirmed; also, that the apparent rhodinol content (detd. by hot formylation) exceeds 40 to 45%, that the rhodinol geraniol ratio is greater than 1, and that all otto of roses contains azulenogenic sesquiterpenes which are present in greater amts in direct oil than in oil from water. The following rapid method is recommended for the detn of stearoptenes to 2-4 g of sample in a 150-cc. Erlenmeyer flask add 50 cc of 75% alc, heat a few min on the water bath, immerse while stirring in an ice-water mixt, filter under suction on a Tramm funnel through a tared filter paper dried at 75°, wash with 200 cc of 75% alc and dry to const wt in a crystal dish at exactly 75°, dissolve in a little  $CHCl_3$  and add a few drops of a 5% soln of Br in  $CHCl_3$ , there should be at most a slight green coloration showing the presence of not more than a trace of azulenogenic sesquiterpenes. A Papineau-Couture.

Opium assay Joseph Rosin and C J. Williams *J. Am Pharm Assoc* 24, 1053-62(1935)—A study of the lime method of opium assay for morphine (I). Dissolving the I obtained in hot  $MeOH$  before titration eliminates (on the basis of I content) about 2% of foreign titratable substances called as I. Assays of pure I by the U S P and B P methods confirmed the "assay loss" of practically 1 mg of I per cc. of lime-I soln as indicated in the B. P., the "loss" fluctuating somewhat unless definite and uniform conditions are maintained in the assay. About half the loss is attributable to the soly of I in the assay solvents, the greater part (if not all) of the balance is caused by the solvent action of the  $NH_4Cl$  on I, adsorption on the lime may be responsible for a small portion of the loss. Increasing the quantity of  $NH_4Cl$  used or the temp during pptn of I increases the loss. It is recommended that: (1) in the U S P assay the quantity of  $NH_4Cl$  be reduced from 1.0 to 0.5 g, which is several times the theoretical quantity required for a 15% opium, (2) the pptn temp (standing overnight) be restricted to about 10°. Satn or near-satn of the lime-I soln with  $NaCl$  before adding  $NH_4Cl$  raises by 1-2% the quantity of I pptd, but also increases cc pptn of other alkaloids. The I pptd in the U S P assay (and probably also in other assays) carries about 3% of non-phenolic by-alkaloids which is counted as I. Since opium contains also other alkali-sol non I alkaloids, these, if present in appreciable quantity, may also produce high results for I. By coincidence of counterbalancing error factors, the U. S. P. assay seems to indicate very close to the true I content. A P-C.

The action of alkalies and alkali salts on antipyrine.

Lloyd E. Harris and Fredd D. Tebow. *J. Am. Pharm. Assoc.* 24, 1069-70 (1935).—On addn of antipyrine to concd. solns. of alkalis or alkali salts there seps an immiscible liquid, which disappears on diln of the mixt with  $H_2O$ , or which on standing gradually changes to a cryst. substance identified as antipyrine. The immiscible liquid is believed to be an isomeric form which changes to the usual cryst. form on standing. A. P. C.

**Phytochemical notes CXII.** The sterols of *Achillea millefolium* Ole. Givold. *J. Am. Pharm. Assoc.* 24, 1071 (1935), cf. C. A. 29, 3805<sup>3</sup>, 4196<sup>4</sup>.—The unidentified sterol, in 134-5°, giving an acetate m. 123-4°, found by Graham in milfoil (C. A. 28, 3185<sup>3</sup>), is shown to be a mixt. of stigmasterol and a stosterol in which the latter predominates. A. Papineau-Couture.

**Comparison of spectrometric method and antimony trichloride test for estimation of vitamin A potency.** W. S. Jones and W. G. Christensen. *J. Am. Pharm. Assoc.* 24, 1072-4 (1935).—Cod-liver oils which had been tested biologically for vitamin A potency were subjected to the  $SbCl_3$  test in 1 lab., and to the spectrometric method in 2 labs. With one exception the  $SbCl_3$  test gave values in fair agreement with those of the bio. assays. By the spectrometric method, with one exception the values for oils of high potency were in reasonable agreement with those of the bio. assays, but for oils of low potency the variations were considerably greater. A. P. C.

**Compound solution of cresol—variation of phenol coefficient when different oils are used for saponaceous base.** P. L. Burrin, A. G. Worion and F. E. Bibbins. *J. Am. Pharm. Assoc.* 24, 1077 (1935).—Several oils (corn, soybean, coconut, linseed) will make a satisfactory compd. soln. of cresol. Peanut and sesame oils are not desirable for use in such a product. Coconut oil is the only one of the group that can be used to manuf. a satisfactory product which at the same time shows an increased PhOH coeff., and for this reason it may be desirable as a base for this prepn. It seems that there may be oils more desirable for this purpose than linseed oil which is now prescribed by the U. S. P. A. Papineau-Couture.

**Improvement in technic in the preparation of three common products.** Edward D. Davy. *J. Am. Pharm. Assoc.* 24, 1079-81 (1935).—The following changes are suggested for facilitating the prepn. and improving the following products: *Sirup of white pine compd. N.F.*—To correct the excessive sassafras oil residue in this sirup, if the oil is added to the menstrum the excess is retained by the drug and satn. is all that should be expected. *Lixir phenobarbital.*—Filtration difficulty is avoided by macerating the cudbear in alc., glycerol and  $H_2O$  in approx. the same ratio as is represented in the finished product, the phenobarbital is dissolved in a small amt. of alc. reserved for this purpose and added to the clear filtrate from the cudbear. *Sapo molles U. S. P. IX.*—Dissolve the alkali in  $H_2O$  (10% of the wt. of finished soap) and immediately add all the oil and stir well, stirring the mixt. at intervals to emulsify the unsapon oil is all that is necessary for sapon. Occasionally when working with small quantities and always when working with 500-800 lb. or more the  $H_2O$  content must be increased by about 20% as sapon. approaches completion. A. Papineau-Couture.

**Advances in disinfectants in 1935.** Emil Klarmann. *Soap (Sanitary Products Sect.)* 12, No. 1, 95, 97 (1936).—A review of testing methods and effectiveness of different types of materials and ultraviolet rays as disinfectants. Twenty-six references. Henry H. Richardson.

**The phenol coefficient as a measure of the practical value of disinfectants.** Jack C. Varley. *Soap (Sanitary Products Sect.)* 12, No. 1, 101, 103, 121 (1936).—Coal tar or cresol type disinfectants when dild. to 20 times their phenol coeff. are equally effective in killing several types of pathogenic microorganisms. V. claims the phenol coeff. when used in this way is a suitable measure of these disinfectants. Five references. Henry H. Richardson.

**Hexargin.** J. K. Gjaldbaek. *Arch. Pharm. Chem.* 42, 615-22 (1935).—*Hexargin* (I), a substitute for the prepn. which is distributed under the name *argolalol* (II) was prepd. from 11 g. 10%  $AgNO_3$  soln. (III) and 98 g. filtered

40%  $(CH_3)_4N_4$  soln. (IV) by shaking III with 20 g. IV and then adding the remaining amt. of IV; it has sp. gr. 1.01. It should be kept in properly closed bottles, protected from light. Its identity with II was established by electrometric titrations with 0.1 N NaCl and 0.1 N KI; the cell  $Ag | 0.01 N AgNO_3, 0.03 N KNO_3 | 3 mol. KNO_3, 0.45 mol. NaNO_3 | I$  or  $II | Ag$  was used. The results are given in tables and illustrated graphically. The therapeutic effects of I and II were also identical. The  $Ag$  ions of I are present mainly in complex combination with  $(CH_3)_4N_4$  (V). The equil. between the  $Ag$  ions, V and the  $Ag-V$  ions shifts and occurs instantaneously. By the consumption of  $Ag$  ions the  $Ag-V$  ions therefore cleave off  $Ag$  ions and I thus acts as an  $Ag$ -ion buffer. It is noteworthy that the concn. of the  $Ag$  ion increases by diln. with water because 1  $Ag$  ion is combined with 2 mol. V (cf. Pawelka, C. A. 18, 3553). The equil. between  $Ag-V$  ion,  $Ag$  ion and V is illustrated by  $AgV_1^+ = Ag^+ + 2V$ , the mass action equation for which is  $a_{Ag^+} a_{V_1^+}^2 / a_{AgV_1^+} = 10^{-11}$ , or  $a_{Ag^+} = 10^{-11} (a_{AgV_1^+} / a_{V_1^+}^2)$ , the concns. (z) being considered equal to the activities (a). The latter equation shows that the concn. of  $Ag$  ion is proportional to the content of  $Ag$  because  $Ag$  is present mainly as  $Ag-V$  ion, but the fact that the  $Ag$ -ion concn. also is inversely proportional to the square of the concn. of V explains that the  $Ag$ -ion concn. of I increases by diln. with water. Excepting very dil. and very concd. solns. a diln. with equal parts of water will double the  $Ag$ -ion concn. The  $Ag$ -ion concn. in I itself is very small and can be calcd. approx. according to  $0.239 = 0.0577 \log(10^{-1/x})$ , where 0.239 is the voltage measured by a cell consisting of the 2 half-electrodes  $Ag/I$  and  $Ag/0.01 N AgNO_3$ . According to this equation the  $Ag$ -ion concn. (x) in I is  $10^{-4.8}$  (cf. C. A. 23, 4298). To det. the  $AgNO_3$  in I weigh 25.00 g. I in an Erlenmeyer flask, dil. with 50 cc. water and acidify with 20 cc.  $HNO_3$ . Add 5 cc. ferric  $NH_4$  sulfate soln. and titrate with 0.1 N  $NH_4CNS$ . One cc. 0.1 N  $NH_4CNS$  corresponds to 0.01699 g.  $AgNO_3$ . D. Thuesen.

**Peppermint oils.** Ernest J. Parry and George Ferguson. *Chemist and Druggist* 124, 37 (1936).—For 23 English oils, 50 American and 6 Kenya oils, results of the detn. of sp. gr., n, [α]<sub>D</sub>, esters as  $AcOMe$ , total menthol and menthone are tabulated. In 13 samples of English oils from the same plant, distd. between 1927 and 1935, menthol varied from 42 to 64%, menthone from 29 to 42% and esters from 2.3 to 6.2%. The other English oils, mostly of 1935, the American (1934-5) and the Kenya oils contained, resp., menthol 52.2-64.9, 46.8-61.7, 52.7-60.3%, menthone 22.0-32.7, 16.9-30.8 (in 17 samples), 10.0-13.7%; esters 3.1-10.1, 4.5-11.0, 24.1-33.3%.

**S. Waldbott.**  
**Determination of oxydimorphine.** R. Drevon. *J. pharm. chim.* 22, 97-106 (1935); cf. Leulier and Dubreuil, C. A. 20, 1826. L. and Drevon, C. A. 26, 3839.—The reagent is pure  $H_2SO_4$  (d. 1.830) 20 cc., with  $Ac_2O$  (98-99%), 1 cc. A mixt. of 2 cc. reagent and 0.05 mg. alkaloid (I) applied in the form of the silicotungstate ppt. gives a characteristic green color of max. intensity, with max. absorption band in the red and orange at 6355 Å. Into a 15-cc. centrifugal tube (cleaned with chromate mixt.) put 5 cc. soln. of I, add 5 drops 5% aq. silicotungstic acid soln. and 0.03 g.  $KHCO_3$ . Put the tube for 30 min. into melting ice, then mix with 1 drop of gelatinous  $BaSO_4$  (radio-graphic type), centrifuge for 3 min. (3000 r. p. m.), again add 1 drop  $BaSO_4$  mixt., rotate for 3 min. and pour off the clear liquid. Dry the inside walls with paper, cool again and add exactly 2 cc. freshly pptd. and cooled reagent. Stir up the clot with a glass rod;  $BaSO_4$  and I dissolve. After 30 min. at ordinary temp. compare the tint in a Baudouin and Besnard colorimeter with standards made up either from known quantities of I, or from an adjusted scale of tints prepd. with solns. of nitrates of Cu, Ni and Co. The best conditions are obtained when 5 cc. of soln. of I contains 0.4-0.10 mg. I, but the reaction is still pos. at a concn. of 0.03 mg. I per 5 cc. The expl. error is ±5%.

**S. Waldbott.**  
**Quinidine thiocyanate; gravimetric and volumetric determination of quinidine.** R. Monnet. *J. pharm.*

*chim* 22, 112-19(1935) —The detn of  $C_{10}H_{15}N_3O_2$  (A) in cinchona preps is usually based on the low soly. of HI A in  $H_2O$  (1:1250 at 15°). M recommends in its place the HCNS A salt (B) which is less sol in  $H_2O$  (1:1477 at 20°). soly is further decreased by excess of KCNS or  $(NH_4)CNS$  used in its prep. or in its detn by wt. To an aq soln of a basic salt of A, neutral to litmus and contg. not more than 0.20 g A, add excess of KCNS, filter, wash first with a little  $H_2O$ , then with a satd aq soln of B, dry at 120° and weigh. In the presence of other cinchona bases, whose HCNS salts are more sol in  $H_2O$  and in alc than B, the same method can be used if in 3rd washing with  $6 \times 1$  cc 96% alc satd with B is applied. Similar ppts with KCNS are formed with emetine, papaverine, strychnine, veratrine and berberine, but none with 22 other alkaloids named, nor with digitalin, strophanthin, acetaminide, adrenaline, antipyrine, procaine, phenacetin, urea, barbital, etc. For the volumetric detn of A (use 0.15 g as nitrate or basic sulfate in aq soln neutral to litmus), evap to 20 cc, add a definite excess of 0.1 N KCNS and det this excess in an aliquot vol with 0.1 N  $AgNO_3$ . The gravimetric method is accurate within  $\pm 1\%$ , the volumetric method is rapid, but gives low results because the pptg excess of KCNS that can be used is limited S W

A new specific reaction for yohimbine M Pescz. *J pharmac chim* 22, 164-5(1935) —By warming a mixt of concd  $H_2SO_4$ , an aq soln of chloral and a soln of yohimbine-HCl salt on the water bath, a blue color is obtained L Rossi, A del Boca and R Lobo *Ibid* 566 —The same reaction has been described before (cf C A 26, 5703) S Waldbott

Chemistry of Australian-grown tobacco N F B Hall and J C Earl *J Council Sci Ind Research* 8, 277-80 (1935) —The smoke from "good" tobaccos is slightly acid or neutral, while that from "bad" tobaccos is usually definitely alk. The N content of a good light tobacco is about 1.0-1.5% (of dry solids), that of the best samples 2.0-2.5% and that of the very bad Australian grown samples 3.0-4.5%. Analyses show that harvesting too early accounts an excessive amt of nitrogenous substances in the leaf, which are not removed during curing. About 25% of the total solids disappear during flue curing. Little of the N is eliminated, and the percentage of N in the total solids increases correspondingly. An extensive degradation of protein substance occurs during curing E D Walter

An acid ester present in the root of the Valeriana officinalis L-mil Congoa *Compt rend* 201, 1152-4(1935), cf C A 29, 3770 —By fractionation of the oil obtained from the roots of the valerian, a product (I),  $b_p$  120-22°,  $d_4^{20}$  1.0214,  $[a]_D^{20}$  4.83°,  $n_D^{20}$  1.43657, was obtained. Hydrolysis of I gave iso- $C_8H_{15}CO_2H$  and  $Me_2CHCH(OH)CO_2H$ ,  $b_p$  124-25°,  $n_D^{20}$  1.43657,  $[a]_D^{20}$  -1.56. Hence I must be  $Me_2CHCH(CO_2H)OCOCH_2CHMe_2$  J White

Isopropylalcohol Council on Pharmacy and Chemistry *J Am Med Assoc* 105, 1684(1935) —Acceptance of isopropyl alc (I) for admission to New and Nonofficial Remedies I is used for the removal of creosote from the skin as a prophylactic agent against creosote burns and has been recommended for the disinfection of the skin and of hypodermic syringes and needles and in the administration of insulin. It is not potable and should not be relied upon to destroy such spore-bearing organisms as *Clostridium tetani*, *Clostridium welchii* or *Bacillus anthracis*. Phys const and tests for aldehyde, acetone and nonvolatile content are given. A dosage form, *Saf-T-Top Isopropyl Alcohol*, 98%, intended solely for the removal of creosote from the skin has been prepd C R Addnall

Sex hormones (Bennett) 18 Esters of amino alcs with 9-acridinecarboxylic acid (Samdahl, Weider) 10 Piperazine (Vinauer) 10 Extg lipoids, etc (Brit pat 435,798) 27. Substituted sulfoxides [used in pharmacy] (Ger. pat. 620,889) 10 Ureides [used as soporifics] (Ger. pat. 620,903) 10 Barbitalic acid compd. (Ger. pat. 620,998) 10. Condensation products of aromatic hydroxy compds with alkenols (U. S. pat. 2,029,539) 10

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Adhesive tape and gauze dressings Warner Eustis and Paul Walter (to Kendall Co) U S 2,029,260, Jan 28 App used and various mfg details are described

Anesthetic Samuel D Goldberg (to Novocoll Chemical Mfg Co Inc) Can 355,246, Jan 7, 1938 A compn for hypodermic injection contains procaine-HCl 20,  $M$   $Na_2HPO_4$  0.022  $M$   $KH_2PO_4$  0.005,  $M$   $NaCl$  0.1,  $M$   $NaHSO_4$  0.01, adrenaline 0.032 g and 0.001  $N$   $H_2PO_4$  2 cc in sufficient  $H_2O$  to make 1 l. The soln has a sp gr of approx 1.0110 and a  $pH$  value of 6.7. Cf C A 29, 5224

Extraction of antipyrine E A Tzofin Russ 38,153, Aug 31, 1934 Antipyrine is extd with hot  $C_4H_9Me$ , from the reaction mass obtained in the methylation of phenylmethylpyrazolone in the usual manner

Barbituric acid derivatives Ernest II Volwiler and

Donalee L. Tabern (to Abbott Laboratories). Can. 354,378, Nov. 26, 1935. Barbituric acids substituted on 1 N atom and doubly substituted on the C atom by said or unsatd. alkyl groups, at least one of which is a secondary or tertiary alkyl group are prep'd by condensing the appropriate disubstituted malonic or cyanoacetic ester with the appropriate substituted urea. The products are sedative, anesthetic or soporific. Can. 354,379. Hypnotic and sedative barbituric acid derivs substituted in the 5-position by  $\text{F}_y\text{CH}(\text{CH}_3)_2$ , in which y is any whole no., are obtained by condensation of malonic or cyanoacetic ester derivs with urea.

**Salts of organic bases** Jean L. Regnier. Fr. 789,156, Oct. 24, 1935. Salts obtained by combining local anesthetic bases such as cocaine and novocaine with particular org. acids, such as phenylacetic, phenylbutyric, benzoic, salicylic, acetic, formic, phenylpropionic and cinnamic acids are found to have greatly increased action over the salts generally used.

**Hydrogen peroxide products** Alexander Wacker Ges. fur elektrochemische Industrie G. m. b. H. (Helmuth Muller and Franz Trinkl, inventors). Ger. 620,574, Oct. 23, 1935 (Cl. 12a 12). Solid storable  $\text{H}_2\text{O}_2$  products are obtained by making  $\text{AcONa}$  or  $\text{AcOK}$  into a paste with aq.  $\text{H}_2\text{O}_2$  soln and evaporating *in vacuo* at 60–70°. The product is used as a disinfectant. Examples are given. Cf. C. A. 29, 859<sup>4</sup>.

**Cosmetics** George H. H. Hardy. Fr. 789,252, Oct. 25, 1935. A cosmetic suitable for painting the lips comprises one or more aniline dyes, e. g., eosin, and a solid powder such as chalk or kieselguhr.

**Massage composition** U. S. Industrial Alcohol Co. Fr. 788,511, Oct. 18, 1935. A semisolid compn. contg. more than 85% by wt. of  $\text{EtOH}$  and which becomes liquid by slight friction with the skin is made by adding a gel-forming agent, such as an alkali salt of a higher fatty acid, and a normally solid wax or waxes, such as carnauba wax, to  $\text{EtOH}$ . Paraffin, lanolin and a fat glyceride may also be incorporated.

**Estrogenic substances** Schering-Kahlbaum A.-G. Brit. 437,051, Oct. 23, 1935. These are extd. from the tubers of *Butea superba* by means of volatile solvents, e. g.,  $\text{FlOH}$ ,  $\text{MeOH}$ ,  $\text{Me}_2\text{CO}$ . Cf. C. A. 29, 7022<sup>2</sup>.

**Hormone extracts** Schering-Kahlbaum A.-G. Ger. 620,990, Oct. 31, 1935 (Cl. 12p 17 10). Crude exts. of genital-gland hormones are refined by treatment with an acylating agent, preferably in the presence of an indifferent solvent, sepp. the esterified from the unesterified constituents, and saponifying the hormone-ester mixt. The acetylation may be carried out by means of acid halide and a  $\text{HCl}$ -binding agent. Thus, a crude ext. from male urine is dissolved in toluene and treated with a soln. of toluene-sulfonyl chloride in toluene and with pyridine. After the pyridine- $\text{HCl}$  is filtered off, the toluene is distd. off and the residue sepd. by fractional crystn. or extn. to obtain the toluenesulfonic acid ester of the hormone, which is saponified to give an oily product. Another example is given. Cf. C. A. 30, 575<sup>4</sup>.

**Vitamins** Charles W. Hooper (to Winthrop Chemical Co.). Brit. 436,713, Oct. 16, 1935. Vitamin D preps. are produced by dissolving an antirachitic vitamin D in a propanediol, preferably 1,2-propanediol.

**Ergot alkaloids** Chemische Fabrik vorm. Sandoz. Fr. 788,812, Oct. 18, 1935. Ergot alkaloids are sepd. and prep'd in the pure state by subjecting solns. of ergot alkaloids to a chromatographic adsorption according to the method of Tawett in indifferent solvents. The crude exts. or solns. of alkaloids are dissolved in indifferent solvents such as  $\text{C}_6\text{H}_6$  and its homologs,  $\text{CHCl}_3$  or dichloroethylene, and the solns. are passed into a column contg. adsorbent insol. in the solvents used, e. g., sugar,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{CaCO}_3$ , fibrous  $\text{Al}_2\text{O}_3$ , etc. By diffusion of the soln. in the ad- or solvent column there appears a chromatogram visible to ultraviolet light. Examples are given of the sepn. of ergotamine and ergotamine, ergotoxin and ergotamine and the constituents of sensibamine.

**Insecticides** Roger C. Ancelet. Fr. 789,205, Oct. 25,

1935. A compn. to be applied to the skin as a protection against mosquitos contains citronella oil, geranium oil, menthol, chloral hydrate, crystal. quassia and alc.

**Antiparasitic charge for gun cartridges** Otto V. Huffman. U. S. 2,029,217, Jan. 21. An explosive is formed into a unitary mass with a binder and an antiparasitic material.

**Antianemia preparation** William J. Horn (to McKesson & Robbins Inc.). Can. 355,214, Jan. 7, 1936. The principal ingredients of the prepn. are an Fe deriv. of protein, such as Fe peptonate, and a Cu deriv. of protein, such as Cu caseinate. The Cu deriv. acts as a catalyst for the action of the Fe in the hemoglobin regeneration.

**Antianemia substance** George B. Walden (to Eli Lilly and Co.). Can. 354,430, Nov. 26, 1935. To stomach and liver tissues are added Fe and vitamin B. 1 g., 10 lb. of whole stomachs are mixed with 20–100 lb. of minced livers, dried and digested under acid, e. g.,  $\text{HCl}$ , at a temp. below 40°.  $\text{H}_2\text{O}$  and fats are removed from the material. To the mixt. is added 1–10 lb. of reduced Fe and 1000–30,000 vitamin B complex.

**Colloidal copper-containing composition for treating anemias** John Torjangan (to Drug Products Co.). U. S. 2,028,575, Jan. 21. Colloidal Cu hydroxide is carried in suspension in an aq. soln. of an org. hydroxy acid salt such as gluconic acid or Na gluconate.

**Sera for treatment of malignant tumors** I. G. Farbenindustrie A.-G. Brit. 436,292, Oct. 4, 1935. Preps. are obtained from the organs, bone marrow or body fluids of animals that have been inoculated with fresh virulent tumor cells, or with exts. or pressed juices thereof prep'd according to Brit. 366,503 (C. A. 27, 3037), but have remained tumor-free. Exts. or pressed juices of the glands or other organs may be obtained, the exts. by means of  $\text{H}_2\text{O}$ , aq. salt solns. or org. liquids, which preferably contain  $\text{H}_2\text{O}$ . The solns. obtained may be purified by addn. of solvents, e. g., alc.,  $\text{Me}_2\text{CO}$ ,  $\text{Et}_2\text{O}$ , to ppt. impurities or by addn. of adsorbents, e. g.,  $\text{Al}(\text{OH})_3$ , active charcoal, to remove ballast substances.

**Therapeutic camphor-group tetrazoles** Karl F. Schmidt (to E. Bilhuber, Inc.). U. S. 2,029,709, Feb. 4, 1936. By treating camphor with at least 2 mols. of hydrazole acid in the presence of a catalyst such as  $\text{SbCl}_5$  or  $\text{SnCl}_4$ , camphortetrazole, m. 212–3°, is obtained. Mired- $\alpha$ - and  $\beta$ -thuyones and pure  $\beta$ -thuyone similarly form tetrazoles m. 86° and 93–5°, resp. Purification of the products may be effected by recrystn. of their double  $\text{HCl}$  compds. and either racemic or optically active forms of the products may be produced, both having therapeutic anesthetic effects relatively free from paralyzing properties. Several examples with details and modifications of procedure are given.

**Medicinal products from chlorophyll and its derivatives** Syngala Fabrik fur chemisch-synthetische und galenische Arzneimittel G. m. b. H. Brit. 436,701, Oct. 14, 1935. Tonics are obtained by treating chlorophyll, porphyrins or like complex compds. derived therefrom, with reagents yielding ions of both Cu and Fe. The products may be filtered and washed, e. g., with dil. acid and alc., and then dispersed in an inert liquid, e. g.,  $\text{H}_2\text{O}$  or an oil, by, e. g., treatment in a colloid mill and (or) with the addn. of an emulsifying agent, e. g., gum arabic or gelatin. They may also be dispersed in fats with lecithin as dispersing agent. Alternatively, dispersions of the crude products may be made by adding an emulsifying agent to the reaction mixt. In an example, an alc. chlorophyll soln. is treated with a soln. of  $\text{FeCl}_3$  and  $\text{CuSO}_4$  acidified with  $\text{HCl}$  and the mixt., heated, the ppt. contg. 2% Fe and 2.5% Cu being filtered off.

**Tobacco** Katadyn A.-G. (formerly Oligodyn A.-G.). Brit. 436,445, Oct. 4, 1935. This corresponds to Ir. 771,194 (C. A. 29, 879<sup>3</sup>).

**Tobacco treatment** Bert Lowenthal (to Federal Tobacco Corp.). U. S. 2,029,494, Feb. 4. Gum, oils, nicotine and mineral matter are removed from the leaf in an acid bath contg.  $\text{HNO}_3$  and  $\text{H}_2\text{PO}_4$ .



## 18—ACIDS, ALKALIES, SALTS AND OTHER HEAVY CHEMICALS

E M SYMMES

Progress in the fine chemical industry during 1935  
Sex hormones and laboratory chemicals R R Bennett  
*Ind Chemist* 12, 13-14, 35(1936), cf C. A. 29, 1893<sup>4</sup>.

E. H

Improvements in the manufacture of sulfuric acid  
M Gurvich *Neft* 2, No 17, 8-11(1931), cf C. A. 29,  
297<sup>4</sup>—A brief discussion on the Selden method for the  
manuf. of  $H_2SO_4$  is presented, and its advantages in com-  
parison with the Pt contact and chamber methods are  
stressed.

Sulfur dioxide and fresh sulfuric acid from refinery  
acid sludge B A Stagner *Ind Eng Chem* 28,  
171-5(1936)—The  $H_2SO_4$  in typical gasoline and kero-  
sene acid sludges is converted by moderate heating into  
 $SO_2$  in yields of 93-4%, based on the acid used in refining  
S in the sludge, not expelled as  $SO_2$ , remains in the coke.  
The coke is neutral and readily pulverized. The  $SO_2$  so  
produced can be easily converted into  $H_2SO_4$  catalytically.  
The rate of spontaneous decompn. of  $H_2SO_4$  to  $SO_2$  in  
cracked gasoline acid sludge is shown E M S

A summary of the lecture of Hugo Peterson, Berlin-  
Stegitz, on problems of sulfuric acid manufacture by the  
nitrogen oxide process Hans Schutt *IX Congr  
intern quim pura aplicada, Madrid* 3, 447-57(1934)—  
A review is given of the theories of the reactions and the  
design of Pb chamber plants E R Rushton

The system vanadic acid-copper oxide-silicic acid as  
multiple catalyst in sulfuric acid manufacture Bernhard  
Neumann, Carl Kroger and Rudolf Iwanowski *Z  
Elektrochem* 41, 821-34(1935)— $V_2O_5$ , CuO and  $SiO_2$  and  
their binary and ternary combinations were studied as  
catalysts for 7%  $SO_2$  mixts in air at temps from 400°  
to 680°. The app has been described by Neumann,  
Pauzner and Goebel (C A 23, 1219). The activity of  
 $V_2O_5$ - $SiO_2$  mixts is directly related to the disocn pressure  
of  $VOSO_4$ , an intermediate in the catalysis. The activity  
of CuO- $SiO_2$  mixts is detd by the position of the equil  
 $CuO + CuO \cdot CuSO_4 \rightleftharpoons 4CuO + SO_2$ . The promoter  
effect of CuO on  $V_2O_5$  in their mixts is attributed to the  
formation of a new solid phase with a higher  $SO_2$  disocn  
pressure. A ternary diagram is shown, relating compn  
and catalytic activity of the 11 mixts studied. The  
highest activity is shown by the mixts having the molar  
compsns 0.4-0.6  $V_2O_5$ , 0.2-0.5  $SiO_2$ , 0.1-0.25 CuO

L W Elder

New methods for removal of pyrite cinders from burners  
N A Baranov *Bumazhnaya Prom* 14, No 7, 25-33  
(1935)—Improvements in the structure and operation  
of mech discharge of pyrite cinders are discussed. An  
exptl device for pneumatic removal and transportation  
of cinders is illustrated and described Chas Blanc

Calculating chromic acid from hydrometer readings  
Nothman S *Hall Platers Guide* 32, 17-18(Jan,  
1936)—A table shows oz  $CrO_3$ /gal for each 0.5° from  
1.5° to 36.0°Bé W H Boynton

Modern methods of producing phosphorus and phos-  
phoric acid C Matignon *IX Congr intern quim  
pura aplicada, Madrid* 3, 139-68(1934)—The heat  
balance of the reactions, conditions of reaction, utilization  
of the slags, methods of oxidizing P and the production  
of  $H_2$  are discussed. At Bex,  $SiO_2$  is replaced by a gneiss  
contn 6%  $K_2O$ , which is cheaper than sand. The product  
contains  $K_2O$  5 and  $H_3PO_4$  90-93% E R R

Potash from polyhalite by reduction process I  
Fraas and Fverett P Partridge *Ind Eng Chem* 28,  
224-30(1936)—Polyhalite was reduced in a continuous,  
rotary Lila by a mixt of CO and  $H_2$  made from natural  
gas. A temp of 830° is best. Higher temp fuses  
polyhalite, lower temp slows the reaction. No K  
compsds are volatilized during the reduction at temps  
up to 900°. At 830°, low S corresponds about to re-  
duction of  $MgSO_4$  to  $MgO$ .  $CaSO_4$  is reduced to  $CaS$ ,  
and an excess of  $H_2O$  vapor causes further S removal,  
leaving some  $CaO$ . The  $K_2SO_4$  was reduced to  $K_2S$  under

all the conditions used. About 16,000 cu. ft. of  $CH_4$  is  
required to reduce 1 ton of polyhalite E M. S

Leucite, a potash and alumina mineral V. Charrin.  
*Industrie chimique* 22, 739-40(1935)—Acid, alk.,  
and NaCl processes for the extn of  $K_2O$  and  $Al_2O_3$  from  
leucite are outlined A Papineau-Couture

Zinc as a chemical raw material I Bruce R. Silver  
*Chem. Industries* 38, 25-8(1936). F H

Burning lime with gas fuel Joseph Kahn *Western  
Gas* 12, No 1, 18-19, 56(1935)—The application of  
off-peak gas heat to lime burning is discussed. The  
presence of H in the gas is undesirable, owing to high  
flame temp, short, sharp flame and nonluminosity, which  
gives severe local heating W. H. Buckner

Determining gypsum and anhydrite G. Bushinskii  
*Mineral'nue Udobreniya Insektifungitsidus* 1, No 5,  
98(1935)—The presence of gypsum and anhydrite in  
their mixts can be detected by optical means. The  $n$   
of gypsum is 1.5208 to 1.5303 and of anhydrite 1.5693-  
1.6138, while the double refraction (measured with a  
Berek compensator) is 0.0097 for gypsum and 0.0437-  
0.0428 for the anhydrite A A. Boethlingk

Drying gel zeolites M G. Larian and Charles A.  
Mann *Ind Eng Chem* 28, 196-200(1936)—Zeolites  
were air-dried under various conditions of temp., humidity  
and velocity. Zeolites of varying compn were also  
prepd and dried under identical conditions. The zeolites  
break up into smaller particles when thrown into cold  
 $H_2O$  after complete drying. The percentage of fine sizes  
(through 28 mesh) depends upon drying conditions.  
The percentage of fines increases with very rapid or over-  
drying. The base-exchange capacity of a gel zeolite  
depends upon drying conditions. The importance of  
compn in base exchange, and in obtaining a rigid, non-  
brittle product, is shown. The amt. of total  $H_2O$  and  
structural  $H_2O$  in zeolites dried under various conditions  
was detd. Hydration also depends to some extent upon  
drying conditions E M. Symmes

The possibility of producing common salt from sea  
water by freezing A G Repa *Bull Far East, Branch  
Acad Sci U S S R* No 13, 3-14(15-16 in German)  
(1935)—In the Soviet Far East, wet and cloudy summers  
make the production of NaCl by solar evapn difficult,  
but in the shallow bays the concn of salt in the water  
under the ice is sometimes 4 times the normal concn of  
sea water. The absence of snow causes the water to  
lose heat rapidly through the ice, since the heat cond of  
ice is 52-56 times as great as that of snow. By freezing  
in a series of 3 basins and pumping off the soln, it can be  
concd sufficiently for economic evapn. E. R. R

Improvements in the manufacture of ammonium sulfate  
Ch Berthelot *Rev chim* and (Paris) 44, 307-10  
(1935)—A description and discussion of the merits of  
the process used at the Mines Finales of the Dutch State  
(cf Berkhoff, C A 29, 5998<sup>4</sup>). A Papineau Couture

Production of ammonium sulfate from gypsum A  
M Polyak and N S Blagoveshchenskaya *Mineral'nue  
Udobreniya Insektifungitsidus* 1, No 5, 7-20(1935).

A review of the following procedures is presented. (a)  
Semicon expts on the conversion of gypsum by gaseous  
 $NH_3$  and  $CO_2$ , (b) semicon conversion of "phospho-  
gypsum" ( $CaSO_4$ , 55.00,  $H_2O$  of crystn 14.56, hygro-  
scopic  $H_2O$  28.10, insol matter 0.48,  $H_3PO_4$  0.68 and  
 $R_2O$ ,  $P_2O_5$ , 1.18%) obtained in the extn of  $H_3PO_4$  from  
apatite subjected to flotation, (c) conversion of gypsum  
by a soln of  $(NH_4)_2CO_3$ , and (d) concn of solns of  
 $(NH_4)_2SO_4$  (semicon scale) Nine references

A A. Boethlingk

Technological layout for the production of ammonium  
sulfate from gypsum E. Granovskaya *Mineral'nue  
Udobreniya Insektifungitsidus* 1, No 5, 20-4(1935)—A  
review of the so-called gypsum and the  $H_2SO_4$  methods  
(Fauser) is presented A A. Boethlingk

Preparation of ammonium sulfate and soda from

mirabilite. I. A. P. Belopolskii. *Mineral'nue Udobreniya Insektofungitsidus* 1, No. 4, 7-20, No. 5, 25-31 (1935); cf. C. A. 29, 1590'.—A detailed description of the prepn. of  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{Na}_2\text{CO}_3$  on a semicom scale is presented. The combined prepn. of  $\text{Na}_2\text{CO}_3$  and  $(\text{NH}_4)_2\text{SO}_4$  from  $\text{Na}_2\text{SO}_4$  is quite feasible by means of the  $\text{NH}_3$ -soda method. The max. amt. of  $\text{NH}_3$  is 0.228 tons and of  $\text{H}_2\text{O}$  1.85 tons per ton of  $\text{Na}_2\text{SO}_4$ , in working with 75-80%  $\text{CO}_2$  (by-product in the prepn. of synthetic  $\text{NH}_3$ ), the max. of  $\text{NH}_3$  is 0.225 tons per ton of sulfate, when gases from lime kilns are used. The av. compn. of the mother liquor at the above concns. and a final carbonization temp. of 29-30° is  $(\text{NH}_4)_2\text{SO}_4$  21.42,  $\text{Na}_2\text{SO}_4$  12.32,  $\text{NH}_4\text{HCO}_3$  5.48,  $(\text{NH}_4)_2\text{CO}_3$  2.0 and  $\text{H}_2\text{O}$  58.78%. In the liquors obtained in the carbonization of  $\text{NH}_3$  solns of  $\text{Na}_2\text{SO}_4$ , a considerable amt. of the latter is still present, together with  $(\text{NH}_4)_2\text{SO}_4$  and  $(\text{NH}_4)_2\text{CO}_3$ . A sepn. of up to 70-74% of  $\text{Na}_2\text{SO}_4$  was effected by circulating a "eutonic" mixt., which at 60° contains  $(\text{NH}_4)_2\text{SO}_4$  36.5,  $\text{Na}_2\text{SO}_4$  16.2 and  $\text{H}_2\text{O}$  47.3%, through the liquor. By cooling the liquor to -10°, a 93% pptn. of  $\text{Na}_2\text{SO}_4$  is obtained, which is returned to the soda plant. In the cycle, the filtered liquor is mixed with "eutonic" soln, chilled to -10°, the mirabilite returned to the soda plant, the soln condensed by evapn. to sep.  $\text{H}_2\text{O}$  (steam), fertilizer and the "eutonic" mixt., which is used again. Various suggestions pertaining to the application of the process on a com. scale are made. A. A. Boechtingk.

Commercial manufacture of aluminum sulfate. Luis Fstrach. *Quimica* 12, 253-7 (1935).—Descriptive.

Manufacture of sodium sulfide. Reduction of sodium sulfate to sodium sulfide at temperatures below 800°. John F. M. White and Alfred H. White. *Ind. Eng. Chem.* 28, 244-6 (1936).—While pure  $\text{Na}_2\text{S}$  does not melt below 1200°, a mixt. contg.  $\text{Na}_2\text{S}$  30-40,  $\text{Na}_2\text{SO}_4$  70-60, melts somewhat below 700°. Reduction of  $\text{Na}_2\text{SO}_4$  by  $\text{H}_2$  proceeds rapidly as long as only the solid phase is present, but is slower in liquid phase.  $\text{CO}$  is not as rapid a reducing agent as  $\text{H}_2$ . Solid C-contg. materials act slowly until a liquid phase forms. Soft coal is the most effective C-contg. agent. Addn. of  $\text{CaO}$  increases the rate of reaction with solid C-contg. materials at temps. below 750°, because it removes  $\text{CO}_2$ , but above 750° the  $\text{CaCO}_3$  is dissolved.  $\text{Na}_2\text{SO}_4$  may be completely reduced in 2 hrs. at 750° by C-contg. materials, or in 1 hr. at 750° by soft coal and  $\text{CaO}$ . Com. practice uses 900-1000°. There are no complicating side reactions at 700°. Graphite is the most satisfactory container.

Influence of mud on the flotation of phosphates. L. I. Stremovskii. *Mineral'nue Udobreniya Insektofungitsidus* 1, No. 4, 61-7 (1935).—The flotation was carried out with ground minerals and synthetic mixts. An excess of mud carries off great proportions of the phosphates, thus lowering their yields in the concentrate. The grain size is therefore of primary importance in the flotation and it should be adjusted to each type of mineral treated.

Discovery of new apatite deposits in Russia. M. P. Fiveg. *Mineral'nue Udobreniya Insektofungitsidus* 1, No. 4, 39-43 (1935).—The Khiby (Kola Peninsula) apatites are of a low grade, but they can be concd. and used commercially. Other deposits are too high in Fe and cannot be worked for the sepn. of  $\text{P}_2\text{O}_5$  on a com. scale.

The total deposits of phosphate raw material in Russia available to January 1, 1934. I. M. Kurman and T. P. Unanyants. *Mineral'nue Udobreniya Insektofungitsidus* 1, No. 4, 44-60 (1935).—Statistical data on the available phosphorites are given and the importance of further prospecting is emphasized.

Insoluble Jules Hamand. *Ann. chim.* [2], 2, 145-63 (1936).—The properties, prepn. and utilization of insoluble as a clarifying agent are shown.

Carbon black. I. A new process for the production of carbon black by thermal decomposition of hydrocarbon gases. L. M. Pidgeon. *Can. J. Research* 13B, 351-69

(1935).—A method is described whereby C black is produced from hydrocarbon gases, such as  $\text{CH}_4$ - $\text{C}_2\text{H}_6$  or  $\text{C}_2\text{H}_4$ - $\text{C}_2\text{H}_6$ , by thermal decompn. of these gases in a heated tube. The essential feature of the process is the use of a porous refractory tube through which a certain amt. of inert gas is allowed to pass. By this means the reacting gas is unable to touch the walls of the system, and deposition of C thereon is prevented. Yields as high as 60% of the C in the gas, corresponding to 65 lb. per 1000 cu. ft., have been obtained from a 70-30  $\text{C}_2\text{H}_4$ - $\text{C}_2\text{H}_6$  mixt., and 19 lb. from  $\text{CH}_4$ . The C black produced by this process is similar to the "soft" blacks in that it has a high d., low sorptive power, it accelerates the cure in a rubber stock and gives similar resistance to aging and abrasion. It imparts greater stiffness to a rubber stock, however, and in many ways occupies a position between the channel and the "soft" blacks. J. W. Shipley.

Preparation and catalytic oxidation of pure, amorphous carbon. Jesse E. Day, et al. *Ind. Eng. Chem.* 28, 234-8 (1936), cf. C. A. 30, 1291'.—In an effort to improve the production rate of amorphous C, suitable for use in oxidation rate studies, many sources of methods were tested. The ash content of C made from sucrose and lactose by  $\text{H}_2\text{SO}_4$  and thermal decompn. was about the same as the original ash content. Phosphates and silicates cannot practically be removed from these sugars. C made in Pyrex glass and glazed porcelain contained 2-10 times more ash than when Pt vessels were used. C made from liquid hydrocarbons contained 0.02-0.13% ash and considerable tar. Unpurified natural gas and  $\text{C}_2\text{H}_2$  gave C contg. high ash, but purified  $\text{C}_2\text{H}_2$  gave C contg. 0.01-0.05% ash. Samples of C so prepd. were oxidized in pure  $\text{O}_2$  under controlled temp. and the gas stream was analyzed continuously for  $\text{CO}_2$ . C samples preheated or activated to remove tars were more resistant to oxidation. The effect of a no. of admixed oxides on rate of oxidation of unactivated C is shown. Some inhibit, others accelerate, oxidation as much as 100-200 times. E. M. Symmes.

Purified wood charcoal and its utilization in gas producers. Jean-Daniel Maublanc. *Industrie chimique* 22, 734-7 (1935).—The prepn. of purified wood charcoal is discussed and its merits for the production of lean gas for use in explosion engines are pointed out. A. P.-C.

Extractable mineral components of active carbon preparations. Jenő Mótusz. *Kísletügyi Közlemények* 38, 169-72 (1935).—Analysis of 4 types of active C (Carbontop-Frankfurt, Iponit-Ratibor, Charbon activ-Pans and Carbo-Merck) showed the following ash contents: 17.64, 11.13, 11.49 and 1.78%, of which 0.165, 0.011, 0.176 and 0.000% was  $\text{MnO}$  and 2.50, 0.77, 3.64 and 0.08% was  $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ , resp. Investigation proved that on exptl. clarification of wines, waters and vinegars much mineral matter was dissolved and the ash content of the clarified liquors much increased. It is proposed that active C with only 1% sol. ash, of which the amt. of Fe and Mn salts should be under 0.01-0.05%, should be used for the above purposes. S. S. de Finály.

Natural and activated decolorizing earths. C. L. Alexanian. *Ann. combustibles liquides* 10, 1097-1127 (1935).—A review, covering the origin, compn., chem. and heat activations, testing and use of decolorizing earths. Sixty references.

Volatilization of silica and Si as Si sulfide (Dolch). 6. Importance of C black to the rubber industry (Garner). 30. Glauber salt (Lavine, et al.). 8. Electrolytic decompn. of caustic phenolates (Drees, Kowalski). 4. Hydrogenation of carbonyl compds. by Ni and platinumized Ni—influence of an alkali (Delépine, Horeau). 10. Revivifying metallic salt solns. used for treating hydrocarbon oils (U. S. pat. 2,023,473) 22.

Wilson, A. P.: Precipitated Chalk: History, Manufacture and Standardization. 2nd ed. Birmingham: John and E. Sturge, Ltd. 62 pp. Reviewed in *Chem. Trade J.* 97, 488 (1935).

Apparatus for cooling and absorbing gaseous hydrogen chloride P. A. Persyannov, Russ. 38,130, Aug. 31, 1934. The app. consists of a tower cooled by spraying with water and a ceramic conduit, connecting the tower with the sulfate kiln, cooled on the outside with water and on the inside with dil. HCl from the tower.

Nitric acid concentration Ingenium Hechenbleikner (to Chemical Construction Corp.), U. S. reissue 19,837, Jan. 28, 1925, 1,921,255 (C. A. 27, 5159).

Concentrated nitric acid Ammoniaque synthétique et dérivés, Soc. anon. Belg. 408,670, April 30, 1935. A first  $\text{N}_2\text{O}$  fraction is shown in the liquid state by absorbing the nitrous gases in highly cooled, concd.  $\text{HNO}_3$ . After the gases are washed with dil.  $\text{HNO}_3$ , part of the resultant soln. is heated to obtain a 2nd  $\text{N}_2\text{O}$  fraction. The  $\text{N}_2\text{O}$  fractions are made to react with O and a soln. of  $\text{N}_2\text{O}$  in dil. acid.

Contact sulfuric acid process Alphons O. Jaeger (to American Cyanamid & Chemical Corp.), U. S. 2,029,530, Feb. 4. For producing  $\text{SO}_3$ , reaction of  $\text{SO}_2$  with O-contg. gases is effected in the presence of a catalyst comprising massive fragments of diatomite brick with which are associated at least one non-base-exchanging catalytically active compd. of V such as Na vanadate and at least one promoter such as alumina. U. S. 2,029,531 relates to the similar use of a catalyst comprising at least one non base-exchanging compd. of V, at least one compd. of an alkali-forming metal, at least one compd. of Al and an inactive siliceous carrier.

Sulfuric acid by the contact process Cyril B. Clark (to American Cyanamid Co.), U. S. 2,028,733, Jan. 21. A gas contg.  $\text{SO}_2$ , together with sufficient O for oxidation to  $\text{SO}_3$  and a gaseous combustible H-contg. material having a H content greater than 3 mg. per cu. ft., is subjected to oxidation in a sulfuric acid converter in the presence of a sulfuric acid contact mass at a reaction temp. so that the  $\text{SO}_2$  is oxidized to  $\text{SO}_3$  and the H content of the gas is oxidized to  $\text{H}_2\text{O}$ , the reacted gases are passed into a sulfuric acid absorber without permitting the temp. to fall below the dew point for acid mist and the exhaust gases from the absorber are subjected to dry pptn. of acid mist. App. is described. Cf. C. A. 30, 578.

Sulfuric acid manufacture by the contact process Cyril B. Clark (to American Cyanamid & Chemical Corp.), U. S. 2,028,739, Jan. 28. Sulfide ores contg. relatively large amounts of impurities including halogens and As are burned under such conditions that a hot  $\text{SO}_2$  gas mixt. is obtained contg. gaseous halogen and As compds. and Fe oxide, and the hot gaseous mixt. is maintained at temps. above  $315^\circ$  for a sufficient length of time to permit the gaseous impurities to react with suspended solids, including the Fe oxide, dust, fume and non-volatile constituents are subsequently removed without material reduction of the gas temp., and the resulting gases are passed through a catalyst of the vanadium type at reaction temps.

Vanadium catalyst Henry Joseph (to General Chemical Co.), U. S. 2,029,576, Feb. 4. A catalyst suitable for use in oxidizing  $\text{VO}$  to  $\text{SO}_3$  is prep'd by forming a mixt. contg. a finely divided carrier such as kieselguhr and an org. gum such as gum tragacanth and adding to this mixt. a soln. of a V compd. such as Na vanadate and then heating the mass to oxidize the org. material.

Hydrolyzing and distilling persulfate solutions Carl W. Tucker and Frank Dorrell (to L. I. du Pont de Nemours & Co.), U. S. 2,028,481, Jan. 21. A soln. such as one of persulfuric acid or a persulfate is divid. in a 1st stage under subatm. pressure to vaporize water together with part of the available  $\text{H}_2\text{O}$ , resulting from the hydrolysis of the persulfate soln., substantially all the vapors thus formed are sep'd from the liquid residue, water is added to the residue and the dil'd residue is dist'd in a subsequent stage. App. is described.

Catalysts for ammonia synthesis S. S. Lachnov, Russ. 38,135, Aug. 31, 1934. Fe is heated in a stream of O with the addn. of activators such as  $\text{Al}_2\text{O}_3$  or  $\text{KNO}_3$ , and after oxidation is complete the product is maintained for some time at the m. p.

Ammonia distillation from aqueous ammoniacal brines George G. Day (to Matheson Alkali Works), U. S. 2,029,467, Feb. 4. The brine is circulated from a distn. column to a reaction vessel in which there is maintained a liquid body contg. free lime, liquor substantially inert with respect to lime is withdrawn from said body, dry lime is introduced into the withdrawn liquor and it is returned to the vessel and thence to the distn. column. App. is described.

Catalysts for the combustion of ammonia W. C. Heraeus G. m. b. H. Pr. 789,599, Oct. 31, 1935. The metal fabric is made of Pt-Rh wires, those for the warp being of greater hardness than those for the wft. The difference may be obtained by using alloys contg. different amts. of Rh or by reheating the wft wires.

Purifying caustic soda solutions Raymon L. Vander Cook and Alexander M. Lawson (to Pennsylvania Salt Mfg. Co.), U. S. 2,028,898, Jan. 29.  $\text{Na}_2\text{SO}_3$  is added to form a substantially insol. complex salt with NaCl and NaOH, the complex salt is removed and  $\text{Na}_2\text{SO}_3$  is recovered from it. For preventing accumulation of  $\text{Na}_2\text{CO}_3$  in the system, recovered  $\text{Na}_2\text{SO}_3$  is dissolved in water,  $\text{H}_2\text{SO}_4$  is added and the complex salt is treated with the soln. to dissolve NaCl and NaOH from it, leaving at least a portion of the  $\text{Na}_2\text{SO}_3$  undissolved, the undissolved  $\text{Na}_2\text{SO}_3$  is sep'd from the soln., and at least a portion of the recovered  $\text{Na}_2\text{SO}_3$  is reused in purifying further amounts of NaOH soln. Cf. C. A. 29, 3790.

Caustic alkali sticks, etc. I. G. Farbenind A-G (Frist. Heintze, inventor), Ger. 620,672, Oct. 24, 1935 (Cl. 12 15). See Brit. 417,465 (C. A. 29, 1216).

Apparatus for hydrating lime V. P. Ponomarev, Russ. 38,475, Aug. 31, 1934. Construction details.

Alkaline earth carbonates H. G. J. Lawarrée, Belg. 407,451, Feb. 28, 1935. The carbonates are ppt'd by the action of  $\text{CO}_2$  on solns. of alk. earth phenolates or cresylates, the phenols being returned to the operating cycle. Cf. C. A. 29, 3790.

Alkali hyposulfites I. G. Farbenind A-G (Rudolf Stroh and Wolfgang Bulow, inventors), Ger. 620,760, Oct. 26, 1935 (Cl. 12: 22). Alkali sulfites are reduced in aq. soln. by means of Zn dust in the presence of sufficient  $\text{CO}_2$  to convert all the Zn to  $\text{ZnCO}_3$ , which is readily sep'd from the hyposulfite soln. Thus, an aq. soln. of  $\text{NaHSO}_3$  is heated in an autoclave with Zn dust and  $\text{CO}_2$  under pressure to give an 80% yield of  $\text{Na}_2\text{SO}_3$ , the Zn forming  $\text{ZnCO}_3$  which is filtered off.

Alkali metal nitrates from ammonium nitrate, Philipp Oswald and Walter Geisler (to I. G. Farbenind A-G), U. S. 2,029,738, Feb. 4. An alkali metal chloride such as NaCl is suspended in a soln., sat'd, with alkali metal nitrate,  $\text{NH}_4\text{Cl}$  and alkali metal chloride, and  $\text{NH}_4\text{NO}_3$  dissolved in a soln. of the same compn. is added to the suspension.

Alkali metal sulfite and bisulfite solutions Gustaf Haglund (to Patentaktiebolaget Gröndal-Kämen), U. S. 2,029,610, Feb. 4. Waste liquor from a cellulose digestion process employing cooking liquor contg. alkali metal S compds. such as that from a sulfite process is treated (suitably by incineration and leaching) to eliminate org. compds. and to form a soln. contg. an alkali metal sulfite and substantially free from sulfides and thiosulfates, the soln. is subjected to the action of  $\text{SO}_2$  and one or more Ca compds. such as  $\text{CaCO}_3$  capable of reacting with  $\text{SO}_2$  to form  $\text{Ca(HSO}_3)_2$ , until the alkali metal salts contained in the soln. have been converted to bisulfites, and the resulting soln. is subjected to the action of one or more alkali metal compds. capable of reacting with  $\text{Ca(HSO}_3)_2$  to form one or more insol. Ca compds. An arrangement of app. is described. Cf. C. A. 29, 894.

Alum M. E. Liffendi and V. F. Nagrev, Russ. 38,137, Aug. 31, 1934. Alunite ore broken up into 2-3 mm. pieces is heated for 35-40 min. at  $700-80^\circ$ , added to water and left for 24 hrs. to cool while being agitated with air.

Aluminum compounds Erik L. Rinman, Brit. 435,870, Oct. 1, 1935. Aluminiferous materials having a high percentage of  $\text{SiO}_2$ , e. g., kaolins, clays, certain

bauxites and not more than 12% of Fe are preferably roasted and then treated with HCl to obtain a soln. of chlorides, which after sepn. from undissolved  $\text{SiO}_2$ , etc., is treated with Ca aluminate (I) to ppt  $\text{Al}(\text{OH})_3$ , and obtain a  $\text{CaCl}_2$  soln. A part of the  $\text{Al}(\text{OH})_3$  is converted into a Na aluminate (II) soln. from which pure  $\text{Al}(\text{OH})_3$  may be obtained, and the remainder is mixed with the  $\text{CaCl}_2$  soln., evapd to dryness and, after optional pulverization, heated in presence of steam to obtain HCl and I for re-use as above. In a modification, the chloride soln. is pptd with a mixt. of I and  $\text{CaCO}_3$ , the resulting mixt. of  $\text{Al}(\text{OH})_3$  and  $\text{CaCl}_2$  soln. dried, pulverized and heated in presence of steam to obtain HCl and I, a part of I is treated with  $\text{Na}_2\text{CO}_3$  to obtain  $\text{CaCO}_3$  and II, the other part of I is re-used for pptg the chloride soln. and  $\text{Al}(\text{OH})_3$  is recovered from the II soln., e. g., by treatment with  $\text{CO}_2$  to obtain  $\text{Na}_2\text{CO}_3$  for treating fresh quantities of I. The undissolved  $\text{SiO}_2$  may be mixed with portland cement to adapt it for use in concrete structures below water.

**Crystals of boron carbide** V. G. Evreinov. Russ. 38,134, Aug. 31, 1934. A mixt. of B and coal is covered with small pieces of an alloy contg. about 10% Ag and 90% Cu for the purpose of dissolving  $\text{B}_2\text{C}$ . The alloy is removed with  $\text{HNO}_3$  after fusion.

**Calcium aluminates** Jean C. Seailles. Fr. 789,323, Oct. 26, 1935. App. is described for purifying crude Ca aluminates by progressive extns. by water or dil. solns. from a previous treatment under such conditions that there is constantly in contact with the solvent an amt. of solid very much above that which corresponds to the normal soly., the contact being maintained for a detd. time preferably with agitation.

**Dicalcium phosphate** Franciscus Visser't Hooft (to Lucidol Corp.). U. S. 2,029,967, Feb. 4. A non-caking, free-running product of sufficient purity to be suitable for use with foods is prepd. by mixing about 5% or less of mono-Ca phosphate and a caking inhibitor such as  $\text{CaO}$ ,  $\text{NaHCO}_3$  or Na benzoate with a dry com. pptd. di-Ca phosphate.

**Iron tannate** M. Tz. Robert-Nik and V. M. Malushev. Russ. 37,703, July 31, 1934. Fe tannate is prepd. by passing an alk. soln. of tannin downward through a tower filled with Fe (rods, sawings, etc.) while air is passed upward through the tower.

**Basic lead carbonate.** A. Rouma and M. Biske. Belg. 407,862, March 30, 1935. Pb hydroxide is pptd. from a soln. of a Pb salt by means of a base, and part of the hydroxide is converted into carbonate; the hydroxide and the carbonate are mixed in suitable proportions to obtain a basic carbonate of the desired compn.

**Magnesium bisulfite from dolomite** rock Clyde D. W. gal (to Inland Lime and Stone Co.). U. S. 2,028,630, Jan. 21. The rock is calcined to produce a mixt. of  $\text{CaO}$  and  $\text{MgO}$ , water is added, and the mixt. is chlorinated to effect selective chlorination of the Ca without chlorinating substantial amounts of the Mg, the residual  $\text{MgO}$  is sepd., and is treated with  $\text{SO}_2$  to produce  $\text{Mg}(\text{HSO}_3)_2$ .

**Magnesium carbonate** Soc. générale du magnésium. Fr. 789,670, Nov. 4, 1935. Neutral anhyd  $\text{MgCO}_3$  is prepd. by calcining dolomite or limestone, treating with a soln. of  $\text{MgCl}_2$  and treating the suspension obtained at 170–200° in an autoclave with  $\text{CO}_2$ , e. g., that obtained in the calcination.

**Magnesium carbonate trihydrate** V. V. Shelyagin. Russ. 34,454, Feb. 23, 1934.  $\text{CO}_2$  is passed through a suspension of  $\text{MgO}$  or dolomite in the presence of 1–2% of a sulfonic acid of the  $\text{C}_6\text{H}_5$ , naphthalene or quinoline series, petroleum sulfonic acid or alizarin oil as catalyst. For example, 6 g. of finely ground  $\text{MgO}$  is mixed with 200 cc.  $\text{H}_2\text{O}$  and 0.12 g. petroleum sulfonic acids and the mixt. is then treated with  $\text{CO}_2$  for 1 hr. 45 min. at 40–50°.

**Natural phosphates** Soc. d'études pour la fabrication et l'emploi des engrais chimiques (Georges Chaudron, inventor). Fr. 789,497, Oct. 20, 1935. Natural phosphates are washed with slightly acidified water to eliminate

parts rich in Al, Fe and Si and org. materials and poor in  $\text{P}_2\text{O}_5$ . The enriched phosphate is attacked by acid.

**Decolorizing phosphate solutions.** George E. Taylor (to General Chemical Co.). U. S. 2,028,632, Jan. 21. A phosphate soln. such as one formed in Na phosphate production and contg. oxidizable impurities is treated with chromic acid to oxidize the impurities, and the Cr is then pptd. from the soln. (suitably by an alkali).  $\text{Na}_2\text{Cr}_2\text{O}_7$  also may be used.

**Phosphates** Kali-Forschungs-Anstalt G. m. b. H. Brit. 436,335, Oct. 9, 1935. This corresponds to Fr. 782,546 (C. A. 29, 7025').

**Treating mixtures of potassium nitrate and ammonium chloride to effect separation.** Carl Kireher, Fritz Mueller and Hermann Suessenguth (to I. G. Farbenind. A.-G.). U. S. 2,029,623, Feb. 4. A mixt. of  $\text{KNO}_3$  and  $\text{NH}_4\text{NO}_3$  (such as a reaction product from KCl and  $\text{NH}_4\text{NO}_3$ ) is subjected to extraction with anhydrous liquid  $\text{NH}_3$  contg.  $\text{NH}_4\text{NO}_3$  and then with anhydrous liquid  $\text{NH}_3$  and the soln. is sepd. from the residue.

**Rare earth compounds** I. G. Chentzov. Russ. 38,139, Aug. 31, 1934. Apatite concentrates are treated with  $\text{HNO}_3$  and soln. is cooled to  $-10^\circ$  to  $-15^\circ$ . The cryst. mass formed (contg.  $\text{Ca}(\text{NO}_3)_2$ ) is filtered off and dissolved in a small amt. of water. Phosphates of the rare earths sep. in flakes.

**Recovery of sodium from its mixtures with calcium and nonmetallic substances** Harvey N. Gilbert (to C. I. du Pont de Nemours & Co.). U. S. 2,029,998, Feb. 4. The mixt. is introduced into a fused salt sepg. bath having a sp. gr. greater than that of Na, contg.  $\text{CaCl}_2$  together with over 30% of  $\text{NaCl}$ , at a temp. of 600–800°, to sep. the Na from associated substances. App. is described.

**Sodium bicarbonate** H. G. J. Lawarrée. Belg. 408,516, April 30, 1935. An amine in the form of carbonate, particularly apt to absorb and give up  $\text{CO}_2$ , is used to catalyze the reaction.

**Sodium bisulfite** G. E. Shnuparkov. Russ. 37,701, July 31, 1934.  $\text{NaHSO}_3$  is prepd. by melting  $\text{Na}_2\text{SO}_3$  with  $\text{SiO}_2$  in the presence of C and passing the gases obtained in the process through a soln. of the previously prepd. melt so as to convert the latter into  $\text{NaHSO}_3$  and  $\text{SiO}_2$ .

**Titanium dioxide** William T. Little (to American Zirconium Corp.). U. S. 2,029,881, Feb. 4. A soln. of a Ti salt such as the sulfate is hydrolyzed by heating in the presence of a solid alkali titanate such as Na titanate which facilitates the process.

**Zinc sulfide** Thomas A. Mitchell and Royal L. Sessions (to Hughes-Mitchell Processes Inc.). U. S. 2,028,162, Jan. 21. In a cyclic process, a 7n compd. such as  $\text{ZnO}$  is treated with HCl to form  $\text{ZnCl}_2$ , the latter is treated in soln. with Na sulfide to ppt. the Zn as a sulfide, the resulting  $\text{NaCl}$  soln. is removed and used for HCl production by treatment with  $\text{H}_2\text{SO}_4$  and the  $\text{Na}_2\text{SO}_4$  thus formed is reacted with a Ba compd. such as  $\text{BaCl}_2$  to form  $\text{BaSO}_4$ , and the latter is heated with a reducing agent such as carbon to form Ba sulfide which is used with  $\text{Na}_2\text{SO}_4$  to form Na sulfide. Numerous auxiliary details of procedure are described. Cf. C. A. 30, 582.

**Metallic barium.** A. A. Ivanov. Russ. 37,849, July 31, 1934.  $\text{Ba}(\text{OH})_2$  is fused with  $\text{Al}(\text{OH})_3$  and the Ba aluminate formed is thermally reduced by known methods.

**Hydrogen** George F. Jaubert. Ger. 620,693, Oct. 26, 1935 (Cl. 12.1.01). App. for producing H from Si, caustic alkali and water is described. Sufficient alkali to convert the Si into  $\text{NaHSiO}_2$  at 15 atm. and 150° is used. Cf. C. A. 29, 6376.

**Iodine, sulfur and phosphates** Eugène J. Burban. Fr. 789,585, Oct. 31, 1935. Ca phosphates are treated with dil.  $\text{H}_2\text{SO}_4$  to produce  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  and  $\text{CaSO}_4$ . The  $\text{CaSO}_4$  is reduced to  $\text{CaS}$  by calcination in the presence of marine algae in the absence of air, and the kelp obtained is used for the extn. of I and pptn. of Ca phosphate with liberation of  $\text{H}_2\text{S}$  which may be decompd. *in situ*.

**Oxygen of high purity.** George J. Boshoff (to The Linde Air Products Co.). Brit. 435,281, Sept. 18, 1935. This corresponds to U. S. 1,985,763 (C. A. 29, 1219').

**Removing phosphorus from blast-furnace gases** Friedrich P. Kerschbaum, William H. Waggaman and Stapleton D. Gooch (to Pembroke Chemical Corp.). U. S. 2,029,663, Feb. 4. Blast furnace gases relatively dilute in P are passed relatively slowly through a series of condensers in which the gases are sprayed with water, at progressively lower temperatures, and the bulk of water in the first condenser is rapidly recirculated for recontact with a given quantity of the gas and is maintained at a temperature of 43-100°, the water in the second condenser being maintained at 18-43° and that in the final condenser at 0-18°.

**Platinum catalyst** P. P. Budnikov. Russ. 37,698, July, 1934. An inert carrier is impregnated with a solution of Pt salts together with reducing agents such as essential oils or turpentine pretreated with S or  $\text{SCl}_2$ . The impregnated mass is ignited.

**The continuous extraction of sulfur by means of carbon disulfide** Ammoniaque synthétique et dérivés, Soc. anon. Belg. 408,374, April 30, 1935. The S-containing material is introduced continuously at one end of the apparatus and the  $\text{CS}_2$  at the other end.

**Sulfur from pyrite and similar materials** A. C. Makovetskii. Russ. 37,699, July 31, 1934. Materials low in S are extracted with an aqueous solution of  $\text{SO}_2$ .

**Sulfur dispersions** Geo. R. Tucker (to Dewey and Almy Chemical Co.). U. S. 2,028,482, Jan. 21, 1935. S is ground in an aqueous solution of a solvent comprising a condensation product of an aldehyde such as formaldehyde with a sulfonic acid of the naphthalene series, to obtain a stable fluid product substantially free from S in a form which would produce caking and showing an especially active Brownian movement.

**Producing agglomerates from phosphate rock** Harry A. Curtis and Armand J. Abrams (to Tennessee Valley

Authority). U. S. 2,020,309, Feb. 4. In preparing charging stock for a P reduction furnace, fine coking coal is added to a hot unsintered mixture of fine phosphate rock and fine silica and the materials are mixed until the mixture contains semicoke.

**Apparatus for producing sulfur dioxide from sulfide ores, etc.** Harold O. C. Ingraham (to General Chemical Co.). U. S. 2,030,021, Feb. 4. A suspension of finely divided material such as Fe or Zn sulfide fines in an oxidizing gas such as air is introduced into an inlet at one end of a reaction chamber having a discharge opening adjacent a wall forming the opposite end of the chamber so that solid materials are separated from reacted gases, and a connection is provided near the outlet end for introducing supplemental oxidizing gas into the reaction chamber, reacted gases being caused to flow through a chamber surrounding the reaction chamber and in contact with the outer walls of the latter.

**Expanding vermiculite** Paul S. Denning (to F. E. Schundler & Co.). U. S. 2,029,521, Feb. 4. The material is suddenly subjected to a high temperature within an enclosed space for not over 15 sec. and is agitated and then suddenly subjected to atmospheric temperature and submerged in water, excess moisture is removed and the mass is immediately exposed to a high temperature within a closed space for not over 15 sec. and then finally suddenly subjected to atmospheric temperature. Apparatus is described provided with an elongated passage through which the material is passed.

**Regenerating used clays** International Hydrogenation Patents Co. Ltd. 1,789,150, Oct. 24, 1935. Clays used for refining lubricating oil fractions, waxes and naphthas are regenerated by treating them with a solvent prepared by destructive hydrogenation in the vapor phase of petroleum distillates. An aliphatic alcohol or ketone may be added to the solvent.

## 19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. E. BARTON AND C. H. KERR

**Specific gravity of glass** Ikutaro Sawai, Morio Nishida and Ikuzo Kubo. IX Congr. intern. quim. pura aplicada, Madrid 3, 343-51 (1934).—A balance, from one arm of which a Pt ball immersed in melted glass is suspended, can be used to determine both sp. gr. and viscosity. Sp. gr. was calculated from the velocity of the ball by Stoke's law. The sp. gr. temperature curve bends sharply at 500° and slightly at 1200°. The viscosity-temperature curve also bends at 1200°. The gain in sp. gr. with rapid cooling increases rapidly between 450° and 600°, the range in which all transition temperatures, determined from several properties, were observed. The effect of annealing on sp. gr. was also investigated.

**Hardness of glasses** M. Pirani and A. Fehse. *Glastech. Ber.* 14, 21-2 (1935).

**Determination of the pendulum hardness of glasses** W. Schmidt and H. F. Gronow. *Glastech. Ber.* 14, 23-6 (1935).—The effect of 3%  $\text{Al}_2\text{O}_3$  on the pendulum hardness of glasses is about the same as 10%  $\text{MgO}$ . There is little difference between 5 and 10%  $\text{MgO}$ . Glasses containing boron oxide show a maximum effect at 12%. The glasses with 5, 10 and 15%  $\text{CaO}$  are harder than the corresponding  $\text{BaO}$  compounds. Lead glasses increase in hardness to 20%  $\text{PbO}$  content and drop rapidly again in going to 25%  $\text{PbO}$ . The results correspond quite well to the results obtained by studying other physical properties such as elasticity, etc.

**Opal glass. I. Silicon, calcium, sodium, oxygen, fluorine series** Henry H. Blau, Alexander Silverman and Victor Hicks. IX Congr. intern. quim. pura aplicada, Madrid 3, 507-34 (1934).—Pure soda lime glasses were prepared by replacing  $\text{Na}_2\text{O}$  with  $\text{NaF}$  and  $\text{CaO}$  with  $\text{CaF}_2$ , and x-ray pictures were taken of the raw materials and of the glasses before and after heat treatment. By comparing the patterns, the crystallinities responsible for opalescence were determined. For glass annealed at 820° for 30 hrs they were low temp. quartz; for 30 hrs at 650°, low-

temp. quartz with traces of low-temp. cristobalite, for 1 hr at 820°, primarily low-temp. cristobalite. Fluoride lines were very weak and approximated closely the lines for quartz and cristobalite. Much  $\text{H}_2$  is lost during the heating. *Cl. C. A.* 29, 560.

**Removal of anisotropy in glass by annealing** Jean Herbert. *Compt. rend.* 202, 214-15 (1936).—The anisotropy and zones structure rendered visible by polarized light, and occurring especially in window glass, is due to layers of slightly different composition, and the method of manufacture. It is removed by annealing for 30-40 hrs (cf. Gaubert, *C. A.* 29, 1221).

**A new method for the application of luminescent screens to glass surfaces** W. H. Kohl. *Can. J. Research* 13A, 126-32 (1935).—An outline is given of the various methods for the application of luminescent screens, and their relative merits. Most widespread in use are methods that utilize a liquid binder that enters into a chemical reaction with the glass surface. In other cases, the powder is partially imbedded in the glass surface by a sintering process. The physical characteristics of the luminescent screen are adversely affected by both of these methods. A process based on the activation of the glass surface by a deposit of S is described. The S coating picks up the luminescent powder in a uniform layer when it is brought into contact with the powder. After being removed by means of a moderate heat treatment, it leaves the screen firmly adhering to the glass surface. Possible mechanisms for the process are discussed.

**The use of coke-oven gas in the glass industry.** R. Allolio. *Glastech. Ber.* 14, 15-20 (1935). J. F. Hyde. **Ceramic art and education** Introduction. Lawrence I. Barringer. *Bull. Am. Ceram. Soc.* 15, 1-2 (1936).—An introduction to the symposium at the Oct. 1935 meeting of the Art Division of the Am. Ceram. Soc. C. H. Kerr. **Ceramic art problems in the colleges** M. E. Holmes. *Bull. Am. Ceram. Soc.* 15, 2-4 (1936). C. H. Kerr.

Art, industry and education in ceramics. R. Guy<sup>1</sup>  
Cowan. *Bull. Am. Ceram. Soc.* 15, 5-13(1936)

Electrical ultramicrometer for measuring the coefficient of thermal expansion of ceramic materials. Adolf Heinz and Horst Kottas. *Spektrum* 68, 49-51, 65-67(1935)

Ceramic firing I The problem and method of procedure Otto Krause and Iherhard Keetman *Spektrum* 68, 1(1935)—Important factors in firing are (1) structural changes, (2) duration of firing and (3) the furnace atm. Changes in ceramic ware during firing result from structural changes and sintering processes. The structural changes consist of decomposition, crystal transformation, fusing and sintering processes. Shrinkage, swelling, and porosity changes belong to the 2nd group. The relation of the above-mentioned factors to the changes in structure, sintering processes and properties were investigated. C. B. Jenni

Earthquakes and Montana clay manufacture Archie C. Bray. *Bull. Am. Ceram. Soc.* 15, 32(1936)

Determination of calcium sulfate, as such, in clays Charles A. Peters. *Bull. Am. Ceram. Soc.* 15, 15-16(1936)—The amt. of sample can be varied depending upon the amt. of  $\text{CaSO}_4$  suspected, but 100 g. is usual. Put this into a liter flask and fill to the mark. Shake and allow to settle. Pour off more than 500 cc. into a 500-cc. wide-mouthed bottle and again allow to settle. Filter off 500 cc. into a beaker and evaporate. As evaporation proceeds the clay particles will coagulate and should be filtered out, the liquid being returned to a 250-cc. beaker. One filtering at the right time is usually sufficient. When the liquid is reduced to a small vol. it is examined under the microscope for  $\text{CaSO}_4$  crystals. When these appear readily, showing that satn. has been reached, add a few drops of  $\text{H}_2\text{SO}_4$ , cool, dil. with 4 vols. of alc., and let stand overnight. Filter on asbestos, wash with alc., gently ignite the ppt. and weigh as  $\text{CaSO}_4$ . If a 100-g. sample was used, multiply by 2 and again by 0.6 to allow for the vol. occupied by the clay.  $\text{MgO}$  can be detd. in the alc. filtrate by the usual phosphate pptn. C. H. K.

Testing the clay from Ordzhonikidze (formerly Viadikavskas) at the "Krasnui Keramik" works at Borovitski in respect to its fitness for the production of sewer pipe T. I. Melnikova and A. K. Freberg. *Siroitel. Material* 1935, No. 7, 36-40.—The clay contains  $\text{SiO}_2$  57.0-62.0,  $\text{Al}_2\text{O}_3$  4.0-5.0,  $\text{Fe}_2\text{O}_3$  5.6-7.6,  $\text{CaO}$  1.15-3.5,  $\text{MgO}$  0.0-1.0,  $\text{SO}_2$  about 0.5%, ignition loss is 3.5-5.6%, refractoriness 1170°, sintering temp. 1080°. The clay proved satisfactory with 20% of refractory clay added. E. E. Stefanowsky

Waste rock from coal pits as a ceramic raw material. L. Ya. Mishulovich. *Siroitel. Material* 1935, No. 7, 40-3, cf. N. Chibunovskii, *Siroitel. Material* 1930, No. 9, 10, 123-6.—Shales from the Donetz basin coal pits are suitable for making brick. The large interval between their sintering and melting points makes possible a dense, durable product of low porosity, especially when finely ground (under 1 mm.). Plastic clay must be added. E. E. Stefanowsky

Promotion of the drying of clay by the coagulating effect of acid H. H. Macey. *Trans. Ceram. Soc. (Engl.)* 34, 396-416(1935).—The 24 fire clays and brick clays treated with increments of HCl varied widely in responding to its power of preventing cracks. A sedimentation test predetermines satisfactorily the behavior of the clays and the quantity of HCl needed to avoid cracking. The action of the acid on plastic clay bodies is due to the opening up of their water channels. The amt. of acid found necessary was less than 0.25% in all cases. Those clays benefitted by HCl treatment showed no tendency to lose this advantage in the firing process. H. F. K.

Potting clays. A. Rigby. *Trans. Ceram. Soc. (Engl.)* 34, 381-91(1935).—General discussion of the geology, prep., characteristics and uses of potting clays. H. F. K.

Pottery printing—the engraving. J. H. Latchford. *Trans. Ceram. Soc. (Engl.)* 34, 417-20(1935).

Pottery printing paper. R. J. Bell. *Trans. Ceram. Soc. (Engl.)* 34, 421-3(1935).

Uses of coal for brickworks. W. H. Gamble and R. F. Paget. *Brit. Clayworker* 44, 311-14(1936).

Frit-kiln bricks. W. Hugill. *Foundry Trade J.* 53, 421, 420(1935).

Effect of oxides of iron and chromium and a reducing atmosphere on the rate of tridymite formation in a silica brick batch T. R. Lynam and W. J. Rees. *Trans. Ceram. Soc. (Engl.)* 34, 507-14(1935). *Bull. Brit. Refractories Research Assoc. No. 28* (Aug., 1932).—Neither of the oxides had much effect on the tridymite formation under reducing conditions. Addition of Siemens' slag and Prussian blue were most effective in open-kiln burning, showing as much as 60-75% tridymite formation. H. F. K.

Effect of the water content of silica brick batches on the porosity of the fired brick T. R. Lynam and W. J. Rees. *Trans. Ceram. Soc. (Engl.)* 34, 500-6(1935). *Bull. Brit. Refractories Research Assoc. No. 24* (Jan., 1931).—A low moisture content is favorable to low permeability, the permeability decreasing more rapidly than the porosity. Moisture below 7.5% is not commercially feasible unless mech. mold filling and heavy machine pressing are used. H. F. K.

Action of alkalis on refractory materials I. Action of potash vapor on refractory materials at 900° and 1000°. F. H. Clews, A. Green and A. T. Green. *Trans. Ceram. Soc. (Engl.)* 34, 436-55(1935). *Bull. Brit. Refractories Research Assoc. No. 32* (Oct., 1933), cf. C. A. 29, 67181.—The aluminous fireclay refractory absorbed more  $\text{K}_2\text{O}$  at 900° and disintegrated more than did the more siliceous fireclay materials which were protected by the formation of an external viscous silicate. With silica brick a liquid silicate formed, which dropped off the specimens and gave no protection. The extent to which chem. decomposition occurred was measured by treatment with dil. HCl. The fireclay test pieces showed extensive chem. decomposition after prolonged exposure to  $\text{K}_2\text{O}$  at 1000° even though they were apparently only superficially altered. Silica refractories impregnated up to 4% with  $\text{K}_2\text{O}$  showed much higher losses when heated to 1000° than did the fireclay materials. A preliminary heating for 5 hrs. at 1200° results in an almost negligible loss of  $\text{K}_2\text{O}$  from fireclay refractories at 1000°. H. F. K.

Problems for research on refractories. Anon. *Bull. Am. Ceram. Soc.* 15, 22-8(1936).—A discussion and listing of problems by the Research Com., Refractories Div., Am. Ceram. Soc. C. H. Kerr

Texture of refractories I. Nonregularity of texture of gas-works fire-clay refractories and its possible effect on durability. T. I. F. Rhead and R. E. Jefferson. *Trans. Ceram. Soc. (Engl.)* 34, 363-73(1935).—Numerous photographs of faulty structures and cross sections of faulty refractories are given to illustrate how inequalities in texture are produced and how these affect their service behavior. II. Pictorial methods of recording the texture of refractories or similar materials. T. F. E. Rhead, J. N. Shorrocks and C. L. Evans. *Trans. Ceram. Soc. (Engl.)* 34, 373-9(1935).—Black wax, forced into the pores of the material under 30 atm. pressure of N, gave the best contrast for photographic record. Ink impressions by means of a resilient gelatin-glycerol mixt. are satisfactory for recording textures of plane surfaces or sections. H. F. K.

Properties of insulating refractories I. The behavior under load at high temperatures. S. M. Phelps. *Am. Refractories Inst. Tech. Bull.* 61, 6 pp (1935).—Strength, wt. and fusion points of various brands are given. The load-test data demonstrate the type of test which should be used in evaluating these materials. The mistake of using a load test in which the brick are under a soaking heat should not be carried over from the testing of fire-clay refractories. Various tests and data are included. E. C. Petrie

Fire-clay refractories S Matthews *Can Mining Met Bull* No 284, 614-18(1935)—General A H E  
Muffle furnace with Kryptol resistance for tests of refractory materials V A Lebedev *Zavodskaya Lab* 4, 1121 2(1935), cf. *C A* 29, 3556—Construction details Chas Blanc

Explanation of the adhesion problems in enameling iron sheets Adolf Dietzel *Sprechsaal* 68, 3-6, 20-3, 34-7, 53-6, 67-9, 84-5(1935)—The adhesion of the enamel is secured by a black inner enamel layer which adheres firmly to the Fe. Empirical results have shown that certain metallic oxides in relatively small amounts (0.5-2.0%) produce the adhering layer. The oxides of Co and Ni seem best, Cu, Mn, Cr and other oxides follow. Disadvantages of the use of adhesive oxides are (1) special methods of application, drying and burning are required and (2) the intense color may be difficult to cover. Phys and chem explanations of adhesion by earlier workers are reviewed. Two enamels were prep'd for the exptl work: I ( $\text{SiO}_2$  55.48,  $\text{B}_2\text{O}_3$  14.60,  $\text{Al}_2\text{O}_3$  7.17,  $\text{Fe}_2\text{O}_3$  0.10,  $\text{TiO}_2$  0.13,  $\text{CaO}$  4.59,  $\text{MgO}$  0.02,  $\text{Na}_2\text{O}$  11.09,  $\text{K}_2\text{O}$  4.84, loss on ignition 125%) was mixed with various amts of adhesive oxides. Forty-two mixts were studied. Enamel II was similar to I with 0.50%  $\text{CoO}$  added. The adhesive oxides were divided into 3 groups: poor, oxides of Fe, Cr, Ce, Ti, V, U and Pb, fair,  $\text{MnO}_2$ , nitrate, nitrite, good,  $\text{CoO}$  and  $\text{NiO}$ . The important reactions taking place in an enamel coated adhesive oxides were investigated. Reactions in which the adhesive oxide is not concerned are: (a)  $\text{Fe} + \text{O}_2$  (from the air)  $\rightarrow$  scale ( $\text{FeO} + \text{Fe}_3\text{O}_4$ ) (b) The scale is dissolved in the soft or liquid enamel (c)  $\text{Fe}_2\text{O}_3$  (from scale)  $+ \text{Fe} \rightarrow 4\text{FeO}$  (d)  $\text{FeO} + \text{O}_2$  (by diffusion)  $\rightarrow \text{Fe}_2\text{O}_3$ ,  $\text{FeO} + \text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4$ , supersatn of  $\text{Fe}_3\text{O}_4$ , and pptn of magnetite (e)  $\text{Fe}_3\text{O}_4 + \text{O}_2 \rightarrow \text{Fe}_2\text{O}_3$ , supersatn of  $\text{Fe}_2\text{O}_3$ , and pptn of hematite. The degree of firing in which reactions a, b and c occur was designated as A, the enamel is mostly green colored. Reaction d occurs in firing B and the enamel is black. In firing C the enamel has a red appearance as a result of the pptn of hematite according to reaction e. The poor adhesive oxides form compds with the  $\text{FeO}$  at the beginning. This oxide layer adheres to the iron like a scale. The enamel clings to these crystals and thereby to the Fe. The adhesion is imperfect and unsatisfactory. The fair adhesive oxides accelerate the formation of the "second scale layer" in firing B according to reaction d and thereby are fair as adhesives. The good and technically important adhesive oxides are reduced to the element by the  $\text{Fe} + \text{CoO} \rightarrow \text{FeO} + \text{Co}$ . The sepn of this metal forms a local current, which at high temp gives a strong corrosion of the Fe surface. The enamel clings in the cavities and adheres. The process was studied microscopically, analytically, electrochemically and thermodynamically. C B Jenni

Lead frits and fritted glazes J H Koenig *Ceram Ind* 25, 131, 136(1936) Herbert S Wilson

Drawing of ground-coat enamels for sheet steel Murray C Gausch *Bull Am Ceram Soc* 15, 14 15 (1936)—In cold weather the enamel broke after drawing while in hot weather it "lost its set." Undersmelting of the frit tended to make the enamel lose set. To overcome ground-coat drawing troubles standards must be maintained in smelting, grinding, aging and temp control. C H Kerr

Design of sheet metal parts for enameled ranges H H Holscher *Ceram Ind* 25, 124, 126, 128, 130, 132 (1936) Herbert S Wilson

Proposals for the standardization of tests of enamel against chemical attack W Dawidl *Chem Fabrik* 1936, 15-18 J H Moore

Service of silica brick in open hearth furnaces (Trubnikov) 9 Examn of the optical and elec properties of didymium glass (Prosdad, et al) 3 Metallurgical stoppers and nozzles (Trakhtenberg, Tatarskii) 9 Red sands of Alpes-Maritimes (Déb) 8 Geologic distribution of fire clays in the U. S (Chelkowsky) 8

Damour, E : Cours de verrerie. Partie 3 Le refroidissement du verre. Paris. Ch. Béranger. 273 pp. F. 55 Cf *C A* 26, 3084

Duncan, Geo S : Directory for the British Glass Industry 3rd revised ed Sheffield Soc. of Glass Technology, 416 pp 4s Reviewed in *Ceramic Abstr.* 14, 275(1935)

Patridge, J. H Refractory Blocks for Glass Tank Furnaces Sheffield Soc. of Glass Technology 128 pp 8s 6d Reviewed in *Ceram Abstr.* 16, 24(1936).

Thorpe, W. A : English Glass. London: A. & C Black 7s 6d Reviewed in *J Soc Glass Tech* 19, 326 (1935)

Ceramic Data Book and Catalogs 8th ed Chicago Industrial Publications, Inc. 330 pp Free

Ceramic Trade Directory, 1935 Edition. Newark, N. J : Ceramics Pub Co Inc 198 pp. \$5 Reviewed in *Ceramic Age* 26, 159(1935).

Glass Württembergische Metallwarenfabrik Ger 620,935, Oct 30, 1935 (Cl 326 10) A method is described of decorating glass by metal in which parts of the glass are made dull or frosted and the metal rubbed thereon. Metal powder mixed with colophony may be used.

Glassmaking Thomas F. Pearson. Fr. 789,066-7, Oct. 22, 1935 Means for supplying molten glass to the molds

Glass sheets N V Maatschappij tot Beheer en Exploitatie van Octrooien Brit 434,992, Sept 12, 1935 Addn to 355,134 (*C A* 27, 2271). The rolls of the sheet-forming machine of 355,134 are set in rotation before the molten glass is poured onto the receiving table and the distance between the rolls is reduced to such an extent that the glass cannot pass between them. As soon as the molten glass has spread evenly across the rolls the distance between them is increased so that the glass passes between them to form a sheet or strip with a substantially square front end.

Apparatus for making sheets of glass N V. Maatschappij tot Beheer en Exploitatie van Octrooien (H E Robinson and F E Eicher, applicants in U S) Fr 789,590, Oct 31, 1935

Glass-rolling apparatus Schlesische Spiegelglas-Manufaktur Carl Tielsch G m b H. Ger. 620,593, Oct 24, 1935 (Cl 32a 18)

Apparatus for the continuous formation of undulating sheets of glass Manufactures des glaces et produits chimiques de St-Gobain, Chauny & Cirey Fr 789,454, Oct 29, 1935

Laminated glass sheets James G McNally (to Eastman Kodak Co) U S 2,029,931, Feb 4 Sheets of glass are united by heat and pressure with an intervening sheet of cellulose acetate coated on both sides with a stable cellulose nitrate having a N content of 10.8-12.5%, a viscosity of not more than 50 sec and an ash of not more than 0.4%, with interposed uniting films of triacetin

Apparatus (with an endless conveyor in a vertical tank) for use in laminated glass manufacture W. Owen (to Pittsburgh Plate Glass Co) U S 2,025,916, Jan 28 Mech features U S 2,028,917 relates to a turnover table for glass sheets

Apparatus (with an endless conveyor in a vertical tank for holding heated liquid) for use in laminated glass manufacture James H Sherts (to Duplate Corp) U S 2,028,927, Jan 28 Structural and operative details U S 2,028,928 relates to a support for use in making curved laminated glass

Laminated glass John C Zola (to Duplate Corp) Can 355,180, Jan 7, 1936 Between glass sheets is interposed a sheet of cellulose nitrate plastic contg a plasticizer (such as dimethyl, diethyl, dibutyl or diamyl phthalate, triacetin, triethyl phosphate or triphenyl phosphate) and a layer of cement comprising a water-sol cement and a high boiling solvent such as monoacetyl diglycerol and diglycerol ether Cf *C A* 29, 4914

Apparatus for bending glass sheets Robert A Miller

(to Pittsburgh Plate Glass Co.) Can. 355,220, Jan. 7, 1936 Various operative and structural details are described

**Antifrost glass** John F. Biesch U. S. 2,020,218, Jan. 28, 1936 Details are described of assembly of two similar panels of glass with an intervening panel of glass such as the windshield of an automobile with a uniting border of a transparent inorganic cement such as Na silicate which provides a dead air space between the panels and is stable against the action of heat and light

**Glass Lampe Yvel Auto-Lampe & Cie., Anciens Etablissements Léo Lévy et Alfred Monnier** Brit. 436,221, Oct. 3, 1935 An incandescent electric lamp for a motor car has a bulb of glass that is made by melting a batch containing at least 0.25 parts of  $\text{CaS}$  and 0.15 of  $\text{S}$  to 10 of sand. The glass absorbs light having a wave length less than about 0.5  $\mu$

**Glass for metal-vapor lamps** Harrison P. Hood (to Corning Glass Works) Can. 354,533, Dec. 3, 1935 A glass of 55–77%  $\text{SiO}_2$ , 10–25%  $\text{B}_2\text{O}_3$  and 10–25%  $\text{Na}_2\text{O}$  resists blackening by alkali metal vapors, is not affected by ordinary atmospheric influences and has a wide range of expansion coefficients

**Crucible for making optical glass** K. G. Kumanian Russ. 38,271, Aug. 31, 1934 Construction details

**Glass** Chémische Fabrik Joh. A. Benckiser G. m. b. H. and Fritz Draibach Brit. 436,420, Oct. 10, 1935 Glass transparent to ultraviolet radiation is made by fusing Be ortho- or meta-phosphate. Ortho- or meta-phosphates of Ca, Mg and (or) Na may be added Cf. C. A. 30, 590\*

**Glass shelf construction for glass-gathering apparatus having glass-gathering molds** Albert F. Tremblay U. S. 2,028,060, Jan. 28, 1936 Structural and mechanical details

**Glass manufacture** Jakob Dichter. Brit. 435,678, Sept. 25, 1935 Vessels, e.g., ampoules, having wide bodies and extended necks are made by narrowing a wide tube at 1 end and fusing thereto a narrow tube forming the neck of the vessel. App. is described

**Machine for forming the necks of bottles that have been blown in a separate mold** Herbert A. Bateson and George E. Bateson Brit. 435,747, Sept. 27, 1935

**Selenium pigment for glass** I. I. Kitaigorodskii. Russ. 38,281, Aug. 31, 1934. A Se pigment is prepared by fusing Zn or Cd selenide with sulfides of one of the above metals in the presence of reducing agents

**Tempering glass** Pilkington Bros. Ltd. and John Wilson Brit. 435,812, Sept. 30, 1935 The glass is heated and cooled indirectly through the medium of a metal in liquid form. App. is described

**Apparatus for tempering sheets of glass** Pilkington Bros. Ltd. Fr. 789,268, Oct. 25, 1935

**Glass furnace** Banks D. Brown (to Capstan Glass Co.). Can. 354,528, Dec. 3, 1935 Cooling jackets are provided at an opening in one end of a glass furnace. These jackets have S-shaped portions extending outward, the ends being secured together to provide a chamber for feeding raw materials to the furnace. A cooling fluid is injected against the curved interior sides of the jackets Cf. C. A. 29, 4536\*

**Glass-melting furnace** Clinton A. Bowen (to Corning Glass Works) U. S. 2,029,052, Jan. 28, 1936 Various structural features

**Apparatus for continuously melting glass** Soc. anon. des manufactures des glaces et Produits Chimiques de Saint Gobain, Chauny & Cirey. Ger. 620,650, Oct. 24, 1935 (Cl. 32a 24.01).

**Device for circulating the molten glass in a forehearth or gathering-basin of a furnace, used in conjunction with a suction fed forming machine** The United Glass Bottle Manufacturers Ltd. and Thomas C. Moorshead Brit. 436,271, Oct. 8, 1935 The device comprises a vertical rotating member which has its lower end immersed in the glass and is reciprocated vertically in addition to being rotated.

**Glass manufacture** Baker & Co., Inc. Brit. 436,250, Oct. 7, 1935 Parts of glass-making app. that come into contact with molten glass as it flows from the furnace

are coated or faced with an alloy containing Pt and Rh, e.g., Pt 90 and Rh 10%.

**Apparatus for facing strips of soft clay for manufacture of bricks or the like** Geo. I. Rogers U. S. 2,028,523, Jan. 21, 1936 Mechanical and operative details

**Refractory materials** Soc. anon. d'isolants & réfractaires (Frédéric C. Le Coultre, inventor). Fr. 788,775, Oct. 10, 1935 A small amt. (2% of the total mass) of the product known as "Kenn Cement" or "English cement" is used as a binder to agglomerate refractory products having a basis of 7r silicate

**Refractory product** The Carborundum Co. Fr. 788,802, Oct. 18, 1935 A refractory material, e.g., for the container of glass furnaces, comprises principally Corundum and a glass placed between the crystals, or Corundum and mullite in the proportion of at least 2 to 1 burned in a vitreous base

**Refractory products containing spinels and suitable for use as coatings with clay** Lucie R. Haglund U. S. 2,020,773, Feb. 4, 1936 Producing highly refractory products consisting mainly of spinels of the type  $\text{RO R}_2\text{O}_3$ , raw materials containing spinel-forming oxides such as Cr ore and silica and lime are fused together so that the solidified molten mass consists of spinels rich in Ca (at least 20% of Ca being present) and Ca compounds are removed at least in part from the solidified mass Cf. C. A. 29, 2315\*

**Basic refractory material suitable for lining basic open-hearth furnaces** Richard L. Lloyd and Reed W. Hyde (to Dwight & Lloyd Metallurgical Co.) U. S. 2,029,627, Feb. 4, 1936 Magnesite or dolomite is mixed with fine sized slugging material such as Fe oxide bearing material and the mix is converted into coarse-sized pieces with an alkali earth oxychloride hydraulic cement binder and is burned by a forced draft in intermix with fuel to form a clinkered hard burned refractory product

**Molding compressed refractory bricks** Société anon. des produits réfractaires & céramiques du Nord. Brit. 435,076, Sept. 13, 1935

**Refractory bricks** Österreichisch Amerikanische Magnesit A.-G. Brit. 437,448, Sept. 23, 1935 This corresponds to Fr. 779,467 (C. A. 29, 5620P).

**Apparatus for molding firebricks and other articles from clay or other plastic material** John A. Biddle, Leslie G. Pitt and Harold J. Haden Brit. 435,840, Sept. 30, 1935

**Fired siliceous tribarium aluminate refractory** John M. McKinley and Willard K. Carter (to National Aluminate Corp.) Brit. 435,348, Sept. 19, 1935 See U. S. 2,017,723 (C. A. 29, 8271P)

**Coatings for furnaces** The Carborundum Co. Fr. 788,801, Oct. 18, 1935. Cement or lime furnaces or other chambers subjected to abrasion, e.g., ball or drum mills, are coated with a cast refractory material having a sclerotic hardness of 8 or more  $\text{Al}_2\text{O}_3$  content, or not a small amt. of Mg, Ca or Na, or a fused mass of  $\text{Al}_2\text{O}_3$  and chromite may be used

**Muffle kiln** Frank M. Hartford. Can. 355,123, Jan. 7, 1936 Structural details

**Muffle tunnel furnace** V. N. Mikhovskii. Russ. 38,040, July 31, 1934 Construction details

**Crystalline abrasive material** Charles R. Walker (to Abrasive Products, Inc.) U. S. 2,029,253, Jan. 28, 1936 In producing uncrushed crystalline abrasive material such as garnet,  $\text{SiC}$  or  $\text{Al}_2\text{O}_3$ , the material is subjected to sudden extremes of temperature change (as by heating to 700° or higher and quenching in water) to produce a reaction effective to cause the material to fracture along natural cleavage planes and to be capable of ready crushing without destruction of the naturally sharp cutting edges at such planes of fracture

**Abrasive wheels** Raymond C. Benner and William G. Soley (to Carborundum Co.) U. S. 2,028,183, Jan. 21, 1936 An abrasive wheel has a metal disk attached to each lateral face by a cement containing a phenolic condensation resin and a granulated filler in sufficient proportion to modify the character of the resin so that the cement has



a higher modulus of elasticity than the abrasive wheel. This serves to reinforce the wheel for high speed use. Cf. *C A* 29, 1600<sup>2</sup>.

**Abrasive wheels** The British Thomson-Houston Co Ltd Brit 436,430, Oct 10, 1935 The wheels consist of a core of Mo or other metal, to which is welded or brazed an abrasive annulus consisting of diamond particles embedded in a cemented carbide compn The method of prep is described

**Abrasive stones** L. I. Ginzburg Russ 38,009, July 31, 1934 In the prep of abrasive stones the binder is made from burned calcium dolomite together with a soln of  $MgCl_2$  and  $Fe_2(SO_4)_3$  soln

Measuring the hardness of abrasive disks or plates

**1** Karl Geppert (to Kugelfischer Erste Automatische Gussstahlwerkfabrik vorm Friderich Fischer) U. S. 2,929,066, Jan 28 A sheet of perforatable material such as sheet metal or cardboard is applied to the surface of the disk or plate, a conical impact pin is driven through the sheet into the disk or plate by a standardized driving force, and the diam of the perforation in the sheet is measured

**2** Enamel insulation for electric resistances I R Ripp and N A Artem'ev Russ 37,753, July 31, 1934 A mixt of water glass and about 5% of metal oxides, asbestos talc, lime stone or refractory clay is melted (heated to about 1000°) and placed on the resistance heated to about 1000°

## 20—CEMENT AND OTHER BUILDING MATERIALS

J C WITT

The fineness of the cement raw mixture I Elutriation of particles using Shōne's apparatus Yoshiaki Sanada and Gunkichi Nishi *J. Soc Chem Ind, Japan* 38, Suppl binding 622(1935) —Some improvements on Shōne's app were made, and exptl data on the elutriation of ground quartz are presented II. Fineness and chemical composition of the raw mixture Ibid 622-4 — Exptl data are given on the analysis of the raw materials used (limestone, clay and volcanic ash), grinding mills used, elutriation tests and chem compns, moduli and fineness compn of 4 raw mixts Conclusions The grinding machinery and grinding conditions greatly influenced the fine grinding of the raw mix, and the chem compn of the fineness fractions greatly differed with differences in raw materials III. Fineness and chemical composition of the clay Ibid 720-1 —Results on quartzite, ganister, shale and clay are presented and show that the  $SiO_2$  content decreased as the size of the fraction decreased IV. The relation between fineness of raw mixture and alite crystals Ibid 721-2 —Ground limestone was sepd into 3 fractions below 15  $\mu$ , 15-58  $\mu$  and above 58  $\mu$ , and the ground clay into 2 fractions below and above 43  $\mu$  Six raw mixts were prepd from the variously sized materials and heated from room temp to 1200° in an elec muffle furnace in about 90 min, and sintered for 15 min at 1460-80° Microscopic clinker examn showed that the size of alite crystals decreased with decreasing grain size of the raw mixt The amt of alite crystals increased as the grain sizes of limestone and clay approached each other Exptl data are given Karl Kammermeyer

Nature of the clay fraction in the raw materials of the German cement industry K Endell, U Hofmann and E Maedgen *Zement* 24, 625-33(1935) —Röntgen ray analysis of the marls and clays commonly used in cement manuf in Germany showed the following clay minerals in order of their abundance (a) "mica clay," in which  $Al_2O_3 + Fe_2O_3 + MgO + FeO$  are to  $SiO_2$  as 1 2 6, (b) montmorillonite (in which  $Al_2O_3$  to  $SiO_2$  as 1 4) and (c) kaolinite A further distinguishing feature observed was the fineness of the accompanying quartz When the 3 clay substances are heated the water losses are gradual for the "mica clay" from 200° to 600°, gradual for the montmorillonite until 200° and the remaining crystn water (about 5%) gradual above 500°, while kaolin loses its water abruptly at 450° The relationship between the clay chemistry and the water content of the fluid cement slurry is shown, with an example of the impracticability of soda adds to a slurry contg kaolinite clay High water content is necessary for slurries with clays consisting largely of montmorillonite Certain colloidal aspects are also discussed, such as the rate of filtration and the water loss on heating the slurry H F Kriege

Importance of the determination of free lime in the manufacture of cement Fernando Pacheco. *Ingeniería* 9, 157-8, 163(1935) —This test is important for controlling the cement mfg process since the free lime content definitely changes the phys properties of the cement It has several advantages over the "indeformability" test:

requires less time, gives better indication of the intermediate transformations during heating and of the compds. formed, of the degree of hydration and consequently of the efficiency Various methods for detg the free lime are described; the need of a more practical, accurate method is emphasized M McMahon

**4** Swelling and contracting of cements G Musgnug. *Zement* 24, 717-21(1935) —Addns of several siliceous powd admixts. such as slags, glass, trass and quartz did not produce great variations in the shrinkage of the cement within the range of good concrete. Transverse tests of concrete again showed the advantage of angular over rounded aggregates H I. Kriege

**5** Composition of portland cement and high alumina cements. A Travers *IX Congr. intern quim pura aplicada, Madrid* 3, 9-75(1934); cf *C A* 28, 2633<sup>2</sup> —A review of optical, x ray and other phys methods of studying cement clinker with photomicrographs, phase diagrams and photographs of spectra Reactions accompanying setting and the theories of hardening are discussed

E R Rushton  
The temperature variation of portland-cement pastes under nonadiabatic condition Tsutomu Maeda *Bull Inst Phys -Chem Research (Tokyo)* 14, 714-19(1935) —The temp variations of the following cement mixt (cement.  $H_2O = 1.5$ ) have been detd at 25° under nonadiabatic condition original cement (I), I +  $CaSO_4 \cdot 2H_2O$ , I +  $CaCl_2$  and I + cane sugar Addn of  $CaSO_4 \cdot 2H_2O$  lowers the second temp rise of the original cement; I N  $CaCl_2$  soln causes the temp rise in 3 steps, the second step being obviously due to the reaction consuming  $CaSO_4 \cdot 2H_2O$  K Konda

**7** Supersulfate metallurgical cements E L Dupuy *IX Congr intern quim pura aplicada, Madrid* 3, 350-69(1934), cf *C A* 28, 7459<sup>2</sup> —Supersulfate cement is an intimate mixt of slag,  $CaSO_4$  and  $CaO$ , with an av compn. of  $SiO_2$  25-30,  $Al_2O_3$  10-18,  $CaO$  40-45,  $MgO < 5$ ,  $FeO$ ,  $MnO < 4$  and  $SO_3 < 10\%$  Phys properties are given, and also diagrams showing the variation of mech resistance with compn A mixt contg 13%  $CaSO_4$  requires only a very small amt of  $CaO$  to give the max crushing resistance, this shows that  $CaO$  acts simply as a catalyst The cements do not crack on setting and are resistant to sea water Test cubes were immersed for more than 2 yr in  $CaSO_4$ ,  $KAl(SO_4)_3$ ,  $NH_4NO_3$ ,  $CaCl_2$ ,  $FeSO_4$ ,  $MgSO_4$ ,  $CaSO_4$ ,  $Na_2SO_4$ ,  $Na_2CO_3$ ,  $Na_3PO_4$ ,  $NaNO_3$ ,  $K_2CO_3$  and  $KOH$  solns without being attacked

E R Rushton  
**9** Testing of cement in plastic mortars G Haegermann. *Zement* 24, 529-32(1935) —A comparison is made between the standard (earth moist) and the plastic condition of cement mortars for test purposes The upper limit of the tensile strength is derived from the compressive strength by the formula tensile strength =  $\sqrt{2/6 \times \text{compressive strength}}$  H F Kriege

Testing of cement in plastic mortars II G. Haegermann. *Zement* 24, 607-9(1935). —Conditions imposed in

America, France, Switzerland, England, Japan, Brazil and Germany in cement tests are compared. H. I. Krieger.

The possibilities of pozzolanas in mortars and concretes. I dw. W. Scripture, Jr. *Eng. News-Record* 115, 163-7 (1935).—A general discussion of pozzolanas and their effect on the properties of mortar and concrete, including workability, vol. change, watertightness, heat evolution, strength and resistance to attack. A pozzolanic material is one that possesses constituents that will combine with lime at ordinary temps. to form insol. compds. of cementitious value. The precise nature of the reaction with lime is not clearly understood. Within limits, the degree of pozzolanic activity is governed more by structure and fineness than by compn. A reliable test of reactivity is required. Absorption of lime from a soln. under fixed conditions seems simplest and most direct. The materials must be carefully selected to secure the desired result. R. E. Thompson.

Design of concrete mixes for Mississippi River dams. Ralph P. Johnson. *Eng. News-Record* 115, 711-6 (1935).—Details are given of the design of the mixes in the construction of 5 locks and 2 dams, involving the placing of 420,000 cu yds. of concrete. The trial-mix method in which the remodeling app. of T. C. Powers (*J. Am. Concrete Inst.* 27, 419 (1932)) was used was employed throughout. R. E. Thompson.

Structural properties of vibrated concrete. Paul Anderson. *Eng. News-Record* 115, 676-7 (1935).—Tests, the results of which are given, indicated (1) that the water-cement ratio law holds for very lean mixes provided a compact concrete can be produced, and (2) that the shrinkage of lean mixes when compacted by vibration is considerably less than for richer mixes of the same water-cement ratio. R. E. Thompson.

Concrete in counterweight made heavy by magnetite. R. H. Rothschild, Jr. *Eng. News-Record* 115, 788-9 (1935).—Magnetite, prep'd from beach sand with the aid of an agitator-drum magnetic separator, was used as fine aggregate in the construction of counterweights for the Park St. bascule bridge between Oakland and Alameda, Cal. The sp. gr. of magnetite is approx. 4.75. The proportions per cu. yd., selected as a result of extensive tests, were: cement 762 lb., magnetite 2700 lb., gravel 1150 lb., water 48 gallons. The wt. of the concrete was 182 lb. per cu. ft. and strengths as high as 4800 lb. in 28 days were obtained. There was a decided tendency to lose strength and a slight excess of water did not have the usual effect on strength as the excess water rapidly rose to the surface. The surface is very uniform and smooth, very hard and highly resistant to abrasion. R. E. Thompson.

Investigating new kinds of froth formers [for concrete]. N. I. Irmolenko and N. A. Abramchuk. *Stroitel. Material* 1935, No. 9, 38-41.—Solns. of surface-active animal and vegetable albuminous materials (hides and lupine albumin, turpentine by-product soaps, etc. from fished) give a froth that is stable on the boundary surfaces of 3 phases, air, water soln., cement. The highest stability of the froth is found in a medium of  $pH$  greater than 7. The physicochemical consts. of froth concretes obtained are in accordance with standard values. The rapidity of setting of cement is higher than that of the destruction of the froths investigated. E. B. S.

Investigation of froth concrete at high temperatures. B. A. Smolyak and T. D. Morozova. *Stroitel. Material* 1935, No. 9, 42-6.—At temps. up to 400° froth concrete is a good thermal insulator and is stable. E. B. S.

Disintegration of concrete by mineral sulfates. R. W. C. Smith. *Water and Water Eng.* 38, 55-7 (1936).—In order to protect the concrete walls against sulfate disintegration they were painted with a mixt. of tar and pitch, the proportions of which were varied to give the proper consistency at the temps. at which the coats were applied. W. A. Moore.

New viscometer for bitumens has extended range. J. O. Rhodes, L. W. Volkman and C. T. Barker. *Eng. News-Record* 115, 714-18 (1935).—An instrument, termed the Koppers Products Viscometer, by means of which the consistencies of all grades of road tar can be det'd. quickly

and conveniently at a single temp. within the range of atm. temps. encountered under service conditions, is described, together with the mode of operation. This instrument, which has a precision of 1% in the lower and a much greater precision in the higher ranges, overcomes the difficulty that the tests usually employed are suitable only for a portion of the consistency range. Although the instrument has been used thus far for road tars only, it should be suitable for testing all grades of road asphalts and many other types of materials. Patents have been applied for. R. E. Thompson.

Mastering a new method of dehydration of gypsum in a suspended condition. L. L. Lipsitz. *Stroitel. Material* 1935, No. 5, 13-22.—The processes and data obtained in previous investigations abroad (cf. Schneider, C. A. 26, 1737) were verified under plant conditions. A detailed description is given of the plant, the technological process and the thermal conditions. The plastering gypsum obtained corresponded to standard requirements. I. I. Stefanovsky.

Preservation treatment of pit-timber. P. F. Dry. *Trans. Inst. Mining Engrs.* 90, 160-4 (1935).—Mine props were treated with cresote, NaCl (8 and 3 1/2% solns.), ZnCl<sub>2</sub> (5 and 2% solns.), NaF (4 and 2% solns.) or Wolman salts (5 and 2% solns.) (compn. NaF 80, dinitrophenol 6, Na dinitrophenate 5, and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> 8%) for 1 hr. at 180°F. and cooled for 16 hrs. Absorption was 7-10 lb. per cu. ft. of timber. Best results were obtained with the most expensive reagents, cresote, Wolman salts and NaF. A 2% soln. of the last 2 is adequate. NaCl gave results far superior to untreated timber. Alden H. Emery.

Investigations on the effectiveness of impregnating wood with thiocyanates. O. Routah and Voitto Virta. *Suomen Kemistilehti* 8A, 125-6, 135-40 (1935).—The sol. murex salts are the more effective in preventing fungus growth. The amt. of HgCl<sub>2</sub> necessary is 0.025% and of KCNS is 2.0-2.1%. A mixt. of the 2 which gives the best results is 0.017% HgCl<sub>2</sub> and 1.45% KCNS. Although KCNS alone is not satisfactory as an impregnating material, it seems to produce a beneficial effect when mixed with HgCl<sub>2</sub> as specimens impregnated with the mixt. withstand molding to a far greater extent than corresponding specimens treated with HgCl<sub>2</sub> alone. E. F. Jukkola.

Studies in old timbers. III. Post neolithic. Ernest A. Rudge and Harold Lewis. *J. Soc. Chem. Ind.* 54, 433-4T (1935), cf. C. A. 29, 6389.—The results of an examn. of submerged forest timbers over 6000 yr. old are reported. The data include extns. by benzene, alc. and hot water, soly. in alkali, cellulose-lignin and ash analyses. The influence of Ie on the decompn. of lignin is again indicated. Alfred L. Kammerer.

Hydrolytic equil. of Ca aluminate hydrates (von Polheim) 6.

Knight, Bernard H.: Road Aggregates. Their Uses and Testing. London: E. Arnold & Co. 264 pp. 21s. Reviewed in *Commonwealth Eng.* 23, 196 (1936).

Leduc, E., and Chenu, G.: Chaux, ciments et plâtres 2nd ed. Paris: Ed. Béanger. 340 pp. F. 45.

Santarella, Luigi: Pontuario del cemento armato. 6th ed., revised. Milan: U. Hoepli. 313 pp.

Santarella, Luigi: Temperatura di presa del cemento. Milan: U. Hoepli. L. 10.

Beton-Kalender. Taschenbuch für Beton- und Eisenbetonbau sowie verwandten facher. Berlin: Ernst & Sohn. 40 pp. M. 0.20.

Internationale Normentabelle für Portland-Zemente. 6th ed. Berlin: Zementverlag. M. 3.60.

Hydraulic cements. Chemische Fabrik Grünau Landshoff & Meyer A.-G. Tr. 788,822, Oct. 18, 1935. An alkali silicate along with one or more other salts the cations of which do not form insol. or difficultly sol. salts with the SiO<sub>2</sub> of the silicate, and the anions of which contain a

metalloid, preferably bi- or trivalent, is added to improve the quality of hydraulic cements. Salts used include alkali salts of acids of P, B, Si, N, HCN and HCNS, as well as bromides and bromates and salts of As, Sb and Se. The advantages of the addition of alkali silicates without the disadvantages are thereby obtained. *Cl C A* 30, 8354.

**Hydraulic cement** "Straba" Straßenbaubedarf A - G Ger 620,980, Oct 31, 1935 (Cl 803 1 06). The cement is obtained in powder form by mixing together the powder hydraulic constituent with powder slag as dust clouds or fine suspensions. Examples mention the blowing together of cement and trass, and cement and fine-ground active  $\text{SiO}_2$  suspended in oil or bitumen.

**Cements** *Bolidens Gruvaktiebolag* Brit 435,015, Sept 12, 1935. A cement or cement mixt contains Al arsenate in admixt with  $\text{CaO}$ ,  $\text{Ca}$  silicate, portland cement, aluminous cement or similar products rich in  $\text{CaO}$ . The Al arsenate may be prep'd by oxidizing  $\text{As}_2\text{O}_3$  together with an aluminiferous material, e.g., bauxite, clay, in air, e.g., by heating a sludge or moist mixt of the ingredients in a counter current of air in a rotating furnace. Catalysts for the oxidation, e.g., Fe, Mn or Cu compds,  $\text{HNO}_3$ , nitrates or nitrous fumes, may be used. *Cl C A* 30, 15391.

**Cements** Soc anon des ciments de Thieu and Léon Blondiau Brit 437,855, Nov 6, 1935. See Belg 400,861 (C A 28, 6269). The calcination takes place below a temp that causes decompos., 900-1200° being preferable.

**Metallurgical cement** Soc anon John Cockerill Belg 406,709, Jan 31, 1935. Clinker, slag and up to 12% of alk. earth sulfate are introduced simultaneously into the pulverizer.

**Hard surfaced cement** Arthur L Smyly U S 2,028,956, Jan 28. A mass of iron material such as scrap iron likely to contain certain impurities capable of forming gas when present in wet portland cement is ground and treated with an alkali soln such as with a 10-30% soln of  $\text{NaOH}$  to decrease the quantity of gas-forming impurities present, the treated material is dried while wet with alkali so as to coat the particles with a protective alkali and the treated iron is used with portland cement.

**Magnesium oxychloride cement** Arnold Hermann U S 2,030,002, Feb 4. A dry mixt formed of  $\text{MgO}$  40,  $\text{BaSO}_4$  40 and chalk 20% is separately formed and then mixed with about two fifths its volume of a liquid mixt formed of a 27%  $\text{Be}$   $\text{MgCl}_2$  soln 60, a 21%  $\text{Be}$   $\text{HCl}$  soln 20 and a 22%  $\text{Be}$   $\text{H}_2\text{SO}_4$  soln 20%.

**Metal lined cementitious pipe** Eric P. Halliburton U S 2,029,369, Feb 4. A pipe with a cementitious body, a lining which may be formed of metal and a casing such as one of metal has ceramic balls or the like embedded in the cementitious material and extending from the liner to the casing.

**Porous concrete** Karl I A Ellund Brit 436,105, Oct 4, 1935. Light concrete bodies are made of portland, slag, aluminous or like cement, a gas-forming substance, e.g., powder Al, aggregate as required and finely divided siliceous material, e.g., clay,  $\text{SiO}_2$ , the mass being heated with steam to cause the  $\text{SiO}_2$  to react with  $\text{CaO}$  liberated in the setting of the cement and also to convert di- and tri-Ca silicates into mono-silicates. An addn of  $\text{CaCl}_2$  gives a product of increased strength.

**Road making compositions** Dezzo Komlos Brit 437,808, Nov 6, 1935. A compn for making roads, pavements, tennis courts, etc., consists of fibrous or granular materials which do not disintegrate in  $\text{H}_2\text{O}$ , e.g., sand, gravel, flint, quartz and 3-5% of a drying oil, e.g., wood, nut, poppy, hemp or linseed oil, to which has been added a small proportion, e.g., 1%, of a Pb, Zn, Mn or Co compd. On applying to the road, etc., an exothermic reaction sets in and the temp rises to 120-150°. The surface is then covered by a layer of loose sand heated to said temp, which is maintained for about 48 hrs when the sand is removed.

**Tar compositions for roads, etc.** Jean L. Chambon and Georges M. Hart. Fr 788,834, Oct. 18, 1935. The

composn. contain, e.g., tar 100, colloidal filler 100 and rubber oil 2 kgs. The colloidal filler contains "administrative" lime 33, colloidal  $\text{SiO}_2$  60, and  $\text{Fe}_2\text{O}_3$  or  $\text{Al}_2\text{O}_3$  6%.

**Bituminous compositions** N. V. de Bataafsche Petroleum Maatschappij Brit 435,732, Sept 26, 1935. Light-colored road and pavement construction, e.g., road coverings, floors, traffic lines and joint and crack fillings, are made by applying a mixt of bitumen, mineral aggregate and powder Al to form a wearing course of required thickness.

**Bituminous compositions** Wm Gooch Brit 436,187, Oct. 7, 1935. Bituminous road, etc., surfacing material is prep'd by mixing together 34-36% of fines, e.g., sand, passing a screen having 8 meshes to the in and retained on 15 meshes, and gravel or granite comprising 7-9% of material passing 1 in and retained by  $\frac{1}{4}$  in, 7-9% passing  $\frac{1}{4}$  in and retained by  $\frac{1}{2}$  in, 25-27% passing  $\frac{1}{2}$  in and retained by  $\frac{3}{4}$  in and 13-15% passing  $\frac{3}{4}$  in and retained by  $\frac{1}{2}$  in mesh, heating the mixed materials until dried, adding about 6-8% of bitumen and mixing at about 380°F.

**Plastic compositions**, William J. Woodfine Brit 435,713, Sept 26, 1935. Slabs for surfacing pavements, etc., are molded from a compn obtained by mixing sawdust and ready-mixed oil paint, rubber latex may be added to the paint.

**Apparatus for coating aggregate with a liquid-vehicle bituminous binder** Wallace M. Hendrick and Gustave Loeffler, Jr U S 2,028,745, Jan 28. Various mech and operative details.

**Manufacture of composite building material such as sheets of wood particles and resinous material** Emil C. Loetscher U S 2,023,616, Jan 21. Various details of app and operation are described.

**Building material from spent tanning wood** R. Z. Taunzhammer, V. I. Elman and B. A. Sumagin Russ 35,994, April 30, 1934. The spent wood is mixed with husks or other vegetable albuminous products as binder, alkali or acid is added at ordinary temp and the mass is pressed and dried at ordinary temp. Acids used may be  $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ ,  $\text{H}_3\text{PO}_4$ ,  $\text{HCOOH}$ ,  $\text{CH}_3\text{COOH}$ , oxalic or citric.

**Asphalt-asbestos mixture suitable for flooring, heat insulation, etc.** Ulric B. Bray, Lawton B. Beckwith and Frederick S. Scott (to Union Oil Co. of Calif.) U S 2,029,289, Feb 4. A fibrous material such as asbestos is mixed with asphalt, water, a stabilizing agent such as Na caseinate, a demulsifying agent such as slaked lime and a finely divided material such as diatomaceous earth which adapts the mixt for trowelling.

**Roofing felt** Pierre Drewsen (to Barrett Co.) U S 2,029,310, Feb 4. Jute fibers and other vegetable fibers such as those of wood are separately cooked and are then mixed with each other and with cereal fibers such as those of wheat or oat straw which have been subjected to a more severe cooking, to form a felt sheet of desired strength and absorptive character.

**Roofing plates** Ya Yu Rontberg Russ 38,480, Aug 31, 1934. Organic fibrous material is made water-resistant by means of hot bitumen and then cemented together with lime-silica soln.

**"Acoustic" partition or wall-covering material** Harold D. Arnold (to Bell Telephone Laboratories, Inc.) U S 2,028,180, Jan 21. A thick, porous and easily compressible layer of material such as felt adapted readily to absorb energy from air waves of low frequencies is used with an exposed thin layer of high density non porous material such as a paint or enamel covering the porous material and serving to prevent excessive absorption of energy from air waves of the higher frequencies.

**Treating wood** Heinrich A. Hellmers and Mersida Veneers Ltd. Brit 435,334, Sept 19, 1935. Pecked wood veneers are seasoned and rendered pliable by steeping cut lengths in an aq soln of formalin, chrome alum, gelatin and glycerol. The treated veneers are adhesively secured to a paper, etc., backing for use as wall coverings, etc.

**Treating wood** Paul von Sonnenthal *Brit. 436,015*, 1 Oct. 7, 1935. Wooden articles of curved and angular configuration are produced by treating veneers or plywood with a soln. contg. 0.1-1% of 1 or more tanning agents, e. g., oak or quebracho bark, sumac, alum, 1e salts, chromates, phenols, aldehydes, until the wood is supple and then bending, folding or winding. Org. solvents, e. g., AcOAm, and sterilizing and fireproofing agents may be added to the soln.

**Preserving wood, etc.** Axel R. Lindblad *Brit. 444,084*, Sept. 12, 1935. Wood or other vegetable matter is

preserved by impregnation with a soln. contg. an alk. earth compd., e. g., a Ca, Ba, Sr or Mg compd.,  $H_2AsO_4$ , or a salt thereof and an org. acid, e. g.,  $AcOH$ ,  $HCOOH$ , so that on evapn. a diffusible sol. alk. earth metal arsenate is left in the wood. In modifications, the  $H_2AsO_4$  or org. acid constituents may be chemically combined with the alk. earth metal constituent.

**Preserving wood, plants and trees** Serge Teliayeff. *Fr. 789,691*, Nov. 4, 1935. A soln. of 5 parts of dinitrophenol in 95 parts petroleum oil is used. The soln. may be emulsified in water contg. an emulsifying agent.

## 21—FUELS, GAS, TAR AND COKE

A. C. FIFLDNER AND ALDEN H. EMERY

The volume changes of mixtures of benzene and petroleum with alcohol and motor benzene. Franz Spaustra *Brennstoff-Chem.* 16, 181-4 (1935). Pyrometer  $d_v$  of motor benzene alk. mixts. are lower than calcd. values at low concns of alk. Variations between detd. and calcd.  $d_v$  are tabulated. Largest differences are with 50 vol. % alc. and become less with increasing  $d_v$  of the petroleum fraction. No relationship with boiling range was found.

I. W. Jung

**Alcohol motor fuel.** Tetsuro Suwa. *J. Fuel Soc. Japan* 14, 1361-79 (1935) (in English 129-32).—The use of 1:10H mixed with com. gasoline as a motor fuel was studied on a Cooperative Fuel Research engine coupled to a 4-kw. Sprague dynamometer. The tests were made with an undil. gasoline with fuels contg. 10, 20 and 30% by vol. of abs. alc. Results: Gasoline contg. alc. up to 20% can be used in gasoline engines without readjusting the carburetor, and the equiv. calorific value of alc. is preferable to the calorific value of gasoline.

F. I. Nakamura

**Compressed gas as a fuel for motor transport.** R. Cook. *Gas J.* 213, 90-3 (1936).—The system of compressed gas compulsion used at present is described, and an analysis made of its economic possibilities in competition with gasoline and heavy oil. Conclusion: Heavy oil constitutes the real competitor to compressed gas. The improvement in the economic possibilities of compressed gas by enrichment with coal gas and by development of a special engine have also been examd.

P. J. Wilson, Jr.

**High speed Diesel-engine fuels.** Ignition characteristics of light oil for high-speed Diesel. Karoku Yamazaki and Sakae Ota. *J. Fuel Soc. Japan* 14, 1473-83 (1935) (in English 131-7).—Y. and O. measured the Diesel and ignition indexes and calcd. the cetene nos. by the methods proposed by Becker and Fischer (*C. A.* 29, 3167), Moore and Kaye (*C. A.* 29, 5257) and W. H. Butler. To verify the actual measurements of these indexes they also carried out engine tests. The distn. characteristics of the oils used, and Diesel index, ignition index and calcd. cetene nos. are given in 2 tables.

F. I. Nakamura

**Applicability of physical constants for determination of ignition behavior (cetene number) of Diesel fuels.** Richard Heinze and Maximilian Marder. *Brennstoff-Chem.* 16, 286-90 (1935).—The parachor,  $d_v$  and  $n_D$  are related to boiling indexes in the comparison of ignitability. Av. differences between detd. cetene no. and that calcd. with  $d_v$  of the parachor is 1.8. Refraction and surface tension appear sufficient for the calcn. of ignitability with Diesel fuels.

I. W. Jung

**Relationship between the ignitability of brown-coal Diesel fuels and their physical properties.** Richard Heinze and Maximilian Marder. *Oel, Kohle, Erdöl, Teer* 11, 724-8 (1935); cf. preceding abstr.—Nearly linear relationships are shown between phys. consts., particularly the parachor and  $d_v$ , of fuels of similar boiling range, and the ignitability. With cresote-contg. brown-coal oils, a correction is needed. The av. difference between the detd. cetene no. and that calcd. from phys. consts. is =1.

I. W. Jung

**Coal-dust motor fuel.** Hans Wahl. *Brennstoff-Chem.* 16, 201-8 (1935).—Various requirements of coal dust have

been examd. Indexes are ash content, abrasive no. and a proposed collective abrasive no.

I. W. Jung

**Fundamentals of coal cleaning.** Henry I. Hixley. *Proc. III Mining Inst.* 1935, 123-35.—A simple description of the principal methods of coal cleaning.

A. H. I.

**The cleaning of Illinois coal.** John Griffen. *Proc. III Mining Inst.* 1935, 33-6.—General.

Alden H. Emery

**Cleaning or betterment of Illinois coal by the dry method.** Edward O'Toole. *Proc. III Mining Inst.* 1935, 42-7.—Data are given on  $H_2O$  retention by coal of various sizes.

Alden H. Emery

**The place of air cleaning in coal preparation.** R. G. Lawry. *Proc. III Mining Inst.* 1935, 37-9.—The upper size for air cleaning is  $1\frac{1}{2}$  in., for washing, 6 in. If the surface moisture of the small coal is less than 5%, dry cleaning should be considered. The advantages of a combined wet and dry plant are considered.

A. H. Emery

**Coal washing in Baum-type washers with selective ash control.** J. W. Wilson. *Proc. III Mining Inst.* 1935, 39-42.

Alden H. Emery

**Coal selection for steam plants.** A. W. Gauger. *Penna. State Coll., Mineral Ind.* 5, No. 5, 1-4 (1936).—The effect of variation of moisture, ash, B. t. u., ash-handling costs, ash-softening temp. and S on the price of coal is discussed.

Alden H. Emery

**Coal utilization (with special reference to sulfur content).** W. D. Langtry. *Proc. III Mining Inst.* 1935, 136-43.—Occurrence of S in coal, its behavior in burning and its effect on heating equipment are discussed.

Alden H. Emery

**Coal friability tests.** A comparative study of methods for determining the friability of coal and suggestions for tumbler and drop shatter test methods.

R. E. Gilmore,

J. H. H. Nicolls and G. P. Connell. *Can. Dept. Mines, Mines Branch Rept. No. 762*, 102 pp. (1935).—The friability of 7 coals was measured by 7 methods: small jar tumbler, A. S. T. M. coke drum tumbler, box tumbler, drum (box) tumbler, Sheffield coke abrasion tumbler, A. S. T. M. coke shatter test and III shatter test. Each method places the coals in approx. the same order of friability and hence all may be considered satisfactory.

Appreciably more breakage occurs in the tumbler than in the shatter tests. The tumbler test should be used to measure comparative friability after a certain amt. of handling has taken place and during preliminary plant crushing. Shatter tests serve better to indicate resistance to breakage prior to plant crushing. The small jar tumbler and A. S. T. M. coke shatter test had sufficient advantages over the others to warrant special consideration.

In the shatter tests, the av. ratio of round to square hole screen sizes varied from 1.07 for the  $\frac{1}{4}$ -in. hole to 1.17 for the  $\frac{1}{2}$ -in. hole. Friability indexes varied directly with size of lumps, the larger being more friable than the smaller.

**Friability index and size stability index (cumulative percentage of dropped coal remaining on screen lower and next in series to the smaller screen used in prep. the sample for test) agreed closely for the  $\frac{1}{4}$ - to 1-in. size of all coals tested and for all sizes of the most stable coal.**

The difference between the size stability index of the least and most friable coals for 2 drops corresponds closely with the friability index for 4 drops. Rate of breakage reached

a max. at the end of the second drop for the different sizes of the least and medium friable coals. A 2-drop modification of the shatter test gave duplicable friability and size stability indices. Dropping the coal on an Fe plate or smooth concrete made little difference. Tentative procedures for shatter and tumbler tests of coal are outlined.

Alden H. Emery

Heating coal-carbonizing equipment. Stief G. u. Wasserfack 78, 905-71(1935).—Coal-carbonizing equipment fired with producer gas should also be equipped to use coal gas, and vice versa, so as to increase the flexibility of gas and coke yields. Calcs. are presented as to the effect of substituting producer gas for coal gas on coal throughput and gas yield, etc. With regenerative heating of the air, the efficiency of the 2 gases is quite similar, as about the same excess of air is required in each case. Coal gas may give too short and hot flames in burners designed for producer gas—this is best avoided by adding sufficient flue gas. If mixts. of coal gas and producer gas are to be preheated before burning, sufficient steam must be added to prevent C and tar sepn. during heating. R. W. R.

Prevention of coal fires during storage. Causes. Avoidance of spontaneous ignition. M. Blanke. Arch. Vormeist. 16, 97-9(1935). Gas u. Wasserfack 79, 13-14 (1936).—Spontaneous ignition of coal storage piles can be avoided by (1) a storage area dry and free from fences, piers, etc. Ashes should be avoided as apt to cause O access. (2) Storage area must be cleaned up before initial use. (3) Coals of different sizes should be stored separately, and breakage avoided. (4) Coals of different origin should be stored separately. (5) The size of the storage piles should be limited, but may be slightly larger in winter than in summer. At least 1 m. should be allowed between piles. (6) In case the fire stops must be filled with coal, this coal should be removed first. (7) The following heights of storage piles are safe: (a) fine coal, over 18% volatile matter, up to 4 m.; large coal, over 18% volatile matter up to 6 m.; fine coal below 18% volatile matter, up to 6 m. and large coal below 18% volatile matter, up to 8 m. (8) Numerous pipes should be inserted for temp. measurements. (9) Coal should always be removed from the outside. (10) Storage in covered bunkers is desirable, storage in sunny corners and discharge of gutters on coal should be avoided. (11) Accumulation of gas above coal in bunkers must be avoided. (12) Storage in cones, etc., should be avoided. (13) Fine coal must not be permitted to reach temps. over 65° in bunkers. Putting out fires.—(14) Bituminous coal starts to burn at about 200°. (15) Small fires are extinguished by opening up the hot "nests" and repiling the coal. (16) Larger fires are difficult to extinguish by dirt or water. Water penetrates with difficulty on account of tar formation and may cause explosions. The safest means is air exclusion, with cooling. CO<sub>2</sub>, N or SO<sub>2</sub> may be introduced into the bottom of the pile and drawn off at the top of the pile by fans. If possible the coal should be removed with grab buckets and extinguished in water for 1-2 mm. and then stored at a new location. R. W. R.

The fusibility of coal ash. Y. Kosaka. J. Fuel Soc. Japan 14, 119-24 (in English) (1935). Cf. C. A. 29, 1204. —K. devised a method embodying improvements on the method of Hubley and Bunte, and examd. about 60 varieties of Japanese coal ashes. The description of the app., its operation and the results of the tests are given.

F. I. Nakamura

Hydrogenations in a tetralin medium. I. Destructive hydrogenation of bitumen and pitch. E. H. Boomer and J. Edwards. Can. J. Research 13B, 323-30(1935). —The importance of a suspension medium in processes of destructive hydrogenation is discussed, with particular reference to the efficiency of the medium as a H carrier. The value of tetralin as a medium, in that it is a good solvent, is stable to heat, and acts as a H carrier, has been detd. in the hydrogenation of bitumen and stable pitch. Tetralin does not cause an increase in yield of oil but does improve the quality somewhat. It increases the rate of hydrogenation of bitumen but does not affect that of stable pitch. II. Destructive hydrogenation of coal

with tetralin and with a mixture of related compounds as media. Ibid. 331-6. —A complex mixt. of polynuclear hydrocarbons obtained in the pyrolysis of natural gas has been hydrogenated. The original mixt. and the product of hydrogenation have been used as suspension media in the hydrogenation of bituminous and domestic coals, and are compared with tetralin as a medium. The original mixt. is much less, and the hydrogenated mixt. only slightly less, effective than tetralin. III. Destructive hydrogenation of cellulose and wood. E. H. Boomer, G. H. Argue and J. Edwards. Ibid. 337-42. —The destructive hydrogenation of cotton wood, cellulose and fir-wood sawdust in a tetralin medium was investigated. When it is present in sufficient excess, tetralin promotes the complete conversion of cellulose and wood to liquids and gases in the absence of a catalyst. The tetralin acts as a H carrier in fulfilling the function of a catalyst. IV. Destructive hydrogenation of grain screenings. E. H. Boomer and J. Edwards. Ibid. 343-50. —The destructive hydrogenation of grain screenings in a tetralin medium has been investigated. With an excess of tetralin, at least 3 to 1 by weight, more than 50% of the screenings can be reduced to oils and about 15% to solid residue. Catalysts are effective by virtue of the fact that they greatly increase the rate of reaction, and also increase the production of hydrocarbon gases at the expense of CO<sub>2</sub>. J. W. S.

Gasoline from coal at Billingham. C. H. S. Tupholme. Chem. Industries 38, 29-32(1935). —See C. A. 30, 268.

E. H.

The results of low-temperature carbonization of some Lower Gondwana Indian coals. Lewis L. Fernor. Records Geol. Survey India 69, Pt. 3, 353-60(1935). —Analyses of 22 coals are given tabularly with yields of coke, oil, liquor and gas and certain data on properties of each. Coals with more than 3% moisture are weakly coking or noncoking, while most of the coals below this yield good low-temp. coke. The high moisture coals yield more liquor than low-moisture coals. The most suitable coals for use in mfg. low-temp. coke are those also suitable for mfg. high-temp. metallurgical coke.

Alden H. Emery

Improvements in low-temperature analysis. W. W. Robinson, Jr., and F. N. Laird. Petroleum World 32, No. 11, 189-91(1935). —Simple procedures and auxiliary equipment are described for the accurate detn. of the liquid and vapor vol. of the residues from wet gases in low temp. analysis of natural gas. Mol. wts. are readily calcd. Chas. Wirth, III.

The Coalite low temperature process. W. A. Bristow. Brennstoff-Chem. 16, 281-6(1935). —The Parker Coalite process of 1906 is described in its present development.

F. W. Jung

Comparison of retort- and waste gas carbonization. E. Groh. Brennstoff-Chem. 16, 221-30(1935). —The Kohlenveredlungs process and the Freiberg and Lurgi processes are compared for costs, yields and quality of products. F. W. Jung.

Press for high cakability indexes. R. Kattwinkel. Brennstoff-Chem. 16, 231(1935). —Addnl. app. for the method proposed previously (C. A. 26, 2847) is described.

F. W. Jung

Carbonization and combustion. David Brownlie. Steam Eng. 5, 137-9(1936). Cf. C. A. 29, 7042. —By carbonization of mixts. of pulverized coal and oil up to 450° and 750-900 lb. per sq. in. a residual product is obtained composed of heavy oil and carbonized coal particles in true colloidal condition. Several of these processes are described briefly along with the Trent process.

Alden H. Emery

The flame "rating" of manufactured gases as a measure of their combustion characteristics. Horst Brückner and Hans Löhr. Gas- u. Wasserfack 79, 17-20(1936). —The combustion characteristics of various manifold gases can be compared on the basis of their "specific flame rating" (C. A. 26, 829) detd. by using a Bunsen burner of 1 sq. cm. cross section (1.28 cm. diam.) with an inner cone height of 0.99 cm. This height of the inner cone corre-

sponds to a flame area of 2 sq. cm., and the "specific flame rating" is expressed in kg. cal./sq. cm./sec. This value is dependent on the rate of flame propagation, heating value, etc. A satisfactory gas should have a max. "specific flame rating" of about  $1400 \times 10^{-4}$  kg. cal./sq. cm./sec.  $\approx 10\%$ . Graphs are given for the variation of this value with various air-gas ratios for a no. of manifold gases. A higher "specific flame rating" can be secured with air-gas mixts somewhat leaner than that used in ordinary appliances of the Bunsen-burner type. Maintenance of the proper value will insure sharp flames and the slight danger of CO in the flue gas.

R. W. Ryan

Combustions in gaseous phase. W. A. Bone. *Inst. intern. chim. Solvay, 5th Conseil 1935*, 119-204, cf C A 28, 876, 879. —A general discussion and review dealing successively with slow combustion, pre-inflammation period, the inflammation period, limits of inflammability, propagation of flame in explosive mixts., "uniform" initial movement, acceleration of initial velocity of the flame by compression waves, detonation, explosions in closed vessels, explosions under high initial pressures, influence of pressure on the spectra of stable H and CO flames in O, and the mechanism of oxidation in combustions in gaseous phase.

A. Papneau-Couture. The dry purification of manufactured gas. K. Bunte. *Gas- u. Wasserfach* 78, 954-9(1935). —Fouling and re-activation of dry purifying material are 2 fundamentally different processes. High capacity for absorbing H<sub>2</sub>S is more important than high rate of absorption (activity) except where purifier capacity is limited. App. for detg. activity is illustrated but not described. The activity of Lux and Lauta purifying materials increases much more rapidly with time than do bog iron ores. A high content of reactive iron is desirable to secure optimum S contents in the oxide in the min. no. of foulings—this reactive iron content is reduced by the formation of iron disulfide, sulfate and various CN compds. While fouling and re-activation may appear to proceed at nearly the same rate with a heavily loaded purifier, this is not ordinarily true, as the H<sub>2</sub>S can find much reactive Fe oxide, while there is much less Fe<sub>2</sub>S<sub>3</sub> available for reaction with O. The optimum moisture contents for fouling and reactivation of a given oxide are generally different. It is believed that the optimum moisture content for fouling corresponds to a thin film of water on the surface of the purifying material, and that increasing thicknesses of this film will decrease the rate of H<sub>2</sub>S absorption due to diffusion processes. Increasing water contents up to the optimum are also believed to increase the amt. of active Fe(OH). Intensively dried purifying material has greatly decreased capacities and activities—partly dried materials can be re-humidified by adding steam to the gas. Optimum temps. for fouling and reactivation differ somewhat. Alkal. addns. to give a higher pH value increase the rate of H<sub>2</sub>S absorption, but have no influence on the reactivation process.

R. W. Ryan

The controlled operation of a carbureted water-gas plant—II. 37th Report of the Joint Research Committee of the Institution and Leeds University. F. J. Dent, W. H. Blackburn and N. H. Williams. *Inst. Gas Engrs. Com. publication* No. 122, 156 pp., *Gas World* 103, 421-2, 431-8, 541-3, *Gas J.* 212, 473-5, 693-5(1935); cf C A 29, 1398. —When using carbureted water gas as a coal-gas auxiliary, the total costs of manuf. per therm are a min. when a plant is operated at a high output. To obtain high output, (a) the blast press should be such that, with the temps. in the fuel bed at the max. allowed by clinker formation and wear and tear of the generator, the heat in the blow gases is no more than is required for carbureting, and (b) the rate of steam supply should be as high as consideration for the quality of the blue water gas will permit, unless the rate of oil supply then required leads to excessive loss of oil efficiency. The relative durations of run and blow are fixed by the necessity of storing sufficient heat in the fuel bed during the blow to decompose the steam during the run. The density of the fuel must be adequate to withstand the blast pressure required to control the heat in the blow gases. When the demand for

carbureted water gas decreases, economies in raw material and maintenance can be secured by modifying operation. Clinker formation and wear and tear are reduced by using lower blast pressure. Improved decompn. of steam and oil and blue water gas of better quality result from slower rates of steam and oil admission. Heat in the blow gases can be increased with advantage until the plant is self-supporting in steam. A large proportion of the steam should be passed down through the fuel bed, for then there is less tendency for heat to leave the generator in the blow gas while there is an improvement in the quality of the gas. With coke of normal grade, nothing is gained by operating with shallow fuel beds under steady conditions. The fuel bed should be sufficiently deep to avoid excessive loss of sensible heat in the gases leaving the top of the generator, without being so deep as to restrict unduly the air supply. A temporary lowering of the fuel bed is a simple means of preventing the temps. of the carburetor from being affected by accidental variations in the moisture content or the size of the coke, or the nature of the ash. The performance of a plant using a normal grade of coke is better than with smaller grades. No mech. difficulties arise with smaller grades, they can be used when their lesser monetary value compensates for lower thermal yield and poorer quality of gas and when the full output is not required. Operation of the mech. grate at a slower speed improves the output, the quality of the gas and the yield of gas per 1000 lb. of coke, mainly because with a slower speed a greater proportion of the steam can be supplied on the back-run. If the grate speed is reduced still more a point is reached when clinker accumulation prevents further gains by disturbing conditions in the fuel bed. A self-clinkering plant has a considerably greater output per sq. ft. of cross section than a hand-clinkering plant. In spite of this, the self-clinkering generator gives a higher thermal yield of gas per 1000 lb. of coke and also of better quality.

A. H. Emery

Importance of the work schedule in gas producer stations. N. A. Migulin. *Ogneupor* 3, 695-700(1935). —Practice at a refractory plant is discussed. E. E. S.

Recently developed chart simplifies computation of flue gas losses. W. F. Schapchorst. *Oil and Gas J.* 34, No. 30, 26(1935). J. R. S.

Gas heating of platens for plastic work. H. R. Hems. *Gas World* 104, No. 2685 *Ind. Gas Suppl.* 3-4(1936).

P. J. Wilson, Jr.

Whessoe-Woodall-Duckham electro-detarrar at Manchester and Margate. K. W. Francombe. *Gas J.* 213, 206-8(1936). —The Manchester plant removes 99.8% of the tar fog from 9-10 million cu. ft. of gas measured under actual conditions per day with a power consumption of 4-5 kw. The Margate plant is designed for 94% tar fog removal from 2,500,000 cu. ft. of mixed coal and water gas.

P. J. Wilson, Jr.

Catalytic oxidation of benzene in gaseous phase. Y. V. Pigulevskii and E. Ya. Varzhemskaya. *J. Gen. Chem.* (U. S. S. R.) 5, 1620-8(1935). —Pure and cracking C<sub>6</sub>H<sub>6</sub> was oxidized in an electrically heated Fe chamber (1.2 cm. X 17 cm.) charged to 2/3 of its capacity with the catalyst prep'd. by pptg. NH<sub>4</sub> vanadate on grog lumps and activating at 450° in a current of air and O. The reaction was carried out with mixts. contg. 3-8% C<sub>6</sub>H<sub>6</sub> and 19.7-89% O by vol. at a temp. interval of 370-450° and contact periods of 0.4-20.6 sec. The reaction gases were conducted through a system of condensers cooled to room temp., 0° and liquid air. Dry air or O was freed from the last traces of H<sub>2</sub>O by freezing with liquid air. A max. yield of 38% maleic acid (I) was obtained at 450° and a contact period of less than 1 sec. in an O atm. The chief products of reaction are I (or maleic anhydride (II)) and 9 H<sub>2</sub>O; the by-products are quinone (4.23%) and some decompn. products probably contg. quinhydrone. When the first (air) condenser was heated to 40-50° to prevent any condensation of the H<sub>2</sub>O formed in the reaction, only pure I collected in the first condenser and I in the second (ice) condenser. It follows that the primary product of the catalytic oxidation of C<sub>6</sub>H<sub>6</sub> is II, which combines with the reaction H<sub>2</sub>O in the condensers with the formation of I. The most probable mechanism of the reaction is alternate

- oxidation of  $C_{12}H_{14}$  to hydroquinone, quinone and II with  
 'cpn of  $CO_2$  and  $H_2O$  Chas Blanc
- Standard Oil of Indiana perfects new treating process  
 for motor benzene Vanderveer Voorhes *Oil and Gas J*  
 34, No 30, 36, 39, 41-2(1938)—Refiners are giving up  
 the method of treating motor benzene with strong acid and  
 its accompanying high cost and large losses V describes  
 methods now used (1) dil  $H_2SO_4$  followed by a water and  
 caustic wash, (2) vapor-phase clay treatment, giving a  
 product that is satisfactory in everything but gum sta-  
 bility This can be remedied by the use of antioxidants  
 Benzenes from certain sources contain excessive amts. of S  
 compds These can be removed by sepg the benzene into  
 several fractions, one of them contg most of the S This  
 fraction can be treated with strong acid Antioxidants  
 are used very satisfactorily in producing a stable benzene  
 made by less severe treating methods J R. Strong
- Separation of acenaphthene from coal tar, and its puri-  
 fication P P Karpukhin and L I Slominskii *Ukrain*  
*Khim Zhur* 10, Wiss-tech Teil, 392-411(1935)(in  
 Russian)—Kamenskii coal tar contains 1.2% acenaph-  
 thene (I), b 277-8° The first fractional distn of heavy  
 oil, b 260-75°, gave 2.52% I A 2nd distn. of the oil and  
 the fraction, b 275-90°, gave addnl 0.14% I The 1st  
 fraction of anthracene oil after 3 fractionations gave  
 5.48% I, of which 1.63% was obtained in the 1st distn  
 Sepn of I from anthracene oil is more profitable, because  
 only 44% of the oil must be distd to obtain the fraction, b  
 279°, as compared with 79% of heavy oil Since the  
 difference between the vapor tensions of fluorene and I  
 increases at lower temps (Everest, *The Higher Coal-Tar*  
*Hydrocarbons*, C A 22, 597), it is advantageous to distil  
 in *vacuo* I crystd from the fraction, b 260-75°, can be  
 used after sepn by centrifuging directly in many synthe-  
 ses I is purified by crystn from benzene, ligroin or alc  
 Plans for a com sepn of I are discussed Chas Blanc
- Low temperature brown-coal tar R Schmidt  
*Brennstoff Chem* 16, 241-7(1935)—A review of work on  
 compn, constituents, analysis, use of fractions, treatment,  
 etc, with extensive bibliography for 1928-1935
- Electricity and the coking industry J F Vazson  
*Gas World* 104, No 2683 Coking Sect., 6-11(1936)—  
 Utilization of electricity in the coke plant is discussed  
 P J Wilson, Jr
- Elimination of sulfur from coke G E Foxwell *Gas*  
*J* 213, 31 3(1936)—F suggests that this is likely to be  
 one of the tech problems of our generation
- Influence of the working schedule of coke ovens on the  
 output and quality of chemical products N A Nikol's-  
 kii *Trans VI Mendeleev Congr Theoret Applied Chem*  
*1932* 2, Pt 1, 678-81(1935)—Rapid working of  
 the ovens does not decrease the output nor impair the  
 quality of the by-products Poor results obtained in  
 practice are due to pyrogenic decompn of the by products  
 in the upper part of the oven and can be eliminated by  
 changing the construction of this part of the oven and by  
 lowering somewhat the temp in the upper part of the  
 oven E E Stefanowsky
- Electrolytic decompn of caustic phenolates (from coke-  
 oven liquors) (Drees, Kowalski) 4 Corrosion-resisting  
 materials for gas appliances (Ward, Fulwider) 9 Use of  
 coke-oven gas in the glass industry (Allolio) 19 Natural  
 and industrial gases (Shagolov) 13 Test for knock  
 characteristics of motor fuels (Anon) 22 Uses of coal for  
 brickworks (Gambile, Paget) 19 Burning lime with gas  
 fuel (Kahn) 18 Kivdo-Rauchikhin brown-coal deposit  
 (Ponomarenko) 8 Sewage disposal and the supply of  
 domestic raw materials V CH from sewage-disposal  
 plants as a motor fuel (Hedmann) 14 The sapropels as a  
 source of high-mol carboxylic acids (Shvedov, et al) 10  
 Lubricating oils and motor fuels (Fr pat 789,567) 22  
 Electrically-heated coke ovens (Fr pat 789,422) 4  
 Product for intensifying combustion (Belg pat 407,586)  
 13
- Dolch, Paul Wassergas Chemie und Technik der  
 Wassergasverfahren Leipzig, J. A. Barth 268 pp  
 M 17 Reviewed in *Gas J* 213, 382 (1936).
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 4 60 Reviewed in *Ind Eng Chem, News Ed.* 14, 81  
 (1936).
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 Einfluss auf den Wirkungsgrad von Vergasermaschinen  
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 Dampfkessel- und Feuerungstechniker. Stuttgart: F  
 Enke. 137 pp M 11; bound M. 12 60
- Alkohol-Gemisch-Kraftstoffe Issued by Oesterreich-  
 isches Petroleum Institut. Vienna: Verlag für Fach-  
 literatur 29 pp M. 1. Reviewed in *Chimie et indus*  
*tr* 33, 1345(1935)
- Apparatus for drying and burning low-grade fuels such  
 as refuse for heating tube boilers Viktor Kolb (to  
 American Lurgi Corp.). U S 2,023,576, Feb 4  
 Various structural and operative details
- Colloidal fuel Erwin Blumner. Brit 436,380, Oct.  
 10, 1935 Light hydrocarbons and fuel consisting of solid  
 carbonaceous particles colloiddally dispersed in oil are ob-  
 tained by pressure cracking a suspension of coal in oil by  
 bringing a thin layer into contact with a heated surface and  
 simultaneously subjecting to centrifugal force sufficient to  
 sep gas and vapor from the suspension but insufficient to  
 sep the coal particles from the oil
- Fuel bnquets P F Turchinskii *Russ* 37,688, July  
 31, 1934 A mixt of coal breeze,  $Ca(OH)_2$  and  $(NH_4)_2CO_3$   
 is pressed and heated to 120-30°
- Fuel oils The Gas Light & Coke Co., Roland H  
 Griffith and Samuel G Hill Brit 436,027, Oct 3, 1935  
 Fuel oils for compression ignition engines are pruned with  
 a small proportion of an org compd that has a halogen  
 atom and a  $NO_2$  group on adjacent C atoms and has an  
 ignition point appreciably lower than the oil In an ex-  
 ample, 0.5-1% of the nitrates of 9,10-bromohydroxystearic  
 acid, or its Et ester, bromohydroxypine, chlorohydroxy-  
 stearic acid or the bromo-nitrate deriv of Et cinnamate  
 is added to a mixt of light creosote oil 80 with gas oil 20 l
- Gas mixer suitable for use with engine intakes Mau-  
 rice Lefebvre U S 2,028,937, Jan 28 Structural and  
 operative details
- Fuel preheating system for internal combustion engines  
 Oscar Summen (to Sulzer Gebrüder Akt-Ges.) U S  
 2,028,527, Jan 21 Various structural and operative de-  
 tails
- Coal flotation Philip M Frantz (to Colorado Fuel and  
 Iron Co.) U S 2,028,742, Jan 28 For recovery of  
 coal from a coal-and-water pulp, by froth flotation, the  
 material is aerated and agitated in the presence of a crude  
 paraffin-base petroleum oil kerosene mixt. and coal tar  
 creosote
- Coal Ernst Bierbrauer and Joseph Pöpperle Fr  
 789,291, Oct 25, 1935 See Ger. 619,239 (C A 30,  
 539)
- Apparatus for cleaning coal on a perovous vibrating deck  
 Mack L Haworth (to Jeffrey Mfg Co.) U S 2,028,-  
 834, Jan 28 Structural and mech features
- Apparatus with a perforated deck for cleaning coal by  
 pneumatic treatment Mack E Haworth (to Jeffrey  
 Mfg Co.) U S 2,028,904, Jan 28 Structural and  
 operative details
- Apparatus for the clarification of coal slurry, etc, that  
 has been treated with a precipitating agent Gesellchaft

für Förderanlagen Ernst Heckel m. b. H., Fritz Vohmann and Erich Trumplemann. Brit. 435,951, Oct. 2, 1935. The app. comprises a no. of downwardly inclined passages of small cross section arranged symmetrically with respect to the axis or a center plane of a receptacle and conveying the liquid from a receiver or receivers at the head of the receptacle to the lower part thereof. The solid matter seeps from the liquid in the passages.

**Retort and associated apparatus for distilling coal, wood, pitch, etc.** Ira H. Derby and Harold R. Horner (to Peter C. Reddy). U. S. 2,029,759, Feb. 4. Various details are described of an app. including a retort in which a vertically movable lifter is mounted. U. S. 2,029,760-1-2-3 also relates to various structural and operative details of app. for similar use.

**Destructive hydrogenation of coals.** International Hydrogenation Patents Co. Ltd. U. S. 2,029,910, Oct. 19, 1935. The fractions freed from hard asphalt and b. between 170° and 400°, preferably 200 to 400°, obtained from the products of destructive hydrogenation of coals, are treated with liquefied hydrocarbons which are gaseous at normal temp., and the refined oil obtained is subjected to destructive hydrogenation by passing it along with H<sub>2</sub> over catalysts under increased pressure and at a high temp., preferably above 350°. Interesting volatile hydrocarbons are obtained. Cf. C. A. 30, 1213.

**Hydrogenating coal, coal distillates, tars, cresols, mineral oils, distillation residues and the like.** Wilhelm Rittmeister (to F. I. du Pont de Nemours & Co.). U. S. 2,029,895, Feb. 4. See Ger. 611,922 (C. A. 29, 6026).

**Combustion gas.** I. G. Farbenindustrie AG. U. S. 2,029,174, Oct. 24, 1935. Monovinylacetylene in the compressed or liquefied state is used for welding, lighting, heating and other industrial purposes.

**Carbureted water gas.** Leslie A. Angus (to Smet-Solvay Engineering Corp.). U. S. 2,029,850, Feb. 4. In operating a water-gas set including a carburetor through which are passed blast gases admixed with an O-contg. gas alternately with the passage of water gas which is enriched with oil forming carbonaceous deposits, the blast gases are burned in the carburetor during the blasting step and concurrent removal of the carbonaceous deposits is effected by the use of sufficient additional O-contg. gas, and during the succeeding water-gas-making step oil is added to the water gas, the cracking of the oil in the water gas making step and the removal of the carbonaceous deposits being effected without introduction of extraneous fuel into the carburetor and so as to avoid maintenance of a substantial body of carbonaceous material in the carburetor. App. is described. Cf. C. A. 30, 1546.

**Fuel gas from hydrocarbon materials such as propane, butane and steam.** Frederick F. Frey, Walter F. Huppke and Jesse A. Guyer (to Phillips Petroleum Co.). U. S. 2,029,657, Feb. 4. In a continuous process for the manufacture of clean fuel gas, in which hydrocarbon reforming reactions and water-gas reactions are caused to proceed simultaneously in the presence of a catalyst such as Ni screens with alumina as a promoter, steam and hydrocarbons such as C<sub>2</sub>H<sub>6</sub> and C<sub>3</sub>H<sub>8</sub> are mixed and passed through a series of externally heated bodies of catalyst, and the mixt. is heated intermediate the catalysts to compensate for loss of heat due to the endothermic character of the water gas reactions, and hydrocarbon material is added at intervals during the passage, so that the resultant gas contains substantial quantities of hydrocarbons and water gas and has a predet. calorific value. App. is described. Cf. C. A. 29, 1619.

**Catalytic apparatus suitable for producing gas from steam and butane, etc.** Jesse A. Guyer, Frederick F. Frey and Walter F. Huppke (to Phillips Petroleum Co.). U. S. 2,029,317, Feb. 4. Various structural and operative details.

**Oil-gas generating apparatus.** Leonard B. Harris (to International Oil Gas Corp.). U. S. 2,029,774, Feb. 4. Various structural and operative details.

**Small chamber furnace for producing coal gas and coke.** Peter Walkersheim. Ger. 620,629, Oct. 21, 1935 (C. A. 29, 67).

**Apparatus for generating gas by partial combustion of powdered coal.** Dmitry Niconoff (to Phelps Dodge Corp.). U. S. 2,028,916, Jan. 28. Various structural and operative details.

**Plant for producing coal gas for heating and lighting.** S. H. G. Incorporated. Ger. 620,700, Nov. 2, 1935 (C. A. 29, 87).

**Upright retort for producing gas and coke.** Heinrich Steinfield. Ger. 620,931, Nov. 7, 1935 (C. A. 29, 2).

**The waste heat is used to produce water gas.**

**Hot gas valve suitable for use in water-gas plants.** Lauritz Ilgard (to Smet-Solvay Engineering Corp.). U. S. 2,029,844, Feb. 4. Various structural, mech. and operative details.

**Purifying gases.** Gustaf H. Hultman and Chies Wilhelm Pilo. Brit. 436,218, Oct. 1, 1935. In purifying gases from H<sub>2</sub>S by washing with an alk. liquid, e. g., alkali carbonate soln., and regenerating the liquid by the introduction of CO<sub>2</sub> and subsequent removal of the H<sub>2</sub>S and CO<sub>2</sub> from a previous cycle of the process is introduced into the spent washing liquid while maintaining the H<sub>2</sub>S therein and 1st the H<sub>2</sub>S and then the CO<sub>2</sub> is removed. The H<sub>2</sub>S may be removed by means of a vacuum after which the soln. may be boiled to drive off the CO<sub>2</sub>.

**Apparatus for purifying producer gas.** A. Ya. Kalmun. Sov. Russ. 31,100, Jan. 31, 1934. A chamber containing an inclined baffle provided with perforations for washing the gas with water, and is equipped with tubes of various diam. for the discharge of water and tar. The upper part contains a vertical gauze for sprinkling the gas with water.

**Removal of hydrogen sulfide and ammonia from gases.** Christian J. Hansen (to Koppers Co. of Del.). U. S. 2,029,262, Jan. 28. A gas such as a coal distn. gas is washed with a thionate soln. to remove H<sub>2</sub>S and the spent soln. is regenerated with SO<sub>2</sub> in sep. chambers, the treatment of the gas in the washing chamber being periodically discontinued and resumed in the chamber theretofore used for the regeneration, so that by alternating the use of the gas treating and regenerating chambers pptd. solids are dissolved from the gas washing chamber. App. is described. Cf. C. A. 30, 1546.

**Tar distillation.** Alexander A. MacCubbin and Joseph Zavernek, Jr. (to Barrett Co.). U. S. 2,029,823, Feb. 4. Vapors and liquid residue from the distn. of coal tar are sep'd from each other, and the residue is heated by indirect heat transfer from heated material passing to the first distn. stage, and the residue is subjected to a second distn. effected under a vacuum. An arrangement of app. is described.

**Fractional condensation of tar vapors.** A. K. Mityurev. Russ. 37,702, July 31, 1934. The tar vapors are passed directly into the rectifying tower to sep. individual fractions in strippers. The tower is flushed with the tar fraction which is obtained by passing tar through scrubbers for the removal of lighter fractions. The app. is illustrated.

**Tar settler.** V. I. Zhunko, L. S. Zagladin and L. F. Lazebnik. Russ. 33,954, Jan. 31, 1934. Construction details of a tar-water separator.

**Purifying tar acids.** Carl F. Hartwig (to The Barrett Co.). Brit. 436,320, Oct. 9, 1935. See U. S. 1,991,079 (C. A. 29, 23407).

**Coking materials such as coal-tar pitch.** Alvan H. Gullaher (to The Barrett Co.). U. S. 2,029,863, Feb. 4. The material is introduced into a hot coke oven heated by the coking of a previous charge, and heat for coking is supplied first to the upper portion of the mass while impeding the flow of heat from the base of the oven into the lower portion of the mass (as by insulating material such as diatomaceous earth or asbestos) and the mass is progressively heated downwardly to effect coking. App. is described.

**Coke ovens working at low or medium temperature.** Carl Still G. m. b. H. U. S. 2,029,048, Oct. 22, 1935.

**Coke-oven battery with upright chambers.** C. Otto & Comp. G. m. b. H. Ger. 620,886, Oct. 29, 1935 (C. A. 29, 101).

**Continuous carbonizing furnace.** Jean Burd. Fr. 788,707, Oct. 16, 1935.



Furnace for low temperature carbonization. M A Erbesfeld Russ 38,114, Aug 31, 1934. Construction details

Apparatus for raising the charging doors of coke ovens V P Kovalev. Russ 34,520, Feb 28, 1934. Construction details

Attachment to the coke-discharge apparatus used in coke ovens V P Kovalev. Russ 34,521, Feb 28, 1934. Construction details

Coke-oven doors Gas Chambers & Coke Ovens Ltd and Arthur H Lynn Brit 436,992, Oct. 22, 1935 Addn to 381,118 (C A 27,5948).

## 22--PETROLEUM, LUBRICANTS, ASPHALT AND WOOD PRODUCTS

W F FARAGHER

Tentative definitions of terms relating to petroleum Am Soc Testing Materials, A S T M Designation D288-35T, Am Standards Assoc, A S A No Z11-38-1935, 3 pp

Probable petroleum shortage in the United States and methods for its alleviation L C Snider and B T Brooks Bull Am Assoc Petroleum Geol, 20, 15-50 (1936) Alden H Emery

Standard method of test for distillation of crude petroleum Am Soc Testing Materials, A S T M Designation D285-33, Am Petroleum Inst Standard, A P I No 525-33, Am Standards Assoc, A S A No Z11.32-1935, 7 pp Alden H Emery

Standard methods of sampling petroleum and petroleum products Am Soc Testing Materials, A S T M Designation D270-33, Am Petroleum Inst Standard, A P I No 528-33, Am Standards Assoc, A S A No Z11.33-1935, 22 pp Alden H Emery

Tentative method of test for color of refined petroleum oil by means of Saybolt chromometer Am Soc Testing Materials, A S T M Designation D156-34T, Am Standards Assoc, A S A No Z11.35-1935, 4 pp Alden H Emery

Tentative method of test for sulfur in petroleum oils by lamp method Am Soc Testing Materials, A S T M Designation D90-34T, Am Standards Assoc, A S A No Z11.38-1935, 5 pp Alden H Emery

Tentative standard Viscosity temperature chart for liquid petroleum products Am Soc Testing Materials, A S T M Designation D341-32T, Am Standards Assoc, A S A No Z11.33-1935, 2 pp and chart Alden H Emery

Standard method of test for water and sediment in petroleum products, by means of centrifuge Am Soc Testing Materials, A S T M Designation D96-35, Am Petroleum Inst Standard, A P I No 520-35, Am Standards Assoc, A S A No Z11.8-1935, 4 pp Alden H Emery

Viscosity temperature relationship of Japanese petroleum oils III, IV Takewo Kurosawa J Soc Chem Ind, Japan 38, Suppl binding 621 2(1935), cf C A 30, 1214.—The working viscosity, i.e., the viscosity at the working temp, was obtained for various kinds of lubricating oils. The working viscosities of heavy crankcase oils were generally lower than those of light spindle oils in normal usage. The concept of transition temp and transition viscosity (at that temp) is developed, as the point of contact at which the line  $V + t = k$  (angle of slope 135°) is a tangent, where  $V$  = viscosity and  $t$  = temp, on the  $V$  vs  $t$  diagram. For Japanese oils having  $V$  of 25-3000, the transition temps lie between 20° and 145° and the transition viscosities between about 50 and 60 sec. Math expressions for the  $V$ - $t$  curves are presented. K Kammermeyer

Theoretical and technical considerations on the refining of mineral oils by physical extraction B Kwal Rechm and (Paris) 44, 282-6(1935).—After outlining the defects of the  $H_2SO_4$  process of refining mineral oils, K presents briefly the theory of refining by solvents. Expts made by K are described that showed the feasibility (on a lab scale) of treating with  $PhOH$  to s.p. the oil into a light fraction and a heavy fraction, which are further refined (after removal of  $PhOH$  by vacuum distn) by extn with  $m$ -nitrobenzyl alc. An app for carrying out the combined  $PhOH$ - $m$ - $O_2NCH_2CH_2OH$  process

continuously is described. Adaptation of the process to com operation is being investigated. A. P.-C.

The bituminous shales of Lozère and Aveyron Jean Barlot Mat, grasses 27, 10683-5(1935); cf C A 30, 12164.—The shale beds of Mende, Canourgue-Campagnac, Severac, Millau, Roquefort, Tournemure, Saint-Jean and Saint Paul are described. Analysis of 12 samples gave the following results: sol in  $HCl$  at 20° 18.2-65.0, org matter 8.1-19.0, oil 4.0-9.6 and gas 1.4-3.1%. Distn of the oil gave: 30-205° (motor spirit) 3°, 205-330° (gas oil) 41, residue (pitch) 20%. The compn of the oil is approx unsatd hydrocarbons 52, aromatic hydrocarbons 12, alcs O, bases 2.0, phenols 0.8 and S 1.15%.

A Papineau Couture  
Problems and progress of the oil industry. Much progress in motor-fuel and lubricating oil production reported Walter Miller. Mining Met, 17, 58-60 (1936) Alden H Emery

Purifying oils with selective solvents The Central Institute of Aviation Fuels and Oils Tekhnika No 33 (1935).—Purifying oils by extracting them with  $PhNO_2$  gives products with a good viscosity-temp curve.

B V. Shvartzberg  
Testing metallurgical fuel oils. John H. Brucka Iron Age 136, No 17, 20-3, 92(1935), cf C A 29, 7630.—The detn of temp-viscosity curves is a valuable supplement to sp-gr data in evaluating fuel oils. The Steiner method measures the time required for a bubble to rise through a 100 mm column of oil. F. G. Norris

Sulfured cutting oils Emde Robbe Mat grasses 27, 10651-2(1935).—A brief discussion of the advantages of the addn of S (preferably in soln in fatty oils) to mineral cutting oils, with a few working formulas for the prepn of sulfured oils. A Papineau Couture

Standard method of test for distillation of gasoline, naphtha, kerosene and similar petroleum products Am Soc Testing Materials, A S T M Designation D86-35, Am Petroleum Inst Standard, A P I No. 507-35, Am Standards Assoc, A S A No: Z11 10-1935, 9 pp Alden H Emery

The toxic properties of ethyl gasoline Gerhard Lund J Ind Hyg Toxicol 18, 37-41(1936).—107 tank attendants, 61 mechanics and 47 chauffeurs of Copenhagen, Denmark, were subjected to a clinical examn. after being exposed to ethyl gasoline for about 1 yr. The gasoline contained 2.4 cc of tetraethyl lead per gal'on. No symptoms of lead poisoning were found in any of the men examd. A. L. Elder

Tentative method of test for gum content of gasoline Am Soc Testing Materials, A S T M Designation D381-34T, Am Standards Assoc, A S A No Z11.36-1935, 4 pp Alden H. Emery

Tentative method of test for knock characteristics of motor fuels Am Soc Testing Materials, A S T M Designation D157-34T, Am Standards Assoc, A S A No Z11.37-1935, 9 pp Alden H Emery

Diesel oil obtained in the benzene synthesis of Franz Fisher and H Tropisch Isolation and chlorination of symmetrical paraffin hydrocarbons H. Koch and G. Ihng Brennstoff-Chem 16, 185-90(1933), cf C A 28, 6291.—Diesel oil, in part dewaxed Kogasin II, was subjected to vacuum distn. Normal paraffins from decane to octane were isolated. The remaining 60-70% of the paraffins consisted of isoparaffins. The  $n$ -paraffins were chlorinated and fractionated by vacuum distn. Bibliography. F. W. Jung

The constitution of lubricants prepared from Kogasin II H. Koch and G. Ibing. *Brennstoff-Chem.* 16, 261-8 (1933); cf preceding abstr.—Alkyl chlorides previously prep'd. were condensed in the presence of active Al or condensed with aromatic hydrocarbons in the presence of  $AlCl_3$ . Reaction products were sep'd by distn and the compn. of the fractions was det'd. Viscosities and viscosity-temp curves are given. F W Jung

Separation and utilization of *o* and *p*-nitrotoluenes from the mononitrotoluenes prepared from the gasoline fraction of Syukkôkô crude oil II Preparation of toluidine from nitrotoluene Masakichi Mizuta *J Soc Chem Ind., Japan* 38, Suppl. binding 629-30 (1935), cf C A 30, 1548—Toluidines were prep'd from *o*- and *p*-mononitrotoluene, sep'd by fractional distn and crystn from crude mononitrotoluene previously described. The yield and quality of the toluidines compared very favorably with those of toluidines obtained from pure nitrotoluene. III Preparation of safranin and magenta Total conclusions *Ibid* 723-4—The mechanisms by which the two compds were prep'd are given in detail. No differences were found between the products made from the toluidenes of Syukkôkô crude oil and those made from pure nitrotoluenes. Karl Kammeyer

Thin layers of tin and other metals I Influence of thin metal layers on the deterioration of technical insulating oils P J Haringhuizen and D A Was *Proc Acad Sci Amsterdam* 38, 1002-6 (1935)—With -ludge formation and increase in acidity as criteria of deterioration of insulating oils, it appears that Cu has the largest influence, Pb less and Sn least. In some cases Sn seems to act as an antioxidant. The Cu is not attacked after 100 hrs, because of formation of a protecting layer. The catalysis is thought to be due chiefly to dissolved Cu. C E P Jeffreys

Standard method of test for dilution of crankcase oils Am Soc. Testing Materials, A S T M Designation D122-35, Am. Petroleum Inst. Standard, A P I No. 524-35, Am. Standards Assoc., A S A No. Z11.29-1935, 3 pp.

The absolute viscosity of crankcase oil for automobiles Benone Anastasiu. *Ann chim anal. chim appl* 18, 5 (1935)—Values for the abs viscosity at 50°, 150° and at a temp. near the flash-point of the oil were det'd with the No 3 capillary tube of the Ubbelohde-Holde viscometer and compared with the values calcd by the formula of C. Walther (cf. C. A. 29, 7090\*) The results show that the calcd. values are slightly higher than the actual exptl. values. W T H

The aromatic constituents of mineral lubricating oils. II. Carl Zerbe and Kurt Folkens *Brennstoff-Chem.* 16, 209-11 (1935), cf C. A. 29, 5643\*—The Edeleanu expt. previously described has been hydrogenated under pressure with and without the catalysts I and  $MoS_2$ . With  $MoS_2$  under 400°, products of improved viscosity characteristics are obtained Above 450°, cracking takes place with formation of low-boiling benzene hydrocarbons. F W Jung

Relationship between the physical properties and chemical constitution of lubricating oils Michael Freund. *Refiner Natural Gasoline Mfr* 14, 486-90 (1935), *Petroleum Z* 31, No 19, Motorenfr. 8—See C. A. 29, 7631\* J. L E

Tentative method of test for color of lubricating oils by means of A S T M Union colorimeter Am Soc. Testing Materials, A S T M Designation D155-34T, Am. Standards Assoc., A S A No. Z11.34-1935, 5 pp.

Standard method of test for precipitation number of lubricating oils. Am Soc. Testing Materials, A S T M Designation. D91-35, Am. Petroleum Inst. Standard, A P I. No. 527-35; Am Standards Assoc., A S A No. Z11.30-1935, 4 pp.

Apparatus and methods for investigating the chemical constitution of lubricating oil, and preliminary fractionation of the lubricating-oil fraction of a Mid Continent petroleum Beveridge J Mair, Sylvester T Schickantz and Frank W Rose, Jr. *J Research Natl Bur Standards*

15, 557-73 (1935) (Research Paper No. 849).—This work describes the preliminary study of the hydrocarbons of lubricating oil. The lubricating fractions of a Mid-Continent petroleum were ext'd. with liquid  $SO_2$ , crystd. from ethylene chloride and fractionally dist'd. in order to sep asphaltic and resinous matter, wax and water-white fractions, resp. The app. is described in detail. Since there was a continuous increase in the viscosity of the fractions of water-white oil with respect to their vapor pressures, blending of fractions for successive distns. was accomplished on the basis of viscosity instead of b p. Although at the end of the fourth distn. no one type of hydrocarbon had been conc'd in one boiling range. It appears likely that the oil can be sep'd. by distn. into const-boiling fractions, which can in turn be sep'd. into hydrocarbons of different types by one of the other phys. methods. P J Wilson, Jr

Lubricating greases R N Smith. *Can Mining J.* 57, 22-4 (1936), cf C A 30, 845\*—A brief description of lubricating greases and their applications W. H. B

Radioactivity of oil waters in Czechoslovakia (Bc-hounek, et al) 3 Geothermal stages and the chemistry of artesian waters [presence of crude oil] (Chelotarev) 8 Infrared absorption spectra of some anthracenic hydrocarbons II Analysis of hydrocarbons in oils (Lambert, Lecomte) 3 Treatment of water for domestic purposes in a Trinidad oil field (Richard) 14 Hydrogenating mineral oils, distn residues, etc (U S pat. 2,029,595) 21 Countercurrent contact of materials such as oils and selective solvents (U S pat. 2,029,687) 9 I Treating lubricating-oil stock with selective solvents (U S. pat. 2,029,690) 1 App for countercurrent contact of materials such as oil stocks and selective solvents (U. S. pat. 2,029,691) 1 App for washing tanks for transporting oil (U S pat. 2,029,788) 1 App for cleaning tanks for transporting oil (U S pat. 2,029,795) 1. Heating fluids such as hydrocarbon oils to be cracked (U. S. pat. 2,029,291) 1. Regenerating used clays (Fr. pat. 789,159) 18 Thermometer and assoc. app. for detg. the temp. of oils in tanks (U. S. pat. 2,028,887-8) 1. Hand pump for oils (U S pat. 2,028,912) 1. Pump and assoc. app. for taking samples of petroleum from tanks (U S pat. 2,029,231) 1. Plastic resins from petroleum residuums (U S pat. 2,029,288) 13 Fractional distn. of petroleum (U S pat. 2,029,528) 1 Thermodynamic properties of mixts of a crude oil and a natural gas (Sage, Lacey) 2

Attwooll, A. W., and Broome, D. C.: Trinidad Lake Asphalt. London: The Baynard Press. 56 pp.

Egloff, Gustav, and Crandal, Emma E.: The Cracking Art in 1934 Chicago: Universal Oil Products Co. 196 pp.

Fussteig, R.: Theorie und Technik des Crackens. Berlin: Allgemeiner Industrie-Verlag. 62 pp. M. 6.80

Gaetz, Halley T.: Water-White Hydrocarbons from Trinidad Asphalt. Los Angeles: Grafton Pub. Corp. 137 pp. \$3.50.

Snider, L. C., and Brooks, B. T.: Petroleum Shortage and Its Alleviation. New York: The Chem. Foundation. 38 pp. Reviewed in *Rubber Age* 38, 217 (1936).

Petroleum-oil distillation. Alexander G. Page (to Union Oil Co. of Calif.). U. S. 2,029,601, Feb. 4. Heat is applied to a petroleum oil in proportion to distill off desired fractions present, and a cooling liquid is circulated through a closed circuit (of a described app.) in contact with the vapors for their fractional condensation (the cooling liquid being circulated under suitable control for this purpose) and the cooling liquid is then passed in heat-exchange relation with the incoming stock to be dist'd. An extensive arrangement of app. is described.

Processing hydrocarbon oils. James M. Whitley and Gustav A. Beiswenger (to Standard Oil Development Co.). Can. 354,567, Dec. 3, 1935. A hydrocarbon oil is fractionated by mixing it with 3-15 vols of a hydro-

carbon contg 1-5 C atoms and heating to within about 75° of the critical temp of the light hydrocarbon and at a pressure of 350-500 lb per sq in. An upper soln of light oil in the light hydrocarbon is sepd from a lower heavy-oil layer, and removed. The light hydrocarbon is distd from its soln and the heavy oil from its soln.

**Fractionating and cracking hydrocarbons from petroleum oils.** John B. Barnes (to Universal Oil Products Co.) U S 2,029,752, Feb 4. Crude petroleum oil contg natural gasoline is heated to vaporize the natural gasoline and the resulting gasoline-contg vapor is sepd from fractions of the crude heavier than gasoline, the heavier fractions are heated to a cracking temp under pressure in a pipe coil or the like and the heated material is thence passed into an enlarged reaction zone maintained under cracking conditions of temp and pressure, and the gasoline-contg vapor is introduced into this reaction zone and subjected to the cracking conditions in it, the reaction products are sepd into vapors and residue, and the vapors are dephlegmated and condensed. App is described.

**Operation of oil stills with tubes and headers.** Clarence H. Thayer (to Sun Oil Co.) U S 2,029,950, Feb 4. An inert gas such as flue gas is introduced into header boxes which surround return bends of a pipe still, in order to take up leakage vapors. Various details of app are described, in which gases from the header boxes are passed to the combustion zone of the still.

**Bubble-tower construction suitable for fractionations.** Loren P. Scoville (to Texas Co.) U S 2,029,277, Jan 28. Various structural details.

**Refining oils.** Hermann Suida, Hans Poll and Alfred Nowak. Fr 780,249, Oct 25, 1945. Mineral oils are sepd into paraffinic, naphthenic and aromatic fractions with simultaneous refining of the paraffinic and naphthenic fractions by treating the oil with a mixt of crude cresol and a more or less hydrophobic liquid solvent having a very selective action between the paraffinic and naphthenic fractions (PhNO<sub>2</sub>, nitrotoluene, PhNH<sub>2</sub>, toluidines, xyldines, PhNH<sub>2</sub>, C<sub>6</sub>H<sub>5</sub>Cl). The solvent may be used to sep the paraffinic fractions, then the naphthenic fractions are sepd by satg with water, the aromatic fractions, resins and asphalt remaining in soln, or the aromatic fractions, etc., may be sepd first by using a solvent said with water.

**Treatment of mineral oils.** Hermann Suida, Hans Poll and Alfred Nowak. Brit 436,194, Oct 7, 1935. This corresponds to Austrian 141,514 (C A 29, 40321). The PhNO<sub>2</sub> may be replaced by other liquid, H<sub>2</sub>O repellent solvents e.g., nitrotoluene, toluidines, PhNH<sub>2</sub>, xyldines, PhNH<sub>2</sub>, dichlorobenzenes, dichlorodethyl ether.

**Treating petroleum.** Frédéric Levi. Fr 783,180, Oct 24, 1935. Petroleum and products from its fractional distn are refined by treatment with certain silicates, infusional earths or animal or activated charcoal.

**Refining hydrocarbon oils.** Roland B. Day (to Universal Oil Products Co.) U S 2,029,250, Jan 28. An oil of approx motor fuel b.p. range is treated at temps between about 95° and 315° with added aq HCl in the presence of a natural Zn-bearing clay, which serves to remove gum forming and S compds. App is described. Cf C A 29, 4372.

**Sweetening mercaptan bearing petroleum oils.** Walter A. Schulze and Lovell V. Chaney (to Phillips Petroleum Co.) U S 2,028,998, Jan 28. The material is treated with a hypochlorite soln in the absence of O carriers so that part of the mercaptans present are converted to disulfides and the rest to alkyl S chlorides which remain in the oil, the material is sepd from the hypochlorite soln and is treated with a strong reducing soln of a water-sol alkali or alk earth sulfite, cyanide or stannite by which the alkyl S chlorides are reduced to disulfides, and the sweetened oil is sepd from the treating soln. Cf C A 30, 8417.

**Refining sulfur containing hydrocarbon oils.** Arnold V. Grosse (to Universal Oil Products Co.) U S 2,029,100, Jan 28. A S-contg oil such as a cracked oil is

treated with H<sub>2</sub> in the presence of a preformed thiomolybdate or thio tungstate of Co or the like under such conditions (suitably by heating under pressure) that the H<sub>2</sub> reacts with S compds in the oil, in the absence of H<sub>2</sub>S other than that evolved in the reaction and from the oil.

**Electric dehydration of petroleum emulsions.** Lyle Dillon (to Union Oil Co. of Calif.) U S 2,029,362, Feb 4. The emulsion is supplied to the surface of an electrode whence it falls freely onto a second electrode, an edge discharge being established from the first mentioned electrode in the direction of flow of the emulsion and an intense elec field being maintained between the electrodes so that the falling stream of emulsion is subjected to the high potential gradient resulting from this elec field. App is described.

**Electrical dehydration of petroleum emulsions.** Harmon I. Fisher (to Union Oil Co. of Calif.) U S 2,029,527, Feb 4. The emulsion to be treated is flowed downwardly through an elongated vertical interrupted elec. treating field and gas is injected into the downwardly flowing material and allowed to rise through it. App is described.

**Revivifying metallic salt solutions used for treating hydrocarbon oils.** Paul C. Rich (to Vapor Treating Processes, Inc.) U S 2,028,473, Jan 21. Used aq solns of heavy metal salts such as ZnCl<sub>2</sub> contg N bases and other org. extracted constituents from the treatment of oil at temps of about 150-230° are commingled with a metallic base such as ZnO and the liberated constituents are sepd from the revivified soln by stratification. An arrangement of app is described.

**Recovering values from acid sludge produced in treating oils, etc., with sulfuric acid.** John C. Bird (to Standard Oil Development Co.) U S 2,028,185, Jan 21. The sludge is contacted with an oxygenated org solvent not completely miscible with the sludge or with water, such as iso Pr alc., and the materials are sepd into a plurality of phases, as by setting and stratification.

**Sulfur dioxide from acid sludge.** James M. Rumble (to Chemical Construction Corp.) U S 2,028,725, Jan 21. See Ir 784,642 (C A 30, 2541).

**Sulfur dioxide production from acid sludge.** Frank J. Bartholomew (to Chemical Construction Corp.) U S 2,028,713, Jan 21. A material such as acid sludge from petroleum treatment is mixed with heated finely divided solids such as hot sand sufficient for supplying heat for decompo of the sludge, volatile products are removed from the zone of heating, carbonaceous residue is removed with the heating medium, and the latter is reheated by burning the carbonaceous residue associated with it, and at least a portion of the reheated material is further used in the process. App is described.

**Coking heavy hydrocarbon oils.** Lev A. Melker (to Universal Oil Products Co.) U S 2,029,783, Feb 4. The oil is discharged onto the exterior surface of a cylinder rotating within a heated zone in which a coking temp is generated exteriorly of the cylinder and the discharge of oil onto the cylinder is continued until a substantial quantity of coke has formed on it, and the coke is then removed from the surface of the cylinder by spraying onto a portion of the coke layer a cooling medium such as water. App is described.

**Apparatus for coking heavy liquid residuums such as those from oil refining.** Clifton J. Pratt (to J. P. Devine Mfg Co.) U S 2,028,166, Jan 21. Various structural, mech and operative details.

**Selectively shutting off flow of water in oil wells.** Eugene F. Ayers (to Gulf Research & Development Corp.) U S 2,029,649, Feb 4. CO<sub>2</sub> is forced into the well and into the water formation under high pressure to penetrate calcareous matter and dissolve it and the pressure is then released to deposit insol calcareous matter of increased bulk in the water formations and plug them. Cf C A 29, 8317.

**Filter for fuel oil or crude oil.** S. I. Chukhrienko. Russ 11,531, Feb 28, 1931. Construction details.

**Centrifugal machines.** Leo D. Jones (to Shupick Specialty Co.) Brit 436,903, Oct 21, 1935. A mixt

of oil, sludge and acid resulting from the treatment of petroleum is sepd. in a centrifugal separator having an outlet for the oil, a 2nd outlet for the sludge and a 3rd outlet consisting of inwardly directed passages which are sufficiently large to permit the discharge of all the acid, but restricted to prevent the flow of any substantial part of the sludge therethrough.

Valve suitable for controlling the flow of hot hydrocarbon oils William R. Kinnaird (to Universal Oil Products Co.) U. S. 2,029,777, Feb. 4 Structural and operative details.

Fluorescent material suitable for addition to hydrocarbon oils Hans Rabe (to I. G. Farbenindustrie A.-G.) U. S. 2,028,472, Jan. 21 A substance giving rise to fluorescence when dissolved in a hydrocarbon oil is produced by condensing a liquid olefinic material or halogenated paraffinic hydrocarbon derivs. with a polynuclear aromatic hydrocarbon in which none of the nuclei is satd with H, such as naphthalene, with a catalyst of the Friedel-Crafts type at a temp. of about 70-230° in the presence of a halide of a metal of the first or second group of the periodic system such as NaCl which does not catalyze the Friedel-Crafts reaction.

Soluble oil Standard Oil Development Co. Ir. 788,858, Oct. 18, 1935 Sol or emulsifiable oils are prep'd. by dissolving in an oil, a sulfonate sol in the oil derived from a petroleum hydrocarbon, a small amt. of a carboxylic acid (oleic acid) and an amino alc. (triethanolamine), the latter being used in at least the amt. necessary for complete combination of the acid.

Mineral oils International Hydrogenation Patents Co. Ltd. Ir. 781,626, Nov. 4, 1935 Comps. which are easily emulsifiable or sol in water for lubricating and other purposes comprise a hydrogenated mineral oil in variable proportions and an emulsifying agent, e. g., ethanalamines or ethylene glycols. The comps. may contain also naphthenic hydrocarbons, sulfonated hydrocarbons, an antioxidant, an antiseptic, water, soaps of vegetable or fat oils, or a fat oil.

Running oils Standard Oil Development Co. Brit. 436,044, Oct. 3, 1935 An oil compn. suitable for flushing the crank case of an internal-combustion engine consists of light lubricating oil and a solvent selected from aliphatic hydrocarbons, alics, ketones, ethers, halogenated hydrocarbons, or aromatic and unsatd. exts. from hydrocarbon oils. Sufficient solvent is added to increase the lauric butanol value of the compn. to above 30. Cf. C. A. 29, 7065.

Highly refined viscous mineral oil suitable for turbine or transformer oil Bertrand W. Story and Everett W. Fuller (to Socomey Vacuum Oil Co.) U. S. 2,028,257, Jan. 21 To stabilize a highly refined viscous mineral oil against acid formation, there are added to it not over 0.1% of benzyl disulfide and about 0.005-0.01% of m-cresol, thymol monosulfide, 2,2'-dihydroxy-1,1'-dinaphthyl sulfide or 4,4'-dihydroxy-diphenyl sulfide.

Fuel oil Carl L. Lauer, Robert E. Manley and Henry D. Loeb (to Texaco Development Corp.) Can. 354,572, Dec. 3, 1935 Acid-oil sludge contg. acid S. compds. is treated with hot oil in a heating chamber at 350-420°. The acid S. compds. are decomp'd. and vaporized and the vapors withdrawn. A purified oil sludge of merchantable fuel-oil grade is recovered from the residual product in the chamber.

Cracking hydrocarbon oils George D. White (to Texas Co.) U. S. 2,029,601, Feb. 4 In a process wherein cracked residual products are withdrawn from the system without recirculation through a heating coil, a combined stream of oil (as hereinafter specified) is passed through a heating coil maintained at a cracking temp. and is maintained in the coil for a time sufficient to effect substantial conversion to lower-b. p. products, the converted products are discharged from the coil into an enlarged sepg. chamber also maintained at a cracking temp. and whence residual products are withdrawn and discharged from the system, vapors are sepd. by fractionation into a vapor fraction, a higher b. p. condensate and a lower b. p. condensate and the vapor fraction is

separately condensed to give the desired final product, such as a motor fuel, the lower b. p. condensate fraction is heated in a sep. heating coil to a cracking temp. materially higher than that of the first-mentioned coil, to effect a substantial conversion, and a portion of the highly heated lower-b. p. condensate is then immediately combined with the higher-b. p. fraction and the combined stream is passed to the first-mentioned heating coil, and the remainder of the highly heated lower b. p. fraction is passed directly to the enlarged chamber to aid in maintaining a cracking temp. in it. App. is described.

Hydrocarbon-oil cracking Albert G. Peterkin, Jr. (to Atlantic Refining Co.) U. S. 2,028,728, Jan. 21 Oil such as a gas oil is heated to a cracking temp. while being passed through a pipe system or the like in zones of a furnace which are, resp., convectively and radiantly heated, the cracked oil is introduced directly into a vaporizing zone whence vapors are passed to a fractionating zone where a series of liquid fractions of progressively increasing volatility are sepd., and from an intermediate point in the fractionating zone there is removed a liquid fraction having lower volatility than gasoline but which will substantially completely vaporize under vapor-phase cracking conditions, this fraction is vaporized and heated to cracking temp. while passing through a heating zone in a furnace where it is heated only by radiant heat, and the cracked products are directly introduced into the vaporizing zone. App. is described.

Refining cracked hydrocarbon distillates Roland R. Day (to Universal Oil Products Co.) U. S. 2,029,758, Feb. 4 A distillate such as one of low b. p. is treated with aq. HCl in the presence of brass, Cu or Zn under conditions suitable for removing gum-forming olefins which are readily reactive with H<sub>2</sub>SO<sub>4</sub>, and desulfurization of the distillate is subsequently effected by treatment with H<sub>2</sub>SO<sub>4</sub>. App. is described.

Inhibiting gum formation in cracked petroleum distillates Elliott B. McConnell (to Standard Oil Co. of Ohio) U. S. 2,030,033, Feb. 4 The distillate has added to it a small proportion (suitably about 0.5% or less) of 2-, 6-, 7- or 8-hydroxy quinoline or of 2-, 6-, 7- or 8-hydroxy tetrahydroquinoline.

Petroleum-oil conversion Ralph H. McKee (to U. S. Hydrogenation Corp.) U. S. 2,028,705, Jan. 21 In treating an oil such as a crude oil, fuel oil or gas oil for production of motor fuel, by hydrogenation below the satn. point, a mixt. of the oil with water is passed through a primary conversion zone subjected to a temp. sufficient to vaporize and crack a substantial portion of the oil, the water being in proportion to retard polymerization of the oil during its heat treatment, resulting vapor and entrained liquid products are passed into a sepg. zone where liquids and vapors are sepd. and whence liquid is passed directly to a secondary conversion zone subjected to a higher temp. than that in the primary conversion zone but below that required for conversion of a substantial portion of the residue into a fixed gas (the treatment being under pressure below 20 atm.), resulting products from both conversion zones are mixed in the sepg. zone, lighter vapors from both conversion zones are together removed from the sepg. zone, and residues from the two conversion zones are recycled with hydrogenous material such as water through the more highly heated secondary conversion zone. App. is described.

Conversion of hydrocarbon oils Joseph G. Alther (to Universal Oil Products Co.) Can. 354,315, Nov. 19, 1935 The oil is heated under pressure to cracking temp. and discharged downward through a reaction zone, and the vapors are sepd. and removed from the unvaporized oil. The unvaporized oil is removed and passed to a second zone for further sepn. of vapors. The vapors from both zones are dephlegmated and the resultant condensate is returned to the heating zone. The dephlegmated vapors are then condensed. Cf. C. A. 29, 7064.

Converting petroleum acid sludge M. I. Khizerovich Russ. 38,453, Aug. 31, 1934 Petroleum acid sludge is added to clay, whereby the emulsion is broken up. The clay-bitumen mixt. can be utilized as a building material,

while the sepd. dil. acid can be worked in the usual manner. Linings for vessels used in hydrocarbon-oil conversion John Carlstrom (to Texas Co.) U. S. 2,028,967-8, Jan. 28 Various structural details are described of vessels with an outer metal shell and linings which may be formed of corrosion resisting materials.

Pyrolysis of hydrocarbon gases Michael Halpern, William B. Logan, Robert E. Manley and Walter Ulrich (to Texas Co.) U. S. 2,030,030, Feb. 4 In the conversion of normally gaseous hydrocarbons such as natural or refinery gases into hydrocarbons of higher mol. wt. such as motor fuel and lubricating oils by a pyrolysis operation to cause substantial cracking of paraffins into olefins and then polymerizing the olefins with use of an adsorptive catalyst, the catalyst used consists essentially of a "high power clay" of the acid treated montmorillonite type 50% or more of which is finer than 200 mesh and a filter aid material such as diatomaceous earth in sufficient amount substantially to reduce the resistance of the clay to the flow of vapors and gases through it. App. is described.

Processing hydrocarbons Boris Malishev (to Shell Development Co.) Can. 355,081, Dec. 31, 1935 Synthetic hydrocarbon mixts. are obtained by reaction of anhyd. unsatd. liquid hydrocarbons in the presence of finely divided anhyd.  $P_2O_5$ . E. g., 5 g. of  $P_2O_5$ , 2 g. lampblack and 0.5 cc. of cresol are added to 1000 cc. of isobutylene and the mixt. is stirred and heated for 2 hrs. at 250°. The product freed of refining agents boils up to 300°. The distd. gasoline has a good color, sweet odor, low gum content and high antiknock value.

Recovering hydrocarbons Carbo Norit-Union Verwaltungen G. m. b. H. Brit. 436,399, Oct. 10, 1935. In the recovery of hydrocarbons by adsorption, they are expelled from the adsorbent by steaming from above downward and the portion thereof obtained in liquid form during the steaming is revalorized by heat-exchange with the gaseous portion, the hydrocarbons being recovered by condensation. During or before the distn., the products may be washed with a reagent adapted to prevent revalorization of injurious constituents, e. g., S compds.

Gasoline Joseph W. Trotter Can. 354,506, Dec. 3, 1935 A mixt. of heavy and light hydrocarbon oils is heated in a high pressure converter to 750-1000°F., the vapors are passed to a second converter and sprayed over a catalytic, electrically heated surface through perforated plates. The vapors are treated with superheated steam at 800-1100°F. and at a pressure of 175 lb. per sq. in. and passed to a third converter and sprayed over a heated catalytic surface at 900-1000°F. and a pressure of 75 lb. per sq. in. The oil is treated with superheated steam and the vapors are condensed.

Refining gasoline Roland B. Day (to Universal Oil Products Co.) U. S. 2,029,757, Feb. 4 For removing S and gum and color forming constituents, gasoline is treated in the vapor phase with  $AlCl_3$  in the presence of Zn at a temp. sufficiently high to decompose the  $AlCl_3$ . App. is described.

Refining gasoline N. V. Nieuwe Oetrouw Maatschappij Fr. 788,742, Oct. 15, 1935 Crude gasoline obtained by cracking oil in the vapor phase above 500-10° and using a gas as heat carrier is purified by isolating the fraction between 70-7° and 118-2°, purifying this fraction and the remaining fractions separately, the treatment of the isolated fraction being more intense, and afterward mixing the treated fractions.

Refining cracked hydrocarbon-oil products of motor-fuel boiling point range Jacques C. Morrell (to Universal Oil Products Co.) U. S. 2,023,785, Feb. 4 The material is subjected, while in the vapor phase, to the action of a soln. contg.  $H_2SO_4$  and  $H_3PO_4$  in order to remove color- and gum-forming constituents and to reduce the S content (the process being suitable for treating a material contg. mono-, di- and tri-olefins).

Refining petroleum distillates such as gasoline Jacques C. Morrell (to Universal Oil Products Co.) U. S. 2,029,115, Jan. 28 The distillate is treated in the vapor

phase with a soln. contg. sufficient  $H_2SO_4$  to effect substantial refining of the vapors and to which has been added a sulfonated hydrocarbon such as benzene sulfonic acid in an amount substantially to dilute the  $H_2SO_4$ .

Rectification of normally gaseous hydrocarbons from refinery operations Paul D. Barton (to Alco Products Inc.) U. S. 2,028,432, Jan. 21 Compressed gases such as those contg.  $C_2H_6$  and  $C_3H_8$  are expanded in a reflux condensing zone (of a described app.) and material from the expansion step is passed into a rectification zone, a selected fraction such as rectified  $C_2H_6$  is withdrawn from the rectification zone and is heated from a material being refrigerated such as a hydrocarbon oil and a portion of the heated fraction is reintroduced into the rectification zone.

Treatment of benzene, gasoline and other hydrocarbons Soc. anon. d'Ougree-Manhaye. Brit. 435,953, Oct. 2, 1935 This corresponds to Belg. 406,192 (C. A. 29, 4555') but an oxidation inhibitor, e. g., cresol, is added to the purified hydrocarbon.

Gasoline like hydrocarbons from ethylene series hydrocarbons Cary R. Wagner (to Pure Oil Co.) U. S. 2,028,686, Jan. 28 Ethylene gases such as those from oil cracking having an ethylene content sufficiently high to produce an exothermic reaction when subjected to the temp. and pressure conditions used are passed through a reaction zone in which temps. of about 350-540° and pressures of 500-1500 lb. per sq. in. are maintained, and the time the gases are in this zone is regulated so as to effect a substantial conversion into gasoline-like aromatic hydrocarbons.

Desulfurizing gasoline Francis M. Rogers (to Standard Oil Co. of Ind.) U. S. 2,028,995, Jan. 28 Difficultly removable S compds. are converted into easily removable S compds. by contacting the gasoline in the vapor phase with solid adsorbent catalytic material of the clay type at temps. of about 340-400° at a flow rate of 12-20 bbl. of oil per hr. per ton of catalyst, the vapors are then condensed and the condensate is further treated to remove the easily removable S compds. (suitably by use of NaOH soln.) App. is described.

Storage of gasoline containing gum- and color forming constituents Harold C. Weber (to Universal Oil Products Co.) U. S. 2,029,743, Feb. 4 For preventing deterioration when the gasoline is stored for a prolonged time, nascent H is generated beneath the surface of the gasoline in storage (suitably by chemical or electrolytic action) and is permitted freely to contact with the gasoline, and an atm. of H is maintained above the surface of the gasoline to exclude oxidizing gases.

Cup grease William P. Hilliker (to Standard Oil Co.) Can. 354,964, Dec. 24, 1935 Oil is processed with a small amt. of  $H_2O$  and 2-35% of a Ca soap of a hydrogenated fat acid by heating to 260-300°F., maintaining the temp. for 1.5 hrs., cooling to about 240°F., adding 1% of  $H_2O$ , and gradually lowering the temp. until a content of 33% soap and 220°F. is reached, and the rest of the oil is worked in. The finished product contains 1%  $H_2O$  and 65-97% oil.

Lubricating oils and motor fuels Siemens & Halske A.-G. Fr. 789,567, Oct. 31, 1935 Deposition of solids or semiholids from lubricants or org. fuels on the walls of motor cylinders is prevented by modifying the surface of the metal of the cylinder by bringing it into contact with S, P or Se or compds. or mixts. of these substances. The modification may be carried out by adding one of the above substances to the lubricant or fuel.

Lubricant Standard Oil Development Co. Fr. 789,595, Oct. 31, 1935 A lubricant is composed of a heavy hydrocarbon oil and a small amt. of an organometallic compd. of the type contg. 2 metal atoms in the mol., e. g.,  $Me_2SnSnPh_4$ , and like compds. of Pb, Bi, Hg and As.

Lubricant Albert Jean-Baptiste Sellier. Fr. 789,588, Oct. 31, 1935 A lubricant having a basis of a vegetable or animal fatty substance contains a substance which automatically neutralizes acids formed during usage. Thus, olive or arachid oil having 5% acidity reckoned as oleic acid is heated in the presence of the proportion of

glycerol which corresponds to this acidity. A lubricant is thus obtained which has no tendency to acidify on using. Lubricating composition. Standard Oil Development Co. Fr. 788,557, Oct. 18, 1935. Deterioration of lubricants composed of or containing polymerized oils is prevented by adding a small amt. (0.01-0.05%) of S, Se or Te.

Lubricants from partial oxidation products of hydrocarbon oils. Joseph H. James (to Clarence P. Byrnes, as trustee). U. S. 2,029,619, Feb. 4. Mixed products of vapor-phase catalytic partial oxidation of a light lubricating oil or the like are separated into fractions of different average mol wt. containing similar compounds in the range of alcohols to oxygenated acids, a heavier fraction is reacted with a relatively small percentage of  $H_2SO_4$  at a temperature materially above  $45^\circ$ , excess free  $H_2SO_4$  is removed and oil-soluble sulfonates are also removed, leaving a lubricant substantially free from gum-forming compounds and oil-soluble sulfonates but containing oxygenated acids and which may be used alone as a lubricant or mixed with other lubricants such as automobile engine oil or with gasoline to serve as an overhead engine valve lubricant.

Lubricant suitable for use on wire rope. Walter D. Hodson. U. S. 2,028,155, Jan. 21. A plastic, jelly-like adhesive lubricant includes a top drier such as  $Co$  oxide and linseed oil in proportions to produce a tough, leathery surface on exposure of the lubricant to air without affecting the body of the lubricant such as Al oleate and mineral and blown oils. U. S. 2,028,156 relates to details of a wire rope having a core containing asbestos fibers ground to different degrees of fineness, with a lubricant, and U. S. 2,028,157 relates to a generally similar lubricated wire rope and core. U. S. 2,028,158 relates to details of lubricated wire rope manufacture.

Lubricating strips for use between spring leaves. Harvey D. Geyer (to General Motors Corp.). U. S. 2,029,366, Feb. 4. A fabric strip is provided on one side with a cushion layer of resilient non-metallic material such as rubber and carries a layer of dry lubricant such as a mix of graphite and cellulose nitrate on the other surface.

Refining south Texas lubricating-oil stocks. Wilson H. Beardsley (to Sinclair Refining Co.). U. S. 2,029,605, Feb. 4. A South Texas lubricating-oil stock having an acid number substantially exceeding 0.2 is subjected to a preliminary neutralization treatment (suitably with NaOH) insufficient to reduce the acid number of the oil to 0.2 and is thereafter treated with  $H_2SO_4$  and with an amount of water about 1.5-3.0% the volume of the oil to assist the separation of the sludge formed.

Solvent fractionation of lubricating-oil stocks. George L. Parkhurst (to Standard Oil Co. of Ind.). U. S. 2,029,680, Feb. 4. A lubricating-oil stock is treated with substantial amounts both of liquid  $SO_2$  and of a vapor-phase cracked naphtha containing at least 25% of olefins, and extract and raffinate fractions are separately recovered. App. is described. Cf. C. A. 29, 6751.

Hydrocarbons of high molecular weight from petroleum oil. David R. Merrill (to Union Oil Co. of Calif.).

U. S. 2,029,382, Feb. 4. A cracked residuum pitch is commingled with a solvent such as lubricating oil capable of dissolving high-mol.-wt. hydrocarbons from the pitch, the soln. formed is separated from insoluble residue and the high-mol.-wt. hydrocarbon material is treated with an aromatic solvent such as  $CaH_2$  and the soln. thus formed is treated with an acid such as 93%  $H_2SO_4$  and the high-mol.-wt. hydrocarbons are separated from the solvent and acid.

Viscosity-responsive devices suitable for controlling engine lubricating systems. Harry T. Booth (to Lubrication Control Corp.). U. S. 2,029,146-7, Jan. 21. Various structural, mechanical and operative details.

Asphalt from residual oil. Ullrich B. Dray and Lawton B. Beckwith (to Union Oil Co. of Calif.). U. S. 2,029,200, Feb. 4. An asphaltic residual oil is oxidized with air to produce an oxidized asphalt, which is then separated into its oil and bitumen constituents by means of a solvent such as liquid  $SO_2$ . The oil constituent is commingled with liquid  $SO_2$  to form a raffinate and an extract, and the extract is commingled with the bitumen and the mixt. is further oxidized with air to produce an asphalt having a lower penetration for the same m. p. and a relatively higher susceptibility to temperature change than the first-mentioned oxidized asphalt. Cf. C. A. 29, 6420.

Asphalt produced from petroleum distillation residuum. Bernard L. Rowe (to Standard Oil Co. of Ohio). U. S. 2,029,922, Jan. 23. A distillate residuum substantially freed from lubricating hydrocarbons is mixed with an oil distillate cylinder stock having a viscosity of at least 190 at  $99^\circ$  Saybolt, and the mixt. is oxidized.

Synthetic asphalt. A. L. Ser and B. Fisel. Belg. 408,393, April 30, 1935. Mud is mixed with tar waste, crude oil waste or natural or artificial bitumens, finely ground products, such as slag, are added.

Oxidizing oils to produce asphalts. Edward G. Ragatz (to Union Oil Co. of Calif.). U. S. 2,029,504, Feb. 4. Oil such as an asphaltic residuum is commingled with an O-containing gas at an elevated temperature (suitably about  $175^\circ$ ) to oxidize the greater part of the oil to asphalt, and the oxidized asphalt is subsequently maintained at a higher elevated temperature (suitably about  $350^\circ$ ) to polymerize oil fractions in the oxidized asphalt and under sufficient pressure (suitably about 25 lb. per sq. in.) to prevent material distillation of vaporizable oil fractions. App. is described.

Bituminous emulsions. Francis V. Lister. Brit. 436,494, Oct. 11, 1935. These are treated with an alk. clay paste and Na or K silicate, added in succession or simultaneously after pre-mixing. Thus, an emulsion is made from bitumen 55 and soap comprising 40% resin or resinous oil and 60% of a 7% NaOH soln. 10 parts; 30 parts of paste comprising bentonite 15,  $H_2O$  78 and 7% of a 7% NaOH soln. are added and then 5 parts of a 35%  $B_6$  Na silicate soln.

Apparatus for the manufacture of acetic acid. G. V. Safonov. Russ. 34,457, Feb. 28, 1934. Construction details are given of an app. for the continuous decomposition of  $Ca(OAc)_2$  with  $H_2SO_4$ .

## 23—CELLULOSE AND PAPER

CARLETON E. CURRAN

Methods of testing cellulose. R. Gabillon. *Rev. chim. ind. (Paris)* 44, 310-15, 330-1 (1935).—A brief description and discussion of the principal tests applied to cellulose, particularly that used in chemical industries.  $H_2O$ , ash, fat, Cl and chlorides,  $H_2SO_4$ -insoluble matter, color, hydrophilic properties,  $\alpha$ -cellulose, Cu no., furfural no., I no., Ag no., KOH soly., methylene blue no., and cuprammonium viscosity. A. Papineau-Couture.

The ripening of cellulose solutions. II. Giulio Tocco and Emilio Cerbaro. *Boll. reparto fibre tessili vegetali, sez. sper. ind. carta e fibre tessili vegetali* 30, 640-2 (1935); cf. C. A. 29, 1623.—The ripening of cellulose acetate dissolved in  $Ca(CNS)_2$  or  $CaBr_2$  is very slow and probably is only complete in a period of several years. The ripening

in  $ZnCl_2$  solns. takes place more readily. The ripening speed of cellulose formate depends on the thiocyanate used: the max. speed is obtained with  $NH_4CNS$  and  $Ca(CNS)_2$ .  $H_2O$  is next, there is a min. of speed with  $KCNS$  and with  $NaCNS$ .  $2H_2O$ . With coned solns of cellulose esters the ripening is accelerated, while with coned solns of thiocyanates or of  $ZnCl_2$  it is retarded. G. A. B.

Processes of solution of cellulose compounds. The structure of colloidal solutions. V. Ya. Kurbatov. *Narodnii Komissariat Tsychelet. Prom. S. S. R. Leningrad, Plastmassy* 1, 3-75 (1935).—Micelles contain polar and nonpolar parts. The ionic or nonionic nature of solvents for them, and hence the chemical structure of the solvents, must be considered. Such rules as those

of Trouton or Ramsay and Young are inexact because they do not consider chain structure. Corrections in these rules are suggested for various org. groups.

H M Leicester

The properties of films of cellulose and its esters I. The properties of cellulose acetate films. D A Fedorov, N Ya Solechnik and A M Kuptzova. *Narodnui Komissariat Tyazhelei Prom. S S S R, Leningrad, Plastmassy 1, 76-92(1935)*—Films of cellulose acetate 0.04-0.07 mm thick have a tearing strength of 7 kg per sq mm and a 10% elasticity. Two days' soaking in H<sub>2</sub>O lowers the strength slightly and raises the elasticity 50%, while 12% H<sub>2</sub>O is absorbed. After 2 months' soaking, however, the film becomes brittle. When plasticizers are added, the hygroscopicity is greatly lowered, and the strength and the elasticity after wetting also fall somewhat. A similar result is attained by increasing the no of acetyl groups on the cellulose. A sharp fall in hygroscopicity is noted when the content of acetyl group is increased from 57 to 58%. The more polar a plasticizer, the more easily it takes up H<sub>2</sub>O, and the less it protects the film from moisture. Nonpolar materials do not plasticize well. Hence those plasticizers which are neither too polar nor too nonpolar give the best results. Such are Ph<sub>3</sub>PO<sub>4</sub>, (MeC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>PO<sub>4</sub>, and di-Et phthalate. When 5-20% of a plasticizer is added to a film, the hygroscopicity drops sharply, and further increase of the amt of plasticizer does not greatly change this property. The amt of H<sub>2</sub>O absorbed by a film is the resultant of the hygroscopicity and the amt of material extd by H<sub>2</sub>O. The application of modern theories to the above facts is discussed. II The technological properties of films made from cellulose. *Ibid* 92-100—The hygroscopicity and H<sub>2</sub>O absorption of films are characteristic of their components, and are almost proportional to the polarity of these. Thus, H<sub>2</sub>O absorption decreases steadily from Cellophane (pure cellulose) through cellulose acetates and nitrates, ethyl- and benzyl-cellulose to polystyrene, which is nonpolar and nonhygroscopic. Hydrophobic plasticizers reduce the hygroscopicity of films, especially of those from cellulose nitrates and benzylcellulose. The latter gives the best of the films studied. The amt. of material extd by H<sub>2</sub>O is practically 0 for benzyl- and ethyl-cellulose, and is 0.5-1% for cellulose acetates and nitrates. The film strength is a max for polar Cellophane and a min for nonpolar polystyrene. For the esters and ethers it is about 6-7 kg per sq mm. Wetting has little effect on the strength of films from Cellophane or polystyrene. It raises the strength slightly for films from hydrophobic compds like cellulose nitrates and lowers it slightly for those from hydrophilic ones like cellulose acetates.

H M Leicester

The viscosity of solutions of cellulose ethers (ethyl-cellulose). S N Danilov and R S Aleksandrova. *Narodnui Komissariat Tyazhelei Prom. S S S R, Leningrad, Plastmassy 1, 100-166(1935)*—The viscosity of ethylcellulose solns depends on the method of prep and degree of ethylation of the ether, as well as on the solvents used. Curves for the viscosity of solns of the ether in mixts of acetates and alcs show a point of inflection at 10% alc, except for AmOAc and EtOH, in which a rise begins at 50% AmOAc. In mixts of aromatic hydrocarbons and alcs there is a min viscosity whose position is not affected by the use of homologous alcs. Mixts of ketones and alcs show no min viscosity. The viscosity of ethylcellulose solns does not rise with diln, as does that of cellulose acetate. When concd ethylcellulose solns in C<sub>2</sub>H<sub>4</sub> are diltd with small amts of alcs, their viscosity falls sharply. The viscosity min is lower than in more dil solns. Ternary mixts of 1 good and 2 poor ethylcellulose solvents give stable solns. The best films are obtained from mixts of C<sub>2</sub>H<sub>4</sub> or PhMe with alcs. C<sub>2</sub>H<sub>4</sub> and acetates are not so satisfactory, and mixts of acetates and alcs are much worse. Ethylcellulose solns are not affected by the dipole moments and similar properties of the solvents as are cellulose acetate solns.

H M Leicester

The acid hydrolysis of cellulose acetates D A

Fedorov and M. M Ratovskaya. *Narodnui Komissariat Tyazhelei Prom. S S S R, Leningrad, Plastmassy 1, 110-82(1935)*—The ordinary methods for detg acidity in cellulose acetates are not satisfactory. It is best to det the loss in wt. on heating to 160°, and, by absorption, the amt of AcOH given off. The acidity in most forms of cellulose acetates is entirely accounted for by AcOH, though H<sub>2</sub>SO<sub>4</sub> may sometimes be present. About 1/2 of the AcOH is absorbed inside the micelle. The rest is either adsorbed on the micelle surface or held mechanically in the ester capillaries. The hydrolysis of cellulose acetates is catalyzed by the adsorbed acid, which is the only form that can be in contact with both H<sub>2</sub>O and the micelle H<sub>2</sub>SO<sub>4</sub>, when present, causes especially strong hydrolysis. The ester can be stabilized by removing the H<sub>2</sub>O from the surface either by drying at 100° or extg with abs EtOH, which also removes 80-90% of the AcOH. Hydrophobic plasticizers are also good stabilizers.

H M Leicester

The synthesis of benzylcellulose S N Ushakov and V. I Gribkova. *Narodnui Komissariat Tyazhelei Prom. S S S R, Leningrad, Plastmassy 1, 158 80(1935)*—A high-quality benzylcellulose contg 2PhCH<sub>3</sub> groups is best prepd from cellulose mercerized with 50% NaOH soln for 24 hrs. This is heated for 8 hrs with 5-6 moles of PhCH<sub>2</sub>Cl at 130°. It is best purified by grinding with abs EtOH to remove the PhCH<sub>2</sub>OH, (PhCH<sub>2</sub>)<sub>2</sub>O and (PhCH<sub>2</sub>)<sub>3</sub>, which are formed as by-products, and then with H<sub>2</sub>O to wash out the NaCl and NaOH. Ag and Ni app show least action on the process. Strong mineral acids attack the ester. The viscosity of C<sub>2</sub>H<sub>4</sub> solns of the compd falls greatly when small amts of EtOH are added. Preliminary attempts to prep xylcellulose by this method give products whose films are of very poor quality.

H M Leicester

Ways of improving the quality of viscose cellulose. V. N Dolivo Dobrovolskii. *Bumazhnaya Prom 14, No. 8, 44-7(1935)*—Improved methods of production of viscose cellulose in U. S. S. R. equal to that of the American and Canadian products are discussed. C. B.

The sorption of lime by cellulose and wood G L Laroque and O Maass. *Can J Research 13B, 380-9(1935)*—The adsorption of CaO on cellulose and wood has been measured. On the basis of mol proportions, the magnitude of the adsorption is shown to be much greater than that of NaOH. It is found that considerable time is required for satn adsorption to take place. The adsorption on similar wood species is shown to be the same and much greater than on cotton cellulose. J W S.

The hemucelluloses II The association of hemucelluloses with lignin. Arthur G Norman and Jagdishwar Gopal Shrikhande. *Biochem J 29, 2250-66(1935), of C A 29, 5651*—The removal of polyuronic hemucelluloses from plant materials and woods was not easily effected by dil sulfite solns unless the material was given a previous chlorination. Probably the lignin and hemucellulose exist in some type of combination since the extn of the latter depended on a treatment effecting a soln of the lignin. E W Scott.

Qualitative and quantitative analyses of sulfite waste liquor F. Roll. *Giesserei 22, 628-30(1935)*—Bundlers for molding sands are briefly discussed. The sulfite waste liquor from paper mills contains inorg constituents such as CaSO<sub>3</sub>, CaO, H<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>O, thio acids, other acids, chiefly lignosulfonic acids or their Ca salts, a little fermentable sugar, dextrose, mannose, as well as gums, albumins, resins, vanillin and sometimes AcOH. The properties are therefore quite variable. The waste liquor is purchased at 32-35°Bé and in some cases as a powder. The latter is much more suitable for mixing with sand. Qual and quant analyses are briefly discussed. The av compn of the dry substance from waste liquor is: lignin 50, sugars 10, SO<sub>3</sub> 10, CaO 8, H<sub>2</sub>O 7, remainder 15%. C. B. Jenni.

The adsorption of carbon dioxide and of water vapor by paper pulp. Donovan D J. *Salley Textile Research 5, 493-508(1935)*—Adsorption isotherms for CO<sub>2</sub> and for H<sub>2</sub>O vapor on slightly beaten and well-beaten paper

pulp were measured over a range of temp. A correlation of the results with the known structure of cellulose and with present ideas concerning the effects of the heating process is presented.

H. P. Ieupold

Projecting and building of machines for paperboard making B. S. Darovskii *Bumazhnyia Prom* 14, No. 9, 60 1 (1935)—Structural details are discussed.

Chas. J. Lane

Asphalt paper, its manufacture, properties and applications Iruz Hoyer *Bitumen* 5, 191-3, 216 20 (1935)  
Asphalt papers are made by (1) impregnating the finished paper with bitumen by the hot process, and (2) adding an emulsion of bitumen and H<sub>2</sub>O in the hollanders or to the wet paper in the mill. Details of these methods are discussed at length. Uses for asphalt paper are roofing, packing or wrapping, paper sacks, insulating material, etc. Twenty-two references.

C. B. Jenni

Chemistry of cellulose formation (Wadano, et al.) 2  
Permanganate no in the evaluation of pulp mill waste water (Haupt-Brutten) 14 Sulfite cellulose tanning extra (Russ) pat. 37,730 29 Decolorizing substances [carbonizing and leaching cooking liquors from the alkali wood-pulp process] (Can pat. 354,360) 18 Improving cellulose materials (Belg. pat. 407,146) 25 Dispersion of resins [product used for sizing papers] (Can pat. 354,905) 13 Centrifugal machines [for paper pulp] (Brit. pat. 436,915) 1 App. for molding strips, etc., of cellulose (Brit. pat. 437,775) 30 Heat recovery from steam and vapors from paper mill digesters (U. S. pat. 2,021,360) 13 Impermeabilizing surfaces such as those of cellophane or its derivs. (U. S. pat. 2,027,776) 13

Meyer, Josef B., and Locard, Edmond. Die Sicherungstechnik der Wertpapiere, unter besonderen Berücksichtigungen der Sicherungstechniken der graphischen und schreibtechnischen Sicherungsmethoden. Biberach-Riss Gunter-Verlag, 168 pp. 1. 31.

Technik und Praxis der Papierfabrikation. Vollständiges Lehr- und Handbuch der gesamten Zellstoff-Fabrikation. Edited by Emil Heuser and Erich Opfermann. Bd. III. Die Heile der Zellstoffe. By Erich Opfermann and Ernst Hochberger. Berlin G. Pöner. 288 pp. M. 35.

Verein der Zellstoff- und Papier-Chemiker und -Ingenieure. Jahresbericht, 1931. Berlin H. Pöner 195 pp. M. 5.

Cellulose. British Celanese Ltd., Walter H. Groombridge and Eric V. Mellers. Brit. 436,090, Oct. 4, 1935. Cellulose is obtained from wood or other lignocellulosic material by dissolving the lignin in org. solvents contg. SO<sub>2</sub> or contg. inorg. basic substances in amt. at least about 0.5% of the wt. of the org. solvent, which may contain considerable H<sub>2</sub>O. SO<sub>2</sub> may be used with the basic substances, which may be present in the form of alkali metal sulfite. In an example, spruce chips are extrd. in stages with an EtOH and H<sub>2</sub>O mixt. contg. SO<sub>2</sub>, then washed with alc. and H<sub>2</sub>O and bleached with Cl<sub>2</sub>. The residue obtained on distn. of the solvent contains lignin and H<sub>2</sub>O-sol. sugars. In another example, the extr. soln. is EtOH and H<sub>2</sub>O contg. NaOH. Cl. C. A. 30, 8014.

Cellulose. Kalle & Co. A.-G. (Julius Voss, inventor) Ger. 620,627, Oct. 21, 1935 (Cl. 39b 14). The swelling capacity of articles of regenerated cellulose such as films, fibers, tubes, etc., is reduced by heating to temps. above 100° with an indifferent org. solvent with a water content of 10-35%. Thus, regenerated cellulose foil contg. 16% glycerol and 10% water is heated for 1 hr. to 106° in an autoclave with 85% EtOH. On cooling, the foil is dried. On steeping in water, the foil shows a water content of 41% as against 62% before treatment with EtOH. Other examples are given.

Estherifying fibrous cellulose. Russel H. Van Dyke, Cyril J. Staud and Harry Leff. Gray (to Eastman Kodak Co.). U. S. 2,023,900, Feb. 4. Fibrous material such

as cotton linters is pretreated with an acid such as glacial HOAc contg. H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> and is subsequently acetylated in a bath contg. a predtd. amount of the acid and of a fatty acid anhydride such as Ac<sub>2</sub>O and an inert diluent such as CCl<sub>4</sub> (the acid content of the pretreated cellulose being reduced to the predtd. amount by washing with the diluent used so that the latter is simultaneously supplied to the cellulose). Cl. C. A. 29, 6269; 30, 6124.

Saccharifying cellulose Henry Dreyfus. Brit. 436,877, Oct. 21, 1935. In saccharifying cellulose material with H<sub>2</sub>SO<sub>4</sub>, the acid is recovered as Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> or other sulfate that evolves SO<sub>2</sub> when heated, by adding Fe<sub>2</sub>O<sub>3</sub> or other suitable oxide to the liquor.

Transparent cellulosic sheets La Cellophane I. 784,027, Oct. 22, 1935. The sheets are impermeabilized by a varnish contg. a cellulose deriv., a plastifier, a wax and "hydroresinates" which are the products obtained by hydrogenating vegetable resins of the colophony type or their derivs. such as esters.

Watermarked pellicles James E. Snyder (to du Pont Cellophane Co. Inc.) Can. 354,932, Dec. 24, 1935. A pellicle of regenerated cellulose is cast, coagulated, regenerated, softened and partially dried, and die-pressed against one of the drier rolls while there is still H<sub>2</sub>O left in the pellicle.

Waterproofing films of cellulosic materials. A. Maurer, Soc. Anon. Brit. 140,300, Oct. 9, 1935. This corresponds to Fr. 770,346 (C. A. 29, 6009).

Cigaret wrappers Société "La Cellophane," Soc. Anon. (to British Cellophane Ltd.) Brit. 435,844, Sept. 30, 1935. A wrapper for a cigaret or a tobacco cartridge is composed of a thin film of regenerated cellulose to which has been added during manu. a liquid and inodorous hydrocarbon in the form of a fine emulsion. The film may be about 0.02 mm. thick and contain 25 g. paraffin oil per 100 g. dry cellulose, with or without small quantities of glycerol, stearic acid and gums, resins or perfumes, e. g., gum mastic or gum elemi.

Cellulose esters. Joseph F. Haskins and Wm. F. Underwood (to du Pont Cellophane Co.). U. S. 2,029,481, Feb. 4. Cellulose, regenerated cellulose or a lowly esterified or etherified cellulose at a temp. of about 35° is treated with a bath contg. an aliphatic monocarboxylic acid anhydride such as Ac<sub>2</sub>O and an addition product of pyridine and HCl together with some uncombined HCl or pyridine. Numerous examples are given. Cl. C. A. 29, 6758.

Cellulose esters such as cellulose acetate Camille Dreyfus and George Schneider (to Celanese Corp. of America) U. S. 2,028,781, Jan. 28. For prepg. an ester contg. little or no S compds. and of good soly. in acetone, an ester such as a cellulose acetate formed by a suspension method in the presence of a S-contg. catalyst such as H<sub>2</sub>SO<sub>4</sub> is treated with an aq. liquid such as fresh water under super-atm. pressure at temps. above 100°, repeatedly, until substantially all of the S compds. are removed and the soly. in acetone is improved. Cl. C. A. 29, 7074. U. S. 2,028,762 relates to the reduction of the viscosity of an ester such as a cellulose acetate which is free from combined S, by treating with an aq. liquid under super-atm. pressure and at temps. above 100°. Cl. C. A. 30, 8027. U. S. 2,028,763 relates to purifying cellulose acetate prepd. in the presence of H<sub>2</sub>O as a catalyst, by heating the cellulose acetate in solid form with water contg. 0.01-0.1% of an inorg. acid such as H<sub>2</sub>SO<sub>4</sub> at a temp. of 103-115° and under a super-atm. pressure of 5-10 lbs. per sq. in.

Cellulose derivatives containing alkoxy-aliphatic acid groups Carl J. Malm and James D. Coleman (to Eastman Kodak Co.). U. S. 2,025,792, Jan. 23. For producing a cellulose deriv. such as a cellulose acetate ethoxyacetate, a cellulose material having free and available hydroxyl groups, such as a cellulose acetate, is treated with an esterifying bath comprising an alkoxy-aliphatic acid anhydride such as ethoxyacetic anhydride and a tertiary org. base such as pyridine as a catalyst.

Nitrocellulose. Milton O. Schur (to Brown Co.).



U S 2,029,547, Feb 4 Sheets of interfelted cellulose fibers are hydrolyzed with a dil. inorg. acid soln so as to reduce the soln viscosity of the cellulose and the size of fiber units in the sheets but without substantial loss of such reduced fiber units, and the pretreated material is then nitrated to produce nitrocellulose of low soln viscosity Cf C A 29, 341<sup>1</sup>

Nitrocellulose composition. Amerigo F. Caprio and Harry E. Smith (to Celluloid Corp.). Can 355,177, Jan 7, 1936 A plasticized compn of nitrocellulose having improved stability and clearness is made by treating with an alc. that has been used to dehydrate nitrocellulose and has been pretreated in the presence of  $H_2SO_4$  or of a permanganate until the alc. passes the concd  $H_2SO_4$  test or the  $KMnO_4$  test and then treating a fresh batch of nitrocellulose with the rectified alc.

Glossy articles of celluloid S A Voevodskii. Russ 38,316, Aug 31, 1934 The articles are etched with  $AcOEt$

Coloring cellulose esters and ethers Franz Ackermann (to Soc pour l'ind. chim. à Bâle). U S 2,028,141, Jan 21 Coloring is effected by use of a product which can be obtained by condensing an aromatic  $p$ -dialkylamino aldehyde such as  $p$ -dimethylaminobenzaldehyde with aliphatic compds contg a reactive methylene group such as benzyl cyanide or a pyrazolone (the double linkage thus formed playing the part of the main chromophore). Such products dye various colors. Numerous examples are given

Saponifying cellulose esters Aceta G m b H. Fr 788,985, Oct 21, 1935 The sapon. of articles, e. g., rayon, made of cellulose esters is accelerated by adding org. bases contg N, P, Sb, As or S to the alk. treating baths. Examples are given using dodecyltrimethylammonium bromide, chloride and thiocyanate, diethylbenzyl-dodecylammonium chloride, ( $\beta$ -phenylethylcarbamylmethyl)trimethylammonium bromide and methylethylthiethylsulfonium methosulfate

Seasoning extruded cellulose ester plastic sheeting. Paul W. Crane and Reuben T. Fields (to du Pont Viscoid Co.) U S 2,028,602, Jan 21 Sheetting such as that of a cellulose nitrate or acetate compn is run through NaCl brine, the temp. of the brine at the point where the sheeting is introduced into it being relatively low and progressively increasing along the path of travel of the sheeting at a rate correlated with the rate of decrease of solvent content so as to avoid distortion of the sheeting, and the sheeting is then further seasoned in air Cf C A 30, 189<sup>1</sup>

Viscose B M Lotarev Russ 37,802, July 31, 1934 The xanthation is carried out with alc. emulsions of  $CS_2$

Rayon from viscose B M Lotarev Russ 37,809, July 31, 1934 To the viscose before spinning is added an alc. soln of starch

Precipitation bath for viscose rayon D I Korneev Russ 37,810, July 31, 1934 To the bath is added  $SO_2$  instead of  $H_2SO_4$  or pyrosulfuric acid to take up excess water that accumulates in the course of the process

Rayon I G Farberinnd A-G Fr 789,563, Oct 31, 1935 Rayon is spun at a speed of more than 90 m per min., the threads being disposed in crossed layers without the aid of a thread guide or spinning funnel, on a support which has a rotary and a to-and-fro movement. The coagulating bath is a rapid one contg  $H_2SO_4$  and  $Na_2SO_4$ , and also salts of metals of higher valency such as Zn, Mg and Al.

Rayon The Calico Printers' Association Ltd Fr 789,363, Oct 29, 1935 Threads or cloth made from regenerated cellulose are weighted in a manner which preserves a soft and pliable touch by using as weighting agents sol. org. compds of high b. p. and sp. gr. (ethylene glycol, glycerol, or their polymers or derivs., mono-, di- and tri-acetin) along with the constituents of a synthetic resin of the  $CH_2O$  urea type, preferably in the form of the primary condensation product and an appropriate catalyst. The impregnated fiber is dried and heated to a relatively high temp. and then washed in slightly alk. water

Rayon yarn or filament. George Schneider (to Camille Dreyfus) Can 354,330, Nov. 19, 1935 Yarns from org. derivs. of cellulose are lubricated with teased oil, oleic acid and  $H_2O$ . E. g., during the winding operation a furnishing roller treats the yarn with 2% of teased oil

Rayon William Whitehead (to Camille Dreyfus). Can 354,973, Dec. 24, 1935 Textile filaments, yarns, and fabrics are lubricated for textile processing purposes with mineral oil, such as petroleum jellies, and stabilized by a phospholipide, such as lecithin. E. g., a spinning charge of 26% cellulose acetate dissolved in 74% of acetone (contg. 5%  $H_2O$ ) is prep'd. To this soln is added 10% (on wt of cellulose acetate) of 90/100 mixt. of mineral oil and lecithin. The charge is then spun by dry or evapn. method.

Artificial threads Heinrich Ziegner. Fr 789,410, Oct 29, 1935 Threads are made from materials which are solidified by the action of solid, liquid or gaseous agents, by passing the material through a tube having porous or gas-permeable walls, the tube being surrounded by the coagulating agent which may be forced through the walls of the tube under pressure. After passing out of the tube the material may be passed through a coagulating agent.

Rayon-spinning apparatus Julius Brenzinger (to Max Ams Chemical Engineering Corp.) U S 2,028,821, Jan 28 Various mech. and operative details

Spinning bucket for use in rayon manufacture Vincent Panoff (to General Elec. Co.). U S 2,029,185, Jan 29. Structural and mech. features.

Multiple spinning nozzles for spinning rayon. Fittore Viviani. Brit 436,435, Oct. 10, 1935

Mat surfaces on rayon Böhme Fettechemie-Ges. m b H. Fr 789,538, Oct. 30, 1935 Mat surfaces are obtained by on the fibers substances yielding cations having surface activity by means of tanning substances or wood exts. and salts of multivalent metals, particularly of the 3rd, 4th, 6th and 8th group. Thus, viscose silk is treated in a bath contg. dodecylpyridinium sulfate and  $FeCl_3$  and then in a bath contg. ext. of chestnut wood. The fiber with mat aspect is subjected to a soaping operation at a relatively high temp.

Paper pulp Sidney D. Wells (one-half to Gerald D. Muggleton) U S 2,029,973, Feb 4 A continuous stream of material such as corn stalks, straw or bagasse is fed into a rod mill, immersed in partially spent, hot cooking lyes from a subsequent digestion step, and fibrous and cellular elements are sep'd by the action of the mill, the pulp material is discharged from the mill continuously, the substantially completely spent lyes are removed, and the material is digested at an elevated temp. with cooking lyes contg. an excess of cooking chemical and the partially spent lyes from this treatment are used in the preliminary treatment mentioned. An arrangement of app. is described.

Charging cellulose digesters with chip wood or the like. Sjune Svensson U S 2,029,086, Jan 28 In an operation in which the speed of charging is accelerated only by means of steam, excess steam is introduced into the downwardly flowing charging material before it is affected by the steam used for speed acceleration, in such a direction that it has no entraining effect upon the charging material and in such quantity that the excess steam prevents air accompanying the charging material from entering the digester. App. is described

Fitting knives and separators of Jordan engine haling sections Harold D. Stuck (to John W. Bolton & Sons, Inc.) U S 2,029,123, Jan 28 Mech. details.

Pulp wood grader. Andrew N. Russell and John D. Robb (to Dominion Engineering Works Ltd.) U S 2,029,125, Jan 28 Various mech. and operative details

Means for spitting pulp in cylinder paper making machines William H. Millsap Brit. 435,472, Sept 23, 1935

Apparatus for treating paper stock Charles P. Tolman (to Noble and Wood Machine Co.). Can 355,072, Dec 31, 1935 A stream of paper making stock is subjected to shearing action under pressure while it is passed in a liquid film through a film shearing gap. Pressure is applied in

advance of the shearing gap, and the freeness of the sheared stock tested. The value of freeness of the stock is adjusted by increasing or decreasing the feed pressure.

Apparatus for testing paper stock. Charles P. Tolman and James T. Coghill (to Noble & Wood Machine Co.) Can. 355,073, Dec. 31, 1935.

Apparatus for determining the concentration of paper pulp. T. M. Titov. Russ. 34,197, Jan. 31, 1934. The concn. of the paper pulp is measured by means of a helical stirring device rotating in a cylindrical container with the pulp and recording the load of an elec. motor rotating at const. speed.

Bleaching wood pulp. Alois Danninger. Fr. 788,916, Oct. 19, 1935. Wood pulp is bleached by treatment with hyposulfite which is produced *in situ*, e. g., by the action of  $\text{SO}_2$  on Zn powder.

Cellulose pulp suitable for esterifications. George A. Richter (to Brown Co.) U. S. 2,028,840, Jan. 28, 1934. A preblended hardwood pulp is suspended in a mercerizing liquor for about 2-8 hrs. or longer, washed free from mercerizing liquor, treated with NaOH to form soda cellulose, and the latter, without aging, may be treated with  $\text{CS}_2$  to form visc. e. Various cellulose esters also may be formed from the treated pulp.

Apparatus for refining fiber. Daniel M. Sutherland, Jr. Can. 354,206, Nov. 19, 1935. The working surfaces of the refining members are kept in such close clearance that the major part of the stock goes through the radially extending grooves of the surfaces.

Pressed fiberboard. Robert T. Pollock (to Respats, Inc.). U. S. 2,029,034, Jan. 28, 1936. For producing hard pressed fiberboard, uncooked wood and vegetable matter such as saw grass or palmetto fibers are reduced to fiber and a mixt. of the fibers contg. an excess of wood fibers is mixed, sheeted, dried and pressed.

Drying vulcanized fiber. G. M. Frolov. Russ. 30,620, May 31, 1933. The fiber sheets to be vulcanized are sprayed with a porous material and are dried while being compressed in closed steam-heated containers.

Sheet vulcanized fiber. Herbert R. Stratford (to Horace B. Fay). U. S. 2,028,932, Jan. 28, 1936. For inhibiting distortive change in sheet vulcanized fiber, moisture is expelled from the fiber, and the surface is sealed with a settable liquid compn. contg. a urea-rosin condensation product together with another synthetic resin such as one formed from a phenol and formaldehyde and a wax such as Japan wax. Cf. C. A. 29, 69571.

Paper manufacture. Philip J. Reimer. U. S. 2,028,952, Jan. 28, 1936. App. is described, suitable for carrying out a method in which an aq. suspension of stock is flowed onto the forming wire of a Fourdrinier type paper machine and liquid is flowed upwardly through the forming wire to maintain the stock on it in suspension, the drainage of water through the wire being gradually regulated so that the formation of two-sided sheets is prevented.

Paper. Elmer C. Schacht (to Behr-Manning Corp.). Can. 354,518, Dec. 3, 1935. A mixt. of comminuted cork and fibers with sufficient  $\text{H}_2\text{O}$  to form a stock has a flexible rubber-like binder incorporated in the web. The rubber-like binder may be rubber latex, gutta-percha latex or balata latex. The amt. used is sufficient to increase the strength and resilience of the web and render it resistant to creasing and flexing.

Paper. Elmer C. Schacht (to Behr-Manning Corp.). Can. 354,517, Dec. 3, 1935. A mixt. of comminuted cork and fibers with  $\text{H}_2\text{O}$  is formed into a web with a flexible resinous binder, such as Glyptal resins, incorporated uniformly. This makes it resistant to creasing and flexing. E. g., 60 parts of alkyl resin together with 20 parts of cork and 30 parts by wt. of fiber satd. with  $\text{H}_2\text{O}$  is placed in a steam-jacketed mixer for 35 min. at 85° and more  $\text{H}_2\text{O}$  added. This is then beaten for 1 hr. and run on a cylinder machine. The paper is then passed through a chamber at 130° to set the resin. This is suitable for manuf. of artificial leather, floor coverings and the like.

Paper. Wilma D. Schumacher. Ger. 620,953, Oct. 30, 1935 (Cl. 55d. 17). App. for watermarking paper is described.

Asbestos paper or cardboard. A. A. Bryushkov and A. D. Bakhtin. Russ. 37,953, July 31, 1934. Low-grade asbestos is disintegrated in a mill, moistened with water, worked in a macerated state in a stamp mill for 2-3 hrs., sepd. in a stream of water from traces of magnetite with the help of a magnetic separator and finally passed through paper or cardboard machines.

Chemically modified paper. George A. Richter (to Brown Co.). Can. 355,155, Jan. 7, 1936. Paper is mercerized with NaOH of less than 18% strength, washed free from chemical soln., and dried.

Paper. George A. Richter (to Brown Co.). Can. 355,156, Jan. 7, 1936. An 8% soln. of cellulose ether is mixed with a 7-8% NaOH soln. and added to 0.5-2% cellulose content with NaOH of sufficient causticity to avoid pptn. of the ether from the soln. An absorptive paper base is impregnated with the dil. soln. to a cellulose ether content of 0.5-2% and treated with an acid reagent to ppt. the ether from soln. *in situ* in the base. Cf. C. A. 30, 2481.

Coating paper. William J. Montgomery and Donald B. Bradner (to Champion Paper and Fibre Co.) U. S. 2,029,273, Jan. 28, 1936. In the manuf. of paper which is at least partially covered with a coating such as a clay mixt. having a cast surface, a fluid aq. coating mixt. is deposited upon a hard non-adhering finishing surface such as one of metal, paper is cemented to the coating and then sepd. with the coating from the finishing surface after hardening of the coating to a non-plastic condition. App. is described.

Conditioning paper. Sturtevant Engineering Co. Ltd. and W. Ardill. Brit. 434,837, Sept. 10, 1935. Paper is cooled and moisture is deposited as "dew" thereon as it passes from the calender rolls to the reel of a paper-making machine, the moisture being compressed into and absorbed by the paper during reeling. App. is described.

Decorating paper. Joseph J. Pohlman (to Beveridge-Marvellum Co.). U. S. 2,028,948, Jan. 28, 1936. Differently colored material is successively applied in irregular and non-conforming areas on paper, so that the areas have no defined boundaries and are formed of dots of color, light in shade and variously spaced, and an overlying unbroken translucent cryst. coating is then applied.

Continuous drying of paper with superheated steam. S. I. Chuvikovskii and A. A. Dubinin. Russ. 29,701, March 31, 1933. The steam used for heating calenders is passed through superheaters together with the steam formed from the moisture present in the paper. The excess of steam may be used for various purposes.

Laminated paper. Lloyd L. Dodge (Rhinelander Paper Co.). Can. 354,301, Nov. 19, 1935. A layer of thermoplastic material is interposed between plies of dried, uncalendered, well-hydrated paper. Water (10-40% by wt.) is incorporated in the paper, and then the paper is calendered under high pressure at elevated temp. The paper is relatively transparent. Cf. C. A. 29, 16391.

Safety paper. Hugo A. Schoeller G. m. b. H. Brit. 434,807, Sept. 10, 1935. Compd. safety paper for checks, deeds, etc., includes 2 or more chemicals that react to give a visible effect when a protective layer sepg. them is tampered with, e. g., by washing with  $\text{H}_2\text{O}$  or alc. or by mech. erasure. The compds. may be (a) salts of Ag or Pb and S, sulfides or chromates, or (b) Fe salts and tannin, tannic or gallotannic acid.

Transparent wrapping paper. Carleton Ellis (to Ellis-Foster Co.). U. S. 2,029,525, Feb. 4, 1936. Glassine paper carries a urea-aldehyde resin and may also carry a plasticizing synthetic resin such as an acetone-HCHO rean.

Transparent paper. Solomon G. Lipsett (to Appleford Paper Products, Ltd.). Can. 354,226, Nov. 19, 1935. A thin sheet of paper is impregnated with about 5% of mineral oil heated to at least 100°, and a coating of 5-30% polymerized vinyl acetate is applied to the surface to seal the liquid.

Transparent paper. Solomon G. Lipsett (to Appleford Paper Products, Ltd.). Can. 354,225, Nov. 19, 1935. Polymerized vinyl acetate is applied to the surface of a

sheet in soln of a concn exceeding 50% by wt. The compd has a refractive index within 0.06 of that of the cellulose fibers. The solvent is evapd. This leaves a smooth coating over the surface.

Typewriter paper of "deferred indelibility." John G. Callan. Brit. 434,852, Sept. 10, 1935. See U. S. 1,994,750 (C. A. 29, 3160<sup>6</sup>).

Waterproof, greaseproof and odorless coated paper. Walter D. Bowly (to Hercules Powder Co.). U. S. 2,028,189, Jan. 21. Paper is coated with starch and with an overlying nitrocellulose coating.

Waterproofed paper suitable for wrapping candy, etc. Earle V. Rodgers. U. S. 2,029,390, Feb. 4. Creped paper is treated with a non-aq soln contg nitrocellulose, a pigment such as Sb oxide, blown castor oil, a stearate such as that of Ca, elem gum, and an org solvent such as toluene and iso Pr acetate. App is described.

Cigaret wrappers. Albert H. Low (one-fourth to Carl Whitehead and Albert L. Vogl). U. S. 2,029,552, Jan. 21. Cigaret wrappers are impregnated with a sol silicate such as Na silicate and with a nitrate such as NaNO<sub>3</sub> or KNO<sub>3</sub>.

## 24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROF AND C. G. STORM

Preparation of explosive substances in the laboratory. I. Preparation of tetryl. Chao Lun Tseng and Ta Chu Lin. *J. Chem. Eng. (China)* 2, 128-32 (1935).—Contrary to the experience of van Duin (C. A. 12, 1554), Langenscheidt's procedure (C. A. 7, 1416-17) is found to give satisfactory results (74-84% yield) on a small scale (20 g dimethylaniline). Van Duin's procedure is easier to carry out and gives a somewhat higher yield (95% yield of crude tetryl), but it consumes a greater amt of HNO<sub>3</sub>. C. L. Tseng

Improvement of the apparent specific gravity of smokeless powder. Yates Y. C. Yen. *Chemistry (China)* 2, 699-709 (1935).—The apparent specific gravity of a smokeless powder is improved by (1) adding alc only during dehydration and not during colloidalization, (2) baking at 45-50° for 10 hrs after cutting to desired size before soaking in H<sub>2</sub>O, (3) sieving to remove powder of irregular sizes, and (4) carrying out the glazing in several steps. By taking advantage of the 2nd point, especially, the smokeless powder produced in the Hanyang powder factory, Hanyang, China, which normally has an apparent sp gr of 0.805, is obtained with an apparent sp gr of 0.830. C. L. Tseng

Detonation limits and velocities of gaseous mixtures. Jean Breton and Paul Lafitte. *Compt. rend.* 202, 316-18 (1936), cf C. A. 28, 5671<sup>1</sup>.—In the same way the lower,  $L_1$ , and upper  $L_2$ , limits of detonation, the max,  $V_M$ , and min,  $V_m$ , velocities (m/sec), of the explosive wave, and the percentage of combustible in various mixts have been detd. C<sub>2</sub>H<sub>4</sub>-O<sub>2</sub>,  $L_1$  3.1-3.2,  $L_2$  36.9-37,  $V_M$  2648 (31% C<sub>2</sub>H<sub>4</sub>), C<sub>2</sub>H<sub>2</sub>-O<sub>2</sub>,  $L_1$  2.8-2.9,  $L_2$  31.3-31.4,  $V_M$  2000 (22%), C<sub>2</sub>H<sub>2</sub>-O<sub>2</sub>,  $L_1$  3.5-3.6,  $L_2$  92-93,  $V_M$  2050 (50%),  $V_m$  1923 (99%), C<sub>2</sub>H<sub>2</sub>-air,  $L_1$  4.0-4.2,  $L_2$  50-51,  $V_M$  2011 (15%),  $V_m$  1722 (35%), NH<sub>3</sub>-O<sub>2</sub>,  $L_1$  25.0-25.4,  $L_2$  75.4-76, C<sub>2</sub>H<sub>6</sub>-O<sub>2</sub>,  $L_1$  2.6-2.7,  $L_2$  greater than equl vapor pressure, Li<sub>2</sub>O air,  $L_1$  2.7-2.8,  $L_2$  4.5-4.6,  $V_M$  1789 (6.1). Increase in pressure widens the limits of detonation, thus for H<sub>2</sub>-O<sub>2</sub> under 1 atm  $L_1$  and  $L_2$  are 15 and 90-90.4, and under 8.7 atm 12.9-14.3 and 91.1-91.3, resp., and for H<sub>2</sub> air  $L_1$  is similarly lowered from 18.5-18.8 to 14.8-15.1. Addn of small amts of H<sub>2</sub> lowers  $L_1$  for CO-O<sub>2</sub> mixts considerably. C. A. S.

Ignition of gases. IX. Ignition by a heated surface. Mixtures of methane and air at reduced pressures. C. A. Naylor and R. V. Wheeler. *J. Chem. Soc.* 1935, 1426-30, cf C. A. 26, 1124, 28, 327<sup>1</sup>.—The ignition temp of CH<sub>4</sub>-air mixts increases with the percentage of CH<sub>4</sub>. The variation of ignition temp with pressure is not uniform over the entire range of proportions of CH<sub>4</sub> and air. Thus for mixts contg less than 10.7% of CH<sub>4</sub>, two pressure limits for ignition exist. Richer mixts have only one pressure limit. At low pressures lag in ignition increases, perhaps because it ceases to be a vessel surface reaction. The lag at a given pressure increases with percentage of CH<sub>4</sub>. B. E. Anderson

Ignition temperatures of hydrocarbons. H. Bruckner and R. Schoneberger. *Brennstoff-Chem.* 16, 290-2 (1935).—App and procedure are described. Ignition temps of propane, butane, pentane, hexane and heptane, benzene, toluene and cyclohexane have been detd in relation to concn, in steam-air mixts. Lowest temps are

with excess of the gas. Temps decrease with increasing no of C atoms, being less with even nos. Review of literature and methods. F. W. Jung

Mechanism of flame extinguishing power of carbon tetrachloride. Charles Dufraisse and Jean Le Bas. *Compt. rend.* 202, 227-30 (1936), cf C. A. 28, 5672<sup>1</sup>.—Ordinary coal gas was burnt at the rate of 30 l/hr by means of 300 l/hr of air contg varying amts of CCl<sub>4</sub>, and the burnt gases were analyzed. The results show the action of CCl<sub>4</sub>, to be complex, a combination of simple diln and consequent cooling, a definite antioxygenic influence, and a combustion supporting effect due to H leaving O to react with Cl (cf Jonsson, *et al.*, C. A. 19, 178<sup>1</sup>). C. A. Silberrad

The danger of explosions in recovery installations. Erik Schwarz. *Kautschuk* 12, 15-16 (1936).—A discussion. C. C. Davis

Explosion waves and shock waves. III. The initiation of detonation in mixtures of ethylene and oxygen and of carbon monoxide and oxygen. W. Payman and H. Titman. *Proc. Roy. Soc. (London)* A152, 418-45 (1935), cf C. A. 29, 4586<sup>1</sup>.—The wave speed camera is used to study the initiation of detonation, the production of shock waves, and the effect of these waves on the flame prior to setting up of detonation. The mixts used were O with C<sub>2</sub>H<sub>4</sub> (easy to detonate) and with CO (difficult to detonate). Speed of flame in a closed-end tube before shock waves are produced depends upon (a) the fundamental speed of propagation of flame by transfer of heat and (b) the motion of the gas in which the flame is moving. The pushing and retarding of the flame by the shock waves may make the flame oscillate. A wave formerly assumed to be from the spark and known to have a pronounced effect on the flame arises in or at the rear of the flame some time after ignition. The speed of shock waves is affected by (a) motion of the gas, (b) varying temps of the gas medium and (c) meeting flames. Detonation may be set up either ahead of the flame front or, more usually, within it, owing to the effect of (a) waves traveling in front of the flame or from behind it, (b) the collision or overtaking of wave and flame or wave and wave, or (c) the collision of a wave with an obstruction or the closed end of a tube. B. E. Anderson

An explosion in the preparation of guanidine nitrate from ammonium thiocyanate (by the method of H. Gockel). C. Schopf and H. Klapproth. *Angew. Chem.* 49, 23 (1936), cf C. A. 29, 6575<sup>1</sup>.—An autoclave built for 50 atm was demolished. Reply. H. Gockel. *Ind.* Karl Kammermeyer

Gas explosions, especially carbon dioxide explosions, in coal mines. Otto Ruff. *IX Congr. intern. quim. pura aplicada, Madrid* 3, 76-103 (1934), cf C. A. 29, 1967<sup>1</sup>. E. R. Rushton

Combustion in gaseous phase (Boze) 21. Starch nitrate (Berl, Kunze) 28. Antiparasitic charge for gun cartridges (U. S. pat. 2,027,217) 17

Pépin Lehalleur, Jean. Traité des poudres, explosifs

et artufices. Paris: J. B. Baillière et fils 500 pp 1  
90, bound F. 105.  
Stettbacher, Alfred. Sprengstoff-Studien. Berlin:  
Pansgrau. 28 pp. M. 4 40.  
Istruzione sui lavori di mina e sugli esplosivi T I  
Esplosivi e mezzi d'accensione Rome 1st poligr  
dello stato, libreria. 178 pp L 8

Propellant explosive powders Fredrick Olsen (to  
Western Cartridge Co.) U S 2,028,900, Jan 28  
Ungranulated nitrocellulose powder grains are treated

with a gelatinizer for nitrocellulose such as dinitrotoluene  
while the grain contains a solvent for the gelatinizer such  
as EtOH Cf C A 29, 6062.

Cartridge case manufacture from brass, etc. Émile  
Sautier (to Manufacture de Machines du Haut-Rhin  
S. A.). U. S. 2,028,990, Jan. 28 Various operative  
details are described

Improvement to safety explosive cartridges. Soc.  
anon d'Arendonck. Belg. 406,968, Jan. 31, 1935. The  
cartridge contains K salts mixed with feldspar Cf  
C A 30, 617.

## 25—DYES AND TEXTILE CHEMISTRY

L A OLNEY

The British dyestuffs industry in 1935 Europe and  
Japanese competition R Brightman *Ind Chemist* 12,  
31-5 (1936)

Azoic dyes in cotton dyeing E B Adams *J Soc  
Dyers Colourists* 52, 5-6 (1936) —Red and blue azoic dyes  
on cotton have produced colors fast to washing and  
bleaching range Matching of a variety of color shades is  
somewhat difficult Difficulties in rubbing, stripping and  
troubles of customers are outlined Azoic dyes on viscose  
rayon C M Whittaker *Ibid* 6-7 —Viscose rayon has  
a very much greater absorptive power for naphthols than  
cotton Rubbing seems minimized in dyed viscose rayon  
Azoic dyes on silk C M Keyworth *Ibid* 7 —Most  
naphthols have a poor affinity for silk, and longer im-  
pregnation than is the case for cotton Diazo compds.  
possess great affinity for silk Azoic dyes are not widely  
used on silk Azoic colors in calico printing R J.  
Hannay. *Ibid* 7-8 —Azoic colors as a class give a  
range of very bright colors and strong shades which tend  
to be harsh in tone. Pale shades from azoic dyes are prone  
to lose more in fastness properties, particularly in light  
fastness, than corresponding shades in the vat dye range  
Azoic dyes and bleaching W Kershaw. *Ibid* 9 —  
Azoic dyes vary in their resistance to kier-boiling with  
NaOH, are resistant to hypochlorites, and mark off with  
certain types of softeners used in finishing, especially on  
old calendaring. Particle size and state of aggregation  
are of fundamental importance. In many instances, goods  
contain dyed yarn which has not been soaped-off properly,  
which is not good practice. Azoic dyes from the dye-  
stuff manufacturer's viewpoint H. Blackshaw *Ibid*.  
9-10. —A brief review Azoic dyeings from the dyestuff  
manufacturer's standpoint A F. Williams. *Ibid*  
10-11.

W. H. Boynton

Cyanine dye series II. Carbocyanines with sub-  
stituents in the three-carbon chain L G S Brooker  
and Frank L. White *J. Am Chem Soc* 57, 2480-8  
(1935); cf. C A 29, 2954 —Ortho esters of higher  
aliphatic, of substituted aliphatic and of aromatic acids  
can be employed for the prepn of thiocarbocyanines  
These esters were prep'd by the action of an alc upon an  
imino ester-HCl, prep'd in turn from a nitrile. *Me*  
*orthopropionate* (I), b. 126-8° (69% yield in 6 days); *Me*  
*orthobutyrate* (II), b. 145-7°, 13%, 23 days; *Me*  
*orthovalerate* (III), b. 167-70°, 12%, 28 days; *Me* *ortho-*  
*caproate* (IV), b. 187-90°, 40%, 5 days; *Me* *orthoiso-*  
*caproate* (V), b. 178-81°, 9%, 35 days; *Et* *orthopheno-*  
*acetate* (VI), b. 99-100°, 30%, 23 days; *Et* *orthobenzo-*  
*ate* (VII), b. 230-5-40-5°, 20%, 43 days The intermediate  
imino ester-HCl salts were unstable, decompg. with evolu-  
tion of gas and were converted directly into the ortho  
esters. 1-Methylbenzothiazole propionide (VIII), m. 173-5°,  
82% on heating 72 hrs, under reflux; butyride (IX),  
m. 186-7°, 63% after 72 hrs, allhydride (X), m. 192-3°,  
88% after 20 hrs; metho-*p*-toluenesulfonate (XI), m.  
183-5-4-5°, 90% after 3 hrs at 100°; 1-methyl-*a*-naphtho-  
thiazole metho-*p*-toluenesulfonate (XII), m. 232-3°, 88%  
after 48 hrs. at 105-10°; 2-methyl-*β*-naphthothiazole  
metho-*p*-toluenesulfonate (XIII), m. 189-90°, 94% on  
heating 3 hrs. at 140° and 3 days at 100°. The quater-  
nary salts were condensed with the ortho esters in boil-

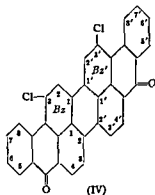
ing anhyd. C<sub>2</sub>H<sub>5</sub>N, the period of heating varying from  
10 to 90 min The yields are of the purified dye, all  
melt with decomp

Thiocarbocyanine iodides —2,2'-di-  
allyl, from X and HC(OEt), (XIV), bright, bronze needles,  
m. 264-6°, 77%, 2,2'-di-*Pr*, from VIII and XIV, prisms  
reflecting green and purple, m. 236-7°, 87%, 2,2'-di-*Bu*,  
from IX and XIV, dark purplish felt, m. 275-7°, 77%,  
2,2'-di-*allyl*-3-methyl, dark crystals with purplish reflex,  
m. 267-8°, 64% from X and MeC(OEt), (XV), 8-  
methyl-2,2'-dipropyl, purplish bronze, m. 295-6°, 64%  
from VIII and XV, 2,2'-di-*butyl*-3-methyl, dark crystals,  
reflecting blue and purple, m. 236-7°, 64% from IX and  
XV, 8-ethyl-2,2'-dimethyl, green reflex, m. 280-7°,  
56% from 1-methylbenzothiazole-MeI (XVI) and Et-  
C(OEt), (XVII), 2,2',8-*tri*-Et, double blue and green  
reflex, m. 236-7°, 41% from 1-methylbenzothiazole-  
EtI (XVIII) and XVII, 2,2'-di-*allyl*-3-ethyl, green, m.  
214-16°, 30% from X and XVII, 8-ethyl-2,2'-dipropyl,  
green, m. 248-50°, 28% from VIII and XVII, 2,2'-di-  
butyl-3-ethyl, dark prisms, reflecting purple and brilliant  
bronze, m. 241-3°, 28% from IX and XVII; 2,2'-di-  
methyl-3-propyl, purple, m. 268-9°, 50% from XI and II;  
2,2'-diethyl-3-propyl, greenish bronze, m. 240-8°, 55%  
from 1-methylbenzothiazole etho-*p*-toluenesulfonate (XIX)  
and II, 8-butyl-2,2'-dimethyl, purple with blue reflex,  
m. 168-9°, 42% from XI and III; 8-butyl-2,2'-diethyl,  
pale bronze, m. 233-4°, 58% from XIX and III; 8-amy-  
l-2,2'-dimethyl, brown needles with very brilliant blue re-  
flex, m. 217-19°, 55% from XI and IV; 8-amy-*l*-2,2'-  
dimethyl, greenish bronze, m. 237-8°, 53% from XIX  
and IV; 8-isoamy-*l*-2,2'-dimethyl, purplish brown with  
green reflex, m. 241-2°, 47% from XI and V; 8-isoamy-*l*-  
2,2'-diethyl, double blue and green reflex, m. 219-20°,  
51% from XIX and V; 8-benzyl-2,2'-dimethyl, reddish  
brown, m. 288-9°, 15% from XI and PhCH<sub>2</sub>C(OEt),  
(XX); 8-benzyl-2,2'-diethyl, emerald-green, m. 242-3°,  
11% from XIX and XX; 2,2'-di-*allyl*-8-benzyl, greenish  
blue, m. 225-7°, 33% from X and XX; 2,2'-dimethyl-8-  
PhOCH<sub>3</sub>, purplish brown, m. 255-7°, 48% from XI and  
VI; 2,2'-dimethyl-8-PhOCH<sub>3</sub>, reddish copper, m. 202-4°,  
39% from XIX and VI; 2,2'-di-*allyl*-8-PhOCH<sub>3</sub>, green,  
m. 211-13°, 41% from XI and VII; 2,2'-dimethyl-3-  
phenyl, bronze, m. 275-7°, 56% from XI and VII; 2,2'-  
diethyl-3-phenyl, greenish bronze, m. 300-1°, 56%  
from XIX and VII. 5,6,5',6'-Dibenzothiocarbocyanine  
bromides —2,2'-di-*Me*, dark purplish felted mat, m. 285°,  
83% from XII and XIV; 2,2'-di-*Et*, dark green, m. 280°,  
68% from 1-methyl-*a*-naphthothiazole etho-*p*-tolu-  
enesulfonate (XXI) and XIV; 2,2',8-*tri*-*Me*, emerald-green,  
m. 278-81°, 42% from XII and XVII; 2,2'-diethyl-3-methyl,  
greenish bronze, m. 261°, 43% from XXI and XV; 8-  
ethyl-2,2'-dimethyl, purple with blue reflex, m. 299°,  
54% from XII and I; 2,2',8-*tri*-*Et*, greenish bronze, m.  
206°, 37% from XXI and I; 2,2'-dimethyl-8-phenyl,  
dull purple, m. 308-10°, 37% from XII and VII; 2,2'-  
diethyl-3-phenyl, dull purple, m. 290°, 38% from XXI  
and VII. 3,4,3',4'-Dibenzothiocarbocyanine bromides —  
2,2'-di-*Me*, greenish bronze, m. 237°, 73% from XIII  
and XIV; 2,2',8-*tri*-*Me*, lustrous green, m. 240-1°,  
19% from XIII and XV, 8-ethyl-2,2'-dimethyl, green, m.  
230°, 24% from XIII and XVII; 2,2',8-*tri*-*Et*, greenish

bronze reflex, m. 217°, 29% from 2-methyl- $\beta$ -naphthothiazole etho-*p*-toluenesulfonate (XXII) and XVII, 2,2'-dimethyl-*S*-phenyl, greenish brown, m. 242-4°, 3% from XIII and VII, 2,2'-diethyl-*S*-phenyl, green, m. 232°, 4.5% from XXII and VII. *Selenocarbocyanine sodides*—2,2',8-tri-Me, bluish purple, m. 270-1°, 36% from 1-methylbenzoxazole metho-*p*-toluenesulfonate (XXIII) and XV, 8-ethyl-2,2'-dimethyl, green, m. 271-2°, 32% from XXIII and XVII, 2,2',8-tri-Et, double blue and brassy-green reflex, m. 146-8°, 16% from 1-methylbenzoxazole etho-*p*-toluenesulfonate (XXIV) and XVII, 2,2'-dimethyl-*S*-phenyl, greenish bronze, m. 271-2°, 18% from XXIII and XIII, 2,2'-diethyl-*S*-phenyl, metallic greenish prisms, m. 280-1°, 13% from XXIV and VII. *Oxocarbocyanine sodide*—2,2',8-tri-Me, garnet-red, m. 230-2°, 6% from 1-methylbenzoxazole metho-*p*-toluenesulfonate (XXV) and XV, 8-ethyl-2,2'-dimethyl, orange-red, m. 280-2°, 11% from XXV and XVII, 2,2',8-tri-Et, orange-red, m. 269-70°, 8% from 1-methylbenzoxazole ethiodide and XVII. Substitution of H in the mol of a thiocarbocyanine dye derived from 1-methylbenzothiazole by an 8-Me group causes the absorption max. in MeOH to shift about 150 Å toward the blue but replacement of 8-Me by 8-Et shifts the max. about 50 Å back toward the red. Replacement of 8 Et by higher aliphatic groupings up to Am and iso-Am causes no further shift. Replacement of H by 8 benzyl causes a shift (75 Å) toward the blue but replacement by 8 Ph causes a slight shift (25 Å) toward the red. Somewhat similar relationships to those summarized above can be traced in the 3,4,3',4'- and 5,6,5',6'-dibenzothiacarbocyanine series and in the selenocarbocyanines. The oxocarbocyanines are exceptional. **III Improvements in the 2'-cyanine condensation** L G S Brooker and G H Keyes *Ibid* 2488-92—Et<sub>3</sub>N is superior to KOH as a condensing agent for the prepn of 2'-cyanines. Thus, 1',2-diethyl-oxa-2'-cyanine sodide, orange-yellow, m. 230-2°, results in 43% yield with Et<sub>3</sub>N and in only 3% yield with KOH, the corresponding seleno compd, red with blue reflex, m. 280-2°, results in 72% yield from I and V; I and IX give 35% of 1,1'-diethyl-2'-pyrido-2'-cyanine sodide, reddish brown with green reflex, m. 237-9°, III and VI give 41% of the same dye, VII and III give the corresponding 4'-cyanine der., reddish brown, m. 194-6° (22%), 1,1'-Diethyl-4'-pyrido-2'-cyanine perchlorate, dark red with double blue and green reflex, m. 211-14°, 12% from I and XI, 1,1'-Dimethyl-2,2'-pyridocyanine sodide, light orange-brown with pale green reflex, m. 315-17°, 14% from II and VIII, the di-Et homolog, m. 237-9°, 12% from III and IX, 1,1'-Dimethyl-2,4'-pyridocyanine sodide, reddish brown with blue luster, m. 214-17°, 10% from II and X, di-Et homolog, dull red, m. 163-6°, 13% from III and XI. The absorption characteristics of the new dyes are given. The nos used above refer to the following: I, 2-iodoquinoline EtI, II, 2-iodopyridine-MeI, III, 2-iodopyridine EtI, IV, 1-methylbenzoxazole-EtI, V, 1-methylbenzoxazole-EtI, VI, quinaldine-EtI, VII, lepidine-EtI, VIII,  $\gamma$ -picoline-MeI, IX,  $\gamma$ -picoline EtI, X,  $\gamma$ -picoline metho-*p*-toluenesulfonate, XI,  $\gamma$ -picoline etho-*p*-toluenesulfonate. **IV L G S Brooker, G H Keyes and F L White** *Ibid* 2492-6—Et<sub>3</sub>N is an excellent condensing agent for the prepn of thiazolo-2'-cyanines. Use was made of the following quaternary salts: I, 2-iodoquinoline EtI, m. 195-7°, II, 2-iodopyridine EtI, III, 2-iodo- $\beta$ -naphthoquinoline EtI, IV, 2-methylthiazole EtI, V, 2,4-dimethylthiazole EtI, VI, 2-methyl-4-phenylthiazole EtI, m. 175-5-6.5°, VII, 2,4-dimethylthiazole etho-*p*-toluenesulfonate, VIII, 2-methyl-4-phenylthiazole etho-*p*-toluenesulfonate, IX, 2,4-dimethylthiazole EtI, m. 157-8°. I and IV give 61% of 1',3'-diethylthiazolo 2'-cyanine sodide, m. 267-9°, 4-Me der., from I and V (66%), m. 255-7°, 4-Ph der., m. 256-8° (76%), 1',3'-diethyl-4-methylthiazolo-2'-cyanine sodide, from I and VII, m. 255-8°, 49%, 4-Ph der., from I and VIII, m. 262-4°, 19%, 1',3'-diethyl-4-methylthiazolo-2'-cyanine sodide, from I and IX, m. 259-60°, 62%, 2'-Pyridocyanine sodides—1',3'-diethylthiazolo, from II and I, m. 260-2°, 31%, 4-Me der., from II

and V, m. 255-7°, 35%, 4-Ph der., from II and VI, m. 247-9°, 30%, 1',3'-diethyl-4-methylthiazolo, from II and IX, m. 242-3°, 24%, 2'-Cyanine sodides—1',3'-diethyl-5',6'-benzothiazolo, from III and IV, m. 268-70°, 24%, 4-Me der., from III and V, m. 278-80°, 33%, 4-Ph der., from III and VI, m. 274-6°, 42%, 1',3'-diethyl-4-phenyl-5',6'-benzoxazolo, from III and VIII, m. 279-82°, 1.5%, 1',3'-diethyl-4-methyl-5',6'-benzoxazolo, from III and IX, m. 275-7°, 41%. All these dyes are photographic sensitizers and details of their action are given. C J West

The vat dyes of the benzanthrone series. **XV Synthesis of 5-methoxybenzanthrone and 5,5'-dimethoxyviolanthrone** Toshio Maki, *J. Soc. Chem. Ind., Japan* 38, Suppl. binding 630-6(1935); cf C A. 30, 867—5-Chlorobenzanthrone (I) was prep'd. from 1-chloroanthraquinone. The constitution of I was det'd by oxidation with CrO<sub>3</sub>. When I was heated with KOH and MeOH in an autoclave, the corresponding 5-methoxybenzanthrone (II) was obtained, i. m. 191°. In the K melt II gave about 65% of 5,5'-dimethoxyviolanthrone which dyed cotton a greenish blue in an alc.-contg, violet red vat. The by-product of the K condensation was a gray-blue vat dye, which was difficultly sol in alk. hyposulfite. When I was condensed with K, the Cl atom was split off giving 5,5'-dihydroxyviolanthrone, which was practically insol in alc. contg. alk. hyposulfite. The 5,5'-substituents of violanthrone (III) in general exerted a rather strong bathochromic action. Exptl. results, procedures and structural formulas are given. **XVI** The constitution of dichlorinated violanthrone and the preparation of some new Bz-3,Bz'-3'-violanthrone derivatives Toshio Maki and Toshisada Aoyama *Ibid*. 636-42—Exptl. proofs are presented for the constitution of the Bz-3,Bz'-3'-dichloro compd, which is given by IV. All vat dyes



prep'd. from II dye a deep blue, independently of the color of the vat dye. All known derivs. of III which are substituted in the anthraquinone ring only, as well as III itself, give violet-red to red violet vat dyes (e. g., light colored dyes), while the derivs. substituted in the Bz rings, as far as known, all give dark colored hyposulfite vat dyes. The prepn and properties of the following dyes are described: Bz-3,Bz'-3'-dimethoxyviolanthrone, Bz-2,Bz'-2'-dihydroxyviolanthrone; Bz-3,Bz'-3'-dichloro-Bz-2,Bz'-2'-dihydroxyviolanthrone, Bz-3,Bz'-3'-diaminoviolanthrone and Bz-3,Bz'-3'-di-*p*-toluenoviolanthrone. **XVII** Preparation and purification of dinitroviolanthrone. Toshio Maki, Yoshio Nagai and Yui Hayashi *Ibid* 710-15—It was found that pure dinitroviolanthrone (V), which is a blue substance, gives the best, clear green cotton dye, and a black dye with hypochlorite oxidation, while mononitroviolanthrone (VI) (a blue-violet substance) dyes cotton a dark blue and upon hypochlorite treatment it gives a drab gray-violet. Violanthrone which possess more than 2-NO<sub>2</sub> groups give dark green to gray-green dyes. The nitration of III was carried out in glacial AcOH. The nitration curve was established and from it the optimum conditions for the

prepn. of V were found to be: 3 parts of III is stirred up in 50 parts glacial AcOH, and a mixt. of 30 parts  $\text{HNO}_3$  (d. 1.43) and 30 parts glacial AcOH is added, and the whole stirred at 60° for 12 hrs. The total ratio of glacial AcOH to  $\text{HNO}_3$  is therefore 8:3. The crude nitration product always contains more or less of a gray by-product dye, which can be removed almost completely with an 80%  $\text{H}_2\text{SO}_4$ , as V is insol in it. Pure V can be obtained by recrystn. from tetrachloroethane. It 10 pts of  $\text{HNO}_3$  only is used in the above directions, VI is obtained. The expts. are described and the nitration curve is presented. Purification of V by means of org. solvents and with 80%  $\text{H}_2\text{SO}_4$ , as well as nitration of III at room temp. (20°) and nitration in concd  $\text{H}_2\text{SO}_4$ , are described. XVIII Constitution of dinitroviolanthrone. *Ibid* 715 20—Expt results which are presented show that V is B: 2, 2'-Bz: 2'-dinitroviolanthrone. Karl Kammermeyer

Aniline black Francis A Newton *Textile World* 85, 2256 (1935).—"Practical hints on preventing streaks, tendering and other defects in printing with aniline black." Ruby K. Wornor

The action of substantive dyes Erik Schurrin *J. prakt. Chem* 144, 63-62 (1935).—A new theory of the action of dyes is proposed, which suggests that in all substantive dyes and intermediate products a many-membered ring system of conjugated double bonds is present, whose residual valence, together with that of the auxochrome group present, is the cause of the substantive action. C. J. West

Preparation and tunctional properties of certain benzoxazole dyes Vladimir J. Mikeska and Marston Taylor Bogert *J. Am. Chem. Soc.* 57, 2713-17 (1935), cf. C. A. 30, 1019.—Azo dyes of 2-p-aminophenyl-5-phenylbenzoxazole and  $\alpha\text{-C}_6\text{H}_5$  give bluish red shades on cotton, the sulfonic acid (I) with  $\text{PhOH}$ ,  $\beta\text{-C}_6\text{H}_5\text{OH}$ ,  $m\text{-C}_6\text{H}_5\text{OH}$ , (OH), and  $\alpha\text{-C}_6\text{H}_5\text{NH}_2$  (II) gives dyes which show yellow, orange-red, orange and purple shades on cotton, the bis-azo dye from II and  $\alpha\text{-C}_6\text{H}_5\text{NH}_2$  gives black shades. 2,7-Diphenyl-5-aminobenzoxazole and  $\alpha\text{-C}_6\text{H}_5\text{OH}$  give a dye yielding a saffron shade on cotton, its sulfonic acid with  $\text{PhOH}$ ,  $\beta\text{-C}_6\text{H}_5\text{OH}$ ,  $m\text{-C}_6\text{H}_5\text{OH}$ , (OH), and  $\alpha\text{-C}_6\text{H}_5\text{NH}_2$  (III) yields dyes and III gives a dye with  $\alpha\text{-C}_6\text{H}_5\text{NH}_2$ , which shows the same color on cotton as those from I. p,p'-(5-Phenylbenzoxazole-2)-o-azobenzene, amorphous brownish red, decompose above 300°, fuming  $\text{H}_2\text{SO}_4$  gives a chloramine yellow,  $\text{C}_{12}\text{H}_5\text{ON}_2\text{S}_2\text{Na}_2$ , deep orange powder, concd.  $\text{H}_2\text{SO}_4$  gives a deep blood-red color, it dyes cotton, wool or silk a direct yellow. Chloramine yellows were also prepd from 2-p-aminophenyl-5-phenylbenzoxazole-sulfonic acid, its 5-Me analog and 2,7-diphenyl-5-aminobenzoxazole-sulfonic acid. Those dyes in which the azo grouping is in the p-position on the 2-Ph nucleus are far better substantive cotton dyes than those having this grouping in position 5 on the benzoxazole portion of the mol. Azo dyes of Ingrain Red type, in the thiazole and oxazole series, on spectroscopic examn. give curves which paralleled each other closely. In general these benzoxazole dyes resemble the analogously constituted benzothiazoles but, as expected, give lighter shades. C. J. West

The dyeing of particularly wide pieces of cotton Siegfried Kosche *Z. ges. Textil-Ind.* 39, 60-1 (1936).—The width of the goods to be dyed should be between 1 and 2 m. Mech. details of the app. employed are briefly described. Conditions and procedures are discussed for dyeing with Sirius and Sirius light dyes, diazo dyes, S dyes and indanthrene or vat dyes, and also for dyeing mercerized goods. Leopold Schellian

Two-tone dyeing as related to the shape and size of silk filaments. Ired A. Mennerich and O. A. Houghton. *Textile Research* 5, 475-82 (1935), cf. C. A. 29, 4948.—Differences in shape (diam. ratio) exceeding 0.03 and in size (mean diam.) exceeding 0.7 micron cause noticeable two-tone dyeing which cannot be corrected by chem. treatment. H. F. Leopold

The fiber-damaging action of vat dyes on cellulose. Hans Frothingham *Monatsh. Seide Kunstst.* 41, 17-20, 22-4 (1936).—A review. Leopold Schellian

Comparison of the light-fastness types for dyed textiles which are in use at present. P. Kraus. *Angew. Chem.* 49, 57-7 (1936).—The German, English and American standards were compared under identical conditions by exposure to sunlight at Dresden, Germany. Expts. were also carried out with the light-fastness tester of J. F. H. Custer. The German and English standards are directly comparable in 7 out of the 8 types, while all of the 6 American standards possess much greater light fastness. Nine references. Karl Kammermeyer

Textile-testing laboratories Robert H. Brown. *Cotton* 100, 48-9, 51 (Jan., 1936).—A discussion covering some of the more recent advanced applications of more accurate control of conditions and recently established and approved principles and methods. W. H. Boynton

Conditioning water for the textile industry. H. H. Morrison *Am. Dyestuff Repr.* 25, P43-7 (1936).—

Conditioning of water may require one or a combination of several processes such as sedimentation, coagulation, settling, clarification, color removal, Fe removal, Mn removal, water softening, etc. W. H. Boynton

The cause of the von Allwörden reaction C. Sturm and H. Collé *Melliand Textilber* 16, 583-6 (1935).—A review. *Ibid* 667-8.—A repetition of the work of von Allwörden (C. A. 10, 1476) gave a compd. in 1915, [α] 57.5°, which is claimed to be glucosazone, [α] 56.5°, instead of galactazone. *Ibid* 795 8.—On treating wool with Br water, 26% of the total cystine and 39% of the total tyrosine goes into soln. Similar treatment with Cl water caused only 1.91% of the total cystine to go into soln. The amino acids, cystine and tyrosine, split off, swell up and dissolve, causing the von Allwörden reaction. F. H. Moser

Influence of various kinds of wool on some of the physical properties of flannel Florence Barr S. Dak. Agr. Expt. Sta., *Ann. Rept.* 1934, 35-8 (1934).—The wool in order of fineness is Rambouillet, Tailless, Southdown, Shropshire and Hampshire. Wool from the Tailless, Hampshire and Shropshire gave high breaking strength, bursting strength and high resistance to abrasion. The wool from Southdown was lowest in these respects. Detailed data are given. C. R. Fellers

The optimum conditions of wool scouring. S. S. Rakhilina and M. I. Piekhan *Sherstyanoe Jelo* 1935, No. 3, 19-23, Nos. 4-5, 17-23.—The fats remaining on the wool after treatment in the successive scouring tanks have lower acetyl values and higher I values, the unsaponifiable matter, the acid value and the sapon value are unchanged. The supposition that the high-melting fats remain in the wool at the end of the scouring is disproved. Removal of fat from wool is best accomplished with soap-soda solns. producing stable emulsions. The soap forms absorption films on the surface of the fat drops and creates in the soln. a concn. corresponding at least to the satn. limit of the surface layer. Soda forms soaps with the free acids of the wool, maintaining the soap introduced into the tank at optimum dispersion at a definite pH value of the medium. The optimum concns. of the scouring liquor are soap equiv. to 0.5-0.9 g. fat acids and soda 3-5 g. per l. B. V. Shvartzberg

Alkalies and wool. Frederick R. Harrison. *Textile World* 85, 2247 (1935).—Up to a pH of 12 at 120°F. for 15 min., the amt. or kind of alkali has no harmful effect on wool, so far as that effect can be measured by tensile strength. Above pH 12, damage is shown by decrease in strength and by yellowing. Ruby K. Wornor

Wool yarn bleaches Walter Bruckhaus. *Deut. Wollen-Graeber* 68, 3-4 (1936).—The compn. of the water used is important, particularly in the case of O bleaches employing  $\text{Na}_2\text{O}_2$  and  $\text{H}_2\text{O}_2$ . In place of wooden tanks it is better to use tank manifolds from V4A steel, stoneware, Havag material, etc. Directions are given for pretreatments and for bleaching with  $\text{Na}_2\text{O}_2$  and bisulfite,  $\text{KMnO}_4$  and  $\text{SO}_2$ ,  $\text{Na}_2\text{O}_2$ ,  $\text{H}_2\text{O}_2$ , S bleaches and combination bleaches utilizing oxidation and reduction methods. L. S.

Deterioration of weighted silk under the conditions of acidity, alkalinity and salinity to which fabrics are subjected in service and maintenance. Rachel Edgar. *Ja.*

Agr. Expt. Sta., *Rept. Agr. Research* 1934, 151(1934); cf. C. A. 29, 7083<sup>4</sup>—Abstract. A decided similarity in behavior was observed among 1-e-weighted black silk crepe fabric, Pb-weighted white silk crepe fabric, Sn-weighted white silk crepe fabric, Zn-weighted white silk crepe fabric and degummed silk crepe fabric when treated with alkali for 10 hrs at 40°. The effect of the alkali at 0.4 N was greater than that of 1.7 N acid. The fabrics were carefully examined chemically and physically, after 33 dry cleanings and launderings. A great decrease in dry elongation of the dry-cleaned silks, low breaking strengths of the dry-cleaned Sn- and Sn-Pb weighted silks, low wet breaking strengths of the Pb-weighted silks and the low breaking strength of the laundered Sn-Pb-weighted silk were noted.

C. R. Fellers  
Too much sheen should be avoided in finishing pure-dye and par-weighted flat crepes. Philip Lebrun *Textile World* 85, 2048(1935).—Finishing operations are described. Formulas for rubbing oil to hide chafe marks and for sizing are given.

Ruby K. Worner  
Spun silk—its manufacture and uses. Noel D. White *Am. Dyestuff Repr.* 25, 27-30, 63-4(1936).—An illustrated description.

W. H. Boynton  
The elimination of the soap from degummed silk. G. Baroni and G. Cola *Boll. ufficiale stes. sper. seta* 5, 53-6(1935).—Acid soaps can be completely eliminated from degummed silk by treatment with alkali, it can be more easily removed by extn. with 10% HCl, or with boiling water followed by CaCl<sub>2</sub>.

G. A. Bravo  
Better boil-off. Philip Lebrun *Textile World* 85, 2257(1935).—A pre-snoaking treatment and the "skein" method of boiling-off silk flat crepes are described. The prevention of chafing is discussed.

Ruby K. Worner  
Serviceability of rayons and silks. I. thelwyn Dodson *Am. Silk Rayon J.* 55, 37-8, 56-7, 58-9, 60(1936).—Data on tensile strength, yarn slippage, shrinkage in cleaning, dry, color fastness for many of the 82 dress goods fabrics are studied.

W. H. Boynton  
Staudinger viscosity law in rayon manufacture. A. Zart *Kunststoffe* 17, 38-42(1935).—This article is a reply to Prof. Staudinger who complained that the detn. of mol. wt. according to his viscosity law (C. A. 28, 6129<sup>4</sup>) has not been given sufficient attention in the rayon industry. Z. reviews Staudinger's investigations of the relation between viscosity and mol. wt., and discusses the possibility of applying this method in the investigation of celluloses used in making rayon and of the intermediate products and the final rayon. Z. concludes that the viscosity law can have only limited application in such studies.

I. Frederic C. Hahn  
Recovery process [for rayon soaking solutions]. Peter C. Wayner *Textile World* 85, 2252(1935).

R. K. W.  
Rayons and linseed-oil sizes. W. Weltzien *Monatsh. Seide Kunststoffe* 41, 32-7(1936).—Although the use of linseed oil for sizing purposes has been deplored, linseed oil is still being used nearly exclusively for strand sizing. Linseed-oil sizes are used particularly for high grade textiles. Advantages of strand sizing with this oil are enumerated, and a review is given of several characteristic properties and relationships dealing with the high smoothness of a good linseed-oil size, the stability of the sizing, the stability test (dry heat at 105° for 3 hrs.), the tropical test (heating for 4 hrs. to 80°, at a relative air humidity of 90%, in an oil bath), ease of desizing, damages to the fibers and strength measurements. Some data are given showing the strength of 2 viscose silks which had been treated with 5 different linseed oil sizes. A schematic drawing shows the various methods of testing unsized, sized and desized rayon. The complete set of tests involves 8 strength detns. of 30 measurements each in addition to various other exams.

Leopold Scheffan  
Warp sizing. III. Paul Seydel *Cotton* 100, 55-7 (Jan., 1936), cf. C. A. 30, 1750<sup>4</sup>.—The gums and glues used in warp sizing and finishing are covered. The gums include gum arabic, tragacanth, cherry tree, sugarbeet, carageen, sea moss, Irish moss, Iceland moss, etc. and some converted starch gums.

W. H. Boynton  
Cotton fiber investigations. Effect of soil types and

seasonal conditions on length and strength of cotton fiber. O. A. Pope *Ark. Agr. Expt. Sta., Bull.* 312, 27(1934), cf. C. A. 29, 8207<sup>7</sup>.—Fiber quality is the composite result of many phys. and chem. properties. A photoelectric method for detg. the fiber length distribution in cotton is suggested.

C. R. Fellers  
Studies of nitrogenous compounds from cotton and linen. Franz Barlet *Monatsh. Seide Kunststoffe* 41, 25-31(1936).—A critical review. In spite of the work of Chilikin (cf. C. A. 24, 3375<sup>4</sup>) the characters of the "black acid" and the "amino acid L" have not yet been explained.

Leopold Scheffan  
A critical study of cotton manufacturing processes. R. L. Lee, Jr. *Textile Research* 5, 167-83(1935), cf. C. A. 28, 7024<sup>4</sup>.—Data are given on the variation which occurs in the uniformity of the strands used in each of the processes of manu. of carded cotton warp yarns. Ibid. 326-36.—Methods of measuring the irregularity of cotton sliver are studied.

H. I. Leopold  
An electrical method for measuring the moisture contents of fabrics. J. L. Spencer-Smith *J. Textile Inst.* 26, T336-40(1935).—See C. A. 30, 1236<sup>4</sup>.

R. K. W.  
Comparative tests for value of sulfur trioxide in different amounts in sulfonated compounds. Albert H. Grimshaw *Am. Dyestuff Repr.* 25, T37-42(1936).—The properties required of textile softening and finishing compds are outlined.

Comparative tests on 8 samples of sulfonated compds with varying percentages of fat and SO<sub>2</sub> are outlined and results tabulated for several. The tests include stability with HCl, stability by Babcock method, stability by Babcock with H<sub>2</sub>SO<sub>4</sub>, stability by Babcock with AcOH, stability of common salt, stability with hard water using CaCl<sub>2</sub>. Some finished tests on dyed skeins, wetting-out tests and emulsion tests are included. While results are somewhat incomplete it appears that in using oils of equal fat value those with higher SO<sub>2</sub> content generally give better results.

W. H. Boynton  
Sources of defects during the washing operation (in the textile industry). A. Foulon *Deut. Wollen-Gewerbe* 68, 49-50(1936).—A survey dealing with the action of soap, mech. and chem. factors causing defects, and the detection of chem. damages by means of the quartz analysis lamp and the microscope. Chem. deterioration is also indicated by the presence of oxycellulose. Tests with the strength tester show whether the washed goods had been weakened only locally or all over.

Leopold Scheffan  
Calgon in textile improvement. Anton Volz *Melliand Textilber* 16, 780-1(1935).—The detergent qualities of Calgon are described.

F. H. Moser  
Cleaning of inexpensive goods before rubberizing (Molnar) 30. Preserving fabrics (Brit. pat. 435,344) 12

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Rusta 10, 453  
Fierz-David, Hans E. *Kunstliche organische Farbstoffe*. Ergänzungsband. Berlin: J. Springer. 136 pp. M. 11. Reviewed in *J. Soc. Dyers Colourists* 52, 24 (1936). Cf. C. A. 29, 1904<sup>4</sup>.

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Verfahren, Normen und Typen für die Prüfung und Beurteilung der Färbungseigenschaften von Färbungen auf Baumwolle, Wolle, Seide, Viskosakunstseide und Acetatknustseide. Issued by "Die Färbungskommission der Fachgruppe für Chemie der Farben- und Textilindustrie im Verein Deutscher Chemiker." 7th issue. Berlin Verlag Chemie. 60 pp. M 3 60

**Dyes.** See pour l'ind chim A Bile. Swiss 174,082, May 1, 1915 (Cl. 37a). Dyes are formed by coupling diazo compds of the general formula  $\text{ROCH}_2\text{CONH}_2$   $\text{C}(\text{CH}_3)(\text{OH})$   $\text{C}(\text{N}(\text{OH})\text{CH}_2\text{CO}_2\text{H})$ , in which R is

an aromatic nucleus of the benzene series, with arylides contg a coupling  $\alpha$  hydroxy carboxylic acid group in the  $\alpha$  position to a OH group, in a medium of pH value 5 to 7. Thus, an aq. soln. of diazotized 4 (methylphenoxyl) acetyl amino 2,5 diethoxyaniline is given an addn. of  $\text{Na}_2\text{PO}_4$  to obtain the correct pH value and is treated with the anilide of hydroxynaphthone acid and  $\text{NaOH}$  to give a dye.

**Dye compositions for printing animal and vegetable fibers.** Intz Grieshaber (to Soc pour l'ind chim A Bile). U. S. 2,029,991, Feb. 1. Printing with dyes of different classes such as indigoid, anthraquinoid and sulfur dyes, also dyes contg methyl in complex combination: azo dyes, di- and triarylmethane dyes, pyrazoline dyes, acridine dyes, quinoline dyes, quinonimine dyes, azine, oxazine and thiazine dyes, is affected with use of auxiliary substances such as alkyl sulfonamides, benzene-sulfonamide, benzenedisalkylsulfonamides, toluene-sulfonamide, toluenemonomethylsulfonamide, toluenedimethylsulfonamide, tolueneethylsulfonamide, mixts. of  $\alpha$  and  $\beta$  toluene-sulfonamide, dibenzene-sulfonamide,  $m$ -aminobenzene-sulfonamide, halo benzene-sulfonamides, naphthalene-sulfonamides, e. g., naphthalenedimethylsulfonamide, naphthaleneethylsulfonamide, naphthalenedisulfonamides, naphthalenesulfonamides, 1-hydroxynaphthalene-8-sulfonamide, tetrahydro-naphthalene-sulfonamides, benzene-sulfonamides, as well as the salts, such as Na and K salts of such compds., further, salts of sulfonamides of the benzene and naphthalene series having in the nucleus sulfo groups or carboxyl groups, such as phthalic acid-sulfonamide or hydroxybenzenecarboxylic acid-sulfonamides. The process may be applied to all kinds of textile printing on webs, yarn or loose materials, for instance in discharge printing or in direct printing, for instance in padding printing or any other kind of printing in which steaming follows the printing. Several examples with details are given.

**Anthraquinone derivatives.** Georg Krametz, Hans Schlichenmayer and Ludwig Schöring (to General Aniline Works). U. S. 2,029,249, Jan. 28. See Ger. 610,580 (C. A. 29, 6074f).

**Acid dyes of the anthraquinone series.** Ernst Dielenbach and Fritz Fischer (to General Aniline Works). U. S. 2,029,258, Jan. 28. Dyes generally drying wool and silk blue shades are produced by heating to about 60–100° a 1-amino-2-sulfo-4-haloanthraquinone compd with an aminocarbonylsulfonamide compd of the general formula  $\text{NH}_2\text{—R—SO}_2\text{NX}(\text{alkyl})$ , wherein X means H or a lower alkyl group, R means an aryl radical of the benzene and naphthalene series and alkyl means a lower alkyl group, at least one of the alkyl groups contg a hydroxy group, in the presence of water, a Cu salt and an acid-binding agent. Several examples with details of procedure are given. Cl. C. A. 29, 3514.

**Thioindigo dye preparations.** Alfred Hagenbäcker and Rudolf Brune (to General Aniline Works). U. S. 2,021,711, Feb. 4. In a suitable phase of a process of pyrazolindigo dyes (e. g., during the oxidation of the hydroxy-thionaphthyls in an alk. medium), a salt, oxide or hydroxide of a heavy metal, such as  $\text{FeSO}_4$ ,  $\text{NiSO}_4$ ,  $\text{CuSO}_4$ , or  $\text{Al}_2(\text{SO}_4)_3$ , is added, in less than an equimol. proportion to the dye being formed. Dye preps thus obtained are readily reduced to the leuco compds. which have a

reduced tendency to sep. from the dye baths at low temps. Several examples with details are given.

**Thioindigo dyes.** I. G. Farbenind. A.-G. Ir. 789,463, Nov. 1, 1915. Dyes are prepd. by transforming a  $\beta$ -hydroxythionaphthene deriv. of the C<sub>10</sub> series, contg an I group in the 1- and halogen in the 6-position, and, if desired, a COOH group in the 2 position, either by treatment with oxidizing agents or by condensing the deriv. or the corresponding 3-anils, 2-oximes or 2-ketones with 4-methyl-6-halo-3-hydroxythionaphthene or their derivs., provided these are suitable for the synthesis of mixed dyes of the thioindigo series. Thus, 1,1'-diethyl-6,6'-dichlorothioindigo (by oxidizing 4-ethyl-6-chloro hydroxythionaphthene-2-carboxylic acid) dyes cotton bright rose shades from the vat.

**Dyes of the triphenylmethane series.** Avery A. Morton and Joseph R. Stevens. U. S. 2,029,830, Feb. 4. In the prepn. of dyes such as crystal violet and malachite green, a halide of a tertiary aryl amine which is  $N$ -disubstituted by alkyl or aryl radicals, such as  $p$ -bromodimethylamine, is condensed in the presence of an alkali metal such as Na, with di- $t$ -carbonate,  $t$ -t benzoate or other suitable carbonyl compd, ester, acid chloride, anhydride, quinone or ketone. Among the starting materials which may be used to produce dyes and related compds are the halides of diethylamine, dimethylammonaphthalene, dimethylammonodiphenyl, methylbenzylamine, methylphenylamine and the like. Other compds contg the carbonyl group, all of which are suitable in place of the di- $t$ -carbonate, are di- $t$ -oxalate,  $t$ -t benzoate, di- $t$ -phthalate, phthalic anhydride, phosgene,  $\text{BzCl}$ , chloroethyl formate, quinone, anthraquinone, xanthone and like compds. Xanthone and anthraquinone react with  $p$ -bromodimethylamine to form strongly fluorescent substances.

**Azo dyes.** I. G. Farbenindustrie A.-G. Brit. 436,390, Oct. 10, 1935. Unsulfonated azo dyes suitable for dyeing cellulose esters are made by coupling the diazo compd of an amine of formula  $2\text{X—C}_6\text{H}_4\text{—N=N—C}_6\text{H}_4\text{—Y}$  in which X is H,  $\text{NO}_2$  or halogen and Y is H or halogen, with an amine of formula  $\text{PhNRR}'$  in which the benzene ring may be further substituted and in which R is alkyl or a hydroxyethyl group, R' is a  $\beta$ , $\gamma$ -dihydroxypropyl or a  $\gamma$ -methoxy- $\beta$ -hydroxypropyl group, in which latter case R may be H. Among examples, (1) 1-nitro-1-aniline  $\rightarrow$  3-chloro-1-( $\beta$ -hydroxyethyl)- $\beta$ , $\gamma$ -dihydroxypropylammonobenzene (fiery yellowish red), and (2) 6-bromo-2,4-dinitroaniline  $\rightarrow$  1-methyl-1-( $\beta$ -butyl)-( $\gamma$ -methoxy- $\beta$ -hydroxypropyl)ammonobenzene (reddish blue).

**Azo dyes.** I. G. Farbenind. A.-G. Ir. 788,920, Oct. 10, 1915. Dyes are prepd. by condensing an aminobenzaldehyde or a nuclear substitution product thereof with a compd. contg. an active  $\text{CH}_2$  or  $\text{CH}_3$  group, diazotization and coupling with a coupling component. Diazotization and coupling may precede the condensation. Thus, 1-aminobenzaldehyde  $\rightarrow$  1-(2'-sulfophenyl)-3-methyl-5-pyrazolone is refluxed with 1 mol. proportion of  $\text{NCCl}_3\text{CO}_2\text{H}$ , giving a dye which dyes animal fiber bright orange-yellow shades fast to light.  $\text{NCCl}_3\text{CO}_2\text{H}$  may be replaced by anilide of acetoacetic acid,  $\beta$ -hydroxythionaphthene, 1,3,3-trimethylindole-4-one or  $\alpha$ -methylindole.

**Azo dyes.** I. G. Farbenind. A.-G. Ir. 789,006, Nov. 4, 1915. Dyes are prepd. by coupling diazotized aromatic amines with pyrazolones of the formula  $\text{RCH—CH(R)—CH(R)—CH(R)—N—CO—CH}_3$ , CR' N. R is H or

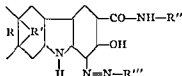
an alkyl group and R' is alkyl, aryl, COOH, COO-alkyl, CONH<sub>2</sub>, CONH-alkyl or CONH-aryl. Thus,  $\text{PhNH}_2 \rightarrow$  1-(3-cloxyethyl-5-methyl-5-pyrazolone, m. 145°, dyes cellulose lacquers in fast yellow shades, sulfanilic acid  $\rightarrow$  1-(4'-methyl-5-cloxyethyl-5-methyl-5-pyrazolone dyes wool in fast yellow shades. Other examples are given.

**Azo dyes.** Soc. pour l'ind chim A Bile. Ger. 620,619, Oct. 21, 1935(Cl. 22a). 1. Fast unsulfonated dyes are prepd. by using coupling or diazo or azo components contg at least one (etherified or esterified) hydroxy-alkylamino group, the coupling being carried out at an



amino or substituted amino group. Thus, diazotized 4-nitroaniline is coupled with the Me ether of *N*-ethyl-*N*-(hydroxyethyl)aniline to give a product coloring acetate silk in light red shades. Many other examples are given.

**Azo dyes** Wilhelm Neelmeier and Heinrich Morschel (to General Aniline Works) U S 2,028,409, Jan 21. Dyes which may be represented by the probable general formula



wherein R stands for a tetramethine chain linked to two vicinal carbon atoms of the benzene nucleus R', R'' stands for a nonsulfonated and noncarboxylated aromatic radical, such as a radical of the benzene, naphthalene and carbazole series, and R''' stands for the radical of a nonsulfonated and noncarboxylated diazotized component suitable for producing azo dyes, in which substituents may occur in the nuclei R' and R''' such as alkyl, alkoxy, halogen and the nitro group, are obtainable by coupling in substance or on a substratum a diazotized nonsulfonated and noncarboxylated amine suitable for producing azo dyes with a 5,6-, 6,7- or 7,8-benzo-2-hydroxycarbazole-3-carboxylic acid arylamide. The shades when produced on the fiber, according to the usual method of producing ice colors, have good fastness. Numerous examples are given, the dyes formed being various shades of brown.

**Azo dyes** Richard Stusser (to General Aniline Works) U S 2,028,450, Jan 21. Dyes of various colors and good fastness are produced in substance or on the fiber by diazotizing 2-nitroaniline or other component free from solubilizing groups and coupling with a pyrazolone such as that of dehydrothiolutidine or the like. Examples and details of procedure are given.

**Azo dyes** Hans Schindhelm and Carl T. Schultz (to General Aniline Works) U S 2,029,591, Feb 4. Various examples are given of the production of dyes of the general formula 4 (RX) 2-HO,SC<sub>6</sub>H<sub>4</sub>CH CHC<sub>6</sub>H<sub>4</sub>(SO<sub>2</sub>H) 2'-(N N R<sub>1</sub> N)-4' wherein X stands for an azo or azoxy group, R means a radical of the benzene or naphthalene series which may be connected with a benzothiazole or arylazo radical or the whole radical attached on the other side to X and R<sub>1</sub> means a radical of the naphthalene series which may contain a sulfonic or carboxylic acid group or may be connected with a further arylazo group, obtained by oxidizing a stilbene compd. of the general formula 4 Y 2-HO,SC<sub>6</sub>H<sub>4</sub>CH CHC<sub>6</sub>H<sub>4</sub>(SO<sub>2</sub>H) 2'-(N N R<sub>1</sub> N)-4', wherein Y stands for a nitro or arylazo or arylazoxy group and R<sub>1</sub> has the aforesaid significance, in an alk. soln., for instance by means of cupric sulfate. In the case of Y standing for a nitro group it is to be converted subsequently into an arylazo or arylazoxy group. These dyes, which may be purified by an aftertreatment usual for stilbene azo dyes, dye vegetable fibers yellow to red to brownish shades of a good fastness particularly to light. Cf. C A 30, 1575<sup>1</sup>.

**Azo dyes** Jose S. Petrus Blumberger (to General Aniline Works) U S 2,029,555, Feb 4. Diazo dyes which in the form of their alkali metal salts are generally dark water-sol. powders, dyeing cellulosic fiber generally bluish shades which by diazotizing and developing with a yellow component, such as methylketol, acetoacetic acid arylamides, pyrazolones and the like, yield green shades of excellent clearness and good fastness, are obtainable by diazotizing a primary amine of the benzene or naphthalene series containing a sulfonic or a carboxylic acid group and coupling with a 1-amino-2-naphthol alkyl ether or a 6- or 7-sulfonic acid thereof, further diazotizing and coupling with a 1-(aminobenzoylamino)-8 naphthol mono- or disulfonic acid which may be substituted in the benzene nucleus of the benzoylamino group by alkyl, alkoxy or halogen, effecting the final coupling with the addition of a

basis of the pyridine or quinoline series, such as picoline, quinaldine and preferably pyridine itself. As initial components there may be used, e. g., aminobenzonic acid, antimesulfonic acid, 1-naphthylamine-4-sulfonic acid, 2-naphthylamine-8-sulfonic acid, *p*-toluidine-*m*-sulfonic acid, chlorotoluidinesulfonic acids, 2-aminidine-4-sulfonic acid (OCN<sub>2</sub>=1), *m*-xylydine-*m*-sulfonic acid and as final coupling components there may be used, e. g., 1-*p*-aminobenzoylamino-8 naphthol-3,6- or -4,6-disulfonic acid, 1-(4'-methoxy-3'-aminobenzoylamino)-8 naphthol-3,6- or -4,6-disulfonic or -4 monosulfonic acid, 1-(4'-methyl-3'-aminobenzoylamino)-3,6-disulfonic acid and 4'-chloro-3'-aminobenzoylamino-4,6-disulfonic acid. Several examples with details of procedure are given.

**Metal compounds of azo dyes** Hans Krzikalla and Walther Kuehne (to General Aniline Works) U S 2,028,981, Jan 28. See Ger. 621,473 (C A 30, 1249<sup>1</sup>).

**Azo dyes** James D. Todd, Laurence F. May and William L. Newbury (to Sherwin-Williams Co.) U S 2,028,958, Jan 28. A dye which is suitable for pigment manuf. is made by coupling diazotized Tobias acid with  $\beta$ -naphthol in the presence of a quantity of phenylmethylpyrazolone up to 25% the quantity of the  $\beta$  naphthol. U S 2,028,959 relates to forming a dye by coupling diazotized Tobias acid with  $\beta$ -naphthol in the presence of a lesser quantity of a diazotized naphthylaminesulfonic acid. Several examples are given.

**Disazo dyes** Heinrich Klingenstein, Hans Roos and Carl Heusner (to General Aniline Works) U S 2,028,479, Jan 21. Several examples are given of the production of diazo dyes of the general formula PyN.NaRCO.NiR(SO<sub>2</sub>H)<sub>2</sub>NI(COAr)NR', wherein Py stands for the radical of a coupling component of the pyrazolone series, such as a 1-aryl-3-methyl-5 pyrazolone which may bear substituents in the aryl nucleus, or a 1-aryl-5-pyrazolone-3-carboxylic acid or a deriv. thereof, such as a carboxylic acid ester, carboxylic acid amide or a carboxylic acid arylamide, which 1-aryl-5-pyrazolone-3-carboxylic acid or deriv. thereof may bear substituents in the aryl nucleus or nuclei respectively, Ar stands for radicals of the benzene series, R stands for a naphthylene radical to which the NH groups have been attached neither in *o*-position nor in *peri* position, *n* stands for the numbers one or two, and R' stands for the radical of a yellow component suitable for preparing azo dyes, such as a pyrazolone coupling component, phenol, phenol ether, salicylic acid, methylketol and acylacetic acid arylamides. These dyes give fast yellow to orange dyeings on cellulosic fiber. Cf. C A 30, 872<sup>1</sup>.

**Metal complex compounds of azo dyes** I. G. Farbenindustrie A.-G. Brit. 430,588, Oct. 7, 1935. See Fr. 787,428 (C A 30, 1237<sup>1</sup>).

**Ethylene azo dyes** Robert Wizinger (to General Aniline Works) U S 2,029,647, Feb. 4. See Brit. 435,449 (C A 30, 1577<sup>1</sup>).

**Indigoid vat dyes** I. G. Farbenindustrie A.-G. Brit. 436,322, Oct. 9, 1935. These are prep'd by condensing 4,7-dichloro-5-methyl-3-hydroxythionaphthene with an isatin  $\alpha$ -deriv., e. g., 5-bromoisatin chloride or 4-methyl-5-chloro-7-methoxysatin  $\alpha$ -chloride in PhCl. Cf. C A 30, 877<sup>1</sup>.

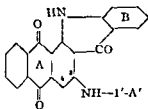
**Vat dyes** I. G. Farbenind. A.-G. Fr. 789,003, Oct. 22, 1935. Dyes of the formula  $\overline{\text{SR}^n\text{N}^m\text{CC}^n\text{N}^m\text{R}^n\text{S}}$  or

$\overline{\text{SR}^n\text{N}^m\text{CR}^n\text{N}^m\text{CZ}^n\text{S}}$  (in which Z is a univalent radical, and R and R' are aryl groups at least one of which belongs to the anthraquinone series) are prep'd by condensing  $\alpha$ -aminoaryl mercaptans with aryl thiazole compds. of the

general formula  $\overline{\text{SR}^n\text{N}^m\text{CC}^n(\text{Y})\text{X}}$ , in which X is H or a halogen, Y is O or 2 atoms of halogen and the group C(Y)X may be in the thiazole or R' ring. Thus, the dye obtained by heating the Na salt of 1-mercapto-2-aminoanthraquinone (I) and 6-chlorobenzothiazole-2-carbonyl chloride (II) in PhNO<sub>2</sub> dyes cotton in fast greenish yellow shades. Dyes are also prep'd from I and 2-methylbenzo-

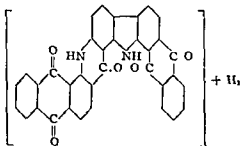
thiazole-6-carbonyl chloride and anthraquinone-2-benzothiazolecarbonyl chloride, 3-bromo-I and II, anthraquinonyl-1,2-thiazole-2'-carbonyl chloride and 1-amino-2-mercapto-4-chlorobenzene,  $\beta$ -aminoanthraquinone and II and 1-chloro-2-aminoanthraquinone and II.

**Vat dyes.** I. G. Farbenind. A-G. Ir 789,551, Oct. 31, 1935. Dyes of the anthraquinone series are prep'd by treating with acid-condensing agents, if necessary in the presence of an org. diluent inert to the condensing agent, anthraquinonyl aminoanthraquinoneacridones of the general formula



in which the anthraquinone radical represented by A has the 4 position free, and that by A' has the 2' position free and B is an aromatic radical. Thus the product obtained by condensing 3 bromoanthraquinoneacridone with  $\alpha$ -aminoanthraquinone is agitated with  $AlCl_3$  in  $PhNO_2$  and heated to 140–50°, the product giving brown shades on cotton. Dyes are also obtained from the condensation products of 3-chloroanthraquinoneacridone (I) with 1-amino-4-benzoylaminoanthraquinone (II), and 3,4',6'-trichloroanthraquinone-2,1(N)-1',2'(N)-benzocridone (cf. Ir. 748,790, C. A. 27, 5549) with II.

**Vat dyes.** I. G. Farbenind. A-G. Fr. 789,561, Oct. 31, 1935. Dyes of the anthraquinone series are prep'd by causing Al or Fe halides to act on anthraquinone-2,1-(N)-benzeneacridones having a 1-aminoanthraquinonyl group in the 6-position of the  $C_{10}H_6$  ring, in the presence of org. solvents nonbasic for the said halides, and, if necessary, causing oxidizing agents to act on the hydrogenated compds., thus formed. The products have the probable formula



the position of the H atoms in excess not being known. The compds. have a low content of  $AlCl_3$ , and give fast brown shades from the vat. The prep'n of the starting materials, anthraquinone-2,1(N)-1',2'(N)-3'-methyl-6'-chlorobenzeneacridone (by causing 4-chloro-2-amino-toluene to react with 1-chloroanthraquinone-2-carboxylic acid (I) and heating with  $BzCl$  for 1 hr. at 150°), anthraquinone-2,1(N)-1',2'(N)-3'-methoxy-6'-chlorobenzeneacridone (by causing 2-methoxy-5-chlorobenzene to react with I and heating with  $BzCl$  at 130–60°), and anthraquinone-2,1(N)-1',2'(N)-3'-phenoxy-6'-chlorobenzeneacridone (by the action of I on 2-amino-4-chloro-aniline) anthraquinone-2-carboxylic acid with agents favoring cyclization). Cf. C. A. 30, 875.

**Vat dyes.** Imperial Chemical Industries Ltd. Ir. 789,570, Oct. 31, 1935. New anthraquinone dyes are prep'd by treating 1,4,5-tris( $\alpha$ -anthraquinonyl amino)-anthraquinone with  $AlCl_3$  as a condensing agent contg.  $AlCl_3$ .

**Vat dyes of the dibenzanthrone series.** Karl Koeberle, Hugo Wolf and Emil Krauch (to General Aniline Works). U. S. 2,029,237, Jan. 28. Nitrodibenzanthrones contg.

about 4–5–6–5% N, violet powders dyeing vegetable fibers from a blue vat green shades which by aftertreatment with hypochlorite become gray to black are obtained by treating dibenzanthrone or its substitution products such as monobromodibenzanthrone, etc., with aq.  $HNO_3$  solns which also may contain  $H_2SO_4$  (suitably by heating to 80–90°). Several examples with details of procedure are given. Cf. C. A. 30, 1069.

**Coloring matters.** Imperial Chemical Industries Ltd. Ir. 789,594, Oct. 30, 1935. These are made by causing an aromatic acid nitrile or amide having a Cl or Br atom in the position ortho to the nitrile or amide group to react with  $CuCN$ . Examples are given of compds. prep'd from *o*-chlorobenzonitrile and *o*-chlorobenzamide which are the same as the Cu compds. of Brit. 322,169 (C. A. 24, 2890).

**Anthrapyridonesulfonic acids.** Klaus Weinand (to General Aniline Works). U. S. 2,029,007, Jan. 28. Products dyeing wool from an acid bath red to violet shades of good fastness are produced by heating a 1,4-diaminoanthraquinone-2-sulphonic acid, the 4-amino group of which has one hydrogen atom replaced by an alkyl, phenyl, chlorophenyl, acetamidophenyl, benzyl, hydronaphthyl or hydrophenyl group, with a compd. having the configuration  $-CO-CH_2-CO-$  selected from the group consisting of malonic acid esters and acetoacetic acid esters of aliphatic alcs. Several examples are given.

**Arylamides of 2,3-hydroxynaphthoic acids.** Wilfred A. Sexton (to Imperial Chemical Industries Ltd.) U. S. 2,029,500, Feb. 4. Compds. suitable for use as ice color coupling components are produced by reacting 2,7-dihydroxynaphthalene-3-carboxylic acid with a primary aromatic amine to produce a 2-hydroxy-7-arylamino-naphthalene-3-carboxylic acid. Aniline, *o*-toluidine, *p*-anisidine and the like may be used as initial materials and several examples with details of procedure are given.

**Color lakes.** Compagnie nationale de matières colorantes et manufactures de produits chimiques du Nord réunies établissements Kuhlmann. Ger. 620,837, Nov. 9, 1935 (Cl. 22/12). Condensation products of the anthraquinone series having the constitution 1-HO-2- $RCH_2$ -4-A- $CaH(CO)_2$ , in which R is a salt-forming group or a radical contg. a salt-forming group and A a OH group or a substituted or unsubstituted amino group, are used for forming lakes. The anthraquinone nucleus may contain other substituents. In an example, sulfonated 2-benzylquinizarin is dissolved in distd. water. A suspension of pptd. washed  $Al_2O_3$  is added to form a bluish red lake. Other examples are given.

**Symmetrical bis(arylamino)phenols.** Leopold Lacka and Oskar Haller (to German Aniline Works). U. S. 2,029,727, Feb. 4. See German pat. 586,806 (C. A. 28, 1546).

**Trifluoromethylphenylazodiaminopyridines.** Max Engelmann (to E. I. du Pont de Nemours & Co.). U. S. 2,029,315, Feb. 4. By diazotizing  $m$ -aminotrifluoromethylbenzene and coupling with an  $\alpha,\alpha'$ -diaminopyridine, products are obtained which are suitable for dyeing cellulose acetate, etc.

**Ketones of the anthracene series.** I. G. Farbenind. A-G. Fr. 789,547, Oct. 23, 1935. Ketones which are intermediates for dyes are prep'd by heating with spongy agents compds. of the formula  $YCOC(R)(R')X$  (Y is a univalent radical of the anthracene series, X is H or an alkali or alk. earth metal, R is a COOH group which may be esterified, R' is H or an acyl radical or a COOH group which may be esterified). Examples are given of the prep'n of 2-acetyl-1- (from the product obtained by condensing anthraquinone-2-carboxylic chloride (I) and acetoacetic ester with  $EtONa$ ), 2-chloro-3-acetyl-1- (m. 154–6°, from the product obtained by condensing 2-chloroanthraquinone-3-carboxylic chloride and malonic ester (II) with  $EtONa$ ), 1,4-dichloro-2-acetyl-1- (from the product obtained by condensing 1,4-dichloro-I with II), 1-nitro-2-acetyl-1- (m. 255–7°, from the product obtained from 1-nitro-I and II), and 1,4-dichloro-6-acetyl-anthraquinone, m. 235° (from the product obtained from 1,4-

dichloroanthraquinone 6-carboxylic acid and II), Bz-1-acetylbenzanthrone, m 174-5°, 2-acetylthiazoleanthrone (from the product obtained from thiazoleanthrone 2-carboxylic chloride and II) and 2-acetylthiazoleanthrone.

**Organic tellurium compounds** Melvin A Perkins (to F I du Pont de Nemours & Co.) U S 2,030,035, Feb 4. Dye intermediates are prep'd by causing a Bz-1-halo-benzanthrone to react with an alkali metal telluride, the halogen in the Bz-1-position being replaced by Te. Various examples are given and among the products obtained is Bz-1, Bz-1-dibenzanthronyl ditelluride, which when fused with alc.-KOH yields isodibenzanthrone.

**Aminodinitrophenylamine** I G Farbenind A-G (Leopold Lanka and Rudolf Heil, inventors) Ger 629,907, Oct 30, 1935 (Cl 12g 1 02). The comp'd 4-amino-3,4'-dinitrophenylamine is obtained by condensing 4-nitro-1-chlorobenzene-2-sulfonic acid with nitro-*p*-phenylenediamine and splitting the HSO<sub>3</sub> group from the resulting 4-amino-3,4'-dinitrophenylamine-2-sulfonic acid by usual methods. The condensation is effected by heating mol amts of the constituents to 130-5° in a pressure vessel in the presence of water and CaCO<sub>3</sub>. The substance m 226-7° and is used for making dyes.

**Condensation products** Durand & Huguenin S A Fr 789,583, Oct 31, 1935. One mol of a naphthol-, naphthylamine- or aminonaphthol sulfonic acid is condensed with at least 2 mols of an ammosalicylic acid in the presence of bisulfite. Thus, 2 mols of *p*-aminosalicylic acid are condensed with 1 mol of 2-naphthol-7-sulfonic acid or 1 naphthylamine-6-sulfonic acid. The products are intermediates for dyes.

**Dyeing** I G Farbenindustrie A G Brit 436,371, Oct 3, 1935. H<sub>2</sub>O insol azo dyes are made on the fiber by applying diazoamino compds and coupling components and developing the dye at a neutral to weakly alk reaction under the influence of an elevated temp. in the presence of moisture, the coupling components, if applied as salts, being transformed by treatment with an alkali binding agent, at least partially, into the free OH form prior to the coupling. The process may be applied simultaneously with the development of vat or aniline black dyes. Among examples, (1) cotton is impregnated with 2,3-hydroxy naphthol aniline in dioxane and pyridine soln, padded with a soln of the diazoamino comp'd from diazotized 4-chloro 2-toluidine and methylaminoethanesulfonic acid and the Na salt of the *o*-amide of 2,3-hydroxynaphthoic acid, padded on the reverse side with aq (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> with or without NaCl and steamed, and (2) cotton is printed with a paste contg the diazoamino comp'd from diazotized 2,5-dichloroaniline and 4-sulfo 2-aminobenzoic acid and diacetoacetyl-*o*-toluidine, padded with aniline black liquor contg NH<sub>4</sub>NO<sub>3</sub>, steamed, chromed and finished. Cf C A 30, 878.

**Dyeing, printing** H Th Bolme A-G Brit 436,410, Oct 10, 1935. In coloring fibrous materials with oxidation dyes, substances that give rise to cations that reduce the surface tension are added to the dyes or printing pastes contg the aromatic bases or their salts to be oxidized. Among examples, (1) unboiled, non-prewetted cotton piece goods are dyed a deep level black by impregnation at room temp with an aq liquor contg PhNH<sub>2</sub>·HCl, PhNH<sub>2</sub>, Na<sub>2</sub>SO<sub>4</sub>, K<sub>2</sub>Fe(CN)<sub>6</sub>, NaClO<sub>2</sub> and decylpyridinium sulfate, dried at 80-90°, steamed 1 min at 108°, treated 5 min in an aq soln of Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> at 40°, rinsed, washed and dried, the goods may be printed with a reserve before the impregnation or steaming, and (2) an unbleached cotton fabric is dyed a level brown by sloppadding in an aq liquor at room temp contg *p*-phenylenediamine, Na sulfoxylate, NaClO<sub>2</sub>, NH<sub>4</sub>Cl, tartar emetic, hexadecylpyridinium chloride and NH<sub>4</sub> vanadate, drying at 80-90°, steaming 1 min at 108° and finishing as usual, a printed reserve may be used.

**Continuous dyeing of undressed fabrics** P P Sazanov Russ 8,111, Aug 31, 1934. The dyeing is effected in a vat by introducing the fabric predried and warmed to the temp of the dyeing vat soln.

**Dyeing textiles with sulfonic ester salts of leuco vat dyes** Alex Wormald (to Imperial Chemical Industries

Ltd) U. S. 2,029,351, Feb. 4. Compds such as dibenzylsulfanilic acid or its Na, K or NH<sub>4</sub> salts (suitably in soln with a polyhydric alc. such as diethylene glycol) are used to facilitate printing, dyeing, padding or stenciling with coloring matters such as Indigosol brown IRRD, Indigosol pink IR extra, Indigosol brilliant pink 13B and the like. Several printing paste formulas are given.

**Printing pile fabrics** Duncan Ferguson and Herbert H Schell (to Sidney Blumenthal & Co.) U S 2,029,768, Feb. 4. A design is applied to the flat side of the fabric by printing (suitably with an engraved Cr-plated Cu roller) and the printing compn is caused to show through on the pile face of the backing fabric without affecting the projecting portions of the pile, the flat side is coated with a binder such as a rubber soln and the material is dried.

**Method of treating spools of textile material with a dyeing or washing liquor** Joseph Annick Brit 436,511, Oct 11, 1935.

**Dyeing animal fibers** Soc pour l'ind chim à Bâle Brit 436,790, Oct 14, 1935. Addn to 433,230 (C A 30, 624). The fibers are dyed with complex metal compds of mordant-dyeing azo dyes contg sulfo groups with the addn to the dye bath of a small proportion, not exceeding 0.5% of the wt of the goods to be dyed, of a sol salt of an org nitrogenous H<sub>2</sub>O insol base or of an org quaternary NH<sub>4</sub> comp'd, which base or comp'd contains at least 1 aliphatic or cycloaliphatic residue comprising at least 8 C atoms but contains no polyethenovy group and is other than a quaternary NH<sub>4</sub> comp'd obtainable by treating with an alkylating or aralkylating agent a *N*-alkyl or *N*-aralkyl benzimidazole substituted at the  $\alpha$  C atom by an aliphatic residue contg at least 8 C atoms. Among ex. amples, wool, 100, is entered into an aq soln at 60-70° contg H<sub>2</sub>SO<sub>4</sub>, the Cr compds of the dye 4-chloro-2-aminophenol-6-sulfonic acid  $\rightarrow$  2,4-dihydroxyquinoline, and the condensation product, *o*-1-*o*-2 part, from stearyl chloride and unsym *N*-diethylethylenediamine, the bath is heated to boiling during 30 min and so maintained 60-75 min and the goods are rinsed and dried. Cf C A 29, 940.

**Dyeing cellulosic material** Imperial Chemical Industries Ltd, Leslie P. Rendell and Harry A Thomas Brit 436,592, Oct 14, 1935. Natural or regenerated cellulosic textile material is dyed with a chrome dye after treatment with an aq soln of a quaternary NH<sub>4</sub> quaternary phosphonium or ternary sulfonium salt of which the mol contains a sat'd, nonhydroxylated aliphatic chain of at least 10 C atoms and contains no acylamino group. The pretreated material may be further treated with a reduced bichromate prior to the dyeing or the dyed material may be aftertreated with a sol bichromate. Among examples, viscose stockinette is immersed 30 min in a 3% soln at 90° of cetyltrimethylammonium bromide, rinsed and placed in a warm dye bath contg Solochrome brown RH, AcOH and Glauber's salt and the temp is raised gradually to 90-95° with an addn of HCOOH after 1 hr and of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> after a further 15 min.

**Dyeing organic derivatives of cellulose such as cellulose acetate** George H Ellis and Frank Brown (to Celanese Corp of America) U S 2,029,312, Feb. 4. The material is treated with an unsulfonated anthraquinone comp'd having as a substituent in the anthraquinone nucleus a phenylamino radical having an alkyl group as a meta substituent, e. g., with 1-amino-4-m-tolylaminoanthraquinone. Numerous examples are given. U S 2,029,313 relates to the similar use of an unsulfonated anthraquinone comp'd having as a substituent in the anthraquinone nucleus an ether substituted arylamino group of the benzene series, such as 1-amino-4-(*o*-methoxyphenyl)aminoanthraquinone. Numerous other examples are given.

**Dyeing acetate silk** I G. Farbenind A-G Ir 789,364, Oct 28, 1935. Acetate silks of various degrees of acetylation or other acylated cellulosics are printed or dyed by 1,4-diaminoanthraquinones slightly sol in water and which contain alkyl radicals in the amino groups, the alkyl radical in the 1-amino group being different from the

alkyl radical in the 4-amino group, or 1 amino group contains 1 or 2 alkyl groups and the other a hydroxyalkyl group or an alkyl group having more than 1 OH group. Examples are 1-methylamino-4- $\beta$ -hydroxyethylamino- (m 190°), by reaction of 1-methylamino-4-aminoanthraquinone and ethylene oxide, 1-methylamino-4-bis( $\beta$ -hydroxyethylamino)-, 1-methylamino-4-butylamino-, 1-hydroxyethylamino-4-hydroxypropylamino-, 1-methylamino-4-phenylethylamino- and 1-methylamino-4-cyclohexylamino-anthraquinone.

Printing acetate artificial silk Wolfgang Jaack and Arthur Schurch (to Soc pour l'ind. chim. à Bâle) U S 2,029,508, Feb. 4. See Fr 779,884 (C A 29, 5674).

Dyeing cellulose esters and ethers Imperial Chemical Industries Ltd. Fr 789,041, Oct. 22, 1935. The esters or ethers are treated with an aq soln of a quaternary ammonium, phosphonium or sulfonium salt, the mol of this salt contg an aliphatic chain, satd or not, of at least 10 C atoms, and afterward dyed with a direct acid or chrome dye. The chrome dyeing is carried out by the chromate or metachrome process of dyeing and subsequently treating with Cr Salts include octadecylpyridinium bromide, ester of oleic acid and *N*- $\beta$ -hydroxyethylpyridinium chloride, cetyl ester of *N*-hydroxymethylpyridinium chloride, etc.

Dyeing wool Imperial Chemical Industries Ltd. Fr 788,991, Oct. 21, 1935. In dyeing wool with acid or chrome dyes, the dye liquor is kept at 60–90° and is maintained in a state of turbulence by introducing bubbles of air, steam or other inert gases which impinge on the material.

Fabrics containing natural silk Leo Wallerstein, Rowland A. Gale and Thomas G. Hawley, Jr. (to Wallerstein Co. Inc.) U S 2,029,968, Feb. 4. In making a fabric contg. natural silk fibers provided with their body-giving gum coatings, some of which coatings in the fabric are in relatively inaccessible condition, while others are in accessible condition to the action of a subsequent decatizing bath, the fibers the coatings of which are relatively inaccessible are treated with a proteolytic enzyme prep. without removing the coating, the impregnated fibers are dried, then combined with unimpregnated fibers, the coatings of which are relatively accessible, to form a fabric. U S 2,029,969 relates to highly twisted silk yarn for weaving and knitting, comprising natural silk in girth, the yarn being rendered more flexible and given lessened tendency toward kinkiness and unevenness by impregnation with a proteolytic enzyme prep. without removal of the gum and being dried while contg. such prep. U S 2,029,970 relates to a generally similar treatment to prepare a yarn for knitting, weaving or other textile operations. U S 2,029,971 relates to fabric formed in part of a similarly treated natural silk and in part of cellulose acetate, capable of being degummed at a temp. below 55° and at relatively low alkalinities such as avoid injury to the cellulose acetate. U S 2,029,972 relates to knitted fabric capable of being simultaneously dyed and degummed in acid, neutral and alk. media, comprising yarn formed of natural silk treated as described with a proteolytic enzyme prep.

Preparing silk for degumming Leo Wallerstein, Thomas Hawley and Rowland A. Gale (to Wallerstein Co.). U S 2,029,350, Feb. 4. Degumming of raw silk is facilitated by a preliminary treatment with a protease prep. such as a papain or pancreatin prep. and drying of the impregnated material.

Apparatus for respinning rayon. Rudolf Etzkorn (to North American Rayon Corp.). U S 2,029,226, Jan. 28. Structural details.

Apparatus for treating thread with fluids such as in applying conditioning oil to artificial silk Henry R. Childs (to Eastman Kodak Co.). U S 2,029,910, Feb. 4. The thread is guided into contact with a portion of a wire mesh screen the interstices of which are filled with a thread-treating fluid.

Treating textiles British Celanese Ltd. Brit 430,703, Oct. 16, 1935. Textile materials, films or foil, of org.

1 derivs of cellulose are treated with a compn comprising a H<sub>2</sub>O-insol. lubricant and a phenol, e. g., olive oil and cresylic acid. Such material may be readily delustered by treatment with a hot aq. delustering medium. Cf. C A 30, 620.

Treating fabrics British Celanese Ltd., Albert Mellor, Ralph J. Mann and Ernest L. Greenwood. Brit 436,463, Oct. 11, 1935. An antierose treatment comprises treating the fabric in open width while substantially tensionless with an aq. liquor, e. g., H<sub>2</sub>O, dil. soap, above 90° for less than 10 min. The treatment may also be used to develop crepe effects in fabrics contg. crepe threads.

Improving textile fibers and cellulosic materials R. Botson. Belg. 407,146, Feb. 28, 1935. Tanning materials are incorporated with the fibrous material and are fixed by means of CH<sub>3</sub>CO, (CH<sub>3</sub>)<sub>2</sub>CO, gelatin or the like, to render the fibers resistant to water and to decay, unshrinkable and undeformable. The process is applicable also to paper.

Retting fibrous plants Cie. Belge des fertilisants. Belg. 408,286, April 30, 1935. The plants are steeped in H<sub>2</sub>O contg. K<sub>2</sub>CO<sub>3</sub>, heated to 75° and cooled to 37°. The vol. is completed with liquid manure or sewer water and air is blown into the tank for several 12 hr. periods. The liquid from this treatment can be used as fertilizer.

Animalization of vegetable or artificial fibers I. G. Farbenindustrie A.-G. Fr 789,331, Oct. 26, 1935. The incorporation of primary, secondary and tertiary amines and quaternary ammonium bases of the general formula (R<sub>1</sub>R<sub>2</sub>R<sub>3</sub>N)OH (at least 1 of the radical R<sub>1</sub>, R<sub>2</sub>, or R<sub>3</sub> contain a chain of 8 or more C atoms, while the others may be replaced by H, aryl or aliphatic chains with less than 8 C atoms) or their salts in vegetable or artificial fibers gives the fibers a strong affinity for wool dyes. Examples of compds. are stearylamine acetate, isopropyldecylamine lactate, *N*-decyl-1- $\beta$ -propylenediamine formate, dimethylstearylamine, methylidibutyldecylammonium bromide and stearylpyridinium chloride.

Bleaching textiles, etc. Adolf Danzinger. Brit 436,268, Oct. 8, 1935. See Austrian 143,295 (C A 30, 8814).

Bleaching cotton goods James A. Clark and Harry G. Smolens (to Buffalo Electro-Chemical Co.) U S 2,029,985, Feb. 4. The goods are moistened with an aq. H<sub>2</sub>O<sub>2</sub> soln and then subjected to a temp. above the b. p. of the soln in an atm. contg. steam for a sufficient time to produce the desired bleaching effect. App. is described.

Fireproof textile material William Whitehead (to Camille Dreyfus) Can 355,242, Jan. 7, 1936. Yarns and filaments of org. derivs of cellulose are treated with an alkylamine salt of an inorg. acid, such as H<sub>2</sub>BO<sub>3</sub>, H<sub>3</sub>PO<sub>4</sub>, HI and HBr. The coating should be 20–50% on the wt. of the yarn.

Increasing the heat resistance of materials such as cellulose acetate fabrics George H. Ellis and Ralph C. Storey (to Celanese Corp. of America). U S 2,028,769, Jan. 28. There is incorporated in the material a small proportion of an insol. metal compd. such as Sn phosphate so that the material on incineration yields a voluminous ash of 0.5–2.0% and so that it has a safe ironing point of at least 300°.

Mercerizing baths Deutsche Hydrierwerke A.-G. Fr 789,582, Oct. 31, 1935. The wetting power of the baths is increased by adding to the lye mixts. of phenols and aliphatic and aromatic alcs, e. g., a mixt. contg. crude cresol 93, benzyl alc. 4 and normal primary octyl alc. 3%.

Mercerizing I. G. Farbenindustrie A.-G. Brit. 436,660, Oct. 16, 1935. See Fr 771,856 (C A 29, 1664).

Preserving textile materials Industriegewerke E. 9 Isakiewicz. Fr. 789,314, Oct. 26, 1935. Clothing, etc., are preserved in an atm. of N.

Dry-cleaning apparatus. The American Laundry Machinery Co. Brit. 436,118, Oct. 4, 1935.

Dry-cleaning apparatus of the drum type. I. G. Farbenindustrie A.-G. Brit. 436,061, Oct. 3, 1935.

Dry-cleaning apparatus with a vacuum or suction operated filter for the used solvent John R. Courtney and Harry Ward. Brit. 436,585, Oct. 14, 1935.

## 26—PAINTS, VARNISHES AND NATURAL RESINS

A. H. SABIN AND CARLTON H. ROSE

Some advances in the field of paints and their future possibilities. Johannes Scheiber. *Beihft Z. Ver dent Chem* No 19, *Angew Chem* 49, 21-22 (1936). K. K.

An objective for the American paint industry. Richard L. Cawood. *Official Digest Fed. Paint Varnish Production Clubs* No 151, 432-6 (1935).—Lecture, stressing the advantages of the pebble mill. Modernizing for profit. *Ibid* 437-9. G. G. Sward.

Trends in the paint and varnish industry. Wayne R. Fuller and M. S. Armstrong. *Chem & Met Eng* 43, 4-9 (1936). E. H.

Flashlights on new European practices. Wilhelm Krumbhaar. *Official Digest Fed. Paint Varnish Production Clubs* No 150, 400-14 (1935). cf. C. A. 29, 632.—A lecture on current practices of the European paint and varnish industry. G. G. Sward.

Bituminous paints for submerged objects. E. Kind-scher. *Farbe u. Lack* 1935, 485-6, 497.—A discussion of a no. of practical points, e. g., testing and selection of suitable bitumens, the advantages of priming with red lead and the superior results obtained with bitumen paints contg. chlorinated rubber. J. W. Perry.

The status of the theory of colors. Ernst Mayer. *Farben Ztg* 41, 31-2 (1936).—The possibility of applying the Ostwald color classification system to the standardization of paints and pigments is discussed briefly. J. W. Perry.

Effect of mill scale on the using of paint. K. G. Lewis and U. R. Evans. *Verfahrenstechnik* 8, 154-5 (1935). cf. C. A. 29, 4716. J. W. Perry.

The evaluation of anticorrosive paints by the swelling test. Hans Wagner and Edgar Pabst. *Farben Ztg* 40, 789-90, 811-13 (1935).—The swelling behavior of linseed-oil paints prep'd with various kinds of chromate pigments and applied to sheet iron was studied. The effect of the pigment on the swelling, which is observed after 100 days' immersion in water, is made clearer by calcg. the swelling factor, i. e., the ratio of the observed swelling to the swelling which the oil in the paint would have experienced if present alone. In all cases superior anticorrosive action was ascribed with firmer adhesion. The swelling factor is an aid in predicting the anticorrosive power of pigments, provided they exert no sp. electrochem. anticorrosive action. The swelling ratio of paint films contg. neutral or basic Pb chromates free from sol. or soap-forming components varied from 0.2 to 0.4. This was reduced to 0.15 and the adhesion and anticorrosive power were improved by the presence of soap-forming components in the pigment. Sol. chromates may improve adhesion and the anticorrosive action even though the swelling factor is thereby increased. H. Wolff and G. Zentler. *Ibid* 1199.—The swelling factor is not entirely det'd by the type of pigment used in linseed-oil paint. The pigment-oil ratio is also an important factor. Changes in the mech. properties, e. g., adhesion, of the paint film brought about by swelling are more important than the magnitude of the latter. J. W. Perry.

Corrosion symposium, 1935. The selection and application of bituminous materials for paints. Walther. *Farben Ztg* 40, 1196-7 (1935).—The properties of various bitumens are briefly discussed with emphasis on products prep'd from Mexican petroleum and anthracite coal. Lab. expts. show that moist O. together with ultraviolet light causes the rapid deterioration of films of blown bitumen. J. W. Perry.

Cold cracking and peeling failures attributable to four causes. E. A. Fluevog. *Paint, Oil & Chem Rev* 97, No 26, 7 (1935).—In order to avoid cold cracking and peeling the finish must (1) have good initial adhesion to the substratum, (2) avoid loss of adhesion due to either reaction with the substratum or penetration of moisture and (3) remain sufficiently flexible so that its coeff. of expansion at low temps. is at least equal to that of the

substratum over which it is applied. An accelerated cold-cracking test is described. J. W. Perry.

Tint retention. C. D. Holley. *Paint, Oil & Chem Rev* 97, No 25, 9-10 (1935).—Exposure tests extending over 2 yrs. and involving over 300 panels are summarized. The desirability of retaining substantial percentages of ZnO (preferably introduced as a high leaded Zn pigment) was again demonstrated. Hydrated Fe oxide pigments and Fe<sub>2</sub>O<sub>3</sub> were superior to chrome yellows and orange chrome, resp., as tinting pigments. Tint retention was not markedly affected by wide variations in type, kind and combinations of drier metal used. Formulation changes involving vehicle compn. indicated that replacing part of the linseed oil with synthetic materials, notably of the alkyl type, improved color retention without loss of durability. Also the substitution of new pigments such as PbTiO<sub>3</sub> gave improved color retention. J. W. Perry.

Developments of titanium pigments. L. Firing. *Official Digest Fed. Paint Varnish Production Clubs* No 151, 440-2 (1935).—A historical lecture. G. G. S.

Zinc yellow and mixed pigments made therefrom. Martin Seidel. *Farbe u. Lack* 1935, 603-4, 615-16.—The properties of Zn yellow and Zn green are discussed and methods of manuf. described. J. W. Perry.

Lead titanate. D. W. Robertson. *Ind Eng Chem* 28, 216-18 (1936).—This new pigment, made from PbO and TiO<sub>2</sub>, is essentially PbTiO<sub>3</sub>. Properties: pale yellow color, sp. gr. 7.3, n<sub>D</sub> 2.7, particle size 0.3-0.2  $\mu$ , ultraviolet absorption almost 100%, chemically inert toward paint vehicles. Paint films contg. it retained a dt. tenacity of 55% for 2 years, chalked very slightly, inhibited rusting, and protected incorporated tinting pigments from fading. Study of PbTiO<sub>3</sub> paints suggests that, contrary to the accepted ideas, chem. inertness and high ultraviolet absorption are necessary for good paint properties, but that variation in particle size and ability to form soaps with the vehicle are not. Arnold M. Collins.

The thickening of red lead ground [in oil] and the production of nonlivering red lead. H. Grohn. *Farben Ztg* 40, 1063-5 (1935).—As shown by G. (cf. C. A. 24, 3396) and others (cf. C. A. 23, 3358, 26, 5436-7) the thickening of red lead-linseed oil paints is due to the pigment's contg. PbO. Oxidation of the latter is most advantageously accomplished by grinding the pigment to an av. particle size of 5  $\mu$  and then heating to 360-380° for 8-10 hrs. This increases the Pb<sub>2</sub>O<sub>3</sub> content of the pigment to 97-108.5% and practically eliminates any tendency toward livering. J. W. Perry.

Lithopone. H. Mills. *Oil Colour Trades J.* 89, 217, 218, 217-18 (1936).—Characteristics of lithopone and other ZnS pigments are discussed. The hiding and reducing powers of several white pigments are tabulated. Lithopone has valuable properties for an inside paint, while for outside use it is well to blend with other pigments. Lithopone or ZnS pigments are remarkably adaptable to formulation. W. H. Boynton.

Color and constitution. L. Dittmar. *Farbe u. Lack* 1935, 555, 568-9, cf. C. A. 30, 629.—The relation between chem. structure and color of synthetic org. pigments is discussed. J. W. Perry.

Systematizing and evaluating emulsified paint vehicles. Hans Wagner and E. Pabst. *Farben Ztg* 40, 1245-6, 1267-8 (1935).—By following, under the microscope, the process of film formation it is possible to classify the film according to type and make general predictions about its properties, e. g., stability. Such a test should be supplemented by reversibility tests by immersion in water and permeability tests, e. g., to CdSO<sub>4</sub> soln. The authors believe that important savings in drying oils can be achieved by application of the emulsion technic. The use of Mittel 100 J with emulsions contg. tung oil is recommended. J. W. Perry.

Reactions in monolayers of drying oils. I. The oxidation of the maleic anhydride compound of  $\beta$ -eleostearic

G. Gee and Eric K. Rideal. *Proc. Roy. Soc. (London)* A153, 116-28(1935).—The film spread on 0.01 N  $\text{H}_2\text{SO}_4$  increased in area at const. pressure owing to oxidation. Fairly consistent force-area and phase-boundary potential curves could be obtained by rapid examn. The limiting area A of the unsaturated monolayer was observed to be 385 Å<sup>2</sup> (calcd 380 Å<sup>2</sup>), the elec moment,  $\mu \times 10^{18}$ , 25.4 e. s. u. (calcd 24.8). Four stages of compression were noted to each of which structural configurations were assigned. At the end A was equal to 120 Å<sup>2</sup> and  $\mu \times 10^{18}$  to 8.2. This state corresponded to submergence of the glyceride mol. and emergence of the outer double bond, both of which were originally in the same horizontal plane. The oxidation velocity varied considerably with pressure. The total increase in area was greater at the higher pressures. Conclusion. The film is oxidized to an unstable primary peroxide, which in turn undergoes polymerization or isomerization. The energy of activation of oxidation was 6500 cal/mol and of isomerization 19,000 cal/mol. II. Polymerization of the oxidized forms of the maleic anhydride compound of  $\beta$ -eleostearin. G. Gee. *Ibid* 120-41, *Trans. Faraday Soc.* 32, 187-95 (1936), cf. C. A. 29, 7162<sup>2</sup>.—Analysis showed that the polymerization is a chain reaction which can be broken by inhibitors like hydroquinone in the substrate or like Ft myristate in the film itself. By compressing a film that had been oxidized at high pressure, polymerization of the stable isomer was studied.  $k$  varied directly with the 3rd power of the pressure as in Semenov's equation for gaseous chain reactions, and the energy of activation was 20,000 cal/mol. Polymerization of the unstable peroxide was studied at temps at which reaction of the isomer was negligible. Pressure and temp had a marked effect on this polymerization, and the stable isomer acted as inhibitor. The energy of activation was 5000 cal/mol.

F. S. Roller. The synthesis of drying oils. A. Ya. Drnberg. *Sovetskii Rekonstruktivnyi i Nauka* 1935, No. 6, 16-25, cf. C. A. 29, 6443<sup>3</sup>.—The esters of unsatd. linseed oil acids and monohydric alcs. cannot form a film; the esters of diat. alcs. form with difficulty a rather unstable film, the films of synthetic glycerol esters are different from the films of the natural linseed oil. The esters of alcs. of high mol wt. form very hard films; possibly these esters can be used to improve vegetable oils. Attempts to obtain unsatd. from satd. acids were practically unsuccessful. The Al salts of hydroxy carboxylic acids are better than the Ca, Zn, Pb and Ba salts and give films stable for 2 yrs. indoors but unfit for outside work. The drying oil from a mixt. of Ca and Al salts of the natural naphthenic acids is somewhat worse. Introduction of asbestos into salt-contg. drying oils lengthens the term of their service and promises to make them fit for outside work. Treatment of chlorinated mineral oil polymers with salts of synthetic hydroxy carboxylic acids to produce a drying oil meets with difficulties due to the great impurity of technical polymers. Unsatd. hydrocarbons obtained by Weitz from the wastes of synthetic rubber, in the presence of naphthenic acids, proved very resilient. By oxidizing the same unsatd. hydrocarbons with oil Ruben obtained inner esters.

B. V. Shvartzberg. Drying properties of mixed oils. Kh. Dorn and R. Frastova. *Masloboino Zhirovo Delo* 11, 487-8(1935).—In the preliminary study of the function of linoleic acid (I) in the drying of oils and the properties of paint films, tests were made with various mixts. of linseed oil (II) with (1) lallelantia and perilla oils contg. more I than in II, (2) makhorka and nut oils with low contents of I, and (3) corn oil with no I. The drying rate of lallelantia and perilla oils is retarded on the addn. of II. With the addn. of makhorka and nut oils up to 70% the drying rate of the mixt. gradually decreases, and then beginning with 70% the curve rises sharply above that for II. The corn-oil mixt. also fails to show an additive function of the II contents, viz., the curve is characterized by the absence of a max and min. The results thus show that the drying power of oil is not directly dependent on the relative proportions of I in an oil. This observation is supported

by the exptl results of Long (C. A. 27, 5557) showing that in the mixts. of triglycerides with increasing percentage of triolein the O nos increase.

Chas. Blanc. A note on the composition of Egyptian linseed oil. M. A. Hummat. *J. Oil & Colour Chem. Assoc.* 18, 312-14 (1935).—An Egyptian linseed oil having the following consts., acid no 5.6, I no (Wijs) 190-2, d<sub>4</sub>, 0.933, n<sub>D</sub><sup>20</sup> 1.481, was analyzed by the methods of Kaufmann (C. A. 23, 2031) and Twitchell (C. A. 15, 5017) and found to contain unaponifiable matter + glycerol 5.30, satd. acids 6.34, oleic acid 7.57, linoleic acid 38.44 and linolenic acid 42.35%. Of the total unsatd. acids, 18.3% yielded insol hexabromide by Coccinaras' method (C. A. 26, 3127). Egyptian and Calcutta linseed oils are closely similar.

J. W. Perry. The oxidation of linseed oil in solution. A. F. H. Ward and G. D. France. *J. Soc. Chem. Ind.* 54, 435-8T (1935).—The oxidation at 70° by O of linseed oil in dil. soln in  $\text{CCl}_4$  was studied. The unsatn was deid by a modification of the Wijs method (C. A. 23, 1518). The I value decreased with time, an induction period being followed by a more rapid reaction. A brown polymerization product, not completely satd., was pptd. This substance could reduce the induction period catalytically. Darkness or an antioxidant increased it. The satn of double linkings had to continue a long way before polymerization proceeded sufficiently for the product to become insol in  $\text{CCl}_4$ . During the time of rapid fall in I value of about 60-70 hrs the oxidation was approx. unimol.

P. S. Roller. The behavior of linolenic acid, linseed oil and wood oil on heating. Karl Meinel. *Naturwissenschaften* 23, 721 (1935).—When linolenic acid (I) or linseed oil is heated in N<sub>2</sub>, the isolated double bonds of I change over into conjugated bonds as shown by decrease in Br no. The Br no. of wood oil increases on heating, the eleostearic acid conjugated bonds move apart. Cf. Kappelmeier, C. A. 27, 4425.

B. J. C. van der Hoeven. The molecular weight of stand oil. (Evaluation of viscosity measurements.) Hans Wolff. *Fettechem. Umschau* 42, 169-71(1935).—Application of the Staudinger equation,  $\eta_{sp}/c = k \cdot M$  (C. A. 28, 6120<sup>4</sup>), to dil. solns. of linseed-oil stand-oil or the acids therefrom in  $\text{CCl}_4$  and in toluene and the interpretation of the viscosity relations of more concd. solns. of the same solutes in linseed oil by means of the Arrhenius equation,  $\log \eta = m \times \log \eta_1 + (1 - m) \times \log \eta_2$  ( $\eta$  is viscosity of mixt.,  $\eta_1$  and  $\eta_2$  are the viscosities of the components, and  $m$  is the quantity of the component with  $\eta_1$  viscosity), showed that the degree of polymerization was the same for both the stand oils and their free acids. This indicates that, for every two fatty acid radicals united during heat treatment by extramol. polymerization, four radicals unite to form dimers by intramol. polymerization. A tendency toward the development of structure viscosity made it necessary to work at low concns. (3 g./100 cc.) when toluene and  $\text{CCl}_4$  were used as solvents. This tendency to aggregate formation was more noticeable with the stand oil gth cides than with their free acids.

J. W. Perry. The trend of polishing varnishes during the past one hundred years. W. M. Jauch. *Official Digest Fed. Paint Varnish Production Clubs* No 150, 398-400(1935).—The uses of natural resins in short oil rubbing varnishes during the past 100 years are reviewed. G. G. Sward.

The weathering of unpigmented varnishes. E. Fonrobert and F. Wilborn. *Farben-Ztg.* 41, 10-12, 32-3(1936).—The selection of synthetic resin for use in oil varnishes by means of outdoor weathering tests is a problem complicated by a no. of factors, e. g., seasonal and other variations in the weather, the proportion of resin in the varnish and the prepn. of the surface to which it is applied. The weathering stand used by the authors was so situated that, depending on the direction of the wind, the samples under test were exposed to an acidic atm. or to the alk. dust of a cement plant. This caused a no. of peculiar effects which are described in detail.

J. W. Perry. The relative values of some weathering tests. A. M. Mees. *Verfkronek* 8, 205-8(1935); cf. C. A. 29,

5287<sup>1</sup>—Eight different unpigmented lacquers and varnishes were tested in several different labs by a variety of methods including accelerated artificial weathering, Florida tide range test, Florida land test, roof exposure and variations of the same. The wide discrepancies between some of the results were probably due to the unusually destructive action of excessive amts of moisture on some of the products under test. Discussion C A Lobry de Bruyn and A M Mees. *Ibid* 209-9.

New work on natural resins H J Loycke. *Forstarchiv* 11, 310-56 (1935).—Improvements in methods of harvesting and purification of resins are reviewed. The yields of oil of turpentine and colophony distd from crude balsamic resin have been increased to about 95%. Small quantities of irritants are present in the harvested balsam which usually are not harmful but which may disturb the esterification of colophony. A trace of mineral acid favors the conversion of resin acid to abietic acid which may be avoided by a 15-min heating to 210°. I. C Feustel.

Congo copal resins. I. Separation of normal nonacosane from the resin. Ferdinando Trost. *Ann chim applicata* 25, 470-2 (1935).— $C_{29}H_{58}$  has been isolated from copal resin by fractional distn, by steam distn and by sepn from paraffin by sapon with alc KOH.

Resins XIX. A copal from the Fiji Islands. Erich Stock. *Farben-Ztg* 40, 1112 (1935), cf C A 28, 3252<sup>1</sup>, 5690<sup>1</sup>.—The copal had the following const: acid no 103.5, sapon no 113.6, sinter point 110°, m. p 135°, ash 0.135%. It leaves a glossy film from alc or turpentine soln and appears to belong to the Mandia copal group. It has, however, a typical microscopical structure, which is described in detail. J W Perry.

Solid-liquid interface (Clayton) Z. Analysis of dry red lead (Anon.) 7. Taste and odor tests of paints for water tanks (Hall) 14. Basic Pb carbonate (Belg pat 407,862) 18. Infusible and insol resins and varnishes (Russ pat. 37,845) 13. Rubber [products used for making lacquers and paints] (Ger pat 620,751) 30. Polymerized oils (Fr pat 789,644) 27. Treating natural gums [for manuf of varnishes] (Fr pat 789,611) 28. Varnishes (Brit. pat 435,270) 13.

Gardner, Henry A. Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors. 7th ed. Washington, D C. Inst of Paint and Varnish Research. 1448 pp. 311. Reviewed in *Ind Eng Chem., News Ed* 13, 483 (1935).

Paints. Beckacite Kunstharzfabrik G. m. b H. Fr 789,645, Nov 4, 1935. Metallic siccatives used for paints and varnishes are obtained by pptg the hydroxides of the metals generally used, e. g., Ca, Zn, Mn, Co, Pb, from aq solns of their resp metal salts, e. g., their sulfates, chlorides, acetates at ordinary or reduced temp. The water used is previously freed from dissolved air by boiling. The paste is mixed with a dispersing agent and caused to react with an org. acid or glyceride at low temp.

Mills for grinding paints, printing inks, etc. Boudewyn C. J. Selier. Fr 788,920, Oct 19, 1935. Cf C A 29, 6445<sup>1</sup>.

Enamel paint. George C Finn (to Sherwin Williams Co.) Can 354,311, Nov. 19, 1935. The pigment material and resin components are suspended in suitable solvent and plasticizing materials and ground and mixed by falling bodies in a rotating mill. The mix is then combined with volatile solvents.

Pigments. A Esser and B Fisel. Belg 408,318, April 30, 1935. Colored mud is emulsified with oil, dried *in vacuo*, heated and ground.

Pigments. Henry A Gardner. Fr 789,594, Oct 31,

1935. Metal salts of phthalic acid when they are relatively insol in water and ordinary org. solvents are incorporated in oil paints, varnishes or cellulose lacquers. The prepn and use of phthalates of Pb, Cu and Ti are given. A pigment may comprise particles of  $TiO_2$  treated on the surface with a relatively small amt of Ti phthalate.

Pigments. Frich Korinth and Georg Meder (to I G Farbenind A-G.) U S 2,028,980, Jan 28. Practically pure ZnO is combined with an oxide of Cd, Mg, Fe or Mn to form mixed crystals or a solid soln by heating the materials together at temps of about 800-1100°.

Red pigments. Ekbert Lederle. U S 2,030,009, Feb 4. Mixed crystals comprising normal Pb chromate, normal Pb sulfate and normal Pb molybdate are pptd by adding to an aq soln contg a sol Pb salt such as the chloride or nitrate a strongly acid soln, contg sulfate, chromate and enough molybdate ions to produce mixed crystals contg at least 4% of Pb molybdate. The Pb salt soln used may also contain a Ba or Sr sol salt. Cf C A 29, 6445<sup>1</sup>.

Calcium sulfate-zinc sulfide pigment. Harold F. Saunders (to Sherwin-Williams Co.). Can 354,310, Nov. 19, 1935. To obtain a pigment contg approx 50% ZnS, 1000 lb of gypsum assaying 80%  $CaSO_4$  is suspended in  $H_2O$ . An aqueous suspension (2400 lb) contg 33 1/3% by wt. of ZnS is added. The mixt is agitated, filtered, dried and calcined at above 600° in an O free atm, quenched, dried and disintegrated.

Zinc-dust paints. Lester D Grady, Jr. (to The New Jersey Zinc Co.). Brit 436,164, Oct 7, 1935. The evolution of H during the storage of Zn-dust paints is limited by the addn of  $CaO$ ,  $BaO$  or activated bauxite in small amt, e. g., 0.25-1%.

Bunder for paints, etc. N. V. tot voortzetting der zaken van Pieter Schoen & Zoon. Fr 789,597, Oct. 31, 1935. A mixt contg one or more crude or polymerized drying oils, a phthalic resin and a chlorinated rubber is used.

Removing dried coatings such as those of paint and varnish from surfaces. Carleton Ellis (to Chadeloud Chemical Co.) U S 2,029,992, Feb 4. A liquid finish remover is applied to the surface and allowed to soften the coating, a pulverulent solid absorbent such as sawdust or kieselguhr is then applied and mixed with the material on the surface substantially to dry up the slush formed, and the mixt. is removed from the surface. Cf C A 29, 5058<sup>1</sup>.

Transfer ink. Frank Solomon (to Beatrice Brier) U S 2,029,204, Jan 28. A transfer ink solid at normal atm temps and flowable into a thin film by low heat from a hot iron is formed of shellac 5,  $TiO_2$  1, tritolyl phosphate 0.5, rosin 5 and varnish 0.5 part, together with coloring matter.

Lacquer for leather. R Collet and J. Coulon. Belg 406,447, Jan 31, 1935. Shellac is dissolved in 90% alc by heating on the water bath and aniline is added to produce the desired shade.

Varnish. Joseph B Dietz and Edmund F. Oeffinger (to E I du Pont de Nemours & Co.). U S 2,028,758, Jan 28. See Can 346,340-1 (C A 29, 2376<sup>1</sup>).

Esters of 2-butyltolanol. Carbide & Carbon Chemicals Corp. Fr 789,407, Oct 29, 1935. See U S 2,014,310 (C A 29, 7343<sup>1</sup>).

Apparatus for fusing and treating organic materials used in making varnishes, etc. Beckacite Kunstharzfabrik G m b H. Fr 789,642, Nov. 4, 1935.

Coating compositions. James A Arvin (to E I du Pont de Nemours & Co.). U S 2,029,851, Feb 4. A compn suitable for coating various surfaces comprises a varnish solvent such as "mineral spirits" and the pentaerythritol ester of an unsatd aliphatic monocarboxylic acid having at least 16 C atoms and an I no. of at least 120, such as the pentaerythritol esters of the acids of linseed, China-wood or soybean oils, with pigments, driers, etc.

## 27—FATS, FATTY OILS, WAXES AND SOAPS

**R. RICHARD L.**

The problems of chemistry in the new Germany XIII  
Auto-oxidation and ketonic decomposition of fats as  
problems in the fat industry. K. Taufel *Angew Chem*  
49, 44-45 (1936). Cf. C 1 30, 775a. The following subjects  
are discussed: (1) synthesis of fats, (2) refining of  
fats, (3) research for fat substitutes, (4) steps to reduce  
fat losses by spoilage, (a) the chem. spoiling of fats and  
(b) the biol. spoiling of fats. Conclusions: Fats can be  
stabilized by keeping bacteria away in the mfg. process,  
by retardation of bacterial growth by cooling means, and by  
the application of bacteria killing substances. Thirty-four  
references. Karl Kammermeyer

The highly unsaturated acids in sardine oil. IX. The constitution of docosahexenoic acid,  $C_{22}H_{40}O_2$ , Yoshikuni S. Toyama and Tomotaro Tsuchiya, *Bull. Chem. Soc. Japan* 10, 1329-43, cf. C. A. 30, 3167. The Am ester of a concd. fraction of docosahexenoic acid still contg. some Am clupanonolinate on ozonolysis yielded  $AcH$ ,  $AcOH$ ,  $CO_2$ ,  $(HOOC(CH_2)_4)_2$ ,  $HOOC(CH_2)_4COOAm$ . These last 3 are derived from the groups  $\cdot CH(CH_2)_4CH$  and  $\cdot CH(CH_2)_4COOAm$ , resp.  $CO_2$ ,  $AcH$  and  $AcOH$  are formed by the secondary decompn. of the products of ozonolysis derived from the group  $\cdot CHCH_2CH$  and the yield of  $CO_2$  indicates at least 3 of these groups in the mol. Hence docosahexenoic acid is composed of one of the following sets of groups  $CH_2$ ,  $\cdot CHCH_2CH$  (3),  $\cdot CH(CH_2)_4CH$  (2) and  $\cdot CH(CH_2)_4COOH$ , or  $\cdot CHCH_2CH$ ,  $\cdot CHCH_2CH$  (4),  $\cdot CH(CH_2)_4CH$  and  $\cdot CH(CH_2)_4COOH$ , and its constitution is probably either  $CH_2 \cdot CHCH_2CH \cdot CHCH_2CH \cdot CHCH_2CH \cdot CH(CH_2)_4CH \cdot CH(CH_2)_4CH \cdot CH(CH_2)_4CH \cdot COOH$  or  $\cdot CHCH_2CH \cdot CHCH_2CH \cdot CHCH_2CH \cdot CHCH_2CH \cdot CHCH_2CH \cdot CH(CH_2)_4CH \cdot CH(CH_2)_4CH \cdot COOH$ . The first of these is the more likely as the yield of  $C_4$  compounds isolated indicates more than one  $\cdot CH(CH_2)_4CH$  group. X. The separation of highly unsaturated  $C_{22}$  acids. *Ibid.*, 1517-7. The residue from the distn. of the highly unsatd. Me esters previously obtained (C. A. 30, 3167) yielded mainly highly unsatd.  $C_{22}$  acids and some polymerized  $C_{22}$ . 1 fractional pptn. of the Na soaps in acetone resulted in the sepn. of isonic acid,  $C_{22}H_{40}O_2$ , from the fraction of the highest degree of unsatn. Indications of the presence of scolondonic acid,  $C_{22}H_{40}O_2$ , and also an alcl.  $C_{22}H_{40}O_2$ , were obtained from the other fractions. XI. The constitution of nisinic acid,  $C_{22}H_{40}O_2$ , in sardine oil. *Ibid.*, 1547-51. Ozonolysis of Am nisinatc yielded  $AcCHO$ ,  $PhCOOH$ ,  $AcH$ ,  $AcOH$ ,  $CO_2$ ,  $HOOC(CH_2)_4COOH$ , and  $HOOC(CH_2)_4COOAm$ . The first 2 are derived from the group  $MeCH_2CH$ ,  $CH$ ;  $AcH$ ,  $AcOH$  and  $CO_2$  from secondary decompn. of malonic acid from the group  $\cdot CHCH_2CH$ ; and the last 2 from the groups  $\cdot CH(CH_2)_4CH$  and  $\cdot CH(CH_2)_4COOAm$ , resp. Yields of ozonolysis products indicate 3  $\cdot CHCH_2CH$  groups and 2  $\cdot CH(CH_2)_4CH$  groups. Relative positions of these are not indicated by these expts, but if double bonds are assumed at 4, 8 and 12 as in moronic acid, eicosatetraenoic acid, and clupanonic acid the constitution of nisinic acid is  $MeCH_2CH \cdot CHCH_2CH \cdot CHCH_2CH \cdot CHCH_2CH \cdot CHCH_2CH \cdot CH(CH_2)_4CH \cdot CH(CH_2)_4CH \cdot COOH$ . Oden E. Sheppard

The preparation of brassic acid starting with rapeseed oil. L. Kefler and A. M. Malcen *Bull. Soc. chim. Belg.* 44, 467-72 (1935).—The oil (100-200 g.) was emulsified with 5%  $\text{HNO}_3$ , treated with solid  $\text{NaNO}_2$  (2% of oil) and allowed to stand 24 hrs. The product was saponified with 10%  $\text{NaOH}$  solution and the saponified oil acids had an i value of 71.35 (theory, 76) and contained about 1% impurity. 1 fractional distn. of the Me esters at 0.5 mm. gave a product of the same purity. F. W. Scott

11. Tetradecenoic and dodecenoic acids in sperm-blubber oil  
12. Tetradecenoic and dodecenoic acids in sperm-blubber oil  
13. Tetradecenoic acid in sperm head oil, Yoshiyuki Toyama  
and Tomotaro Tsuchiya, *Bull. Chem. Soc. Japan* 10,  
163-9, 570-3 (1935).—See *C. A.* 30, 3154<sup>g</sup>, G. G.

Yoshiyuki Toyama and Taniotaro Tsuchiya *Bull.*  
*Chem Soc Japan* 10, 573 (1935) Sec. C, A, 30,  
1191 G. G.

Hexadecenol in sperm blubber oil Yoshiyuki Toyama  
and Goroku Akiyama *Bull. Chem. Soc. Japan* 10,  
659-81 (1935). See C. A. 30, 316.<sup>3</sup> G. G.

The oil, fat and soap industries in 1935 R. Furness  
Ind. Chemist 12, 21 A (1936) E. H.

**A survey of modern fatty oil processes** T Andrews  
*Oil Colour Trades J* 80, 367-71 (1956) — A brief summary  
of the processes. Sluiming, filtering, bleaching and neutral-  
ization methods and decolorizing are discussed and  
changes in plant design indicated. W H Boynton

Fatty oil from the seeds of *Xanthium strumarium* L. (Yun. A. Brinke and F. F. Gutt, *Bull. Lar. Lasi Branch Acad. Sci. U. S. S. R. No. 13*, 17, 29 (in German) 30) (1935). The air-dried seeds, with the hull removed, contg. 6.7% H<sub>2</sub>O, gave about 10% of a semisolid oil, contg. palmitic 1.5-2.0, stearic 7.0-7.5, oleic 28.7,  $\alpha$ -moleic acid 30.1 and  $\beta$ -moleic acid 31.4%, d<sub>4</sub><sup>20</sup> 0.9107, n<sub>D</sub><sup>20</sup> 1.4757, and value 0.6 sapon value 189.70, esterification value 181.13, I value 137.01, At value 3.77, Reichert Meissl value 1.01, Polenske value 0.18, Helmer value 0.52 and unsaponifiable matter 0.85. Even in the unacidulated state, the yield of this plant is 220 kg of oil per hectare, almost as much as for sunflowers.

A method for the study of the change of vegetable oils during oxidation (and polymerization) V. M. Hretnian, N. A. Rodnolud Komussariz, I. Lazhel' I. Prom S. S. K., Leningrad, *Plastmasses* 1, 391-27(1945) Math equations are derived connecting various properties of the oils with time and temp. H. M. Ickester

**Chemical studies of cottonseed and its products.** W. D. Gallup. *Okla Agr Expt Sta, Rept* 1932-4, 177 pp. (1931). — Cottonseed having a high oil content also had a high content of gossypol. For seeds of low oil content the oil:gossypol ratio was 55:1, and for oil rich seeds the ratio was only 35:1. The percent of gossypol in the crude oil reduces the alkali refining loss of the oil. The nutritive values of cottonseed meal are discussed. C. R. F.

What is the most economical process for extracting oil from decorticated cotton seed? J. de Raadt, *Matsiro grasses* 27, [1061] 3(1955)—from a comparison of the relative costs of (1) discontinuous hydraulic pressing; (2) continuous mech. pressing and (3) preliminary continuous pressing followed by solvent extrn., it is concluded that (3) is the most economical, particularly under the conditions prevailing in the Argentine Chaco.

**The fatty oil of Chanoschanos (Forsskal)** Kinze  
Kafuku and Chuta Hata. *J. Soc. Chem. Ind., Japan* 33  
Suppl. (1934) 19(15).—*Chanoschanos* (Forsskal) is a  
fish indigenous to tropical waters of the western Pacific.  
The body, liver and intestines contained fatty oils amount-  
ing to 1.8, 30 and 1.5%, resp., which showed a great  
similarity to herring oil in their properties. The following  
properties are reported:  $d_4^{20}$ , 0.93; acid no., sapon. no.,  
I no. and unsaponifiable matter. The chief constituents  
proven were, myristic, palmitic, stearic, zoömaric,  
tetradecenoic, oleic, linolic and linolenic acids, and a  
highly unsat'd acid  $C_{21}H_{39}O_2$ . The unsaponifiable  
portion, amounting to 1.1%, consisted chiefly of cholesterol  
and squalol. The color reaction for vitamin A was  
vivid with liver oil, but not with the other oils.

Characteristics of halibut liver oils. R. T. M. Haine and J. C. Drummond, *Analyst* 61, 27 (1936). Norman Piers, A. C. Jones and Wilfred Smith *Ibid* 71 (1937); cf. C. A. 29, 1060.—The increasing use of this oil in medicine makes it important that the analyst should know the characteristics of a pure sample of oil to detect adulteration. In the first of these 2 independent papers, the values obtained in the analysis of 18 samples of West



Greenland oils, 9 samples of Labrador oils and 3 samples of Iceland oils are given. In the second paper, the values obtained from 41 samples of Iceland, Faroes and West Greenland oils and of 5 samples of Norwegian oils are tabulated. The "blue values" of West Greenland oils, indicating the vitamin A content, varied from 623 to 12,930. The lowest blue value in any sample of pure oil was 405. The sp. gr. of all the oils was 0.928 or a little less. The I values ranged from 114.0 to 161.0. Apparently there is some relation between a high I value and a high content of vitamin A but it is not quite clear what this is. The refractive indices varied from 1.47 to 1.49, the unsaponifiable matter from 6.34 to 17.6%. W. T. H.

Viscosity and plasticity of disperse systems. VIII. Viscosity of hydrogenated fats (Volarovich, Ravich) 2. Treating fat-contg. waters (Belg. pat. 407,621) 14. Detergents (Brit. pat. 436,866) 13. Surface tension of soaps of K and Li salts of higher aliphatic acids (Lottermoser, Giese) 2.

Vidal y Martí, Juan. Fabricación de jabones y lejas en pequeña y grande escala. Revised ed. Madrid. Espasa Calpe. 268 pp. Ptas 5.

British Standard Specifications. No. 627—1935 Sampling of Fats and Fatty Oils in Packages or in Bulk. No. 628—1935 Coconut Oil. No. 629—1935 Ground Nut Oil. No. 630—1935 Olive Oil. No. 631—1935 Rape Seed Oil. No. 632—1935 Raw Linseed Oil. London. Brit. Standards Inst. 2s. 2d. each. Reviewed in *Chem. Age* 33, 454 (1935).

Extracting lipoids, etc. I. G. Farbenindustrie A.-G. Brit. 435,798, Sept. 27, 1935. Lipoids and other substances having the character of fats are sepd. from animal or vegetable tissues or the crude fluids therefrom by treatment with an alc. having 6-10 C atoms, or a mixt. thereof. The process is also applicable for the elimination of the extractive substances, as when therapeutically active soaps of albumin or carbohydrates are being purified. In examples, (1) juice expressed from liver and of  $\text{pH}$  6.8-7.2 is mixed with octyl alc., N being passed through the mixt. for 3 hrs. while it is cooled with ice, the mixt. of lipoids and alc., which seps. in a solid or semi liquid condition, is caught on a filter and the solvent removed by evapn. under reduced pressure, and (2) blood serum is agitated at room temp. with octyl alc. and the mixt. of lipoids and alc. that seps. is removed by centrifuging or filtering, a serum nearly free from lipoids being obtained.

Consistent greases. Produits chimiques Becoline (Gustave Beckmann, inventor). Fr. 789,633, Nov. 4, 1935. Greases are obtained by mixing deglycerolated oils or fats with sulfonated oils or fats, incorporating other animal or vegetable or mineral oil in the cold and saponifying with caustic lye. Thus, a fatty acid of high mol. wt., a sulfonated animal fat and deglycerolated coals oil are intimately mixed at 70°, then mixed with a mineral oil in the cold and saponified by NaOH.

Apparatus for refining oils and fats. Felipe Inda Jimenez. Fr. 788,840, Oct. 18, 1935.

Oils. N. V. Maschinenreien-en Apparaten Fabriek. Ger. 620,703, Oct. 25, 1935 (Cl. 23a 3). Animal, vegetable and mineral oils and fats are rendered odorless, after the usual refining, by allowing them to trickle in a fine stream over a series of inclined surfaces in a high vacuum

at 100-150°. The gases or aromatic vapors evolved are sucked off above the inclined surfaces.

Extracting fatty oils. Francis M. Hamilton. Brit. 436,482, Oct. 11, 1935. Oil is extd. from fish livers or carcasses by disintegrating the material with revolving or rotating knives without adding oil, steam or solvents and then expressing the oil from the resulting mass in a filter press. App. is described.

Nickel catalyst suitable for use in hydrogenating oils. William W. Myddleton (to Robinson-Bindley Processes Ltd.). U. S. 2,029,789, Feb. 4. Ni oxide is mixed with ethyl orthosilicate, coagulation is effected by the addition of  $\text{NH}_3$ , the material is broken up into granules and the dried granules are heated in H or other suitable reducing gas.

Polymerized oils. Beckacite Kunstharzfabrik G. m. b. H. Fr. 789,644, Nov. 4, 1935. Body is given to a drying, semi- or non-drying oil capable of being polymerized, by electrically dispersing a heavy metal such as Co, Mn or Fe in an org. liquid miscible with the oil, e. g., BuOH in turpentine oil, and heating the oil with the colloidal dispersion until a viscous oil is produced.

Refining oils such as carrot oil or palm oil. Vernon Jersey (to S. M. A. Corp.). U. S. 2,029,722, Feb. 4. Oils contg. appreciable amts. of carotene and a substantial amt., but not over about 10%, of free fatty acids, are treated with an aq. soln. of an alk. material such as NaOH or  $\text{Na}_2\text{CO}_3$  in the presence of air at substantially normal atm. pressure, in an amount approx. just sufficient to combine with the free fatty acids present, with slow stirring to avoid inclusion of air, and after neutralization is effected the mixt. is warmed to about 50-60°, allowed to stratify, and the refined product is sepd.

Purifying fish oil. Weaver L. Marston (to Sharples Specialty Co.). Can. 354,307, Nov. 19, 1935. The impure oil obtained by expression from fish is heated directly by steam and part of the steam is condensed. Nearly all the remainder of the steam is evapd. The oil is then continuously sepd. from impurities by centrifuging. Apparatus for separating albumin and mucilage from oil. A. L. Tepeev. Russ. 34,267, Jan. 31, 1934. Albumins and mucilage are sepd. from oil by passing it through a heating coil in the upper part of a vessel and a cooling coil in the lower part of the same vessel.

Apparatus for steaming oil seeds. S. S. Il'in. Russ. 34,672, Feb. 28, 1934. Addn. to Russ. 33,243 (C. A. 28, 3009). Construction details.

Sunflower seed fat of definite melting point and consistency. D. A. Nechaev, A. D. Lebedev and I. A. Oberhard. Russ. 37,787, July 31, 1931. Hydrogenated sunflower-seed oil is melted at 60-75° and then crystallized. The nonsolid part is pressed out at 75 atm., the solid part melted again at 60-75°, recrystd. and pressed. The fraction obtained by pressing at 300 atm. and 37° is finally left at 15° until solidified.

Synthetic wax for the manufacture of wood, metal or leather polishes. S. Kamarovsky. Belg. 410,633, Aug. 31, 1935. Fatty oils are cooked or saponified with  $\text{Ca}(\text{OH})_2$  and mixed with candleilla wax.

Thermostatic wax-melting pot. Harry E. Dow (to United Shoe Machinery Corp.). U. S. 2,023,533, Jan. 21. Structural details.

Stabilizing soap products against discoloration or rancidity. Robert M. Reed (to Procter & Gamble Co.). U. S. 2,029,506, Feb. 4. About 0.01-1.0% of *p*-tert-amyphenol is added to soaps as a stabilizer.

## 28—SUGAR, STARCH AND GUMS

J. K. DALE

Efficiency of sucrose extraction in cane grinding mills. M. Ch. Varona. *Proc. Asoc. Tec. Azucareros Cuba* 8, 141-4 (1934). O. W. Wilcox.

The caramelization of sucrose. A. Joszt and S. Mohácsi. *Biochem. Z.* 282, 269-70 (1935).—On heating sucrose *in vacuo* there is no gas formation but about 10% is lost

in the form of a liquid distillate which has about 20% solid residue. The latter consists almost entirely of furfural compds., but probably no furfural. S. M.

Oliver filter performance in Hawaii. S. A. Wickey. *Facts About Sugar* 31, 53-4, 59 (1933).—Sugar losses in filter cake have been reduced 60% on the av. by replacing plate

and frame presses with Oliver filters; the purity drop between clarified and filtered juice has also been very much smaller. There has been a considerable reduction in cost of labor, filtering medium and fuel. Less floor space and less supervision are required, while the filter station is easier to keep clean. The av. mud eliminated by the Olivers was 0.585 ton per 100 sq ft per hr. If the filters must be operated at high speed, the clarity of the juice suffers. As little as possible of the filtrate should be recirculated, especially when thin muds are being handled.

F. W. Zerban

**High-speed centrifugals** Ralph B. Johnson *Facts About Sugar* 31, 55-9 (1936).—The results of factory tests with centrifugals running at 1600-1700, instead of 1100-1200 r. p. m., are shown in tables and graphs, and discussed. The high speed machines purge more sucrose to the same purity in the same time, and handle massecuites at considerably greater viscosity. Best results are obtained if the temp. of the massecuite and of the sugar is kept at a max.

F. W. Zerban

**Hydrolysis of oat hulls with hydrochloric acid** Loren C. Bryner, L. M. Christensen and Ellis I. Fulmer *Ind. Eng. Chem.* 28, 206-8 (1936).—In the HCl hydrolysis of oat hulls a practically constant yield (about 40%) of xyllose is obtained at each pressure (varying from atm. to 7 kg./sq. cm.) with an appropriate concn. of acid (varying inversely with the pressure, 1.49 to 0.42 N HCl). Under these optimum conditions there is little charring. About 2.2% of volatile products and about 5.5% of lignins are lost during the hydrolysis.

O. W. Wilcox

**Net density of starch** Bunsuke Takei *Mem. Coll. Sci., Kyoto Imp. Univ.* A18, 169-72 (1935), cf. Yoshida and Takei, *C. A.* 26, 3916.—Purified starch reaches a constant concn. and after 5 hrs. pumping with an oil pump at 70-90°. The net ds. of rice and potato starches after this treatment are 1.646 and 1.650, resp., considerably higher than previous values.

C. D. West

**Starch nitrate** E. Berl and W. C. Kutzke *Ann.* 520, 270-89 (1935).—Starch (I) is nitrated with  $\text{HNO}_3\text{-H}_2\text{PO}_4$  mixts. giving a starch nitrate (II) with a max. N content of 13.0% as against 14.14% for trimitate. The viscosity of II shows a flat max. after 12 hrs. of nitration. Decreasing the  $\text{H}_2\text{O}$  content of the mixed acids decreases the stability of II without increasing its percentage of N. With 6% or more  $\text{H}_2\text{O}$  in the acid, the product does not delaminate below 180°.  $\text{H}_2\text{O}$ , MeOH and particularly AcOH act as stabilizers. The potato starch used shows by x-rays about 9 interference rings. This no. is reduced to 3 by nitration, and raised again but not quite up to the original no., when II is converted into sol I by sapon. Under the microscope, II shows a transparent shell, surrounding an opaque center. By freezing the moistened II, suspending in water and fractionating by settling, 2 products are obtained contg. 11.6 and 12.1% N, resp. Treated with  $\text{Me}_2\text{CO}$ , the outer shell swells, while the inner material gives a clear soln. This proves the presence of 2 different materials, amylose nitrate and amylopectin nitrate. As with cellulose nitrate, the use of  $\text{H}_2\text{PO}_4$  gives a product which is less broken down, and thus shows a higher viscosity (about 10 fold) than when  $\text{H}_2\text{SO}_4$  is used. The viscosity is also a function of the origin of I, of the strength of acid used in nitration and of the time and temp. of nitration. Sep. nitration of amylose and amylo-

pectin show higher N content and much lower viscosity in the case of the latter. Preheating of I in autoclave up to 180° progressively decreases the viscosity of II.

G. Calingaert

**Colloid chemistry of rice starch** (Sakurada, et al.) 2. **Sugar-cane physiology** (Beauchamp, et al.) 15. **Curly-top-resistant beet variety** (Skuderna, et al.) 11D. **Registration of density** (Spengler, et al.) 1. **Milk-of-lime scale** (Raven) 1. **Dilatometer method for following the hydrolysis of sucrose** (Hitchcock, Dougan) 2. **Heat savings through insulation of flanges** (Forschungs Heim fur Wärmeschutz) 1. **Sugar feeding stuff expts** (Koch) 12. **Heat transmission in [sugar] evaporators** (Claassen) 1. **Heart rot disease of sugar beets** (Brandenburg) 15. **Cu deficiency in sugar beets** (Van Schreven) 15. **Starch preps** (Brit. pat. 436,028) 12.

**Ghandi, M. P.** *Indian Sugar Industry, 1935*. Calcutta: Indian Sugar Mills Assoc. 79 pp. 7s. 6d. Reviewed in *Facts About Sugar* 31, 30 (1936).

**Sandera, K.** *Surovárenské Cukroviny*. Prague: Zentralverein der Czechoslovakischen Zuckerindustrie. 136 pp. Kc. 35.

**Adressbuch für die Zuckerindustrie Europas.** 65th annual vol. Berlin: Zuckerliteratur. 504 pp.

**Annuaire sucrier, 1935-36** 62nd ed. Paris: J. fab. sucre. 712 pp. F. 30. Reviewed in *Bull. assoc. chim.* 52, 889 (1935).

**Apparatus for straining sugar-cane juice.** Andrew C. I. Coulter. U. S. 2,029,655, Feb. 4. Structural details.

**Apparatus for obtaining sugar crystals from crude sap.** Mtg. Mühlenbau und Industrie A.-G. Ger. 620,882, Oct. 29, 1935 (Cl. 89d. 6). The crude material is run onto an endless perforated band where it crystallizes, drains and moves to a collecting chamber.

**Apparatus for determining sugar in condensation waters** I. I. Shepko and I. P. Tolochko. Russ. 38,060, July 31, 1931. Construction details.

**Preserving sugar juice.** V. I. Kundzhulyan and N. E. Loginov. Russ. 38,058, July 31, 1934. The juice is treated with water glass, satd. with  $\text{CO}_2$  or  $\text{SO}_2$ , to a pH of 10-11, and the solidified mass is dried.

**Starch** Max Rusch and Michael Siebert. Ger. 620,884, Oct. 29, 1935 (Cl. 89k. 2). Pure starch is obtained by swelling, grinding and pasting grain, especially rye. During the paste-forming operation, the mass is strongly stirred and the starch sep'd from the suspension, e.g., by centrifuge. The starch is purified by treatment with org. solvent nonmiscible with water, such as  $\text{C}_2\text{H}_5\text{Cl}$  or  $\text{CHCl}_3$ .

**Rye starch** Hugo Kühl and Gustav Soltan. Ger. 620,883, Oct. 29, 1935 (Cl. 89k. 2). Rye meal is made into a paste with a 0.4% water-glass soln. and worked up by centrifuge.

**Treating natural gums** Becklerte Kunstharzfabrik G. m. b. H. Fr. 789,641, Nov. 4, 1935. Natural gums, particularly copal gums, are made more suitable for the manuf. of varnishes, etc., by kneading or beating the heated gum to render it more sol. and more reactive, dissolving it and neutralizing its acidity once it is in soln. The kneading or beating may be carried out in the presence of a solvent, the neutralization being then effected first.

## 29—LEATHER AND GLUE

ALLEN ROGERS

**Characteristics of vegetable tanning materials.** VII. The amount, velocity and intensity of combination of vegetable tanning materials with animal hide substance. Fritz Stather and Reinhold Lauffmann. *Collegium* 1935, 420-33, 470-1; cf. C. A. 29, 2774.—Strips of fresh beef hide, from which the outer layers had been split, were tanned with 10 com. vegetable tanning exts. The velocity of tanning was det'd by removing part of the hide from the tanning ext. after 1, 2, 4, 8, 16, 32 and 64 days, washing it

10 times with water and analyzing it. The amt. of tanning was measured by the max. degree of tannage (parts combined tannin per 100 parts hide substance). The tanning curve followed the equation  $B = K \log t$  (erroneously given as  $B = K \sqrt{t}$  in the first paper), in which B is the amt. of combined tannin, K a const. characteristic of the tannin and t is time. Values for K and amt. of tanning, resp., were: chestnut 36.3, 46.2, quebracho 32.5, 53.3, sulfited quebracho 31.7, 52.2, myrobolans

31.6, 51.3, mimosa 30.2, 51.9, sumac 27.2, 40.9, valonia 26.0, 45.0, oak bark 22.5, 41.8, gambier 22.4, 42.1, pine bark 10.8, 30.5. The intensity of tanning was detd by washing the leather for 28 days, rotating constantly, with one change of water per day. Afterward the residue was washed 16 days with 70% alc but the alcohol soln bore no relation to the water soln. and was of no value in detg intensity. The first 8 aq. exts. were considered to give the "free H<sub>2</sub>O sol. matter," and the sum of extns. 9-13 the "fixed H<sub>2</sub>O-sol. matter," and matter going into soln in extns 14-28 was considered to result from hydrolysis of the leather. "Free H<sub>2</sub>O-sol. matter" was fairly const at 13-14%, but was a little higher for gambier, myrobolans and valonia. "Fixed H<sub>2</sub>O sol. matter" varied from 1.5 to 2.6. The percentage decrease in degree of tanning from the 13th to the 28th washing was taken as a measure of intensity. The values ranged from 19 for oak and sumac to 25 for pine and gambier.

## I D C

Bating and dyeing XXIX Theory of mineral tanning I Elöd, Th Schachowsky and M Weber-Schafer *Collegium* 1935, 400-20, cf *C A* 29, 83921.—On untanning Cr leather a small part of the Cr compd. (about 0.3% Cr) is found to be more "intensely" combined than the major portion, this is probably the amt of Cr necessary to render gelatin films stable to boiling. During tanning a change in chem. compn., state of aggregation, etc., of the Cr compds is probable, there is no proof that this is not true. Expts in which Cr solns and proteins were allowed to react under such conditions that changes in the Cr compds would be minimized showed that these conditions were not realized if the  $pH$  of the pickled protein was equal to or lower than the  $pH$  of the Cr soln. The optimum tanning action of Cr(NO<sub>3</sub>)<sub>3</sub> and CrCl<sub>3</sub> was at  $pH$  4.5-5.0, of Cr sulfate (28% basic) at 3.5-4.0, therefore, at the optimum, the sulfate has a more intense tanning action than the chloride or nitrate. In general, the condition of the Cr compd and not the isoelectric point of the protein is the dominant factor in tanning. As the basicity of CrCl<sub>3</sub> solns was increased (by dialysis) their tanning action decreased, probably because when the particle size increases in the absence of protein, active centers are closed.

## I D C

A further study of the fat liquoring process for chrome-tanned leather. Shoo Tze Leo and Tao-Yu Cheng *J Chem Eng (China)* 2, 106-11 (1935), cf *C A* 29, 42041.—Fat liquors with sulfonated oils have less penetration power than other types of fat liquors, while fat liquors with egg yolk have greater penetration power than others. In the extn by solvent before fat liquoring, the bound fats and acetone ext. will be low if the petroleum ext. is high, and vice versa.

## C L Tseng

The prevention of acid damage to vegetable tanned leather. Gerhard Otto *Collegium* 1935, 449-55.—Sets of leathers, tanned with a quebracho-chestnut-valonia mixt with or without addn of sulfite cellulose, Tanigan F C or Tanigan O was electrometrically titrated before and after aging 2 years. During aging one set was exposed to air and another had air excluded. Leathers congt Tanigan F C or O retained the original buffering capacity of the vegetable tanning material for 2 years, while pure vegetable leather had entirely lost its buffering power. The hide substance of the latter was noticeably attacked, as was shown by sol. N data, while the Tanigan leather was not attacked. Treatment of hide with tanning sulfonic acids of high mol wt decreases or prevents absorption of acid gases. Sulfite cellulose has a similar but lesser protective action. The quinhydrone and glass electrodes gave the same  $pH$  values up to  $pH$  5.7, so the former can be used for detg free acid and buffer capacity of leather by titration.

## I D C

The liming process in the manufacture of leather. Shoo-Tze Leo and Cheng-Tan Chang *J Chem Eng (China)* 2, 89-101 (1935).—The liming effects of calcsols with CaO alone and with CaO + As<sub>2</sub>S<sub>3</sub> or Na<sub>2</sub>S are studied. As<sub>2</sub>S<sub>3</sub> usually produces a tighter grain than Na<sub>2</sub>S, while pure CaO produces a loose grain. The greater the loss of hide substances during this process, the higher are the ash

and lime contents and the looser the grain of the finished leather. A leather of good quality should contain 63-72% of hide substance. The leather is too soft if the hide substance content is below 63%, and too stiff if above 72%.

## C L Tseng

Action of vegetable leather on metallic iron I Influence of leather fats on iron V. Kubelka, V. Nemec and S. Zuravlev. *Collegium* 1935, 531-41.—See *C A* 30, 321<sup>1</sup> I D C

App for glue extn (U S pat 2,028,935) 1 Lacquer for leather (Belg. 400,447) 26 Building material from spent tanning wood (Russ. pat 35,904) 20

The Boot and Shoe Maker. Edited by E. Bordoli. London: The Gresham Pub. Co., Ltd. 50s. Reviewed in *J. Intern. Soc. Leather Trades' Chem* 20, 43 (1936).

Handbuch der Leder-Industrie. Edited by Ludwig Jablonski. Bd. II. Treibmaschinenfabriken und technische Lederartikel. 2nd ed. Berlin: Atlas-Verlag M 8. Reviewed in *J. Intern. Soc. Leather Trades' Chem* 19, 576 (1935). Cf *C A* 29, 3871<sup>1</sup>.

The Leather Trades' Year Book, 1935. London: Leather Trades' Pub. Co., Ltd. 28s. pp. 10s. Reviewed in *J. Intern. Soc. Leather Trades' Chem* 19, 576 (1935).

Shins and leather. I. G. Farben und A.-G. Fr. 789,676, Nov. 4, 1935. These are degreased by treating them with sulfuric esters of aliphatic alcs of high mol wt, fat amides of high mol wt. congt sulfonyl groups, fat esters of high mol wt. sulfonated in the alc group or salts of these compds. Examples are given of the use of the sulfuric esters of dichlorostearic ethanalamide, propanolamide of copra fat acid, oleic ethanalamide, tetra-chloropalmitic ethanalamide and dibromoleic butanolamide. Cf *C A* 30, 322<sup>1</sup>.

Tanning. Harold G. Turley and Ian C. Somerville (to Rohm & Haas Co.). U. S. 2,029,088, Jan. 28. Hides or skins such as pickled kid, sheep or calf skins are treated with an aldehyde such as formaldehyde capable of reacting with the hide protein, a salt of Al such as the sulfate and a synthetic org. tanning material. Numerous examples are given.

Synthetic tanning materials. Alphons O. Jaeger (to American Cyanamid & Chemical Corp.). U. S. 2,029,322, Feb. 4. In the production of a synthetic tanning agent of the sulfonated diarylmethane type, at least one reaction step is carried out in the presence of a decolorizing absorbent such as activated carbon so that a synthetic tanning agent is obtained having a light color and producing lighter shades of leather than would otherwise be obtained. Cf *C A* 30, 322<sup>1</sup>.

Sulfite cellulose tanning extracts. L. Ya. Reznik and M. I. Khaduk. Russ. 37,790, July 31, 1934. Sulfite cellulose solns are prep'd by introducing NH<sub>3</sub> and Na<sub>2</sub>SO<sub>3</sub> into the heated sulfite soln, and concg to a solid consistency.

Sulfite cellulose tanning extracts. L. Ya. Reznik. Russ. 37,797, July 31, 1934. NH<sub>3</sub>, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and NH<sub>4</sub>HSO<sub>3</sub> are introduced into heated sulfite soln and the product is evap'd to the consistency of dry ext.

Sulfite cellulose extract. L. Ya. Reznik. Russ. 37,798, July 31, 1934. The sulfite cellulose soln is treated, before concn, with NH<sub>3</sub> and acid salts of Cr, Al or of Fe and Ni.

Impermeabilizing leather. André F. L. Long. Fr. 789,095, Oct. 22, 1935. The color of the leather is preserved by applying a protective coating of a trialkyl ether of glycerol before immersing the leather in the impermeabilizing bath.

Impregnating leather. Clément Dupure and Marcel Dupure. Fr. 789,051, Oct. 22, 1935. Crude or tanned rubber is made more resistant to wear and to the action of moisture, while not made brittle, by impregnation with nonhygroscopic resinous substances not decompd. by water, e.g., aldehydic resins and more particularly PhOII-CH<sub>2</sub>O resins and natural and artificial gums.

Synthetic leather. Engelbert Windelen Fr. 789,131, 1 Oct. 23, 1935. Fibers of waste leather are subjected to a fermentation whereby the more easily decomposable parts are dissolved. Vulcanizing agents and an accelerator are beaten up with the fibers, and latex, e. g., Revertex, stabilized by casein, is added. Water is added to give the mass the form of sheets. Untreated leather fibers, cellulose fibers, etc., may be added to give greater permeability to the product.

Decorative patent leather. Benjamin J. Kaplan U. S. 2,029,377, Feb. 4. Various details are described for the manuf. of leather having the usual patent surface coating with a decorative discontinuous layer of contrasting color incorporated in the coating.

Apparatus for applying finely divided material such as nebulized dyeing material to leather. Matthew M. Merritt (to Tanning Process Co.) U. S. 2,028,796, Jan. 23. Various structural and operative details.

### 30—RUBBER AND ALLIED SUBSTANCES

C. C. DAVIS

The laws and the mechanism of the electrophoresis of rubber latex. Industrial applications and the manufacture of articles by electrophoresis. Georges Génin *Revue caennaise* 12, No. 117, 11-15 (1935).—An illustrated review and description. C. C. Davis

Acetone extraction of raw rubbers. V. Effect of heating of acetone extract at 100° and storing the acetone extract on the saponification value. Hidemaro Endô *J. Soc. Rubber Ind. Japan* 8, 964-8 (1935).—See C. A. 30, 323.

Methods of extraction of rubber from tau-saguz. V. Zhuravlev and V. Provorov *Sovetskii Kauchuk* 1935, No. 12, 42-7.—The roots of tau-saguz (I) contained water 5.5, ash 6.06, tars 2.4, rubber 23.5 (av. 20%). They were treated by 2 methods. (1) The *Gintzelmetla* method involved consecutive disintegration of the raw material to insure flotation in app. The roots of I were cleaned, cut into small pieces, disintegrated in a Gelfry machine, and milled to crush bark and wood pulp, the mass was treated with hot water to swell the wood pulp. The wood pulp was removed from the rubber in a Rozenfeld and Lebedev app., and the material was fed into a Rettinger classifier. The floated rubber was fed into the receiving screens, the rubber concentrate was washed and the "skins" of I were dried and packed. The yield of "skins" was 67-69%, and they contained  $H_2O$  1.5, ash 0.98, tars 4.5, rubber 70-83 (acetone plus  $CHCl_3$  ext. 80-88%). (2) The *microbiol. method* (in the exptl. stage) consisted in fermenting the roots of I. The roots were cut into pieces, treated with hot water and left for 7 days in a fermentation soln. contg. urine,  $(NH_4)_2CO_3$  or  $(NH_4)_2HPO_4$  (no difference in results was observed), the amts. of salts being detd. daily by the acidity. After washing, "skins" of the following compn. were obtained:  $H_2O$  1.16, ash 0.93, tars 5.11, rubber 88.87, insol. matter 5.09%. The yields from the dry distn. of tau-saguz rubber were: fraction up to 150°, 10.19; 150-200°, 6.77; 200-300°, 46.35; 300-310°, 7.59; residue, 24.33; losses, 4.98%. The reaction of the tar water was alk. By treatment of I rubber with  $Cl_2$  and  $Br_2$ , the products corresponded to those obtained with Para rubber. The dipentene fraction gave with  $Br_2$  a violet coloration. I rubber contains isoprene units. A. P.

Simplified method of estimation of the proportion of rubber in kok-saguz for purposes of selection. P. Stolbin. *Sovetskii Kauchuk* 1935, No. 2, 19-21.—Boil kok-saguz root (1.2 g.) and 50 cc. of 3% NaOH soln. 30 min., decant, roll down the root on glass to a flat piece, wash the flat piece with water acidified with  $HCl$ , treat with cold  $H_2SO_4$  (d. 1.84) for 4 hrs. to destroy the cellulose, wash, leave overnight in 50 cc. of 3% NaOH, wash out NaOH, dry at 70-80° and weigh. Multiply the wt. by 0.38 (The flat piece which was treated in this manner contd. 38% of substances extd. with  $CHCl_3$ ). This factor must be checked for each large series of analysis. A. Pestoff

The newest principles of formulating rubber mixtures. B. Fabritzev. *J. Rubber Ind. (U. S. S. R.)* 12, 757-62 (1935).—The free S in rubber is oxidized by the air to  $H_2SO_4$  or  $H_2SO_3$ . These deteriorate the rubber, therefore formulas should contain a min. of free S. Vulcanization in the future will be with polysulfide accelerators like tetramethylthiuram disulfide (I), e. g., 3% in the presence of a very small proportion of S, or combinations of S 0.1-0.5% and Se 3.0%. The effects of various ingredients,

including  $ZnO$ , magnesia alba, ground asbestos, etc., in a formula with 3% tetramethylthiuram disulfide and no S, on the mech. properties of rubber are shown. A. Pestoff

The plasticization of rubber and rubber mixtures with tau-saguz and with light crepe, and the mechanical properties of the vulcanized rubber. N. Chesnokov. *Sovetskii Kauchuk* 1935, No. 2, 34-41.—The mixts. tested contained rubber (tau-saguz (I) or light crepe (II)) 100, mercaptobenzothiazole 0.8, stearic acid 2.0,  $ZnO$  5.0,  $CaCO_3$  50 or lamp black 35, S 3.5. The rubber mixts. were milled for different times and cured 10, 20, 30, 40 and 50 min. at a temp. corresponding to 2.5 kg. per sq. cm. The plasticity of I was higher than that of II, and it did not change with the time of plasticization, whereas that of II increased. I can be used in rubber mixts. without plasticization. The plasticities of rubber mixts. with  $CaCO_3$  and lamp black were higher than the plasticity of pure rubber, lamp black mixts. increased the plasticity to a smaller degree than did  $CaCO_3$ . I and II in mixts. with  $CaCO_3$  gave the same plasticities after calendaring. The tensile strengths of rubber mixts. with II were higher than with I (189 kg. per sq. cm. and 151 in mixts. with lamp black). The residual elongations of rubber mixts. with I were higher than with II (28 and 22%). The reverse relationship between the plasticity of rubber mixts. and tensile strength was observed, a decrease in the plasticity of lamp-black mixts. compared with  $CaCO_3$ , corresponded to the same percentage increase in tensile strength.

A. Pestoff  
The plasticization of rubber on the Gordon plastometer. N. Krulov. *J. Rubber Ind. (U. S. S. R.)* 12, 739-46 (1935); cf. C. A. 29, 6464.—Descriptive. A. P.

Thermal plasticization of rubber. Technological characteristics of the product of thermal plasticization. M. Farberov and A. Suslyakov. *J. Rubber Ind. (U. S. S. R.)* 12, 715-22 (1935); cf. C. A. 29, 7696; Gusev, Gruzevich, Khovrina and Zelikov, C. A. 29, 6797.—Rubber was plasticized in an autoclave at 150°, cooled to 50° and passed 3 times through a mill (65-70°). The plasticity increased directly with the time of thermal treatment. The energy used was 15-50% less than that with mech. treatment. With a high proportion of rubber and a high plasticity, the economy is greater with thermal plasticization. The mech. properties of vulcanizates prepd. from mech. and thermally plasticized rubber aged the same. The thermally treated vulcanizates adsorbed less water and showed less calender effect; at plasticities of 2.5-2.75, their solns. gave lower viscosities and a plasticity of 1.85 a higher viscosity than solns. of mechanically plasticized rubber. Thermally plasticized rubber was used to prep. sponges, rubberized fabrics and frictions. Thermally plasticized rubber required a higher concn. of alkali for its water dispersion.

A. Pestoff  
Aging of rubbers by exposure on top of Mount Fuji and on level land. Shohai Saito. *J. Soc. Rubber Ind. Japan* 8, 631-93 (1935).—Rubber sheet was exposed for 319 days at the top of Mt. Fuji and below on level land. Contrary to the expected there was greater aging effect on top of the mountain because of more ultraviolet light; the deterioration was less than below. This may have been due to more cloudy days and the low temps. K. Kitsuta

Determination of free sulfur in rubber. E. W. Oldham, L. M. Baker and M. W. Craytor. *Ind. Eng.*



ethylenediamine turned pale brown, and those containing phenylenediamine pale yellow-green. Among compounds of the phenylnaphthylamine type, phenyl- $\alpha$ -naphthylamine did not show consistent colors, but varied from dark green to green, depending on the particular kind of antioxidant used. However, the substances belonging to this type can easily be identified by their outward appearance. Phenyl  $\beta$ -naphthylamine turned yellow or yellow-green.  $\beta$ -Naphthols turned sepia or dark gray. Color reactions which are believed to be characteristic are those of Parazonal, aldol- $\alpha$ -naphthylamine resin, Agertite white, Neozone A and C, Akroflex C and Fictol white.

**Means of vulcanizing rubber** S. M. Shulman, L. G. Dontz and B. G. Yashkov *Setsel's Rekonstruktsiya*, Nauka 1935, No. 4, 160—Electro heating is used.  
B. V. Shvartzberg

**Film forming compounds containing chlorinated rubber** (U. S. pat. 2,029,588) 13 Binder for paints [contg rubber] (Fr. pat. 799,597) 26 Compound sheet materials [of rubber, etc.] (Brit. pat. 435,088) 13 Automatic electrolytic vulcanizer (Fr. pat. 789,515) 4

**Freundlich, Herbert** The Chemistry of Rubber London Methuen & Co. Ltd. 72 pp. 2s. 6d. Reviewed in *Ind. Chemist* 11, 511 (1935), *J. Am. Chem. Soc.* 58, 381 (1936)

**Noble, Royce J.** Latex in Industry New York Palmerton Pub. Co., Inc. 384 pp. \$7. Reviewed in *Rayon and Melliand Textile Monthly* 17, 66 (1936), *Chem. Warfare Bull.* 22, 46

**Porter, Jermain D.** The Chemcraft Rubber Chemistry Manual Hagerstown, Md. The Porter Chem. Co. 76 pp.

**Treating latex** International Latex Processes Ltd. and Mario Faldini Brit. 435,636, Sept. 25, 1935. Vulcanized aq. dispersions of rubber are clarified by centrifuging in a clarifier. The vulcanization before the centrifuging is preferably effected in the presence of excess vulcanizing agents, the excess being removed by the clarifying, which may be in 1 or more stages.

**Concentrating rubber latexes** Alfred E. Petersen and Wilhelm Gensecke (to Metallgesellschaft A.-G.) U. S. 2,029,531, Feb. 4. Latex to be concentrated is heated while avoiding the formation of steam (suitably in a tube and header app.) and is introduced in a state presenting a large surface, such as a spray into a vacuum evaporator at a temperature which is higher than the b. p. of the latex under the pressure prevailing in the evaporator. App. is described. Cf. C. A. 30, 1001

**Concentrating latex** Rubber Producers Research Association, Geo. Martin, Wilfred S. Davey and Herbert C. Baker Brit. 437,788, Nov. 5, 1935. Rubber latex is creamed by adding a  $H_2O$  sol. alkylated deriv. of starch or of other food reserve polysaccharide. The rate of creaming may be accelerated by restricting the amt. of  $NH_3$  in the latex and by warming. A sol. methylated starch may be obtained by heating an aq. dispersion of potato starch with NaOH and  $Me_2SO$ , and treating the cooled reaction mixt. with dil.  $H_2SO_4$  until it is only slightly alk. The product may be used directly or the alkylated starch may be 1st isolated by pptn. with alc. or by evapng to dryness under reduced pressure, extrg. with  $CHCl_3$ , dehydrating with anhyd.  $MgSO_4$  and evapng the  $CHCl_3$ . Cf. C. A. 29, 7122

**Synthetic rubber** I. G. Farbenindustrie A.-G. Fr. 788,973, Oct. 21, 1935. Vulcanizable products of good quality are prep'd by polymerizing butadiene and its homologs in an emulsified form, as emulsifying agents nonvolatile org. compounds are used and these are allowed to remain in the products after their coagulation. Agents used include transformation products of oil fms. of high mol. wt., drying or semidrying oils, such as linseed oil, wood oil, olive oil, fish oils, and alkali metal or  $NH_3$ , salts of carboxylic acids of high mol. wt.

**Rubber substitutes** Herbert Vohrer. Fr. 789,172, Oct. 24, 1935. Elastic objects are made by forming a paste in the cold with water and polyvinyl alc. insol. in water and emollients, the paste being pressed to any desired shape and heated to 80–100°. The ratio between the polyvinyl alc. and fillers on the one hand and water and emollients on the other is between 1 and 1.5, the fillers being glue, dextrin, gelatin, starch, etc., which are insolubilized by adding tanning substances.

**Halogenating rubber, etc.** Metallgesellschaft A.-G. Brit. 435,729, Sept. 26, 1935. See Fr. 785,257 (C. A. 30, 647)

**Chlorinated rubber.** Deutsche Gold- und Silber-Scheideanstalt vorm. Roeder. Brit. 435,726, Sept. 26, 1935. Addn. to 425,769 (C. A. 29, 61004). The process of 425,769, in which chlorinated rubber is sep'd from its soln. by spraying in the presence of a condensable vapor, e. g., steam, at or above the b. p. of the solvent, is carried out in the presence of an alkali for stabilizing purposes. The alkali, as vapor or finely divided solid, e. g., NaOH, may be blown in by the atomizing medium for the chlorinated soln. or may be added to liquid into which the soln. is sprayed.

**Rubber compositions** Siemens & Halske A.-G. Brit. 435,027, Sept. 9, 1935. An elec. conductor has an insulation consisting of a mixt. of unvulcanized rubber and waxes falling in the classes montan waxes freed from resin and bleached and hardened by oxidation, chlorinated  $C_{18}H_{34}$  and wax obtained from the higher alcs. of the fat series, wax acid and vegetable oils hardened by hydrogenation. The proportions are preferably rubber (0 and wax 40. Balata and gutta percha may be added, as may other waxes, e. g., carnauba, ceresin, "Superla". Cf. C. A. 29, 7692

**Rubber compositions** Franklin H. Lushington Brit. 436,509, Oct. 14, 1935. A self-vulcanizing compo. for use on roads, walls and floors comprises corr. dust, peat dust, wood flour or sawdust and rubber latex in the proportions of 1–5 of dry rubber to 1 of corr. dust, together with the following vulcanizing agents: pptd. S 3–15, an accelerator, e. g., diphenylguanidine, quinodine, piperidine, 0.5–1, an activator, e. g.,  $ZnO$ , 2–3 and, if necessary, an anti-coagulant, e. g.,  $NH_3$ , NaOH, 0.5–1%, all calc'd on the solid content of the latex. The compn. is left to stand to effect partial vulcanization and then applied to the surface to be covered.

**Rubber compositions** Henry P. Stevens, W. H. Stevens and Clayton and Stevens Ltd. Brit. 435,401, Sept. 23, 1935. The rubber compns. of Brit. 390,820 (C. A. 27, 6020), comprising rubber mixed with a high proportion of an oily softener, are used for the production of textile machinery parts, e. g., rollers, belts, bands and pickers, liable to be contaminated with the oil naturally occurring in the fibers or used in the treatment thereof.

**Rubber derivatives** I. G. Farbenindustrie A.-G. Fr. 788,840, Oct. 19, 1935. Rubber, products obtained by polymerizing isoprenes, butadienes and rubber substitutes such as factice are treated with dil. F to give products contg. up to 30% of F. The products are particularly suitable for the production of insulators in electrolytic cells serving for the production of F.

**Chlorinated rubber compositions** I. G. Farbenindustrie A.-G. Brit. 436,393, Oct. 10, 1935. Alkylated naphthalenes, in which the alkyl groups preferably contain 3–4 C atoms, are incorporated with chlorinated rubber fillers, color, resins, softeners and, to produce a lacquer, org. solvents may be added.

**Rubber threads or strips** Carl L. Beal (to American Anode, Inc.) Brit. 436,259, Oct. 8, 1935. These are made by shearing along a plurality of parallel lines a sheet of  $H_2O$ -contg. latex coagulum, whereby a rounded edge is produced with a longitudinal "seam" along the shear line. App. is described.

**Rubber threads** Thomas L. Shepherd. Fr. 788,944, Oct. 21, 1935. A very fine stream of coagulant (e. g., 5–25% soln. of  $AcOH$ ) is introduced into a bath contg. latex of suitable concn. and a divided material, e. g., cotton

rayon or wool flock. The thread formed is drawn out and subjected to further necessary treatments. Cf C A 30, 1608\*.

Apparatus for producing rubber fibers Lorenz Börner Ger 620,412, Oct. 30, 1935 (Cl 39a 10 03)

Apparatus for molding strips, etc., of rubber or cellulose by dipping Siegfried Baul Brit 437,775, Nov. 5, 1935

Finely divided rubber Rubber-Latex-Poeder-Compagnie N. V. 1 r 789,058, Oct. 22, 1935 One or more substances which deposit in the form of an insol or slightly sol coating, preferably nonhygroscopic, are added to rubber latex which is then pulverized into a drying atm. Suitable addns are solubilized flour or starch and a phenol or aldehyde, preferably  $\text{CH}_3\text{O}$ , or a protein and a tanning agent, or substances producing an artificial resin on drying or a sol glass, etc. 1 r 789,059 App for pulverizing rubber latex

Sponge rubber Glen S Hiers (to Collins & Aikman Corp.) U S 2,029,617, Feb. 4 A sponge rubber is formed from an aq rubber dispersion contg borax and a vegetable gum, such as locust kernel gum, which is coagulable by the borax, and a cell-forming agent such as  $(\text{NH}_4)_2\text{CO}_3$

Rubber articles Karl Lesberg Brit 446,288, Sept. 30, 1935. This corresponds to 1 r 770,812 (C A 29, 959\*). The latex is preliminarily agglomerated, e. g., by alum, Ca formate, etc.

Hollow rubber articles Paul Iaber Brit 436,424 Oct. 10, 1935 These are produced from aq dispersions of rubber by depositing the undissolved constituents of the dispersions on the inner walls of hollow porous molds that are not completely filled with the dispersions, the molds being given simultaneous rotational movements about 2 axes preferably disposed at right angles. Variegated articles may be obtained by using several colored dispersions

Hollow rubber articles such as balls or inner tire tubes. Levi M. Rosenthal U S 2,028,808, Jan. 24 Various operative details

Rubber-coated articles I G Farbenindustrie A-G Brit. 436,532, Oct. 14, 1935 See Ger 606,041 (C A 29, 1622\*)

Rubber shoe soles resembling leather. Walter R. Hiebler (to Hood Rubber Co.). U S 2,029,371, Feb. 4 Articles such as rubber shoe soles contg a high proportion of fiber carry a surface layer of wax through which the fibers extend and this surface is burnished so that it resembles sole leather

Surfacing rubber articles such as portions of rubber shoes with comminuted material such as flock Alvin L. Diller and Leslie H. L'Hollier (to Hood Rubber Co.) U S 2,029,361, Feb. 4 A layer of adhesive liquid comprising a coagulant for rubber such as a soln. contg  $\text{ZnCl}_2$ , glycerol, etc., is deposited upon a support and a layer of comminuted material is deposited upon this layer; coagulable rubber material is applied over these layers, and the resulting layered product is removed from the support

Ornamented rubber articles Leslie H. L'Hollier and Alvin L. Diller (to Hood Rubber Co.). U S 2,029,379, Feb. 4 In forming bathing caps or other ornamented articles, a liquid rubber dispersion is ejected from a nozzle onto a supporting surface to form a gossamer design, and upon this there is deposited a layer of rubber of a contrasting appearance and this layer is then removed with the rubber of the design adhered to its surface Cf C A 29, 4633\*

Apparatus for producing shirred margins on rubber articles Arthur E. Collins (to B F. Goodrich Co.). U S 2,029,359, Feb. 4 Various mech details

Rubber to metal bonding method George Livings and Bernard W. D. Lacey (to Dunlop Tire and Rubber Goods Co. Ltd.) Can. 354,930, Dec. 24, 1935 The surfaces of a metal sheet and a sheet of uncured rubber compn are bonded together by exposing the rubber sheet compn to Hg vapor lamps for 15 min at 18 in distance, and then vulcanizing the sheet rubber *in situ* upon the metal surface

Apparatus for coating wires with rubber Charles J

Moody and Earle A. Mitchell (to Phelps Dodge Copper Products Corp.). U. S. 2,029,435, Feb. 4. Various structural, mech and operative details.

Apparatus and method for coating the wires of vehicle wheel tires with rubber latex. Hubert H. Burton and John Bull Rubber Co. Ltd Brit 436,844, Oct. 16, 1935

Treating raw rubber passed through calendars 1. T. Kataev Russ 36,002, Apr. 30, 1931 The shrinkage of rubber obtained from the calendars is eliminated or lowered to 0.1 to 0.2% by passing it through a vat of boiling water (time of exposure 1 min)

Preserving rubber, etc Wingfoot Corp Brit 436,153, Oct. 7, 1935 Rubber or other deteriorable compn is vulcanized in the presence of an antioxidant consisting of a tertiary dialkyl naphthylamine, the naphthyl radical being unsubstituted and the alkyl radicals, which may be the same or unlike, connected only through the tertiary N atom. The tertiary amines may be prep'd by causing an alkyl halide to react with a naphthylamine in the presence of  $\text{Na}_2\text{CO}_3$ , or a dialkyl sulfate, an alkyl ester of toluene-sulfonic acid or a secondary dialkylamine with a naphthylamine Cf C A 30, 619\*

Rubber Imperial Chemical Industries Ltd and William Baird Brit 435,024, Sept. 6, 1935 Divided on and addn to 431,951 (C A 30, 900\*) To prevent oxidation and flex-cracking of rubber there is incorporated in the mix an alkoxy- or polyalkoxy-diaryllamine in which the alkoxy group or groups are in the *m*- or *p*-position to the imino group and the aryl radical is Ph, tolyl or xyllyl, 4,4'-dimethoxydiphenylamine is excluded. Examples are given of the use of *p*-methoxydiphenylamine, 3-methoxy-4'-methylphenylamine and 3,4'-dimethoxydiphenylamine

Rubber William Patter ter Horst (to Dominion Rubber Co. Ltd.) Can 354,278, Nov. 19, 1935 To improve the resistance and retard the deterioration, there is incorporated in rubber a S deriv of the reaction product of a ketone with a secondary amine

Plasticizer for rubber Julius Hyman (to Velocel Corp.) Can 354,960, Dec. 21, 1935 Rubber is mixed with 0.1-25% of a viscous, sticky, liquid polymer of reddish yellow color derived from the high pressure liquid-phase polymerization at 450-750° F of cracked gasolines and having an iodine no. of 100-200

Rubber Irma Louis Blumer Ger 620,751, Oct. 25, 1935 (Cl 39b 3) A softening agent for rubber transformation products consists of ethers of PhOH and polyhydric alcs. Thus, PhOH, cresol, guaiacol,  $\alpha$ -resorcinol, naphthol, etc., are etherified with glycerol, etc. The products are used for making *laqueurs*, *pastes*, etc.

Process and apparatus for softening raw rubber and compositions thereof Società italiana Pirelli Brit. 435,974, Oct. 2, 1935 A rolling mill is used which comprises 2 oppositely revolving rollers, the rubber being worked as an endless loop by feeding the forward end of the issuing sheet again to the rolls, the loop accumulating in superimposed folds upon a support at the feed side of the mill as its length increases with continued working to the desired thickness and the rollers drawing continuously from the accumulation

Rubber solutions J. R. Geigy A-G. Brit 436,512, Oct. 11, 1935 The viscosity of solns. of rubber, gutta-percha, balata, etc., is reduced by treating the rubber, etc., with a sulfonic acid, before, during or after the dissoln or swelling, at a moderate temp., e. g., 50°. The acid may be *p*-toluene-, ethane-, benzene-, xylene-, cymol-, nitrobenzene- or nitrochlorobenzene-sulfonic acid Cf C A 29, 769\*

Utilizing waste rubber. Ernest Bemelmanns Brit. 435,890, Oct. 1, 1935 Waste rubber contg. fibers is heated in a closed container to carbonize the fibers and render the rubber capable of being plasticized by passage between rollers. Thus, a motor tire cover is heated at 220° for an hr. in a closed container and allowed to cool thereon. On milling, the carbonized fiber is converted into a powdery filler

Vulcanizing press for tires, inner tubes, etc Giuseppe

Cozzo (to Società italiana Pirelli). U. S. 2,029,060, Jan 28, 1935. Mech features.  
 Vulcanizing press suitable for vulcanization of tire casings, etc. Colin Macbeth U. S. 2,023,618, Jan 21, 1935. Mech features.

Vulcanization apparatus. Lewis J. Clayton (to Viceroy Manufacturing Co. Ltd.) Can. 354,318, Nov. 19, 1935. A strip of rubber is passed through and supported centrally in a tunnel having reflective walls. Radiant heat elements are disposed at intervals, and wire mesh screens are placed between the heater and rubber. Cf. C. A. 29, 4212<sup>1</sup>.

Pneumatic tire casings. Ralph M. Reel (to Pharis Tire and Rubber Co.) U. S. 2,027,248, Jan. 7, 1935. A tire is formed comprising a fabric carcass and a compd. outer covering of rubber composed of an outer tread and side wall layer of relatively hard high abrasive resisting properties, an intermediate cap layer of a more resilient compn. than the tread layer, and an inner layer adjacent the cap layer and of softer and more resilient compn. than the cap layer, the inner layer contg. Sb sulfide, rubber and ZnO, the intermediate cap layer contg. about 44% as much ZnO as rubber and being relatively thin as compared to the outer tread.

Rubber substitute. I. G. Farbenind. A-G. Fr. 787,467, Sept. 23, 1935. Products obtained by polymerizing butadiene, its homologs or their derivs. or mixts thereof among themselves or with other polymerizable compds., in the form of emulsion, are purified by extr. or pptn. and to these are added org. compds. not or only slightly volatile or their mixts in amt. of 10-50, preferably 20-30%. Org. compds. include vegetable, animal or mineral oils, fats or waxes, resins, resinic acids, bitumens, olefins of high b. p., drying or semidrying oils and asphalts. The vulcanized products have good resistance to rupture and elasticity.

Rubber-like compositions. I. G. Farbenind. A-G. Fr. 788,670, Oct. 14, 1935. Isobutyl vinyl ether having a high degree of purity is polymerized either alone or in admixt. with other polymerizable compds. at temps. below 10°, e. g., -40° to -80°, by means of BF<sub>3</sub> used as such or as a product of addn. with an org. compd. contg. O.

Vulcanization accelerators. Wilhelm Lommel and Rudolf Schroter (to I. G. Farbenind. A-G.) U. S. 2,027,184, Jan. 7, 1935. See German 578,673 (C. A. 28, 932<sup>2</sup>).

Vulcanization accelerator. Arthur W. Sloan and Robert C. Goodwin (to B. F. Goodrich Co.) Can. 354,142, Nov. 12, 1935. As accelerator is specified the salt of a bivalent metal and a diaryldithiocarbamic acid, e. g., Zn diphenyldithiocarbamate.

Vulcanization accelerators. Wm. C. Calvert and Howard I. Cramer (to Wingfoot Corp.) U. S. 2,028,086, Jan. 14, 1935. As vulcanization accelerators there are used compds. of the general formula R<sub>2</sub>NHCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, in which R is a hydrocarbon radical, such as *o*-tolylethylene-diamine or various other specified similar compds. (details for making some of which are given).

Vulcanization accelerators. Wingfoot Corp. Fr. 788,621, Oct. 14, 1935. A salt of a secondary furyldithiocarbamic acid (I) is caused to react with a haloaryl thiazole, preferably in the presence of a solvent. Thus, 5-nitro-1-benzothiazyl di- $\alpha$ -furfuryl-dithiocarbamate, m. 93-5°, is made by refluxing 1-chloro-5-nitrobenzothiazole (II), di- $\alpha$ -furfurylamine, CS<sub>2</sub> and NaOH in a solvent such as EtOH. Results are given of the use of this accelerator. Fr. 788,622. A secondary hydrogenated furyldithiocarbamic acid is substituted for I in the process of Fr. 788,621. Thus, 5-nitro-1-benzothiazyl bis(tetrahydro- $\alpha$ -furfuryl)dithiocarbamate, m. 116-8°, is made by refluxing II, bis(tetrahydro- $\alpha$ -furfuryl)amine, CS<sub>2</sub> and NaOH in EtOH.

Sulfenamides. I. G. Farbenind. A-G. Fr. 785,990, Aug. 23, 1935. See Ger. 615,580 (C. A. 29, 8405<sup>9</sup>).

Thiazyl monosulfides. Jan Teppema (to Wingfoot Corp.) U. S. 2,023,082, Jan. 14, 1935. A  $\mu$ -halothiazole is caused to react with a mercaptobenzothiazole (as by heating the materials together with solvents) to form a dithiazyl

monosulfide, which may be used as a vulcanization accelerator with rubber compns. Starting materials which are mentioned include 1-chlorobenzothiazole, 1-chloronaphthothiazole, the 1-chlorotolylthiazoles, the 1-chloroxylythiazoles, 1-chloro-5-nitrobenzothiazole, 1-chloro-4-chloro-5-nitrobenzothiazole, 1,4-dichlorobenzothiazole, 1,5-dichlorobenzothiazole, 1-chloro-4-nitrobenzothiazole, 1-chloro-5-nitrobenzothiazole, 1-chloro-3-phenylbenzothiazole, 1-chloro-3-phenylthiazole, 1-chlorothiazole, 1-chloro-5-ethoxybenzothiazole and other ring-substituted nitro, alkoxy, halo, alkyl and aryl 1-chloroarylenethiazoles, 1-bromo and other halo thiazoles. If the sulfide starting material is a mercaptobenzothiazole, either the hyposulfite or corresponding metal sulfhydrate thereof may be employed. There may be used 1-mercaptopthiazole, 3-butylmercaptobenzothiazole, 1-mercaptop-3-phenylthiazole, 1-mercaptop-3,5-dimethylbenzothiazole, 5-nitromercaptobenzothiazole, 5-aminomercaptobenzothiazole, 4-chloromercaptobenzothiazole, 5-chloromercaptobenzothiazole, 1-mercaptaphthothiazole, 1-mercaptop-5-methoxybenzothiazole, 5-methylmercaptobenzothiazole, 1-mercaptop-3-phenylbenzothiazole, the mercaptotolylthiazoles, and other ring-substituted nitro, amino, halogen, alkyl, alkoxy and aryl mercaptoarylene thiazoles. Various details of procedure are given.

Carbamyl disulfides. Jan Teppema (to Wingfoot Corp.) U. S. 2,024,613, Dec. 17, 1935. By reaction of a dithiocarbamate with a nitro-substituted haloarylenethiocarbamate, rubber-vulcanization accelerators are produced such as 2-benzoyl-4-nitrophenyl dimethylthiocarbamyl disulfide, the corresponding di-Et, di-Bu, dicyclohexyl and  $\Gamma$ -cyclohexyl derivs., 2-benzoyl-4-nitrophenyl pentamethylenethiocarbamyl disulfide, *o*-nitrophenyl ethylcyclohexylthiocarbamyl disulfide, and the corresponding dicyclohexyl, di-Me and di-Am derivs., *o*-nitrophenyl morpholythiocarbamyl disulfide, 2-nitro-4-chlorophenyl morpholythiocarbamyl disulfide, 2-nitro-4-chlorophenyl pentamethylenethiocarbamyl disulfide, 2-nitro-4-chlorophenyl difurfurylthiocarbamyl disulfide, *o*-tolyl ethylphenylthiocarbamyl disulfide, *o*-tolyl dibenzylthiocarbamyl disulfide, 4-nitrophenyl dibenzylthiocarbamyl disulfide, 2,4-dinitrophenyl dibenzylthiocarbamyl disulfide, 2,4-dinitrophenyl dimethylthiocarbamyl disulfide and 2-nitro-3-methylphenyl dimethylthiocarbamyl disulfide.

Mercaptobenzothiazyl aryl disulfides. Max Bögemann (to I. G. Farbenind. A-G.) U. S. 2,026,873, Jan. 7, 1935. Rubber vulcanization accelerators are formed by condensing together (suitably by heating in C<sub>6</sub>H<sub>6</sub>) 1-mercaptopbenzothiazole or its 3,5-di-Cl or 5-chloro-3-methoxy derivs. or other compds. such as are described by Sebrill and Boord (C. A. 17, 3376), or by Teppema and Sebrill (C. A. 21, 2688-9), with compds. of the formula aryl-S-X, such as PhSCl, *p*-MeC<sub>6</sub>H<sub>4</sub>SCl, *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SCl, 1-ClC<sub>6</sub>H<sub>4</sub>SCl, 2-*o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SCl and the like. Details are given of the production of benzothiazyl *p*-nitrophenyl disulfide, in 137-8°, 4-methoxy-6-chlorobenzothiazyl *p*-nitrophenyl disulfide, m. 172-3°, and 4,6-dichlorobenzothiazyl *p*-nitrophenyl disulfide, m. 181-2°.

Vulcanizing rubber. Imperial Chemical Industries Ltd. Fr. 787,621, Sept. 26, 1935. Accelerators are made by causing a quaternary ammonium halide (other than tetramethylammonium halide) to react with a metal deriv. of a mercaptobenzothiazole. The reaction is carried out in a medium in which at least one of the reagents is sol. and at least one of the reaction products is insol. Examples are given of the prepn. of hexadecylpyridinium (m. 102-5°), hexadecyltrimethylammonium (m. 120-30°), ( $\beta$ , $\gamma$ -dihydroxypropyl)pyridinium (m. 101-7°) and  $\beta$ -hydroxyethylpyridinium-1-benzothiazyl sulfide and a product, m. 85-6°, from benzyltrimethylammonium chloride and Na salt of 1-mercaptopbenzothiazole.

Vulcanized rubber sponge. Fabbriche Riunite Industri Gonina Torino. Ger. 622,238, Nov. 22, 1935. (Cl. 3<sup>1</sup>/<sub>2</sub> 11 01). See Fr. 768,905 (C. A. 29, 640<sup>1</sup>).



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